

**Process-Product Characteristics of Refractance Window
Dried Turmeric Powder and Golden Milk Products**

**Thesis submitted in partial
fulfillment of the requirement for the degree of**

DOCTOR OF PHILOSOPHY

by

Preetisagar Talukdar



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Indian Institute of Technology Guwahati
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December, 2022

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Dedicated to
my Parents (Mr. Sanatan Haloi and Mrs. Debajani Haloi),
my Husband (Dr. Pankaj Jyoti Barman)
and
my Brother (Mr. Jyoti Sagar Talukdar)





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CERTIFICATE

This is to certify that the Ph.D. thesis entitled **“Process-Product Characteristics of Refractance Window Dried Turmeric Powder and Golden Milk Products”** (submitted by Mrs. Preetisagar Talukdar) was carried out under my supervision at the Department of Chemical Engineering, Indian Institute of Technology Guwahati.

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DISCLAIMER

The experimental, modelling and characterization related data presented in this Ph.D. thesis was carried out by me and is reported after due verification. To the best of my knowledge, the work summarized in this Ph.D. thesis has not been submitted elsewhere for the award of any Degree or Diploma.

(Mrs. Preetisagar Talukdar)



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The Ph.D. thesis targets the sensitivity and optimality of the novel refractance window drying (RWD) process for the drying of *Curcuma longa* and associated product development. The conducted research work has been addressed in five phases of research namely comparative efficacy of RWD, tray and oven drying process for the drying of paste and sliced *Curcuma longa* samples; sensitivity and optimality of mylar film thickness, water bath temperature and drying time during RWD of *Curcuma longa* sliced sample; response surface methodology (RSM) based optimization of bath temperature, drying time and air velocity; NaFeEDTA and folic acid fortification of refractance window (RW) dried *Curcuma longa* powder and associated characterization and analysis; storage and sensory studies of fortified and non-fortified RW dried *Curcuma longa* powder and golden milk product system and nutritional and sensory analysis of RW dried Lakadong turmeric variety powder and golden milk product system.

Targeting the first objective of the Ph.D. thesis, the drying characteristics of slice and paste *Curcuma longa* samples have been addressed using three alternate drying methods namely refractance window drying, oven drying and tray drying. The comparative assessment of the alternate drying process has been targeted in terms of desired properties such as moisture content (MC), antioxidant activity (AA), curcumin content (CC), total phenolic content (TPC), total flavonoid content (TFC), and colour. Thereafter, a comparative conceptual economic assessment of laboratory scale RWD, tray and oven drying processes has been as well targeted. Notable findings of the work include simplicity of the RWD process, significant reduction in drying time and best performance of sliced sample at 60 °C bath temperature.

For the fixed choice of 1 mm thick *Curcuma longa* sliced sample, the following thesis objective targeted the sensitivity of the RWD process with respect to alteration in mylar film thickness (125 – 350 µm) and bath temperature (65 – 95 °C). Drying kinetics and nutritional characteristics of the process-product system have been considered. Equilibrium drying time, moisture diffusivity, fitness of thin layer

models, MC, TPC, TFC, CC, AA and colour indices have been considered as the responses to judge upon the associated optimality and sensitivity. Thereby, mylar film of 250 μm has been inferred to be relevant along with drying temperature and drying time.

In the following objective, targeting the sensitivity and the optimality of the RWD process parameters such as water bath temperature (65 – 95 $^{\circ}\text{C}$), drying time (80 – 360 min) and air-velocity (0.5 – 1 m/s), RSM based experimental design approach was followed to achieve best combinations of MC, TPC, TFC, CC, AA and colour indices. Thereby, best fit model that accounts upon the influence of independent variables on dependent variables and analysis of variance (ANOVA) have been considered. The relevant findings confirmed greater sensitivity of water bath temperature and drying time but not air-velocity. Thus, optimal choice of process parameters has been achieved to venture into fortified and golden milk product development.

The next objective involved the fortification of RW dried turmeric powder with folic acid and NaFeEDTA constituents. The RW dried turmeric powder and the folic acid fortified and NaFeEDTA fortified RW dried turmeric powder samples were subjected to physical characterization such as bulk density, swelling power, solubility, dispersion time, hygroscopicity, water binding capacity and colour. Also, other analysis such as FTIR, XRD, TGA, DSC, FESEM and particle size analysis have been considered. The findings confirmed non – significant influence of fortification on the desired properties of the folic acid and NaFeEDTA fortified RW dried turmeric powder product. In vitro digestion study was undertaken to assess upon the bio-availability of curcumin, folic acid and NaFeEDTA.

The eventual objective involved further characterization of RW dried turmeric powder and folic acid fortified and NaFeEDTA fortified RW dried turmeric powder sample in terms of moisture isotherm, permeability of packing material, shelf life and storage parameters. A sensory analysis was conducted to obtain the best constitution of RW dried turmeric powder in milk (golden milk product). Thereby, for the best formulation of golden milk, characteristics of the product under unrefrigerated and refrigerated conditions have been evaluated in addition to the storage study. Other characterization such as MC, TPC, TFC, AA, CC, colour indices, bulk densities, water binding capacities etc., have also

been assessed with time. Marginal alterations have been affirmed for the RW dried fortified and unfortified turmeric sample.

Further delineating upon even better product development, the last objective of the thesis involved characterization of Lakadong turmeric variety based RW dried product and golden milk formulation. The considered characterization involved MC, TPC, TFC, AA, CC, colour indices, FTIR analysis. Thereafter, the sensory analysis was carried out to assess upon the optimal constitution of Lakadong turmeric and milk. Thereby, the optimal milk formulation was studied for its nutritional characteristics. The findings conveyed marginally better nutritional profile but significantly higher CC profile of the RW dried Lakadong turmeric product.

These findings of the Ph.D. thesis further substantiate upon the need to further research in the scale up, design and formulation research of RWD based turmeric product development. The thesis especially emphasizes on the possible scope for fortified turmeric tablets and golden milk product systems that have wider application in the naturopathy and ayurveda based nutritional support for the good health of humankind. Interesting preliminary trends of Lakadong turmeric variety can be further promoted towards product commercialization avenues of the produces of North-East India.



The subjective novelty of the Ph.D. thesis is as follows:

- Economic efficacy of the refractive window drying (RWD) process for small scale processing systems for rural application.
- Sensitivity and optimality of mylar film thickness for the RWD of sliced turmeric samples in terms of the Moisture content (MC), Total phenolic content (TPC), Total flavonoids content (TFC), Anti-oxidant activity (AA) and Curcumin content (CC) constitution.
- Response surface methodology (RSM) based optimization of the process parametric choice (drying temperature and time and air velocity) of the RWD of sliced turmeric samples.
- Functional, storage and shelf characteristics of folic acid and NaFeEDTA fortified refractance window dried turmeric powder.
- Formulation and characterization of refractance window dried turmeric fortified golden milk product using conventional and Lakadong turmeric varieties.



| | Page No. |
|--|-----------------|
| Dedication | v |
| Certificate | vii |
| Disclaimer | ix |
| Acknowledgement | xi |
| Abstract | xiii |
| Thesis Novelty | xvii |
| Contents | xix |
| List of Tables | xxxix |
| List of Figures | xxxvii |
| Nomenclature | xli |
| Chapter 1: Introduction and Literature Review | 1-38 |
| 1.1 Preamble | 1 |
| 1.1.1 Significance of Horticultural Produces | 1 |
| 1.1.2 Relevance of Horticultural Preservation Technologies | 2 |
| 1.1.3 Search for Thermal Processing Technologies Prompting Nutritional Retention of Produces | 2 |
| 1.1.4 Horticultural Produce Based Functional Food Development | 3 |
| 1.1.5 Turmeric Production and its Usage | 5 |
| 1.1.6 Turmeric Based Customized Products | 6 |
| 1.2 An Overview of Refractance Window Drying process | 8 |
| 1.2.1 Working Principle | 8 |
| 1.2.2 Alternate RWD Operational Schemes | 9 |
| 1.2.3 Heat Transfer Characteristics of the Contact Film | 11 |
| 1.2.4 Technical and Economic Efficacy | 11 |

| | | |
|---|--|--------------|
| 1.3 | Prior Art | 12 |
| 1.3.1 | Comparative Assessment of RWD Processes in Conjunction with Other Low Cost Drying Methods | 12 |
| 1.3.2 | Sensitivity of Mylar Film Thickness on the Process-Product Characteristics of the RWD Process | 16 |
| 1.3.3 | Statistical Design based RWD Process Parametric Optimality Associated to the Drying of Horticultural Produces | 18 |
| 1.3.4 | Fortification of Food Products | 21 |
| 1.3.5 | Storage and Shelf-life Studies of Dry Turmeric Powder Product and Turmeric based Liquid Products | 25 |
| 1.4 | Research Gaps | 29 |
| 1.4.1 | Technical and Economic Efficacy of Turmeric Drying using Refractance Window, Oven and Tray Drying | 29 |
| 1.4.2 | Sensitivity and Optimality of Mylar Film Thickness and Temperature during Refractance Window Drying of Turmeric System | 31 |
| 1.4.3 | Optimality of Refractance Window Drying Process Parameters based on Design of Experiments Approach | 32 |
| 1.4.4 | Need for Refractance Window Drying Fortified Turmeric Product Development | 33 |
| 1.4.5 | Storage Study and Shelf-life Characteristics of Fortified Turmeric Powder and Turmeric Fortified Liquid Product | 35 |
| 1.5 | Objectives of the Ph.D. Thesis | 36 |
| 1.6 | Organization of the Ph.D. Thesis | 37 |
| Chapter 2: Materials and Methods | | 39-70 |
| 2.1 | Techno-economic Efficacy of Refractance Window Dried <i>Curcuma longa</i> | 40 |
| 2.1.1 | Raw Materials | 40 |
| 2.1.2 | Chemicals | 40 |

| | | |
|------------|---|-----------|
| 2.1.3 | Sample Preparation | 40 |
| 2.1.4 | Oven, Tray and Refractance Window Drying | 40 |
| 2.1.5 | Determination of Nutritional Parameters | 41 |
| 2.1.6 | Statistical Analysis | 45 |
| 2.1.7 | Yield and Rehydration Ratio | 45 |
| 2.1.8 | Sensitivity Analysis | 46 |
| 2.1.9 | Energy Consumption | 46 |
| 2.1.10 | Cost Efficacy | 46 |
| 2.2 | Process and Product Characteristics of Refractance Window Dried | 47 |
| | <i>Curcuma longa</i> | |
| 2.2.1 | Raw Materials and Chemicals | 47 |
| 2.2.2 | Sample Preparation | 48 |
| 2.2.3 | Refractance Window Drying | 48 |
| 2.2.4 | Drying Characteristics Curves | 49 |
| 2.2.5 | Fitness of Drying Curve Models | 50 |
| 2.2.6 | Determination of Moisture Diffusivity and Activation Energy | 50 |
| 2.2.7 | Determination of Nutritional Parameters | 51 |
| 2.2.8 | Statistical Analysis | 51 |
| 2.2.9 | Fourier Transformed Infrared Spectroscopic Analysis | 52 |
| 2.3 | Statistical Design Based Sensitivity and Optimality of Process-Product | 52 |
| | Characteristics of Refractance Window Dried <i>Curcuma longa</i> | |
| 2.3.1 | Raw Materials and Chemicals | 52 |
| 2.3.2 | Sample Preparation | 52 |
| 2.3.3 | Optimization of Refractance Window based Drying Process | 53 |
| 2.3.4 | Experimental Design | 53 |
| 2.3.5 | Nutritional Analysis | 55 |
| 2.3.6 | Statistical Analysis | 55 |

| | | |
|------------|--|-----------|
| 2.3.7 | Optimization of Process Variables | 56 |
| 2.3.8 | Validation of Optimum Values | 56 |
| 2.4 | Fortification of the Optimal Refractance Window Dried <i>Curcuma longa</i> | 56 |
| | Powder Product | |
| 2.4.1 | Raw materials, Chemicals and Sample Preparation | 56 |
| 2.4.2 | Refractance Window Drying | 57 |
| 2.4.3 | Fortification with Sodium Ferric Ethylenediaminetetraacetate and Folic Acid | 57 |
| 2.4.4 | Characterizations of Refractance Window Dried <i>Curcuma longa</i> Powder Products | 58 |
| 2.4.5 | In-vitro Analysis | 61 |
| 2.4.6 | Statistical Analysis | 63 |
| 2.5 | Storage and Shelf-life Studies | 63 |
| 2.5.1 | Sample Preparation | 63 |
| 2.5.2 | Evaluation of Sorption Characteristics | 63 |
| 2.5.3 | Shelf-Life Characteristics | 64 |
| 2.5.4 | Nutritional and Physical Characteristics of Stored Dry <i>Curcuma longa</i> Powder Samples | 65 |
| 2.5.5 | Sensory Analysis and Nutritional Characteristics of Refractance Window Dried Turmeric Powder Based Golden Milk Product | 66 |
| 2.6 | Preparation and Characterization of Lakadong Turmeric Based Golden | 69 |
| | Milk Product | |
| 2.6.1 | Raw Materials | 69 |
| 2.6.2 | Sample Preparation | 69 |
| 2.6.3 | Tray and Refractance Window Drying | 70 |
| 2.6.4 | Characterizations of Dried Lakadong Turmeric Samples | 70 |

| | | |
|--|---|---------------|
| 2.6.5 | Preparation and Characterization of Lakadong Turmeric Based Golden Milk | 70 |
| Chapter 3: Techno-economic Efficacy of Refractance Window Dried <i>Curcuma longa</i> | | 71-94 |
| 3.1 | Introduction | 71 |
| 3.2 | Drying Time | 73 |
| 3.3 | Physical and Nutritional Characteristics Oven, Tray and Refractance Window Dried <i>Curcuma longa</i> Samples | 73 |
| 3.3.1 | Yield and Rehydration Ratio of Refractance Window Dried <i>Curcuma longa</i> Samples | 73 |
| 3.3.2 | Moisture Content | 75 |
| 3.3.3 | Total Phenolic Content | 76 |
| 3.3.4 | Total Flavonoid Content | 77 |
| 3.3.5 | Antioxidant Activity | 79 |
| 3.3.6 | Curcumin Content | 80 |
| 3.3.7 | Colour Indices | 81 |
| 3.4 | Sensitivity Analysis of Alternate Drying Process | 82 |
| 3.5 | Statistical Analysis of Oven, Tray and Refractance Window Drying Process | 85 |
| 3.6 | Energy Consumption of Drying Processes | 87 |
| 3.7 | Comparative Economic Competence | 88 |
| 3.8 | Literature Comparison | 92 |
| 3.9 | Summary | 92 |
| Chapter 4: Process and Product Characteristics of Refractance Window Dried <i>Curcuma longa</i> | | 95-116 |
| 4.1 | Introduction | 95 |
| 4.2 | Drying Characteristics Curve | 96 |
| 4.3 | Fitness of Drying Models | 98 |

| | | |
|---|--|----------------|
| 4.4 | Moisture Diffusivity and Activation Energy | 100 |
| 4.5 | Physical and Nutritional Characteristics | 102 |
| 4.5.1 | Moisture Content | 102 |
| 4.5.2 | Total Phenolic Content | 104 |
| 4.5.3 | Total Flavonoids Content | 105 |
| 4.5.4 | Antioxidant Activity | 106 |
| 4.5.5 | Curcumin Content | 107 |
| 4.5.6 | Colour Indices | 107 |
| 4.6 | Optimal Mylar Film Thickness | 108 |
| 4.7 | Fourier Transformed Infrared Spectroscopic Analysis | 110 |
| 4.8 | Statistical Analysis | 110 |
| 4.9 | Literature Comparison | 113 |
| 4.10 | Summary | 114 |
| Chapter 5: Response Surface Methodology Based Optimality of RWD Process Parameters | | 117-138 |
| 5.1 | Introduction | 117 |
| 5.2 | Control Data Set | 119 |
| 5.3 | Box Behken Design Based Experimental Design | 119 |
| 5.4 | Effect of Drying Temperature on Responses | 119 |
| 5.5 | Effect of Drying Time on Responses | 120 |
| 5.6 | Effect of Air-Velocity on Responses | 123 |
| 5.7 | Optimal Data Set based on Experimental Design | 124 |
| 5.8 | Model Selection | 124 |
| 5.9 | Variance Analysis | 124 |
| 5.9.1 | Anti-oxidant Activity | 124 |
| 5.9.2 | Total Phenolic Content | 125 |
| 5.9.3 | Total Flavonoid Content | 125 |

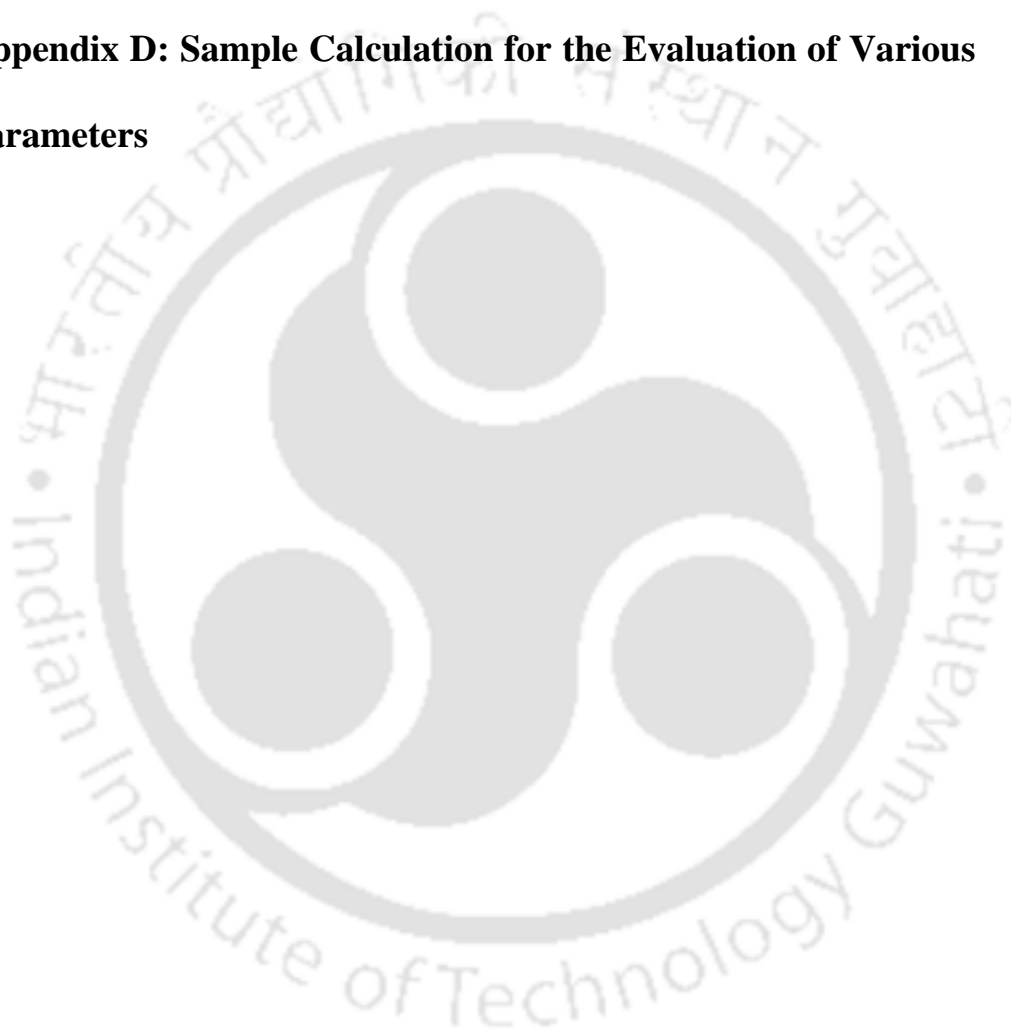
| | | |
|--|---|----------------|
| 5.9.4 | Curcumin Content | 126 |
| 5.9.5 | Moisture Content | 126 |
| 5.9.6 | Colour Indices | 127 |
| 5.10 | Response Surface Analysis | 128 |
| 5.11 | Numerical Optimization and Validation | 128 |
| 5.12 | Comparison of Experimental and Statistical Design based Optimal Data Set | 135 |
| 5.13 | Literature Comparison | 135 |
| 5.14 | Summary | 137 |
| Chapter 6: Fortification of Refractance Window Dried <i>Curcuma longa</i> Powder and Its Associated Studies | | 139-164 |
| 6.1 | Introduction | 139 |
| 6.2 | Physical Characteristics | 140 |
| 6.2.1 | Bulk Density | 140 |
| 6.2.2 | Hygroscopicity | 141 |
| 6.2.3 | Solubility and Swelling Power | 142 |
| 6.2.4 | Dispersion Time | 143 |
| 6.2.5 | Water Binding Capacity | 145 |
| 6.2.6 | Colour Indices | 145 |
| 6.3 | Statistical Assessment of Physical Properties | 146 |
| 6.4 | Fourier Transform Infrared Analysis | 148 |
| 6.5 | Thermogravimetric Analysis | 149 |
| 6.6 | Differential Scanning Calorimetry | 151 |
| 6.7 | X-Ray Diffraction | 153 |
| 6.8 | Field-Emission Scanning Electron Microscopy | 155 |
| 6.9 | Particle Size Distribution, Polydispersity Index and Zeta Potential | 157 |

| | | |
|---|--|----------------|
| 6.10 | In-vitro Digestion based for the Bio-accessibility of Curcumin, Folic Acid and NaFeEDTA in Turmeric Powder Products | 158 |
| 6.11 | Literature Comparison | 161 |
| 6.12 | Summary | 162 |
| Chapter 7: Storage and Associated Studies of Unfortified and Fortified Refractance Window Dried <i>Curcuma longa</i> Powder and Golden Milk Products | | 165-192 |
| 7.1 | Introduction | 165 |
| 7.2 | Powder Sample Preparation | 166 |
| 7.3 | Sorption Characteristics of Powder Products | 168 |
| 7.4 | Permeability of Packaging Material | 168 |
| 7.5 | Shelf-Life Assessment | 168 |
| 7.6 | Sensory Assessment of Powder Product | 169 |
| 7.6.1 | Colour and Appearance | 170 |
| 7.6.2 | Aroma | 170 |
| 7.6.3 | Taste | 171 |
| 7.6.4 | Aftertaste | 172 |
| 7.6.5 | Consistency | 172 |
| 7.6.6 | Mouthfeel | 173 |
| 7.6.7 | Overall Acceptability | 173 |
| 7.7 | Nutritional Properties of the Optimal Golden Milk Formulation | 175 |
| 7.8 | Storage Assessment based Nutritional and Physical Characteristics of Powder Products | 177 |
| 7.8.1 | Moisture Content | 177 |
| 7.8.2 | Anti-oxidant Activity | 178 |
| 7.8.3 | Total Phenolic Content | 179 |
| 7.8.4 | Total Flavonoid Content | 180 |

| | | |
|--|---|----------------|
| 7.8.5 | Curcumin Content | 180 |
| 7.8.6 | Colour Indices | 181 |
| 7.8.7 | Folic Acid | 182 |
| 7.8.8 | Iron Content | 182 |
| 7.8.9 | Bulk Density | 182 |
| 7.8.10 | Solubility | 183 |
| 7.8.11 | Swelling Power | 184 |
| 7.8.12 | Water Binding Capacity | 185 |
| 7.8.13 | Dispersion Time | 185 |
| 7.8.14 | Hygroscopicity | 186 |
| 7.9 | Statistical analysis of data associated to storage studies | 186 |
| 7.10 | Literature Comparison | 189 |
| 7.11 | Summary | 190 |
| Chapter 8: Nutritional Characteristics of Fresh, Tray and Refractance Window Dried Lakadong Turmeric and Golden Milk Products | | 193-206 |
| 8.1 | Introduction | 193 |
| 8.2 | Moisture Content of Turmeric Powder Product | 194 |
| 8.3 | Total Phenolic Content of Turmeric Powder Product | 195 |
| 8.4 | Total Flavonoid Content of Turmeric Powder Product | 196 |
| 8.5 | Anti-oxidant Activity of Turmeric Powder Product | 196 |
| 8.6 | Curcumin Content of Turmeric Powder Product | 197 |
| 8.7 | Colour Indices of Turmeric Powder Product | 198 |
| 8.8 | Statistical Analysis | 199 |
| 8.9 | Fourier Transform Infrared Spectroscopy Analysis | 200 |
| 8.10 | Sensory Data of Turmeric based Golden Milk Product | 201 |

| | | |
|-------------|--|----------------|
| 8.11 | Nutritional Characteristics of Lakadong Turmeric Based Golden Milk Product | 204 |
| 8.12 | Summary | 205 |
| | Chapter 9: Conclusions and Future Work | 207-212 |
| 9.1 | Conclusions | 207 |
| 9.1.1 | Techno-economic Efficacy of Refractance Window Dried <i>Curcuma longa</i> | 207 |
| 9.1.2 | Process and Product Characteristics of Refractance Window Dried <i>Curcuma longa</i> | 208 |
| 9.1.3 | Response Surface Methodology Based Optimality of Refractance Window Drying Process Parameters | 208 |
| 9.1.4 | Fortification of Refractance Window Dried Turmeric Powder and its Associated Studies | 209 |
| 9.1.5 | Storage and Associated Studies of Unfortified and Fortified <i>Curcuma longa</i> Powder and Golden Milk Products Samples | 210 |
| 9.1.6 | Nutritional Characteristic of Fresh, Tray and Refractance Window dried Lakadong Turmeric Powder and Golden Milk Product | 210 |
| 9.2 | Future Research Directions | 211 |
| | References | 213-226 |
| | List of Publications | 227 |
| | Appendix A: Calibration Curves for Determination of Total Phenolic Content, Total Flavonoid Content, Curcumin Content, Folic Acid Content | 229-230 |

| | |
|--|----------------|
| Appendix B: Summary of Conceptual Cost and Economic Assessment Model for Laboratory Scale Turmeric Oven, Tray and Refractance Window Drying Methods | 231-234 |
| Appendix C: Images of Equipment and Samples used in this Ph.D. Studies along with the Products Obtained | 235-238 |
| Appendix D: Sample Calculation for the Evaluation of Various Parameters | 239-240 |





List of Tables

| Table No. | Table Caption | Page No. |
|---------------|--|----------|
| Table 1.1 | Literature data summary of vegetables and fruit samples subjected to refractance window drying. | 15 |
| Table 1.2 | Literature data summary on the sensitivity of mylar film thickness. | 17 |
| Table 1.3 | Literature data summary of modelling approaches based system analysis. | 19 |
| Table 1.4 (a) | Literature data summary of mineral fortified powder products. | 24 |
| Table 1.4 (b) | Literature data summary of turmeric fortified liquid food products. | 24 |
| Table 1.5 | Storage and shelf-life data summary of dried vegetable produces. | 28 |
| Table 2.1 | Summary of fitness models deployed for the investigation of thin layer drying kinetics. | 49 |
| Table 2.2 | RSM design based data set summary for various combinations of temperature, air-velocity and drying time. | 55 |
| Table 2.3 | The 9-point hedonic scale for sensory analysis. | 67 |
| Table 3.1 | Yield and rehydration ratio data of oven, tray and refractance window dried turmeric powder products. | 75 |
| Table 3.2 | Moisture content data of dried <i>Curcuma longa</i> with alternate drying methods. | 75 |
| Table 3.3 | Colour indices of oven, tray and refractance window dried turmeric samples. | 82 |
| Table 3.4 | Sensitivity characteristics of responses for (a) refractance window (b) tray and (c) oven dried turmeric samples. | 83 |
| Table 3.5 | The ANOVA based statistical data summary for various cases (a) AA of slice, (b) AA of paste, (c) TFC of slice, (d) TFC of paste, (e) TPC | 85 |

| | | |
|-----------|--|-----|
| | of slice, (f) TPC of paste, (g) CC of slice, (h) CC of paste, (i) MC of slice, (j) MC of paste, (k) colour indices of slice and (l) colour indices of paste samples of <i>Curcuma longa</i> . | |
| Table 3.6 | Energy consumption data of oven, tray and refractance window drying of turmeric system. | 88 |
| Table 3.7 | A summary of best data of tray, oven and refractance window dried turmeric products. | 91 |
| Table 4.1 | Fitness and model parameters of best fit model to represent refractance window drying kinetics. | 100 |
| Table 4.2 | Moisture diffusivity and activation energy data of refractance window dried <i>Curcuma longa</i> for various combinations of temperature and film thickness. | 101 |
| Table 4.3 | Equilibrium moisture content data of refractance window dried 1 mm thick <i>Curcuma longa</i> slices. | 103 |
| Table 4.4 | Colour index data summary of dried <i>Curcuma longa</i> samples at various combinations of drying temperature and mylar film thickness. | 108 |
| Table 4.5 | ANOVA based statistical analysis data summary for various parameters and mylar film thickness cases: (a) AA, 150 μm , (b) AA, 250 μm , (c) AA, 350 μm , (d) TFC, 150 μm , (e) TFC, 250 μm , (f) TFC, 350 μm , (g) TPC, 150 μm , (h) TPC, 250 μm , (i) TPC, 350 μm , (j) CC, 150 μm , (k) CC, 250 μm (l) CC, 350 μm , (m) MC, 150 μm , (n) MC, 250 μm and (o) MC, 350 μm , (p) colour indices, 150 μm , (q) colour indices, 250 μm and (r) colour indices, 350 μm . | 110 |
| Table 4.6 | Comparative summary of optimal process-product characteristics of <i>Curcuma longa</i> dried samples. | 115 |
| Table 5.1 | Box-Behken Design based data summary of refractance window dried <i>Curcuma longa</i> system. | 122 |

| | | |
|-----------|--|-----|
| Table 5.2 | ANOVA data summary of best fit model to represent anti-oxidant activity of BBD-RSM based RWD of <i>Curcuma longa</i> . | 129 |
| Table 5.3 | ANOVA data summary of best fit model to represent total phenolic content of BBD-RSM based RWD of <i>Curcuma longa</i> . | 130 |
| Table 5.4 | ANOVA data summary of best fit model to represent total flavonoid content of BBD-RSM based RWD of <i>Curcuma longa</i> . | 131 |
| Table 5.5 | ANOVA data summary of best fit model to represent curcumin content of BBD-RSM based RWD of <i>Curcuma longa</i> . | 132 |
| Table 5.6 | ANOVA data summary of best fit model to represent moisture content of BBD-RSM based RWD of <i>Curcuma longa</i> . | 133 |
| Table 5.7 | ANOVA data summary of best fit model to represent colour indices of BBD-RSM based RWD of <i>Curcuma longa</i> . | 134 |
| Table 5.8 | Control, RSM based experimental and RSM based numerically optimized data summary of <i>Curcuma longa</i> samples. | 136 |
| Table 5.9 | Literature and best results data summary for RSM design based RWD of <i>Curcuma longa</i> . | 136 |
| Table 6.1 | Bulk density data of unfortified and fortified turmeric powder products. | 141 |
| Table 6.2 | Hygroscopicity data of unfortified and fortified turmeric powder products. | 142 |
| Table 6.3 | Solubility and swelling power data of unfortified and fortified turmeric powder products. | 143 |
| Table 6.4 | Dispersion time data of unfortified and fortified turmeric powder products. | 144 |
| Table 6.5 | Water binding capacity data of unfortified and fortified turmeric powder products. | 144 |

| | | |
|---------------|---|-----|
| Table 6.6 | <i>L</i> , <i>a</i> , and <i>b</i> values of unfortified and fortified RW dried turmeric powder samples. | 146 |
| Table 6.7 | Statistical analysis data of dried turmeric products (a) solubility, (b) swelling power, (c) hygroscopicity, (d) dispersion time, (e) water binding capacity, (f) bulk density and (g) colour indices. | 146 |
| Table 6.8 | Particle size, polydispersity index and zeta potential data summary for unfortified and fortified refractance window dried turmeric powder samples. | 158 |
| Table 6.9 | Comparative data summary of fortification and associated characteristics of dried turmeric products. | 162 |
| Table 7.1 | GAB model fitness parameters for refractance window (RW) dried, folic acid fortified RW dried and NaFeEDTA fortified RW dried turmeric powder samples. | 167 |
| Table 7.2 | A summary of nutritional characteristic of (a) unrefrigerated golden milk product at 24 hr and (b) refrigerated golden milk products at 24 and 48 hr. | 176 |
| Table 7.3 (a) | Time dependent moisture content (%) data of stored refractance window dried turmeric powder products. | 178 |
| Table 7.3 (b) | Time dependent anti-oxidant activity (%) data of stored refractance window dried turmeric powder products. | 178 |
| Table 7.3 (c) | Time dependent total phenolic content (mg GAE/g sample) data of stored refractance window dried turmeric powder products. | 179 |
| Table 7.3 (d) | Time dependent total flavonoids content (mg quercetin/g sample) data of stored refractance window dried turmeric powder products. | 179 |
| Table 7.3 (e) | Time dependent curcumin content (% w/w) data of stored refractance window dried turmeric powder products. | 180 |

| | | |
|---------------|---|-----|
| Table 7.3 (f) | Time dependent alteration of colour indices of refractance window dried turmeric powder products. | 181 |
| Table 7.3 (g) | Time dependent alteration of folic acid content (mg/100 g sample) of refractance window dried turmeric powder products. | 181 |
| Table 7.3 (h) | Time dependent alteration of iron content (mg/100 g sample) of refractance window dried turmeric powder products. | 183 |
| Table 7.3 (i) | Time dependent alteration of bulk density (g/mL) of refractance window dried turmeric powder products. | 183 |
| Table 7.3 (j) | Time dependent alteration of solubility (%) of refractance window dried turmeric powder products. | 183 |
| Table 7.3 (k) | Time dependent alteration of swelling power (g/g) of refractance window dried turmeric powder products. | 184 |
| Table 7.3 (l) | Time dependent alteration of water binding capacity (%) of refractance window dried turmeric powder products. | 184 |
| Table 7.3 (m) | Time dependent alteration of dispersion time (s) of refractance window dried turmeric powder products. | 185 |
| Table 7.3 (n) | Time dependent alteration of hygroscopicity (g/100g) of refractance window dried turmeric powder products. | 185 |
| Table 7.4 | ANOVA based statistical analysis data for various cases (a) moisture content, (b) anti-oxidant activity, (c) total phenolic content, (d) total flavonoids content, (e) curcumin content, (f) colour indices, (g) iron content, (h) folic acid, (i) bulk density, (j) solubility, (k) swelling power, (l) water binding capacity, (m) dispersion time and (n) hygroscopicity | 187 |
| Table 7.5 (a) | A summary of storage data of various dried products and turmeric powder products. | 191 |
| Table 7.5 (b) | Data summary of best findings of fortified liquid food products. | 191 |

| | | |
|-----------|---|-----|
| Table 8.1 | Moisture content data of fresh, tray dried and refractance window dried Lakadong turmeric slice samples. | 194 |
| Table 8.2 | Total phenolic content (mg GAE/g sample) data of fresh, tray dried and refractance window dried Lakadong turmeric slice samples. | 195 |
| Table 8.3 | Total flavonoid content (mg quercetin/g sample) data of fresh, tray dried and refractance window dried Lakadong turmeric slice samples. | 196 |
| Table 8.4 | Anti-oxidant activity (%) data of fresh, tray dried and refractance window dried Lakadong turmeric slice samples. | 197 |
| Table 8.5 | Curcumin content (% w/w) data of fresh, tray dried and refractance window dried Lakadong turmeric slice samples. | 198 |
| Table 8.6 | Colour indices data of fresh, tray dried and refractance window dried Lakadong turmeric slice samples. | 198 |
| Table 8.7 | ANOVA based statistical data for various cases of Lakadong turmeric samples (a) moisture content, (b) total phenolic content, (c) total flavonoid content, (d) anti-oxidant activity, (e) curcumin content and (f) colour index (<i>L</i> values). | 199 |

List of Figures

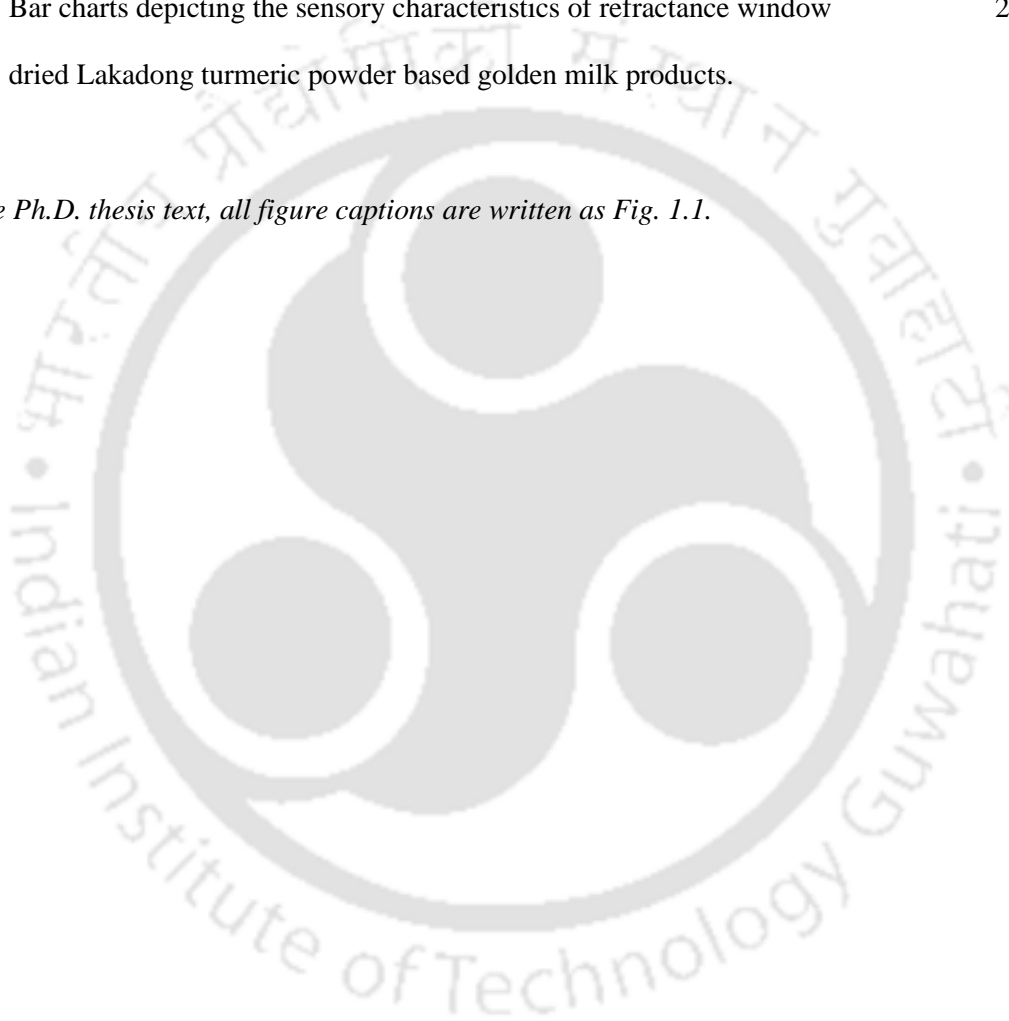
| Fig. No. | Figure Caption | Page No. |
|----------|--|----------|
| Fig. 1.1 | Representation of the production of garlic, turmeric, chilies and pepper from the year 2012 to 2018 in North Eastern States of India. | 3 |
| Fig. 1.2 | A schematic of conventional turmeric powder production process. | 7 |
| Fig. 1.3 | A schematic of refractance window drying set up. | 7 |
| Fig. 3.1 | Total phenolic content of tray, oven and refractance window dried turmeric slice and paste samples. | 77 |
| Fig. 3.2 | Total flavonoids content of tray, oven and refractance window dried turmeric slice and paste samples. | 78 |
| Fig. 3.3 | Antioxidant activity of tray, oven and refractance window dried turmeric slice and paste samples. | 79 |
| Fig. 3.4 | Curcumin content of tray, oven and refractance window dried turmeric slice and paste samples. | 81 |
| Fig. 3.5 | Total annualized conceptual cost (Rs per kg of dried sample) of laboratory scale dried turmeric system and with oven, tray and refractance window drying process | 89 |
| Fig. 3.6 | Bar diagrams depicting percentage contributions of electricity cost, manpower cost, equipment cost, maintenance cost and depreciation cost to the total conceptual annualized cost of alternate drying methods | 91 |
| Fig. 4.1 | Moisture ratio (MR) vs drying time profiles of RWD <i>Curcuma longa</i> samples for various mylar film thickness cases (a) 125, (b) 250 and (c) 350 μm . | 99 |
| Fig. 4.2 | Fitness plots of the logarithm of moisture ratio versus drying time for various cases of mylar film thickness (a) 125, (b) 250 and (c) 350 μm . | 101 |

| | | |
|----------|---|-----|
| Fig. 4.3 | Arrhenius plots for moisture Diffusivity estimation for various cases of mylar film thickness (a) 125, (b) 250 and (c) 350 μm . | 102 |
| Fig. 4.4 | Effect of drying temperature and mylar film thickness on the total phenolic content of refractance window dried <i>Curcuma longa</i> . | 104 |
| Fig. 4.5 | Effect of drying temperature and mylar film thickness on the total flavonoids content of refractance window dried <i>Curcuma longa</i> . | 105 |
| Fig. 4.6 | Antioxidant activity characteristics of refractance window dried <i>Curcuma longa</i> . | 106 |
| Fig. 4.7 | Curcumin content variation with drying temperature and mylar film thickness. | 107 |
| Fig. 4.8 | The FTIR spectra of dried turmeric powder products obtained at various water bath temperature values. | 109 |
| Fig. 5.1 | Response surface plot of anti-oxidant activity of refractance window dried turmeric samples and for various binary combinations (a) temperature and air velocity, (b) temperature and drying time and (c) air velocity and drying time. | 129 |
| Fig. 5.2 | Response surface plot of total phenolic content of refractance window dried turmeric samples and for various binary combinations (a) temperature and air velocity, (b) temperature and drying time and (c) air velocity and drying time. | 130 |
| Fig. 5.3 | Response surface plot of total flavonoid content of refractance window dried turmeric samples and for various binary combinations (a) temperature and air velocity, (b) temperature and drying time and (c) air velocity and drying time. | 131 |
| Fig. 5.4 | Response surface plot of curcumin content of refractance window dried turmeric samples and for various binary combinations (a) temperature | 132 |

- and air velocity, **(b)** temperature and drying time and **(c)** air velocity and drying time.
- Fig. 5.5 Response surface plot of moisture content of refractance window dried turmeric samples and for various binary combinations **(a)** temperature and air velocity, **(b)** temperature and drying time and **(c)** air velocity and drying time 133
- Fig. 5.6 Response surface plot of colour indices of refractance window dried turmeric samples and for various binary combinations **(a)** temperature and air velocity, **(b)** temperature and drying time and **(c)** air velocity and drying time. 134
- Fig. 6.1 FTIR spectra of unfortified and fortified turmeric powder product samples. 150
- Fig. 6.2 Mass loss vs temperature plots for **(a)** refractance window (RW) dried, **(b)** folic acid fortified RW dried and **(c)** NaFeEDTA fortified RW dried turmeric powder samples. 150
- Fig. 6.3 Heat flow vs temperature plots for **(a)** refractance window (RW) dried, **(b)** folic acid fortified RW dried and **(c)** NaFeEDTA fortified RW dried turmeric powder samples. 152
- Fig. 6.4 XRD spectral diagrams of **(a)** refractance window (RW) dried, **(b)** folic acid fortified RW dried and **(c)** NaFeEDTA fortified RW dried turmeric powder samples. 154
- Fig. 6.5 Scanning electron microscope image of **(a)** RW dried, **(b)** folic acid fortified RW dried and **(c)** NaFeEDTA fortified RW dried turmeric powder products. 156
- Fig. 7.1 Moisture content alteration and water activity for **(a)** refractance window (RW) dried **(b)** folic acid fortified RW dried and **(c)** NaFeEDTA fortified RW dried turmeric powder samples. 167

| | | |
|----------|---|-----|
| Fig. 7.2 | Permeability plot for moisture gain by silica gel with time. | 167 |
| Fig. 7.3 | Bar chart depicting sensory characteristics of refractance window dried turmeric powder based milk products for varied turmeric constitution cases (a) 0 g, (b) 0.5 g, (c) 1 g, (d) 1.5 g and (e) 2 g in 100 mL milk. | 174 |
| Fig. 8.1 | FTIR spectra of fresh, tray and refractance window dried Lakadong turmeric powder samples. | 201 |
| Fig. 8.2 | Bar charts depicting the sensory characteristics of refractance window dried Lakadong turmeric powder based golden milk products. | 203 |

Note: In the Ph.D. thesis text, all figure captions are written as Fig. 1.1.



Abbreviations

| | |
|----------|---|
| AAS | Atomic Absorption Spectrophotometer |
| BBD | Box-Behnken Design |
| BET | Brunauer-Emmett-Teller |
| CCD | Central Composite Design |
| DSC | Differential Scanning Calorimetry |
| FESEM | Field-Emission Scanning Electron Microscopy |
| FTIR | Fourier Transform Infrared |
| GAB | Guggenheim-Anderson-de Boer |
| HDPE | High Density Polyethylene |
| LDPE | Low Density Polyethylene |
| NaFeEDTA | Sodium Ferric Ethylenediaminetetraacetate |
| PET | Polyethylene Terephthalate |
| PP | Polypropylene |
| RSM | Response Surface Methodology |
| TGA | Thermogravimetric Analyzer |
| XRD | X-Ray Diffraction |

Nomenclature

| | |
|-----|------------------------------|
| AA | Anti-oxidant Activity |
| CC | Curcumin Content |
| db | dry basis |
| EMC | Equilibrium Moisture Content |
| MC | Moisture Content |

| | |
|-----|--------------------------------|
| MR | Moisture Ratio |
| O | Oven dried |
| OP | Oven dried Slice |
| OS | Oven dried Paste |
| RP | Refractance window dried Paste |
| RS | Refractance window dried Slice |
| RW | Refractance Window |
| RWD | Refractance Window Drying |
| T | Tray dried |
| TFC | Total Flavonoids Content |
| TP | Tray dried Paste |
| TPC | Total Phenolic Content |
| TS | Tray dried Slice |
| wb | wet basis |

Notations

| | |
|-----------------|---|
| W_1 | Weight of fresh sample (g) |
| W_2 | Weight of dried sample (g) |
| A_1, A_2 | Absorbance of control and sample respectively |
| W_f | Weight of fresh sample (g) |
| W_s | Weight of RW dried sample (g) |
| a, b, c, k, n | Drying model parameters |
| M_t, M_e, M_o | Moisture content at any time, equilibrium time and initial time (min) |
| D_{diff}, D | Moisture diffusivity and effective moisture diffusivity (m^2/s) |
| x | Sample thickness (mm) |
| t | Drying time (min) |

| | |
|--------------------|--|
| E_a | Activation energy (kJ/mol) |
| R | Gas constant |
| T | Temperature |
| A, B, C | RSM based independent variables: water bath temperature, air velocity and drying time respectively |
| W_s, W_d, W_{sd} | Weight of original sample weight, dried residue and dried sediment mass (g) |
| M_w, M_d | Weight of a wet and dry weight basis of the powder (g) |
| x | Enhancement in powder sample (g) |
| a_h | Powder sample amount being used for the measurement (g) |
| W_i | The water content of the powder being exposed to the humid environment |
| C, K | Moisture isotherms constant parameters |
| M_o, M | Moisture isotherms monolayer moisture content and EMC respectively |
| θ | Shelf-life (days) |
| W_s | Weight of dry solids (g) |
| P^* | Saturated vapour pressure of water at ambient temperature (Pa) |
| K | Water vapour permeability of the packaging material (kg/m ² dayPa) |
| A | Area of the package (m ²) |
| RH | Relative humidity of the environment in which the package is placed (%) |
| a_w | Water activity of the powder |
| X_i | Initial moisture content (% db) |
| X_c | Critical moisture content (% db) |





Chapter 1:
Introduction and Literature Review



Introduction and Literature Review

In this chapter, the first section (section 1.1) provides a brief overview on the significance of horticultural produces and their associated preservation. Following this, the section devotes towards the prominence of thermal processing and horticultural produced based functional food product development. Thereafter, the section devotes towards the turmeric product, its usage and the associated customized products. In the following section (section 1.2), the available prior-art with respect to four primary areas of research namely overview of the refractance window drying process, alternate refractance window drying schemes, heat transfer characteristics of the contact film and the technical and economic efficacy of refractance window drying process. Thereafter, section 1.3 details upon the prior art in the field of turmeric processing, fortification and product development. Eventually, in section 1.4, the thesis summarizes the associated lacunae in the chosen fields of study and augments towards the possible scope for further research. Based on such wider subjective analyses of available prior art, the thesis objectives have been set as mentioned in section 1.5. Finally, a brief account of the thesis organization has been presented in section 1.6 of the chapter.

1.1 Preamble

1.1.1 Significance of Horticultural Produces

Sustainable economy and holistic well-being of humankind translates into adequate nutrition based horticultural produces. Being rich resources of carbohydrates, protein, dietary fiber, vitamins and minerals, the vegetables and tubers regulate and nourish human health. With about 14 % of total vegetable produces, India is next to China in terms of vegetables production volume. Thereby, the vegetables exports accounted to 55 % of total exports of India in 2015 – 2016 (Indian horticulture database 2015 – 2016, National Horticulture Board Ministry of Agriculture, Government of India; Sahni and Shere, 2017).

1.1.2 Relevance of Horticultural Preservation Technologies

Due to the perishable nature of vegetables, their shelf life is often enhanced through techniques such as canning, drying and irradiation. These aim to reduce microbial activity through the moisture removal or the hosting modification of environment (Nindo et al., 2003).

Among several methods, thermal methods such as drying have promising features such as elimination of refrigeration cost, packaging materials and preservatives; ease of transportation and affordable processing cost. However, a detrimental issue in drying is with respect to associated trade-off in nutrition, as heat sensitive desired constituents do get deteriorated in due course of the long drying duration (Karam et al., 2016).

1.1.3 Search for Thermal Processing Technologies Prompting Nutritional Retention of Produce

The North-East India with its rich biodiversity and agro-climate zone is a host to varieties of endemic and conventional horticultural produce. Spices constitute a substantial produce in the region (Fig. 1.1). The region's vegetables produce in the winter season are comparatively higher than those in the summer and monsoon months. Contextually, the most inexpensive sun drying preservation process is disadvantageous and irrelevant from the perspective of region's sub-tropical winter climate, long durations (> 30 h) of sun drying, poor nutrition of the sun-dried product quality, labour intensive process and possible microbial infestation (Mondol et al., 2020). As an alternative, hot air drying through oven or tray drying are often adopted to achieve good quality dried horticultural produce at an affordable cost. However, even tray drying process consumes about 4 – 10 h drying time to achieve dried vegetables with appropriate shelf-life

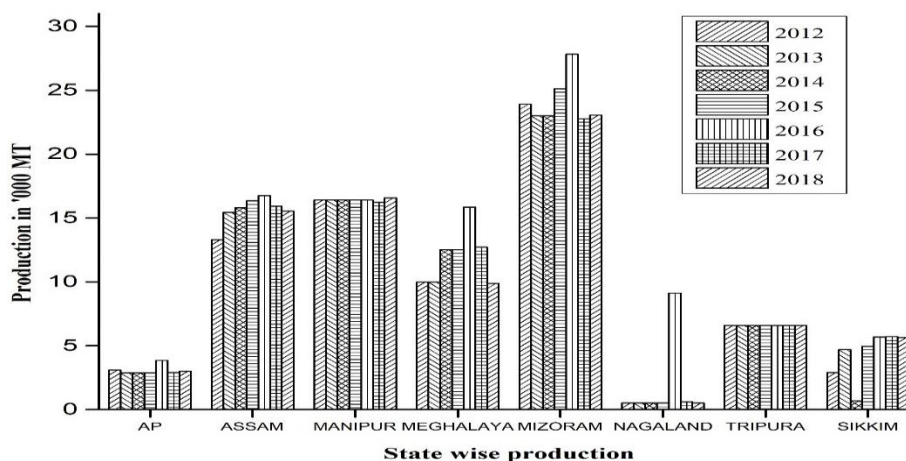


Figure 1.1: Representation of the production of garlic, turmeric, chilies and pepper from the year 2012 to 2018 in North Eastern States of India (Source: <https://nhb.gov.in/>).

(< 8 % moisture content). Thus, quicker thermal schemes with processing duration by about 1.5 – 2 h would be beneficial to achieve dried products with comparatively better nutritional characteristics (Abbssaid et al., 2013).

1.1.4 Horticultural Produce Based Functional Food Development

Through their adequate consumption, functional foods refer to those being fortified with specific constituents that enhance human nutritional and physiological markers. Technology based constitutional variations of a food product thereby surpasses traditional nutritional value of its major yet individual ingredients. In general, functional foods often target either improving general bodily conditions (pre and probiotics) or reducing disease risks (cholesterol mitigating products) or curing illness or customizing products making life easier (gluten free). Greater acceptance of functional foods in the society has been due to ever increasing healthcare costs, steady enhancement in life expectancy, and sustained desire of old aged community to secure quality life style and habitat (Khan et al., 2013).

Early functional food product development targeted fortification with vitamins (C/E/folic acid), iron and calcium. Thereafter, the functional food product research emphasized on the fortification with

micronutrients such as omega-3 fatty acid, phytosterol and soluble fiber and thereby promote good health or prevent disease. Recent emphasis on functional foods attempt to achieve food products with multiple health benefits (Jones and Jews, 2007; Iwatani and Yamamoto, 2019). Horticultural produce based functional foods aim to optimize constitution of local and exotic vegetables, spices and herbs and thereby achieve sensory as well as nutritionally rich value-added product. Invariably, the processing scheme such as drying will have its influence upon the product quality. Hence, individual, collective and hierarchical processing and formulation research need to be simultaneously addressed in due course of horticultural produce-based food product development. Laboratory and bench marks trails need to be evolved as cost competitive industrial process-product schemes. Bio-active constituents are often desired in such products. Needless to convey, aromatic and medicinal plants of North-East India provide huge scope for product development, improvised standardization and formal acceptance in the consumer market.

Food product intermediates can be broadly classified as seeds, juices, pickles, fermented foods, powder foods, dried foods etc. These are often not studied for their compatibility with horticultural produces. Hence, relevant technologies are to be adopted and optimized from a product quality assurance perspective. Considering such pedagogy, anti-oxidants, bio-active constituents and minerals rich natural produces are often being targeted for integrated products such as soups, pickles, instant powder, chips chutneys, sauces and dips. Sensory, taste, texture, flavour and acceptability are usually targeted in such product analyses along with suitable control samples.

Notable research methodologies in the field of food product intermediates and food products refer to **(a)** the role of processing scheme and its parameters optimality to achieve desired products (for example, Mulet et al., (2007) used hot air drying method to dry turmeric rhizomes while Pedreschi and Moyano, (2005) studied the effect of pre drying on the texture of potato chips), **(b)** enriched formulation research (for example, Ahouagi et al., (2021) developed a strawberry-enriched ketchup sauces with higher constitution of phenolics, total flavonoids, antioxidant activity), **(c)** modelling studies associated to optimal process-product characteristics of horticultural products (for example, Shishir et al., (2016) used RSM modelling to optimize the spray drying parameters for pink guava powder). Dry powder formulation

constitutes an important class of horticultural produce-based food products. These include baby food, cheese/whey products, coffee, coffee substitutes, tea extract, egg powder, flavours, soup mixes, soy based food powders, spices/herb extracts, vegetable protein, vegetable powders etc.

To a large extent, the vegetable based powder food formulations require thermal processing. Hence, thermal processes facilitating shorter drying time tend to enhance bio-active and nutritional constitution of dried product. These shall be investigated from the perspective of process-product characteristics i.e., optimal choice of processing parameters of such processes for desired optimality in terms of sensory nutrition, appearance and their combinations. In summary, vegetable based functional powdered food products cannot ignore the process parameters that substantiate upon product quality and do not negate in terms of the nutritional aspects. Refractance window drying (RWD) is one such process that promotes the high retention of nutritional characteristics. Studies carried out till date affirm very good quality products. In the notable prior art, the RWD based tomato powder possessed better quality than that being produced through the convective drying method (Abul and Ghanem, 2011). Similarly, Hernandaz-Santos et al., (2016) confirmed better quality of RWD dried carrot slices in comparison with those being obtained with the convective drying method.

1.1.5 Turmeric Production and its Usage

Among various horticultural produces of North-East India, *Curcuma longa* (Turmeric) is one of the promising produces that can be effectively transformed into customized value added product. While India produces 83 % of world turmeric, the North-East India produced about 13 % (De, 2017). Further, the North-Eastern region also produces the Lakadong variety of turmeric that possesses very high curcumin content (7.4 – 8.6 %). Apart from direct utility as a spice, turmeric has numerous applications in food, cosmetic and medicinal products. Turmeric is primarily utilized as a medicinal constituent for its pharmacological effects such as antioxidant (Quiles et al., 2002), anti-inflammatory, antimutagenic, anticarcinogenic and antimicrobial characteristics (Prasad and Aggarwal, 2011). Turmeric is also used in

alternate medicinal systems for rheumatoid arthritis, chronic anterior uveitis, conjunctivitis, small pox, chicken pox, wound healing urinary tract infections, and liver ailments (Dixit et al., 1988). Thereby, herbal medicine recommends its usage as a key ingredient. Turmeric is the recommended choice for digestive disorders; reduction of flatus, jaundice, menstrual difficulties, and colic mitigation of abdominal pain and distension (Bundy et al., 2004); and for dyspeptic conditions being symptomized with loss of appetite, postprandial feelings of fullness, and liver and gall bladder malfunctioning scenarios (Mills and Bone, 2000). Clinical utility of turmeric is towards the mitigation and treatment of several diseases such as familial adenomatous polyposis in the intestines (Cruz-Correa et al., 2006), inflammatory bowel diseases (Hanai and Sugimoto, 2009), colon cancer (Naganuma et al., 2006), arthritis (Fetrow and Avila, 2001) and dyspepsia (Blumenthal et al., 2000).

1.1.6 Turmeric Based Customized Products

Turmeric rhizomes are often converted into intermediate products. These are eventually utilized in the development of high end value added products. Alternate intermediate products based on turmeric rhizomes primarily refer to (a) extracts such as oils and curcumin (b) turmeric juice (c) turmeric paste and (d) turmeric powder. Various extraction and processing techniques are often adopted towards the production of these intermediate products. These include (a) extraction methods like low pressure solvent extraction, soxhlet extraction and hydro-distillation for oils and curcumin extract products (b) blender based pressing for turmeric juice products (c) grinding in a mixer to achieve turmeric paste products and (d) drying processes such as hot air oven, tray, cabinet and convective drying for turmeric powder production (Braga et al., 2003). Several processes are followed hierarchically in the development of a turmeric product. For example, turmeric powder is produced from turmeric rhizomes through the hierarchy of curing, drying (sun drying) and grinding. Conventional method to produce turmeric powder from *Curcuma longa* rhizomes involves curing in water or 0.05 - 0.1 % alkaline water for 40 - 45 min and subsequent sun drying for 10 – 15 days and grinding (Fig. 1.2).

The intermediate products are often further customized towards desired product palette. Fresh, powdered and juice intermediate products have further customization towards turmeric juice, milk fortification, fruit and vegetable juice fortification, constituent and/or fortificants in curry powder and pickles, butter and cheese, milk latte and tea. Further, turmeric intermediate products also have applications in traditional cosmetics. Turmeric paste is also used as a key ingredient in medicines and anti-inflammatory hair oils, skin oil etc. In the recent past, turmeric has been considered as an important constituent in the development of enriched value added functional and nutraceutical products. From food product perspectives, the choice ranges from golden milk, milk latte, tea, high quality turmeric powders, essentials oils, seasonings, chips, crackers, turmeric powder and juice blend, pickles, health drinks, turmeric drops, curcumin powder, candies, cookies and soup mix. On the other hand, in the field of cosmetics and medicinal products, these have been customized as beauty creams, body creams, ointments, medicinal creams, soaps, band-aids, capsules, tablets, toothpaste, gel, serum, mask, oils etc., (Lantz et al., 2014; Pawar et al., 2017; Bhowmik et al., 2009).

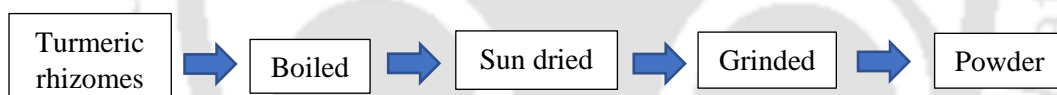


Figure 1.2: A schematic of conventional turmeric powder production process.

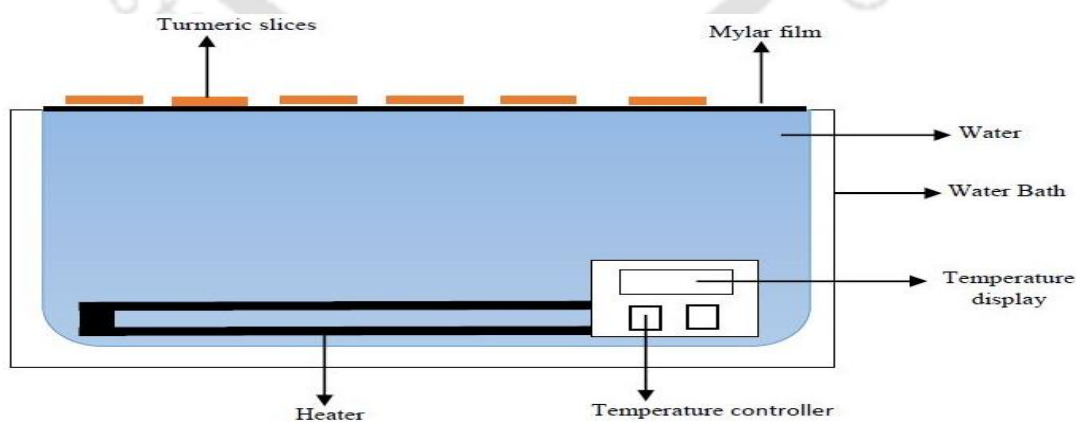


Figure 1.3: A schematic of refractance window drying set up.

1.2 An Overview of Refractance Window Drying process

1.2.1 Working Principle

Being a novel drying method, the refractance window drying (RWD) process involves a hot medium such as water that is kept in contact with a plastic interface to facilitate heat transport as per conduction and radiation modes of heat transfer. In the RWD, conduction due to the direct contact of hot water with the plastic interface is facilitated in the RWD process. Typically, hot water at 90 – 95 °C serves as the hot medium to provide heat to the interface that hosts the material to be dehydrated. Thereby, sample temperature can be kept below 70 °C (Abonyi et al., 2002). Juices, purees, sliced foods and suspensions can be dried using RWD by placing them on the plastic film in contact with hot water at its temperature just below the boiling point (Abul-Fadl et al., 2011).

During RWD process (Fig. 1.3), all three modes of heat transfer namely conduction, convection and radiation are active (Ochoa Martinez et al., 2012) and thereby enhance and intensify moisture removal from the sample in short drying duration (Hernandez-Santos et al., 2016). While conduction is apparent due to the material being touch with the plastic film placed on hot water, it is interesting to note that the plastic film (Mylar film) has specific propensity to absorb electromagnetic radiation in the wavelength range of 3.0 to 15.3 μm . Thereby, the sample being kept on a plastic film such as a mylar film facilitates minimal refraction at the sample-mylar film interface. This in turn enables and creates a heat transfer window due to the selective passage of the infrared radiation through the sample. Thereby, other segments of the film that do not host the sample do not get radiated.

According to the inventors of the RW drying system, the infrared transmission is stronger when the plastic interface is in intimate contact with water on one side and a moisture-laden material is kept on the other side. Thus, at the sample portion, the refraction is minimized and radiant thermal energy passes through the film and reaches the product (Nindo and Tang, 2007).

The dried sample's thickness and moisture content do influence its absorptivity of heat. Further, the thermal energy transfer from the product to the ambient air is primarily by convection and through

evaporative cooling of the dried product. Such an evaporation is very intense and constitutes a major portion of the consumed energy during RWD. In other words, in due course of time, as the moisture content of the dried sample gets reduced, the infrared window eventually closes due to diminished infrared radiation transport to the sample. Thereby, for the dried sample, the refracted infrared radiation enhances refractive index of the material. Eventually, conduction overtakes radiation as the predominant mode of heat transfer for the sample with minimum moisture content (Karadbhajne et al., 2019). Thus, all three heat transfer modes are active and dominant at the hot water-film interface. However, conduction and radiation occur through the film and convection occurs at the air-film surface.

Due to these coupled influences, the RWD process fosters a significant reduction in drying time from about 6 – 10 h to 1 – 2 h. Such shorter drying time facilitates better retention of nutritional parameters in the dried product due to effective drying at lower processing temperature (Nindo and Tang, 2007).

During the final drying phase of the RW dried product, due to the product being almost dry, heat transfer by conduction becomes predominant and the rate of heat transfer to the product slows as the product dries further. A cooling section at the discharge end of the RW dryer is often facilitated to reduce the product temperature and preferably reach a state below the glass transition temperature of the product. This is a promising feature to facilitate dried product with better nutritional retention.

1.2.2 Alternate RWD Operational Schemes

Magoon, (1986) patented a RWD apparatus for the effective moisture removal from pulp and fruit juice samples. It constituted a water reservoir being shaped as an elongated trough. The trough was supported with a polyester film on its surface and a conveyor belt. The dried samples were carried out by the film for effective drying being facilitated through the hot water. A heater regulated the water temperature at a desired set point. Bolland, (2000) improved Magoon's patented RWD design by including a cold water tank at the outlet end of the dryer for dried product cooling. Thereby, final product degradation can be mitigated. Further, the design also includes a system for the generated vapour removal during drying.

For their customized food processing research, Nindo et al., (2003) deployed a commercial full-scale RW dryer (Model 2, MCD Technologies Inc., Tacoma, WA, USA). The unit was about five times the length of a pilot scale RW dryer. Mylar film plastic endless belt (1.41 m width and 0.2 mm thickness) was deployed. Prior to the water heating section that constituted four water circulation compartments, puree samples were placed at the entrance of the drier. Ocoro-Zamora and Ayala-Aponte, (2005) fabricated a prototype RW dryer for the drying of papaya puree. The configuration referred to a water tank (0.6 x 0.4 x 0.1 m) with a water heater to achieve water temperature of 70 °C. Mylar™ type polyester membrane being transparent to infrared radiation slid over the water surface at 1 mm/min speed. Water re-circulation was also considered to facilitate effective reuse of the heated water and thereby save energy. Hernandez-Santos et al., (2016) developed a laboratory scale RWD setup (for carrot slices RWD studies) by placing 0.017 cm thick mylar plastic film on the surface of a stainless-steel water bath. Two electrical heaters (840 W each) were used as the heating elements and the water bath temperature was controlled. The plastic surface temperature was as well monitored. Ochoa Martinez et al., (2012) deployed a 5.5 L stainless steel thermostatic control equipped water bath being set at 92.5 °C. During each experimental run, the mylar film of 1730 cm² and 0.26 mm thickness was deployed to evenly place 18 samples.

Far infrared assisted refractance window drying was investigated for the drying of apple slices. Comparatively, hot air oven drying characteristics were also assessed by the authors. The findings reported that the drying time and energy consumption respectively reduced by 69 % and 46 % in comparison with the hot air oven drying. Also, higher retention of the TPC, TFC and ascorbic acid were noted (Rajoriya et al., 2020). Baeghbali et al., (2019) conveyed that the combination of ultrasound and infrared with RWD facilitated higher drying rates and lower moisture content in the dried sample. For the case, the flavonoids content (1.72 – 1.86 mg catechin/g d.m), phenolic content (6.28 – 6.51 mg GAE/g d.m) and anti-oxidant activity (5.52 – 6.37 %) were high and were comparable to freeze drying methods. The research studies of Puente Diaz et al., (2020) inferred that the infrared assisted RW drying was able to reduce the processing time by 60 % with respect to the RWD process. Thus, the EMC was easily achieved. Invariably, the drying kinetics were influenced with the assistance of the infrared heating technique.

1.2.3 Heat Transfer Characteristics of the Contact Film

During RWD, the relative role of each heat transfer mode namely conduction, convection and radiation is dependent upon the heat transfer resistance being offered by the water (Ortiz-Jerez et al., 2015). The thermal radiation transport across the plastic film can be evaluated using the polyester film optical properties. Further, most such films are poor conductors of thermal energy.

Tsilingiris, (2003) reported that most polymer films have low transmittance with values starting from 7×10^{-12} mm but some polymer films have high transmittance with values greater than 12 mm. The minimum total transmittance of mylar film within the wavelengths of 6.5 and 14.5 mm has been evaluated by Ortiz-Jerez et al., (2015). With 0.24 W/mK thermal conductivity, 1.39 g/cm³ density and 0.28 cal/g/°C specific heat at ambient conditions, mylar is the most promising and optimal material for the RWD process (DuPont Teijin Films, 2003; Ortiz-Jerez and Ochoa-Martínez, 2015). At wavelengths of 3×10^{-8} mm, water exhibits high absorptivities and high transmissivity (~90%) to infrared radiation through Mylar films (Sandu, 1986).

1.2.4 Technical and Economic Efficacy

Several investigations affirmed that the RWD process facilitated higher retention of product quality (colour, vitamins, total phenolic content, ascorbic acid and anti-oxidants) in comparison with other conventional drying methods. According to Hernandaz-Santos et al., (2016), the RWD facilitated only 9.9 % loss of beta-carotene in carrot puree samples in comparison with the tray drying process (57 % loss). For pureed asparagus, Nindo et al., (2003) reduced moisture content from 90 to 4 % in only 4.5 min using the RWD. Nindo et al., (2003) affirmed reduced microbial ability in RWD processed pumpkin puree samples with atleast 4.6, 6.1, 6.0, and 5.5 log reductions for total aerobic counts, coliforms, *E. coli*, and *L. innocua*, respectively (latter three counts got reduced to undetectable levels). For pomegranate juice, Baeghbali et al., (2016) analysed that the RWD dried products possessed better or equal anthocyanin, colour and anti-oxidant activity those obtained with the freeze drying method. In this regard, it shall be noted that the cost

of the RWD is significantly lower than that of the freeze drying process. While tray and oven drying is effective to yield good results for the kinetics, water activity etc., they are not as effective as the RWD in terms of the retention of carotene, total phenolic, antioxidant, ascorbic and colour indices and texture.

While RWD systems have a comparable thermal efficiency with the drum drying (52 – 77 %), the hot air drying systems only have 50 % efficiency (Adamiec et al., 1995). Despite having much lower capacities than the rotary dryers, the RWD systems have comparable operational capacity with spray dryers and drum dryers (Nindo and Tang, 2007).

Evaporation refers to a major fraction of the total energy being consumed in the RWD system. Nindo and Tang, (2007) inferred that in comparison with the freeze drying process, the RWD processes incur 50 – 70 % lesser capital and 50 % lesser energy costs. Compared to the spray drying, Baeghbali et al., (2016) reported 33 % lower energy requirements for the RWD. Thus, the RWD system is a viable low cost scalable option for less intensive process operation from both capital and energy cost perspectives and achieve good quality intermediate powdered food products.

Compared to the oven drying system, Abbasid et al., (2015) inferred that the RWD system was comparatively cost effective. Similarly, Baeghbali et al., (2010) reported that the RWD dried tomatoes consume about 375 – 525 W for a batch of 150 g tomatoes. This is significantly lower than the higher energy being consumed in the freeze drying process (70 – 84 kW) for the same product. Further, for the RWD and convective drying processes, Abul-Fadl and Ghanem, (2011) inferred that the production cost of dry tomato powder was about Rs. 99.6 and Rs. 280.3 per kg for RW and convection drying respectively. In the following section, relevant prior art is detailed in various sub-themes.

1.3 Prior Art

1.3.1 Comparative Assessment of RWD Processes in Conjunction with Other Low Cost Drying Methods

Alternate drying methods include hot air oven, convective, tray, freeze, spray, vacuum, microwave and fluidized bed drying methods. Among these, due to longer drying period and lower drying rates, hot-air oven drying is not highly promising to retain taste, colour and nutritional content of the product (El-Safy, 2014). On the other hand, tray drying is an economical and a commonly deployed methods. However, it suffers from the limitations of product quality. From processing time perspective, tray drying requires about 2.5 to 5.5 h of moderately higher temperature (70 – 50 °C) operation. Comparatively, the freeze drying process demands upto 18 – 24 h at a reduced processing temper above of 20 °C (Nindo et al., 2003). From the cost perspective, it is well known that vacuum (Drouzas et al., 1999) and freeze (Nindo and Tang, 2007) drying are significantly expensive methods and are not recommended towards the production of high volume dried products at an affordable cost.

To overcome the limitations of the mentioned drying processes such as high processing time, higher cost, scalability and retention of nutritional parameters, a novel drying method such as RWD can be targeted. Ochoa-Martinez et al., (2012) targeted mango slice drying using RWD and hot air drying methods. In another study, the RWD processed haskap berry powder has been analyzed to retain 93.8 % of anthocyanin with respect to original frozen berry samples (Celli et al., 2016). Considering loquat slice system, El-Safy, (2014) evaluated the efficacy of RWD, hot air drying, solar drying and halogen oven drying. Compared to the tray drying, Abonyi et al., (2002) inferred significant reduction in beta-carotene loss of 9.9 % using the RWD process as opposed to 57 % in drum dried process for carrot puree samples. Similarly, Nindo et al., (2003) concluded that the RWD reduced the moisture content of pureed asparagus from 21 to 0.04 % db in only 4.5 mins.

However, limited investigations have been conducted with respect to RWD of vegetable resources and their comparative assessment with other conventional processes such as tray drying. Abbasid et al., (2015) inferred that for 2 mm tomato slices, the RWD process ensured higher resemblances of nutritional characteristics with respect to those being evaluated for the fresh sample. Akinola et al., (2014) concluded that the RWD process effectively reduced the moisture content of red onion slices of 1 mm from 7.19 to 0.2 kg water/kg dry solids in a short span of 40 min. Ghanem, (2010) considered similar quantities of

processed materials and indicated that the RWD based drying of liquid strawberry (strawberry juices) samples involved a reduced time span of 0.77 - 0.90 h. This was significantly high in comparison to a conventional hot air dryer (5 h and 60 °C). For the tomato powder system, Abul-Fadl and Ghanem, (2011) conducted experimental investigations using RWD and conventional drying methods and inferred that the RWD processed tomato powder possessed higher retention of antioxidant activity constituents and total flavonoids. For the carrot slices system, the RWD and convective drying processes have been compared by Hernandez-Santos et al., (2016). The authors opined that, in comparison to the convective drying process, the RWD enabled better retention of colour index, TPC and AA values in the processed product. Similarly, for the kiwi fruit system, Jafari et al., (2015) targeted a comparative assessment of RWD and oven drying systems. Thereby, the RWD has been inferred to be the best in terms of highest values associated to textural hardness, colour, rehydration index and organoleptic characteristics.

Dadhaneeya et al., (2023a) studied that the RW dried dragon fruit possessed higher nutritional quality and required lower drying time in comparison to vacuum drying and hot air oven drying processes. The RWD process took 960 min to attain a moisture content of 6.50 % (dry basis) along with the retention of higher TPC (182 mg GAE/100 g) and crude fiber content (0.98 %). Zamani et al., (2023) obtained higher combinations of total phenol content (2.7 mg GAE/g dry weight), total flavonoid content (2.26 mg QE/g dry weight), antioxidant activity (79 %), and essential oil content (0.65 %) in the RW dried *Dracocephalum kotschyi* samples. The RWD was also successful to effectively reduce the microbial contamination along with the assurance of higher colour quality, acceptable green colour and lesser browning in the dried sample. In another study, it was reported that the RWD dried broccoli, kale and spinach sample had higher total phenolic content of 8.50, 18.77 and 12.40 mg/g respectively (Vargas et al., 2022). Reporting the drying characters of dragon fruit pulp, Gautam and Abdul, (2023) conveyed that only 42 min was required to dry the sample with 3.5 mm thickness at 95 °C. The RWD study of banana sample affirmed a drying time of 760 min which is much lower in comparison to the hot air oven drying and vacuum drying processes (1125 min and 975 min drying time respectively). Also, the high retention of colour indices, total phenolic content (441mg GAE/100 gm) and crude fiber (1.92 %) was assured in the dried sample (Dadhaneeya et al., 2023b).

A critical summary of the fruits and vegetables samples being subjected to RWD processes has been presented in Table 1.1.

Till date, the RWD of *Curcuma longa* (turmeric) has not been targeted. However, few studies have been conducted by targeting the oven drying of the turmeric tuber. Among unblanched and blanched turmeric slices that were subjected to hot air drying at 55 °C, Pradeep et al., (2016) inferred superior quality of unblanched oven dried samples. Similarly, Raza et al., (2018) inferred drying process optimality in terms of prior boiling for 1 h followed with drying at 70 °C for 21 h to achieve rhizomes with a maximum curcumin content of 2.97 %.

The table clearly affirms that banana slices, tomato slices and dragon fruit pulp have been studied till date for the RWD system but not tubers such turmeric. Also, in many reported prior art, control samples were not considered.

Table 1.1: Literature data summary of vegetables and fruit samples subjected to refractance window drying.

| S. No. | System | Parameters | Independent variable range | Dependent variable range | Best results | Reference |
|--------|------------------------|------------------------|---------------------------------------|---|--|----------------------------|
| 1. | Tomatoes slices | 0.25 mm film thickness | 65 – 95 °C 2 - 3 mm pulp thickness | 4.1 – 6.5 °Brix 60 - 89% Solubility 10 – 20 min Drying time | T = 95 °C Pulp thickness = 3 mm Brix of pulp = 5.2° Solubility = 80% t = 17 mins | Castoldi et al., (2015) |
| 2. | Dragon fruit peel pulp | 0.15 mm film thickness | 95 °C 3.5 mm pulp thickness | Colour (a* value) Drying time | T = 95 °C Colour = 19.02 t = 42 mins | Gautam and Abdul, (2023) |
| 3. | Banana slices | 0.09 mm film thickness | 70 °C Slice thickness: 5- 7 mm | Drying time 4.97 – 5.63 p ^H | Thickness: 5 mm t = 780 mins 4.97 p ^H | Dadhaneeya et al., (2023b) |

1.3.2 Sensitivity of Mylar Film Thickness on the Process-Product Characteristics of the RWD

Process

During RWD, a Mylar film is deployed as a film or medium to facilitate the transport of thermal energy to the dried materials. Akinola and Ezeorah, (2016), Castoldi et al., (2015), Zotarelli et al., (2015), Ochoa-Martínez et al., (2012), Akinola et al., (2014) considered a fixed choice of mylar film thickness (0.25 mm or 10 mil) to carry out their investigations.

Ochoa-Martínez et al., (2012) studied mango slices (1 and 2 mm thickness) by targeting the RWD process being operated at 92 °C water bath temperature. The authors determined drying kinetics, water activity, and colour change and compared the obtained values with those being achieved with a tray drier being operated at 62 °C and 0.52 m/s air velocity. While the tray dryer took 4 h to achieve the dried product with similar characteristics, the RW drying method took a lower time duration of only 1 h.

Deploying 0.17 mm mylar film thickness, Hernandez-Santos et al., (2016) studied the RWD of dry carrot slices (3 cm diameter; 0.2 and 0.4 cm thickness) at 74 and 94 °C. The moisture loss, colour, total polyphenol content, and antioxidant activity were determined in conjunction with those being obtained from the tray drying process (control sample). Compared to the convective drying, the RWD facilitated a significant reduction in drying time (26 – 51 %) and took only 75 min for a substantial moisture reduction. Using mylar film thickness of 0.25 mm, Castoldi et al., (2015) analysed the influence of RWD process variables on the drying rates and characteristics of dried tomato pulp. The investigations involved variant combinations of water bath temperatures (65, 75, 85, and 95 °C), and pulp thickness (2 and 3 mm) associated to 4.8 – 5.2 °Brix tomato pulp samples. The dried powder characteristics were evaluated in terms of water activity, solubility, dispersion time, and water sorption isotherm.

Table 1.2: Literature data summary on the sensitivity of mylar film thickness.

| S. No. | System | Parameters | Independent variables range | Dependent variables range | Best results | References |
|--------|------------------|---|--|--|--|-------------------------------|
| 1. | Mango slices | T = 92 °C Film thickness = 0.25 mm | t = 40 – 75 min Slice thickness = 1mm, 2 mm | 4.56 ×10 ⁻¹⁰ - 6.83 ×10 ⁻¹¹ m ² /s Diffusivities 0.34 - 0.048 kg water/kg dry solid Moisture content 0.5 – 0.7 water activity | t = 75 mins Slice thickness =2 mm Optimized Diffusivities: 2 mm = 6.83 ×10 ⁻¹¹ m ² /s Moisture content: 0.048 kg water/kg dry solid Water activity: 0.5 | Ochoa-Martínez et al., (2012) |
| 2. | Red onions slice | T = 92°C t = 1 hr. Air flow rate: 0.52m/s Film thickness = 0.05 mm | Slice thickness: 1- 3 mm | 0.02 – 0.05 kg water/kg dry solid Moisture content | Thickness: 2 mm Moisture content: 0.02 kg water/kg dry solid | Akinola et al., (2014) |
| 3. | Kiwi slices | | T = 80 - 100°C 100 – 300 µm Slice thickness = 0.8 – 2.4 mm | 4.1 – 8.5 % Moisture content 56 – 65 L values Colour 8 – 10 Rehydration ratio | 4.1 % Moisture content 65 L values Colour 10 Rehydration ratio | Azizi et al., (2017) |

Using 2 mm thick mylar film, aluminium sheet and aluminium foil as conducting medium, Kaur et al., (2017) conducted RWD studies of mango pulp (2 mm thickness) in a water bath being operated at 95 ± 1.5 °C. For both sheets and foil, a final moisture content of 2.5 % (db) was achieved after 35 min of drying time.

The only available literature with respect to the efficacy of mylar film thickness in the RWD process is that of Azizi et al., (2017) for kiwi slices (0.8 - 2.4 mm) in the parametric range of 80 – 100 °C and 100 – 300 µm mylar film thickness. Studies were carried out to analyse the drying rate, colour index, rehydration ratio. In this study the nutritional properties were not analysed, only physical characterizations (colour index and rehydration ratio) and drying kinetic studies were conducted. The research found that the mylar film thickness did not potentially alter the properties of the kiwi slices.

Table 1.2 summarizes the relevant data associated to RWD investigations that considered the mylar film thickness either as a parameter or a degree of freedom. It is apparent that for only kiwi slices, the mylar film thickness was varied and therefore indicates limited insights.

1.3.3 Statistical Design based RWD Process Parametric Optimality Associated to the Drying of Horticultural Produces

Drying studies involving food systems often target the evaluation of drying constant and moisture diffusivity for a useful analysis of associated transport characteristics. Thereby, suitable drying conditions can be predicted using various alternate drying models. An alternate approach towards greater understanding of the role of process conditions in influencing process-product characteristics is the response surface methodology (RSM). This involves a statistical approach based analysis of measured data and subsequent criticality of process parameters and measured responses. Comparing both drying kinetics and response surface methodology approaches, it is inferred that the statistical tool is comparatively better than the drying kinetics approach (Shishir et al., 2015).

Several studies addressed till date indicate the relevance of response surface methodology for the optimization of two or three decision variables or degrees of freedom and thereby indicate that the methodology is effective to evolve a mathematical relationship between a limited number of input variables and output responses. Thereby, RSM reduces the number of experimental trails being conducted to evaluate the complex interactions of multiple decision variables for a critical response. Thus, compared to trial and error based approaches, laboratory tests can be judiciously studied and be made more efficient through the RSM optimization methodology.

Usually, response surface methodology implementation has three major hierarchical procedures namely (a) design of experiments using either Box-Behnken design (BBD) or Central composite or mixture model design and collection of experimental data (b) statistical and regression analysis to identify most competent

Table 1.3: Literature data summary of modelling approaches based system analysis.

| S. No. | System | Drying Method | Design | Independent variables | Dependent variables | Best findings | References |
|--------|--------------------|-------------------|--|---|--|--|----------------------|
| 1. | Mushroom Slices | Vacuum | Central composite rotatable design | 46 – 74 °C 20 – 580 mbar | Total solids 65- 90 % water activity 0.32 – 0.76 | 57.1 °C and 100 mbar 85.40 % total solids 0.615 a _w value | Sumic et al., (2016) |
| 2. | Sweet potato flour | Convection drying | Box-Behnken design | 55 – 65 °C 1 – 3 w/v citric acid 1 – 3 soaking time | Anthocyanin 15 – 40 mg/100g TPC 50 – 70 mg/g | For anthocyanin 62.91°C, 1.38%, 2.53 min For TPC 60.94°C, 1.04% and 2.24 min Anthocyanin 19.78 mg/100 g TPC 61.55 mg/g. | Ahmed et al., (2011) |
| 3. | Jackfruit | Drum drying | Face centered central composite design | Steam Pressure: 300-400 kPa Rotational speed: 1-3 rpm | 5.6 – 7.9 % moisture content 0.01 – 0.04 water activity | Steam pressure:336 kPa Rotation speed 1.2 rpm M.C: 6.767% Water activity:0.24 | Pua et al., (2010) |

algebraic model representing the response surface of the system and (c) optimization of decision variables through the developed best fit algebraic model.

A critical summary of the application of the RSM towards food process development, analysis and optimization is as follows.

Ahmed et al., (2011) carried out RSM based experimental investigations for the extraction of anthocyanin from purple sweet potato. Four decision variables namely temperature, ethanol to ammonium sulfate, extraction time and pH were varied in the range of 60- 90 °C, 34 – 76 ml, 30 – 75 min and 8 - 14

respectively. Box-Behnken design approach was followed by the authors involving 27 runs (24 factorial experiments and 3 repeated runs at the center point). The analysis of variance (ANOVA) eventually enabled the identification of second order polynomial expression as the best fit model with good fitness characteristics.

Deploying central composite design approach, Shishir et al., (2015) optimized spray drying process variables (inlet temperature, maltodextrin concentration and feed flow) associated to the production of pink guava powder. Thereafter, ANOVA enabled the identification of quadratic expression as best fit model to describe all critical responses (final moisture content, particle size and lycopene) with R^2 of 0.9749, 0.9616, and 0.9505, respectively. The p value for all cases were less than 0.01 and affirmed very good fitness. It was analyzed that the final moisture content reduced with inlet temperature and maltodextrin concentration but not the particle size.

Deploying Box-Behnken design approach, Obajemihi et al., (2020) targeted the RSM based analysis for the improvisation of colour characteristics of dried tomato fruits. Sixty eight triplicate samples were collected by varying air temperature, slice thickness, cultivar and pre-drying treatment approaches. The ANOVA affirmed that quadratic models best described the drying characteristics of tomato in terms of the huge angle colour value and colour difference.

Targeting RSM based optimization of shiitake mushroom drying, Sumic et al., (2016) addressed the optimization of vacuum drying process being operated in the range of 46 –74 °C and 20 – 580 mbar. Thereby, optimized process variable and critical response variables were obtained as 57.1 °C, 100 mbar, 85.40 % total solids and 0.615 water activity.

A critical summary of the above mentioned prior art have been presented in Table 1.3 of the Ph.D. thesis. As indicated, RSM based investigations have not been targeted for the RWD of turmeric samples.

1.3.4 Fortification of Food Products

1.3.4.1 Fortified Product Development

Vitamin and mineral deficiencies cause learning disabilities, mental retardation, low work capacity, blindness and even premature death. To overcome these issues, food fortification has been the best choice in comparison with the pharmaceutical supplements. Fortification of food products involves enhancing essential micronutrients such as vitamins, minerals and trace elements in foods. Thereby, multiple mineral deficiencies can be addressed to enhance health benefits without potential health risks. Cereals, flours, rice and milk are often fortified to reduce deficiencies. Fortification also has potential challenges in terms of bio-availability of added nutrients, unacceptable organoleptic changes and subsequent rejection of a developed product by the consumers and targeted population (Prinz-Langenohl et al., 2001).

Three most common micronutrient malnutrition have been identified for human beings. These are iron, iodine and vitamin A (Allen et al., 2006). The micronutrient folate received significant global attention (Kam et al., 2012) due to its critical ability to address and mitigate issues associated to early embryonic brain development, malformation of embryonic brain and spinal cord or neural tube diseases (Boeneke and Aryana, 2008). With 79 % of children between 6 – 35 months and women between 15 – 49 years of age being anemic in India (Krishnaswamy, 2009), iron deficiency has been opined due to consumption of foods with lower bio-availability of iron. Thus, iron fortified product research needs greater emphasis (Glinz et al., 2017).

1.3.4.2 Development of Mineral and Vitamin Fortified Dry Food Products

A brief account of the prior art associated to the mineral and vitamin fortified dried food product development is as follows.

Modupe et al., (2008) targeted the utilization of folic acid, iron and iodine salts and addressed their potential influence towards mitigating anemia, goiter, neural and spinal disease. Rebellato et al., (2006) fortified whole wheat flour with various iron compounds. Berry et al., (2009) fortified flour with folic acid and inferred that an additional intake of 100 – 150 µg/day of folic acid through the consumption of fortified

flour is effective towards the reduction of neural tube deficit at birth through enhanced blood folate concentrations. Deploying spraying and drying methods, Jahan et al., (2001) fortified chickpea seeds and flour using ferrous sulfate hepta-hydrate, ferrous sulfate mono-hydrate and sodium ferric ethylenediaminetetraacetate (NaFeEDTA) fortificants.

Similarly, Tripathi et al., (2012) deployed finger millet and sorghum flours as double fortifications vehicles with ferrous fumarate, zinc stearate and ethylenediaminetetraacetate (EDTA) fortificants. Thereby, bio-availability studies were conducted.

1.3.4.3 Associated Studies on Mineral Fortified Dry Turmeric Powder Product

The influence of fortificants on the mineral fortified dried fortified products is often targeted through associated studies. Karn et al., (2018) fortified Nepalese curry powder with alternate iron compounds. The authors inferred that the deployed fortificants ferrous sulphate (30 mg), ferrous fumarate (25 mg), Elemental iron H-reduced and electrolytic (25 mg) and sodium ferric ethylenediaminetetraacetic acid (NaFeEDTA) (20 mg) did not trigger adverse effects in terms of the physical, chemical and sensory qualities of the curry powder product. Among the chosen fortificants, both electrolytic (16 mg) and ferrous sulphate (21 mg) have been opined to be the most economical fortificants. Alam et al., (2010) fortified whole wheat flour with a fortificants premix of ferrous sulfate, ethylenediamine tetraacetic acid and folic acid (20:20:1.5 ppm). Thereafter, the sample was stored for 60 days in ambient temperature condition. The associated study of the authors ensured that the iron levels potentially influenced few sensory characteristics such as colour (56 - 69 L values), texture (7 – 8), flexibility (6 – 8), chewability (6 – 9) and overall acceptability (7 – 9) of the prepared naan products but not their taste (8 – 9) and flavour (8 – 9).

Modupe et al., (2019) fortified salt with folic acid, iron and iodine. For this purpose, a solution containing 2 % iodine, 0.5 or 1 % folic acid was sprayed onto the salt containing encapsulated ferrous fumarate. Thereby, the triple fortified salt possessed 1000 ppm iron, 50 ppm iodine, and 12.5 or 25 ppm folic acid. Subsequently, the spray solution and salt were stored for 2 and 6 months respectively and at variant temperature (25 – 45 °C) and humidity (60 – 70 %) conditions. The associated study ensured that

even at 45 °C, more than 70 % iodine and folic acid were retained in the salt. The best fortified salt product in terms of stability referred to that with 12.5 ppm folic acid, 50 ppm iodine and 1000 ppm iron. Thereby, the conducted research affirmed promising scope for simple spraying technology to achieve triple fortification of the salt product.

A critical summary of fortified product development and associated studies of mentioned mineral fortified dry food products is summarized in Table 1.4 (a – b). In summary, fortification was primary targeted for maize meal, wheat flour, salt etc., For dry turmeric powder, fortification was not targeted. However, this is also the need of the hour with products such as turmeric tablets being consumed abundantly in the consumer market.

1.3.4.4 Development of Turmeric Fortified Liquid Food Products

In the field of turmeric fortified liquid food products, limited investigations have been addressed and reported. Deploying tulsi and ginger juices and turmeric powder, Gaur et al., (2019) developed a fortified milk product. The authors inferred that for a constitution of 25 % tulsi juice, 3 % ginger juice and 0.1 % turmeric powder, the sterilized herbal milk fortified product affirmed best sensory scores. Compared to the milk (control) sample, the fortified product had significantly higher antioxidant activity, viscosity, fat and total polyphenol contents. The optimized product had been analyzed for its proximate characteristics. For a cost of the newly developed herbal milk of Rs. 14.17/200 mL, the antioxidant and total phenolic content have been affirmed to be 50.14 % DPPH activity and 96.25 mg GAE/100gm respectively.

Park et al., (2019) reported a colloid food system constituting turmeric extract powder and milk. Sonication followed with spray drying was adopted by the authors to produce a nanoemulsion extract powder of the turmeric. The fortified colloidal system was analyzed for colour, storage stability and volatility characteristics. The fortified milk product exhibited good stability for 21 days at 4 °C.

Table 1.4 (a): Literature data summary of mineral fortified powder products.

| S. No. | System | Fortification Method | Independent variables | Dependent variables | Best findings | References |
|--------|-----------------------|----------------------|--|---------------------------------|--|----------------------------|
| 1. | Nepalese Curry powder | Dry mixing | Ferrous sulphate, ferrous fumarate, NaFeEDTA | Stability of fortificants | NaFeEDTA (20 – 18 mg) | Karn et al., (2018) |
| 2. | Finger millet flour | Dry mixing | Iron (6 mg /100 g) | Bio-accessibility of folic acid | Bio-accessibility of folic acid: 2.39 mg /100 g | Tripathi and Patel, (2011) |
| 3. | Wheat and maize flour | Dry mixing | Folic acid | Bio-accessibility of folic acid | 100 to 150 µg/day reduced the prevalence of NTDs | Berry et al., (2009) |

Table 1.4 (b): Literature data summary of turmeric fortified liquid food products.

| S. No. | System | Fortification Method | Independent variables | Dependent variables | Best findings | References |
|--------|------------------|---|--|-----------------------|---|------------------------|
| 1. | Whey beverage | Curcumin extract added into 50 ml whey | Curcumin extract and commercial curcumin | Anti-oxidant activity | Curcumin extract 60.56 % AA, whey beverage 43.51 % AA | Ankitha et al., (2018) |
| 2. | Lassi | Direct mixing | Beta-cyclodextrin | Curcumin retention | 50 to 90 % curcumin retention | Maurya et al., (2020) |
| 3. | Plant based milk | Direct mixing of curcumin-NaOH solution | Curcumin | Bio-accessibility | 60 % more bio-accessible | Zheng et al., (2021) |

Palthur et al., (2014) reported the efficacy of ginger flavoured herbal milk. To do so, the authors added ginger extract to milk and conducted proximate, organoleptic, antioxidant activity and iron chelating activity studies. Thereby, the fat, protein, total solids and ash content of the developed product were evaluated as 2.05, 3.48, 17.57 and 0.67 % respectively. For the same product, the organoleptic parameter, anti-oxidant activity and iron chelating activity were determined as 13.5, 55 and 58 % respectively. Thus, it is apparent that few studies addressed gold milk product development but not with RWD processed turmeric product that may possess good nutritional characteristics.

1.3.5 Storage and Shelf-life Studies of Dry Turmeric Powder Product and Turmeric based Liquid Products

1.3.5.1 Storage and Shelf-life Studies of Dry Turmeric Powder Product

Shelf-life studies facilitate an insight into the desired characteristics in terms of sensory, chemical, physical and microbiological properties of a food material. Thereby, compliance of the product towards label declaration of nutritional data in due course of storage is sought for consumer acceptability.

To a significant extent, food preservation methods target moisture content control in the processed dry foods. Often for such dry food products, moisture content related shelf-life studies target the determination of equilibrium moisture content (EMC) and food water activity (a_w) i.e., in terms of a moisture sorption isotherm. Literature reported isotherm models have been categorized into various kinetic models. These include absorbed water monolayer (Brunauer-Emmett-Teller model), multi-layer and condensed film (Guggenheim-Anderson-de Boer model), semi-empirical (Halsey model) and purely empirical models (Oswin and Smith models). The best fitness of either of these models shall be addressed experimentally. Further, the influence of temperature, humidity, oxygen and light need to be addressed in terms of the stored food product characteristics.

For environmental conditions of India (high relative humidity of 90 % and temperature of 38 °C), the accelerated storage study methodology suggested by Potter, (1978) is relevant to foster quicker insights

into the ingress and storage time relationships. Early investigations targeting storage studies of processed food products were based on static gravimetric method for mango powder (Jaya and Das, 2005) and garlic (Moreno et al., 2006) produces. Also, Singh et al., (2001) targeted carrot powder storage studies through a rigorous analysis of the time dependent moisture and protein content of the system.

In the specific research theme of storage and shelf-life studies of dried tubers and roots, very few investigations exist. A brief account of these has been presented as follows.

Deploying commercial potato varieties OP-1, Kufri Sutlej and Kufri Ashoka, Misra and Kulshrestha, (2003), prepared potato flour and stored the flour samples at room and refrigerated conditions. The authors evaluated moisture content, ascorbic acid, protein, minerals, vitamins, total dietary fiber, total starch and in vitro protein digestibility for a storage period of 3 and 6 months. For both storage period cases, the authors inferred that while significant changes did not exist in the nutritional composition, the moisture content enhanced (5.6 – 17 %) and ascorbic acid content decreased (34 – 12 mg/g) with time.

Debnath et al., (2002) conducted a moisture sorption study of onion powder being produced through alternate processing schemes such as freeze drying, vacuum shelf drying and flow drying processes. Thereby, the authors analysed the powder samples in terms of flowability and moisture sorption. The Brunauer-Emmett-Teller (BET) method was followed to determine the minimum moisture levels in the onion powder. For freeze dried, vacuum shelf dried and flow dried onion powder samples, the monomolecular layer of adsorbed water and flowability were determined as 2.09, 1.96 and 1.94 %, and 78, 83, 88 %, respectively.

Under ambient and refrigerated environment conditions, Lahari et al., (2020) conducted storage studies of cured (0.05 % sodium bicarbonate) and non-cured dried turmeric rhizome (10 mm thickness) based turmeric powder samples. These samples were prepared using a hot air oven and a pulveriser. For the cured dried turmeric powder, the variation in moisture, curcumin and oleoresin content after 180 days under ambient environment condition varied as 14.60 – 16.77 % (db) (enhanced), 3.11 – 1.72 (reduced) and 10.30 – 7.01 % (reduced) respectively. However, for the non-cured turmeric rhizome based powder sample, these responses varied as 14.48 – 16.99 % (db) (enhanced), 2.82 – 0.526 (reduced) and 11.55 – 9.78 %

(reduced) respectively. Similarly, the samples subjected to storage study for 180 days under refrigerated conditions have been analysed to indicate a variation of moisture, curcumin and oleoresin content as 14.60 – 16.99 % (db) (enhanced), 3.12 – 1.77 (reduced) and 10.30 – 8.06 % (reduced) for the cured dried turmeric powder respectively. Corresponding variations for the non-cured dried turmeric powder under refrigerated conditions were 14.48 – 17.23 % (db) (enhanced), 2.81 – 1.39 % (reduced) and 11.55 – 9.89 % (reduced) respectively. Thus, from both quality and cost affordability perspectives, the best case can be analysed to be cured turmeric powder being kept under ambient storage condition.

Deploying alternate packaging materials and storage under ambient and refrigerated conditions, Sidhu et al., (2013) conducted a storage study of turmeric rhizome and turmeric powder samples. Gunny bag, plastic bag, cloth bag and polyethylene terephthalate (PET) jar were chosen as alternate packaging materials for the turmeric rhizome samples. The turmeric powder was stored in high density polyethylene (HDPE), low density polyethylene (LDPE), polypropylene (PP), laminated aluminium foil and PET jars. Under ambient and refrigerated conditions and for a storage period of 90 days, highest moisture content (5.38 – 22.30 % and 20.63 % respectively) existed in the samples stored in gunny bags. Similarly, the PP based packaging material was detrimental for the turmeric powder samples (14.55 – 16.76 % and 16.21 % respectively). For these samples, the colour of the turmeric rhizome and powder did not reduce significantly (24.26 – 20.00 and 20.50 for the rhizomes kept in gunny bag and 27.63 – 22.51 and 23.34 for the turmeric powder kept in the PP under ambient and refrigerated condition respectively). Thus, for rhizome and turmeric powder samples, gunny bags and PP respectively indicate highest deterioration. The PET jars have been analysed to be the best packaging material for both rhizomes and dried turmeric powder samples due to minimal variation in the oleoresin and curcumin content with storage time (5 % and 8 % for oleoresin of rhizome and turmeric powder and 7 % and 9 % for curcumin content of rhizome and turmeric powder kept under ambient and refrigerated condition respectively).

A critical summary of the literature reported findings has been presented in Table 1.5. As indicated, while the methodology is rich to provide very useful insights into the considered product storage issues, the

Table 1.5: Storage and shelf-life data summary of dried vegetable produces.

| S. No. | System | Parameters | Independent variables | Dependent variables | Methodology | Best findings | References |
|--------|-----------------------------|-------------------------------------|-----------------------|---|--------------------|--|-------------------------------|
| 1. | Potato flour | Tray dryer 55°C for 24 h | 3 – 6 months | Moisture content, protein content, minerals, vitamin, ascorbic acid | Proximate analysis | 5.6 – 17 % moisture content; 34 – 12 mg/g ascorbic acid | Misra and Kulshrestha, (2003) |
| 2. | Garlic powder | Freeze dried Stored at 30 °C | 0 – 14 days | Anti-oxidant activity | ORAC assay | No change in anti-oxidant activity | Moreno et al., (2006) |
| 3. | Turmeric rhizome and powder | Ambient and refrigerated conditions | 90 days | Oleoresin content Moisture content Curcumin content | Isotherm model | Oleoresin: 5 – 7 % Curcumin: 8 – 9 % | Sidhu et al., (2013) |

literature is scanty towards oven/tray dried *Curcuma longa* powder. No prior art addressed the storage characteristics of RWD processed *Curcuma longa* powder, which is being targeted in this Ph.D. thesis.

1.3.5.2 Storage Study of Turmeric Fortified Golden Milk Products

Ankitha et al., (2018) developed a curcumin fortified whey based beverage product. Direct addition of standard commercial curcumin and ethanol extracted curcumin was targeted by the authors to achieve the fortified beverage product samples. Among both cases, the ethanol extracted curcumin product was analyzed to be the best and affirmed a variation in the antioxidant activity from 82 – 61 % and 82 – 69 % for a period of 180 days and for cases without and with preservatives respectively.

Maurya et al., (2020) studied lassi enhancement through curcumin supplementation with β -cyclodextrin. The β -cyclodextrin enhanced curcumin retention in lassi from 50 to 90 %. The sample with 25 ppm curcumin (250 ppm) and its conjugation with 1:4 ratios of β -cyclodextrin was found to be optimum

for lassi supplementation. The developed product upon storage in LDPE pouches or PET bottles had a shelf-life of 20 days at 4 ± 1 °C and 90 - 95 % RH. Corresponding control sample had a stability of 12 days.

Without causing adverse effects on oil body properties or stability, Zheng et al., (2021) successfully loaded curcumin into all milk analogs namely coconut, cashew, almond, and oat milks samples. Due to the localization of curcumin inside the oil bodies, the curcumin had a high encapsulation efficiency (> 86 %) in all milk analogs. The curcumin-loaded plant-based milk had greater than 60 % bio-accessibilities which were higher than those being evaluated for the crystallized turmeric (5 %).

Turmeric based fortification was targeted and storage study was conducted in terms of the anti-oxidant activity of the whey-based beverage, lassi and vegetable milk-based products. However, no investigation exists for the turmeric fortified milk, which is being targeted in the Ph.D. thesis.

1.4 Research Gaps

1.4.1 Technical and Economic Efficacy of Turmeric Drying using Refractance Window, Oven and Tray Drying

The RWD of carrot system was widely investigated by Abonyi et al., (2002) and Akinola et al., (2016). Other researchers addressed the RWD of yam (Akinola and Ezeorah (2016)), and potatoes (Zalpouri et al (2020)) respectively.

Abonyi et al., (2002) considered belt speed as a parameter and varied water bath temperature for the evaluation of carotene content, moisture content and ascorbic acid content of the RW dried carrot samples. Akinola et al., (2016) considered carrot slice thickness as a parameter and varied water bath temperature and drying time for the evaluation of bulk density, rehydration ratio and moisture content. For the yam samples, Akinola and Ezeorah, (2016) varied slice thickness and water bath temperature for the evaluation of moisture content and rehydration ratio. Zalpouri et al., (2020) considered sample thickness as a parameter and varied °Brix and water bath temperature for the evaluation of moisture content, bulk density and porosity. In summary, no investigation has been targeted till date for the RWD of turmeric samples.

However, for the turmeric samples, few investigations have been conducted for conventional drying systems.

Considering peeled and unpeeled turmeric rhizomes, Mulet et al., (2005) conducted drying kinetic studies (only moisture content as the dependent variable or response) in a convective drier at variant temperature conditions (chosen based on trial-and-error basis) along with fitness of measured data with Fick's 2nd law. Targeting blanched and unblanched turmeric rhizomes, Blasco et al., (2006) conducted a trial and error-based kinetics study (moisture content as response variable) in a convective dryer for various combinations of air flow rate and temperature. Thereafter, the authors carried out fitness investigations with diffusion model based on the slab geometry and determined the drying rates. Borah et al., (2016) investigated the drying kinetics (moisture content as the only response variable) of sliced turmeric in a solar biomass integrated drying system. The authors specified the temperature range as per the operational characteristics of the system for heat generation. Thus, it can be inferred that no investigations exist till date in the confined research theme of the comparative technical assessment of RWD, oven and tray drying. Such studies are important from the perspective of the possible scope to reduce the processing time and retention of the desired product characteristics of the dried product.

With regards to cost and economics efficacy, Abbasid et al., (2015), Baeghali et al., (2010) and Abul-Fadl and Ghanem, (2011) inferred that the RWD method facilitated lower processing cost in comparison with oven drying, freeze drying and convective drying respectively. For all three cases, the processing cost was evaluated through the determination of costs associated to equipment, sample, electricity and maintenance for oven, freeze, convective and RW drying processes respectively. Thus, it can be inferred that no investigations exist till date in the confined research theme of the comparative economic assessment of RWD, oven and tray drying. Such studies are important to visualize the cost efficacy of RWD and thereby foster its sustainability to serve agro-horticultural processing needs.

In the near future, economic efficacy of the RWD process needs to be investigated for small scale systems that are affordable for the rural community of India. The literature elaborates significantly with respect to technical efficacy. However, process economics in comparison with the tray and oven drying

processes are to be considered. Thus, it is anticipated that for the turmeric (*Curcuma longa*), similar research directives would be applicable.

1.4.2 Sensitivity and Optimality of Mylar Film Thickness and Temperature during Refractance Window Drying of Turmeric System

Ochoa-Martinez et al., (2012), Hernandez-Santos et al., (2016), Akinola et al., (2014), Akinola and Ezeorah, (2016) and Zotarelli et al., (2015) investigated the influence of mylar film thickness on mango slices, carrot slices, red onion slices, yams slices, and mango pulp respectively.

For mango slices, Ochoa-Martinez et al., (2012) considered both mylar film thickness and drying temperature as parameters and slice thickness and drying time as independent variables for the evaluation of RWD processed mango slices in terms of drying kinetics, water activity, and colour change responses. For carrot slices, Hernandez-Santos et al., (2016) also considered fixed choice of mylar film thickness and varied water bath temperature and drying time for the evaluation of moisture loss, texture, colour, total polyphenol content, and antioxidant activity of RW dried carrot slices.

For red onion samples, Akinola et al., (2014) considered mylar film thickness sample weight and mylar film thickness as parameters and varied slice thickness, temperature and drying time for the determination of responses such as bulk density, rehydration ratio and moisture content. For the yam samples, Akinola and Ezeorah, (2016) deployed a transparent plastic film of specified thickness and considered slice thickness and water bath temperature as well as parameters for the evaluation of moisture content and rehydration ratio of the dried samples. For mango pulp, Zotarelli et al., (2015) considered mylar film thickness as a parameter and varied water bath temperature and sample thickness for the evaluation of the drying rate of RW dried samples.

In the above mentioned literature, the mylar film thickness being considered by Ochoa-Martinez et al., (2012), Hernandez-Santos et al., (2016), Akinola et al., (2014), Akinola and Ezeorah, (2016) and Zotarelli et al., (2015) were 0.26, 0.26, 0.05, 0.15 and 0.25 mm respectively.

Azizi et al., (2017) investigated the effect of different temperature (80 – 100 °C) and mylar film (100 – 300 µm) on kiwi slices. Adopting trial and error method, the authors considered various choices of water bath temperature, slice thickness, mylar film thickness and drying time for their combinatorial influence upon the colour and rehydration ratio of dried kiwi slice samples. The authors affirmed that while slice thickness and temperature influenced the colour and rehydration ratio, the mylar film thickness only had a marginal influence on the mentioned responses. The methodology adopted in the article is highly tedious and complex and generalized trends could not be deduced despite carrying out investigations on a trial and error basis.

Thus, it can be inferred that the sensitivity and optimality of mylar film thickness has not been studied till date for the RWD of turmeric samples. Such an investigation would provide useful insights into the associated sensitivity of the film thickness towards desired critical responses.

1.4.3 Optimality of Refractance Window Drying Process Parameters based on Design of Experiments Approach

The response surface methodology (RSM) based design of experimental approach was addressed for the alternate drying processes based drying of red currants, apple slice, persion shallot, beetroot juice powder, sapota bar, mango pulp by Sumic et al., (2016), Majdi et al., (2019), Chayjan and Fealekari, (2014), Jalgaonkar et al., (2020) and Shende and Datta, (2020) respectively.

Sumic et al., (2016) processed red currants through vaccum drying and considered drying time, temperature and pressure as degrees of freedom. The authors analysed moisture content, water activity, total colour change, firmness, rehydration power, total phenols, total flavonoids, monomeric anthocyanin, ascorbic acid and antioxidant activity content as desired responses. Majdi et al., (2019) optimized process conditions associated to the convective drying of apple slices by considering air temperature, air velocity and apple slice geometry as independent variables. The authors evaluated the process performance in terms of drying time, energy consumption, and shrinkage. For the persion shallot, Chayjan and Fealekari, (2014)

optimized convective drying performance by targeting the RSM based optimization of drying air temperature, air velocity and slice thickness. Thereby, the authors targeted effective moisture diffusivity, specific energy consumption, shrinkage and colour change as desired responses.

The application of RSM for the RWD is confined to few research articles. For the mango pulp product being produced through the RWD, Shende and Datta, (2020) deployed the RSM method by considering pulp thickness and drying temperature as independent variables. The authors targeted drying time, ascorbic acid, total phenolics content and mango leather hardness of mango leather as desired responses. Similarly, for the sapota bar processing through the RWD, Jalgaonkar et al., (2020) deployed RSM for the optimization of temperature, pulp thickness and pectin concentration. Thereby, the authors considered drying time, ascorbic acid content, colour, hardness, cohesiveness, gumminess, and chewiness of the bar as dependent variables.

In summary, the investigations addressed by Sumic et al., (2016), Majdi et al., (2019), Chayjan and Fealekari, (2014), Jalgaonkar et al., (2020) and Shende and Datta, (2020) refer to vacuum drying, convective drying, spray drying and RWD respectively. Thus, no investigations have been addressed towards the optimization of the RWD processing of turmeric samples using the RSM. Such investigations can provide needful insights into the associated sensitivities of desired responses with respect to the degrees of freedom of the RWD process. Thereby, optimal process parametric choices can be obtained for the desired set of critical responses. Such optimal data will serve as a useful guideline towards the scalability and benchmarking of the turmeric processing and product development through the RWD process.

1.4.4 Need for Refractance Window Drying Fortified Turmeric Product Development

1.4.4.1 Fortification of Dried Turmeric Powder

Fortification research methodology was investigated for salt fortification with folic acid, iron folic acid and iodine, whole wheat flour fortification; iron compounds based fortification of salt and Nepalese curry powder; curcumin fortification of whey beverage and sorghum flour double fortification with iron

and zinc. These were respectively addressed by Li et al., (2011), Modupe et al., (2019), Karn et al., (2011), Ankitha et al., (2018) and Tripathi et al., (2012).

Considering alternate fortification methods such as direct blending as a powder, spraying onto the carriers as aqueous solution or suspension and blending as a microencapsulated method, Li et al., (2011) investigated salt fortification with folic acid fortificants through the determination of storage characteristics, stability test, and micronutrients retention of the fortified salt samples. Targeting triple fortification of salt with folic acid, iron and iodine, Modupe et al., (2019) considered their constitution as an independent variable for the evaluation of colour and stability of the fortified samples. Karn et al., (2011) conducted fortification of Nepalese curry powder by targeting the constitution of ferrous sulfate, ferrous fumarate, and NaFeEDTA fortificants. Thereby, the authors evaluated colour, peroxide value, thiobarbituric acid reactive substances, moisture, water activity, iron, sensory qualities, phenolic content, phytate content and iron bio-availability as desired responses. In due course of the curcumin fortified whey beverage, Ankitha et al., (2018) altered curcumin, sweetener and flavourant constitution for the evaluation of the anti-oxidant activity and shelf-life characteristics of the processed product. Targeting iron and zinc fortificants constitution, Tripathi et al., (2012) studied sorghum flour fortification through the evaluation of the bio-accessibility and storage characteristics of the processed product.

Thus, among fortified powder products, while iron and folic acid fortifications were targeted for products such as flour, no investigations have been targeted for the RWD processed turmeric powder product using iron and folic acid. Similarly, among the liquid product formulations, while curcumin fortified whey product was targeted, no investigations addressed the RWD processed turmeric powder for the turmeric fortified milk product i.e., golden milk which is being consumed as a health supplement product in the contemporary food consumer market.

1.4.4.2 Turmeric Fortified Milk Product Development

The pertinent prior art refers to milk fortification with tulsi and ginger juices and turmeric powder (Gaur et al., 2019), milk with ginger juice (Palthur et al., 2014) and milk fortification with turmeric extract

(Park et al., 2019). Among these, Gaur et al., (2019) addressed optimization of tulsi juice, ginger juice and turmeric powder in the product for the critical optimality of fat, total solid, carbohydrate, anti-oxidant and total phenolic content in the fortified milk product. Similarly, Park et al., (2019) considered colour measurement, volatility analysis and storage stability of turmeric extract nanoemulsion powder for a colloidal milk food product. For the ginger fortified milk product, Palthur et al., (2014) addressed proximate analysis, organoleptic analysis, anti-oxidant and iron chelating activity of ginger fortified herbal milk product. In summary, while one of the prior art literatures addressed the turmeric milk product, it was produced through sonication and spray drying techniques which are significantly expensive than the RWD process to produce high quality dry turmeric powder product. Thus, there is a need to investigate RW dried turmeric powder based golden milk product.

1.4.5 Storage Study and Shelf-life Characteristics of Fortified Turmeric Powder and Turmeric Fortified Liquid Product

The storage and shelf-life characteristics of potato flour, curcumin fortified lassi, turmeric rhizome and turmeric powder, turmeric powder, dried garlic and carrot powder were addressed by Misra and Kulshrestha, (2003), Maurya et al., (2020), Sidhu et al., (2013), Lahari et al., (2020), Moreno et al., (2006) and Singh et al., (2003) respectively.

For the potato flour system, Misra and Kulshrestha, (2003) conducted storage studies for various combinations of conventional storage conditions (room and refrigeration) and storage time (3 and 6 months). Thereby, the authors evaluated moisture, protein, minerals, vitamins, total dietary fiber, total starch and in vitro protein digestibility as critical responses. For the curcumin fortified lassi product, Maurya et al., (2020) considered storage temperature, relative humidity and storage time as degrees of freedom for the evaluation of sensory, physio-chemical (fat, solid non-fat, total solids and ash content), titratable acidity, curcumin retention and microbiological quality of the fortified product. For the turmeric rhizome and turmeric powder being produced through conventional drying method, Sidhu et al., (2013)

targeted the influence of storage conditions and packaging materials on the moisture content, colour, curcumin and oleoresin content of the rhizome and powder samples.

Lahari et al., (2020) investigated the storage and shelf-life of turmeric powder by considering processing conditions (boiled and un-boiled turmeric rhizomes), storage time, storage conditions (normal and refrigerated) and packaging materials as degrees of freedom. Thereby, the authors evaluated the independent variables values by targeting the moisture, curcumin and oleoresin content of the samples. Targeting storage characteristics of dried garlic system, Moreno et al., (2006) analysed the criticality of storage time and storage conditions (combination of different water activity and temperatures) through the evaluation of anti-oxidant activity and amadori compounds. For the hot-air oven dried carrot powder, Singh et al., (2003) considered storage time as the degree of freedom to evaluate the moisture content, protein content, ascorbic acid, iron content of the processed samples.

In summary, for the mentioned powdered food products such as potato flour, turmeric powder, dried garlic and carrot powder, storage studies have not been conducted for those being obtained through the RW drying method. In this regard, it shall be noted that Sidhu et al., (2013) and Lahari et al., (2020) did address storage study of turmeric powder system being obtained through traditional drying method. Thus, it is apparent that storage study and shelf-life estimation are to be addressed for the fortified RWD based turmeric powder product.

Also, in the field of turmeric fortified liquid products, lassi was targeted but not the milk (golden milk product).

1.5 Objectives of the Ph.D. Thesis

Considering the above cited lacunae in the mentioned prior art, the following objectives have been chosen for the Ph.D. thesis.

- a) Comparatively, assessment upon the RWD, oven and tray drying characteristics of *Curcuma longa* dried product nutritional characteristics (anti-oxidant activity, total phenolic content, total

- flavonoid content and curcumin content) and their conceptual processing cost characteristics for the drying of *Curcuma longa* paste and slice samples.
- b) Sensitivity analysis of mylar film thickness on the RW drying characteristics and nutritional properties (colour, anti-oxidant activity, total phenolic content, total flavonoid content and curcumin content) of RW dried *Curcuma longa*.
 - c) RSM based sensitivity and optimality analysis of RWD process parameters (temperature, drying time and air velocity) to achieve dried *Curcuma longa* product.
 - d) Folic acid and iron fortification of RW dried *Curcuma longa* product and subsequent characterization based analysis of the dried product (physical and in-vitro characterizations).
 - e) Accelerated storage study of optimally fortified RW dried turmeric powder product in terms of shelf life, adsorption isotherm and nutritional properties along with sensory, nutritional and storage characteristics of the RWD based turmeric powder based fortified golden milk product.
 - f) Nutritional, sensory and storage characteristics of the RWD based Lakadong turmeric powder fortified golden milk production.

1.6 Organization of the Ph.D. Thesis

Addressing the mentioned objectives, the Ph.D. thesis has been organized into nine chapters. A brief account of these chapters is as follows:

Chapter 1 briefly summarizes relevant investigations being targeted till date on alternate drying methods and products being formulated in conjunction with dried *Curcuma longa* product and its fortification and subsequent food product development. Thereafter, the chapter customizes towards associated lacunae and subjectively defined objectives of the Ph.D. thesis.

Chapter 2 presents a brief overview of the deployed materials and adopted methods to achieve the mentioned objectives. Adopted methods include associated to alternate drying processes, optimization,

dried product characterization, fortification, storage, shelf life and golden milk product development and characterization.

Chapter 3 addresses the comparative technical efficacy of the RWD in conjunction with the oven and tray drying methods for the sliced and paste samples of the *Curcuma longa*. Nutritional (AA, TFC, TPC, CC), moisture content and physical characterization (colour) outcomes have been discussed along with best findings reported in the relevant literature. Finally, conceptual cost, energy and exergy analyses were conducted for the chosen alternate drying methods.

Chapter 4 details upon the sensitivity characteristics of mylar film thickness and drying temperature on the RW processed *Curcuma longa* samples. Drying kinetics of the alternate case studies were targeted and the product quality was evaluated based on nutritional, physical and moisture content characterization.

Chapter 5 reports findings associated to the RSM based optimization of the RWD processing of *Curcuma longa*. Thereby, criticality of process parameters was judged and best process parametric and product characteristics were identified.

Addressing fortification of the RW dried turmeric powder, Chapter 6 details upon the relevant characterization outcomes in terms of physical characterization and in vitro digestion studies. Research findings were expressed in terms of the hygroscopicity, solubility, wettability, swelling power, thermal analysis, FTIR, XRD and surface morphology.

The storage and shelf-life characteristics of fortified and unfortified RW dried turmeric powder have been addressed in Chapter 7. These as well include sensory analyses and addresses investigations associated to the development of turmeric fortified milk product and its storage characteristics. Subsequently, Chapter 8 addresses the nutritional and sensory studies of RWD processed Lakadong turmeric powder and golden milk product. Finally, conclusions and possible scope for future work have been addressed in Chapter 9 of the thesis.



Chapter 2:
Materials and Methods



Materials and Methods

In this chapter, details with respect to the targeted experimental, analytical and optimization methodologies have been delineated for the realization of mentioned objectives of the Ph.D. thesis. Section 2.1 presents a brief summary of relevant materials and sample preparation procedures along with the methods used for determination of nutritional properties. Also, the section addresses methodologies associated to the identification of optimal drying method based on nutritional content of the dried samples. Following this, section 2.2 details upon the procedures associated to kinetic studies and trial and error-based approach to analyse the process and product characteristics of refractance window dried Curcuma longa with respect to mylar film thickness. These refers to drying kinetics, fitness of drying models, determination of diffusivities and activation energies of dried samples and variation of nutritional parameters with drying temperature and time. Section 2.3 elaborated upon statistical design-based approaches for the evaluation of optimal drying process parameters. The section presents useful details with respect to the response variable models based on the analysis of variance (ANOVA) approach and subsequent numerical optimization studies. Section 2.4 deliberates upon the procedures followed for the fortification of the dried turmeric sample and studies targeted for the evaluation of associated characteristics. The section 2.5 provides details on the storage study of RW dried turmeric sample, folic acid fortified turmeric sample and NaFeEDTA fortified turmeric sample and their shelf-life characterization. Also, nutritional and physical characteristics of stored powder samples along with the sensory analysis and nutritional characteristics of the RWD processed turmeric based fortified golden milk product have been delineated. Thereafter, section 2.6 details upon the procedures involved in the preparation and characterizations of Lakadong turmeric based golden milk product.

2.1 Techno-Economic Efficacy of Refractance Window Dried *Curcuma longa*

2.1.1 Raw Materials

Turmeric rhizomes were procured from market complex, Indian Institute of Technology Guwahati, Kamrup, Assam, India and were packed in polythene pouches to avoid contamination in due course of their transportation.

2.1.2 Chemicals

Sodium carbonate, aluminium chloride, potassium acetate, gallic acid and absolute methanol were procured from Merck India. 2,2-Diphenyl-1-Picrylhydrazyl (DPPH), extra pure quercetin and Folin-Ciocalteu reagent were purchased from SRL Pvt. Ltd., India. Curcumin (99 %) and ethanol were obtained from Asper, India and MTedia, India respectively.

2.1.3 Sample Preparation

The sample preparation procedures firstly involved preliminary washing of the raw turmeric rhizomes with running water to get rid of dirt and other contaminants. Thereafter, the washed turmeric rhizomes were thoroughly wiped to remove excess water and peeled. The peeled turmeric was sliced into 1 mm thick slices using a slicer (Model: VDNSI) for drying experiments using oven dryer, tray dryer and refractance window drying (RWD) system. Further, paste samples were also prepared by grinding the sliced turmeric samples with an electric grinder (Make: Phillips, Model: Classic). During drying studies, 1 mm thick paste samples were deployed.

2.1.4 Oven, Tray and Refractance Window Drying

During hot air oven and tray drying studies, the prepared turmeric slices and paste samples (both 1 mm thick) were arranged on several trays in a laboratory scale hot air oven (Make: REICO) and in a tray drier (Make: International Commercial Traders, Kolkata, India). Thereby, sliced or paste samples (weighing

150 g as either 1 mm sliced or 1 mm thick paste sample) were dried at 60 °C. For the RWD process, a water bath (Make: Jain Scientific Glass Work) and a Mylar film (Make: Lamtex Solution) was used and the mylar film was made to float on the surface of the water bath. Thereafter, the RWD drying was conducted by placing a layer of slice or paste sample on the film at 60 °C. The dried samples were eventually subjected to grinding with a dry portable electric grinder. Thereafter sieving with an 80 mesh sieve (Make: Shakewell) was carried for the ground powder to achieve 0.177 mm average particle size of the sieved samples (Daulay et al., 2019). The powdered sieved samples were eventually subjected to nutritional evaluation studies.

2.1.5 Determination of Nutritional Parameters

2.1.5.1 Moisture Content

AOAC, (2010) method was followed to determine the sample moisture content (MC). The procedure first involved weighing a known quantity of sample in a glass petri dish. Thereafter, the sample was dried in an oven for 14 h at 105 °C. Subsequently, the sample was transferred to a desiccator for cooling and weighed. Based on the initial and final weights of the sample, the moisture content of the dried turmeric slice/paste samples was determined using the expression:

$$MC (\%) = \frac{W_1 - W_2}{W_1} \times 100 \quad (1)$$

where W_1 and W_2 are the weight of fresh sample (prior to drying) and dried sample respectively.

2.1.5.2 Antioxidant Activity

To determine the antioxidant activity (AA) of the fresh and dried turmeric samples, the literature reported 2,2-diphenyl-1-picrylhydrazyl (DPPH) assay method (Barimah et al., 2017; Sochor et al., 2010) was adopted and meticulously followed. The sequential steps in the assay method were as follows. Firstly, 10 mg sample and 20 mL absolute methanol were mixed and subjected to sonication for 30 min in a sonicate bath (Model: Elmasonic S 30 H, Make: Elma). Thereby, Whatman No. 1 filter paper was used to filter the

mixture and obtain its extract. The following step involved the addition of 3 mL 0.002 % methanolic DPPH solution to 1 mL filtrate. During this step, a control sample was also duly prepared by mixing 1 mL absolute methanol with 3 mL 0.002 % methanolic DPPH solution. The third step involved thorough shaking of both samples and subsequent incubation in a dark environment for 30 minutes. Thereafter, the fourth and final step involved the measurement of absorbance at 517 nm wavelength using a UV spectrophotometer (Make: Shimadzu, Model: UV-2800). Based on the measured absorbance values, the antioxidant activity of the sample was evaluated using the expression:

$$\% AA = \frac{A_1 - A_2}{A_1} \times 100 \quad (2)$$

where A_1 and A_2 are the absorbance of control and evaluated samples respectively.

2.1.5.3 Total Phenolic Content

The sample total phenolic content (TPC) was determined using Folin-Ciocalteu reagent (FCR). The working principle of the method refers to an alteration in the colour of the titration system from yellow to blue-black colour. The colour change mechanism was due to the reduction of tungstate-molybdate prevalent in the mentioned reagent with the phenolic compounds of the sample (Barimah et al., 2017). Often, TPC is expressed in terms of gallic acid equivalence. Hence, a calibration chart was prepared using gallic acid standard. To do so, firstly, gallic acid stock solution was prepared by mixing 0.5 g of dry gallic acid, 10 mL of 50 % methanol and water in a 100 mL volumetric flask. Thereafter, 50 - 500 mg/L gallic acid solutions were prepared through appropriate dilution of 5 mL stock solution. Subsequently for each solution, 0.1 mL was mixed with 6 mL distilled water and 0.5 mL of the said reagent. After mixing for about 5 min, the mixture was added with 1.5 mL of 20 % sodium carbonate solution and was diluted to 10 mL solution volume. Eventually, the standard gallic acid solutions were subjected to absorbance measurements at 765 nm wavelength using UV-VIS spectrophotometer. The absorbance of the fresh and dried sample extracts was measured for the evaluation of their TPC characteristics. For the solid turmeric sample, extraction

procedure was followed that involved mixing of 0.5g of sample with 10 mL of 50 % methanol and subsequent filtration using Whatman paper No.1. Thereby, the solution absorbance was measured at 765 nm. Using the absorbance value and calibration chart, the TPC of the sample was evaluated in terms of mg gallic acid per gram of the dry sample weight.

2.1.5.4 Total Flavonoid Content

Aluminium chloride (AlCl_3) colorimetric method (Demla & Verma, 2012) was adopted to determine the total flavonoid content (TFC) of various samples. The working principle of the method involved the realization of a yellow coloured solution to quantify the TFC. Usually, TFC is expressed in terms of the equivalent quercetin content. Thereby, a standard curve was prepared through the dissolution of 100 mg of quercetin in 100 mL of 80 % ethanol. To do so, and thereby, achieve quercetin solutions with variant concentrations, 1 - 5 mL of the prepared quercetin solution was taken with a pipette for subsequent dilution with distilled water to achieve standard quercetin solutions of 100 - 500 mg/L. Thereafter, 0.5 mL of stock solution was mixed with 1.5 mL of absolute methanol, 0.1 mL of 10 % aluminium chloride, 0.1 mL of 1 M potassium acetate solution and 2.8 mL of distilled water. The final solution after thorough mixing was kept at room temperature (27 °C) for 30 min. After this step, the absorbance of the standard solution was measured at 415 nm using a UV-VIS spectrophotometer. Similar procedures were followed for fresh and dried turmeric samples after conducting TFC extraction from the solid samples into the liquid media. This involved a thorough mixing of 1 mL of 80 % ethanol with 10 mg of the turmeric sample in a vortex mixer (Make: Spinix) followed with the addition of 9 mL of 80 % ethanol. Subsequently, the solution was sonicated for 10 min to achieve a uniform sample concentration of 1 mg/mL (1000 ppm). Thereafter, the solution was filtered using Whatman filter paper No. 1. The extract solution eventually was subjected to absorbance measurement at 415 nm. Using the measured absorbance of the extracted sample and calibration chart, the TFC of the dried sample was determined and expressed as mg quercetin/g dry weight.

2.1.5.5 Curcumin Content

The curcumin content of the fresh and dried sample was determined by adopting the procedure summarized in the relevant literature (Pawar et al., 2014). Thereby, standard or calibration curve had to be prepared for the curcumin. To do so, firstly, 10 mg of curcumin standard was dissolved in 10 mL of 95 % methanol and was subjected to sonication for 10 min. Thereafter, 10 mL of the solution was mixed with 90 mL of 95 % methanol to achieve a standard solution of 100 mg/L. Subsequently, 0.5 - 5 mL of 100 mg/L standard solutions were taken with a pipette and were mixed with 95 % methanol to obtain 0.5 - 5 mg/L standard solutions. The absorbance of the samples was measured at 425 nm using UV-VIS spectrophotometer.

For both fresh and dried turmeric samples, extraction procedures were duly followed using appropriate liquid media. These were as follows. Firstly, 10 mg of fresh or dried turmeric sample was mixed with 5 mL of 95 % methanol and the mixture was subjected to sonication for 10 min. Eventually, using a 10 mL volumetric flask and 95 % methanol, 5 mL of extract volume was adjusted to 10 mL. Thereafter, the solution was filtered using Whatman filter paper No. 1. Subsequently, 0.4 mL of the filtrate was added to 5 mL of 95 % methanol. Thereby, the extract solution absorbance was measured at 425 nm. Using the measured absorbance and calibration chart, the curcumin content of the fresh or dried sample was determined and expressed as % curcumin content (w/w).

2.1.5.6 Colour Indices

The colour indices of both fresh and dried samples were determined using a colorimeter (Make: Data color, Model: 250) set up (L , a , b) (Caparino et al., 2012). In the mentioned nomenclature, ' L ' value represents lightness (0 for black and 100 for white) and the ' a ' scale represents the red/green dimension (positive values for red colour and negative ones for green colour). Further, the ' b ' scale represents the yellow/blue dimension, (positive values for yellow colour and negative ones for blue colour). For each

case, the measurements for L , a and b were conducted for three different sample spots. Thereby, the data have been reported as the mean of these three measurements.

2.1.6 Statistical Analysis

One-way ANOVA methodology was applied for the statistical evaluation of the associated variations in the analyzed parameters. Based on the analysis of variance approach, the methodology involved the evaluation of the differences between the mean values and associated significance among independent parameters (Pytlakowska et al., 2012). Origin Pro 9 software was deployed to conduct one-way ANOVA analysis. Thereby, the achieved responses have been evaluated for their confidence levels and subsequent mitigation of errors beyond acceptable range in experimental investigations was ensured. Each experiment was carried out in triplicates and the repeatability/reproducibility of the data were less than the permissible limit of 5 % standard deviation from the average value.

2.1.7 Yield and Rehydration Ratio

The yield of RW dried turmeric powder, folic acid fortified and NaFeEDTA fortified turmeric powder samples were determined in terms of the ratio of the sliced samples before and after RWD. Thereby, the yield was determined using the expression:

$$Yield(\%) = \frac{W_s}{W_f} \times 100 \quad (12)$$

where W_f and W_s are the weights of fresh and RW dried samples respectively (Prakash et al., 2004).

The rehydration ratio was evaluated by placing 5 g (initial weight) of the RW dried turmeric slice sample in a 500 mL beaker loaded with 150 mL of distilled water. The system was boiled for 5 min. After rehydration, the sample was taken out and weighed after wiping with tissue paper. Thereby, final weight of the rehydrated sample was achieved. The rehydration ratio was evaluated as a ratio of the rehydrated sample weight to the initial sample weight (Prakash et al., 2004).

2.1.8 Sensitivity Analysis

To quality upon the percentage of alteration in the desired property, the sensitivity analysis was conducted. Thereby, the following expression was used:

$$\% \text{ change} = \frac{\text{Values of dried sample} - \text{Values of fresh sample}}{\text{Values of fresh sample}} \quad (3)$$

2.1.9 Energy Consumption

The energy needed for the drying process was calculated using the expression:

$$\text{Energy} = \text{Power (kW)} \times \text{amount of time (h)} \quad (4)$$

2.1.10 Cost Efficacy

Considering operating, equipment, maintenance, depreciation and manpower costs, the cost efficacy studies targeted the annualized total processing costs associated to slice and paste turmeric samples being processed with oven or tray or RW drying processes. The total annualized cost was evaluated as the sum of electricity, equipment, manpower, maintenance and depreciation costs. Cost evaluations have been supplemented with other auxiliary parameters such as power (kW), power tariff, time taken for 1 batch (h), cost of electricity per batch, batch per day, number of days per year, cost of electricity per year, amount of sample per batch, no. of days per year, amount of sample per year (kg). Using these and other cost parameters, electricity costs associated to drying process per one kg of fresh and dried samples were determined. Similar calculations have been considered for the electrical costs associated to grinding operation.

The equipment costs for RWD, tray drying, oven drying, slicer and grinder were determined by considering factors such as equipment purchase cost, life span, personal interest loan for year, equal monthly instalments (EMI) per year. While equipment maintenance costs were evaluated to be about 10 % of overall equipment cost, the depreciation was calculated to be 10 % of process cost. The manpower costs

were evaluated based on number of persons, salary for each personnel and overall salary per year for the appointed manpower of a small batch process. Thereby, annual production costs have been determined. The reported cost efficacy in the thesis could serve as a reference or guideline for commercial turmeric drying with laboratory or pilot scale systems. The methodology followed for the economic evaluation was conceptual in nature and as per the procedure outlined in the relevant literature (Chakraborty et al., 2020). Accordingly, normalized equipment, maintenance and depreciation costs have been determined for all processes by considering uniform processing of about 1090 kg of turmeric on an annual basis. In the cost expression, Guthrie correlation exponent factors have been taken as 0.65 and 0.25 for equipment and manpower costs respectively (Biegler et al., 1997).

Various economic indices such as annualized processing cost per kg fresh and dry sample and percentage contribution of various costs (electricity, manpower, equipment, maintenance and depreciation costs) have been considered to gain useful insights into the conceptual processing cost of alternate drying processes (RWD, oven and tray drying). Both slice and paste samples have been considered to gain useful insights. Appendix B details upon the relevant expression and evaluations.

2.2 Process and Product Characteristics of Refractance Window Dried *Curcuma longa*

In the previous sub-section, among hot-air oven, tray and RWD process, the RWD process with 1 mm slice sample provided good performance. In this section, experimental investigations of RWD processed *Curcuma longa* were targeted for variant constitution of water bath temperature and mylar film thickness. The following sub-sections delineate upon the conducted studies.

2.2.1 Raw Materials and Chemicals

Sections 2.1.1 and 2.1.2 respectively delineate upon the adopted raw materials and chemicals for the carried out investigations.

2.2.2 Sample Preparation

Section 2.1.3 of this Ph.D. thesis details upon the sample preparation process to achieve 1 mm slice sample thickness of turmeric rhizomes. These samples were used for the RWD process studies.

2.2.3 Refractance Window Drying

An indigenous batch RWD system was customized and deployed for the drying investigations. A pictorial representation of the RWD process has been illustrated in Fig. 1.3. The batch RWD process was arranged as a water bath system in which mylar film was placed on water surface for the RW drying of *Curcuma longa*. The temperature of the water bath was controlled using a thermostat regulated digital controller. Periodically, the bath temperature was measured using a thermometer for the achievement of uniform bath temperature during the drying process. Thereby, manual control facilitated utmost variation of the water bath temperature by ± 1 °C. Using a spatula, sample loading and unloading was facilitated on the mylar film in due course of the experimental investigations.

During the experimental investigations, the water bath was set to a bath temperature of either 65 or 75 or 85 or 95 °C. The parametric values for the temperature and slice thickness have been chosen based on the available data trends in Ochoa-Martinez et al., (2012). Further, the efficacy of alternate mylar film thickness systems were targeted for three cases i.e., 125, 250 and 350 μm . Thus, RWD experiments were conducted for each combination of water bath temperature and mylar film thickness. The experimental procedure during the batch RWD process has been elucidated in the following paragraph.

After reaching the set or desired bath temperature, the mylar film was first placed to float on the water surface of the water bath. Thereafter, a layer of turmeric slices was placed on the film. During the drying process, the sample weight was periodically weighed by transferring the sample to an analytical balance (Make: Mettler Toledo) for a very short period of time (5 – 7 s). Thereby, the RWD was carried out until a constant sample weight could be achieved that corresponds to the equilibrium moisture content (EMC) of the sample. The dried samples were thereafter subjected to grinding using a dry portable electric

Table 2.1: Summary of fitness models deployed for the investigation of thin layer drying kinetics.

| S. No. | Model | Equation |
|--------|----------------------------|--|
| 1. | Newton | $MR = \exp(-kt)$ |
| 2. | Page | $MR = \exp(-kt^n)$ |
| 3. | Henderson and Pabis | $MR = a \exp(-kt)$ |
| 4. | Logarithmic | $MR = a \exp(-kt) + c$ |
| 5. | Two Term | $MR = a \exp(-k_0t) + b \exp(-k_1t)$ |
| 6. | Singh et al., (2014) | $MR = \exp(-kt) - akt$ |
| 7. | Approximation of Diffusion | $MR = a \exp(-kt) + (1 - a)\exp(-kbt)$ |
| 8. | Silva et al., (2012) | $MR = (-at - bt^{1/2})$ |
| 9. | Verma et al., (1985) | $MR = a \exp(-kt) + (1 - a)\exp(-gbt)$ |

grinder and subsequent sieving using a 80 mesh sieve (average particle size of 0.177 mm particle size for the sieved powder samples) (Daulay et al., 2019). Thereby, the powdered sieved sample was subjected to various analyses being delineated in the following sub-sections of the Ph.D. thesis. The shortest duration of the RWD investigations was 90 min and at 95 °C water bath temperature. In the conducted studies, the sample weight was measured for a very short period of time (5 – 7 seconds). During the period, the moisture loss in the sample and temperature was presumed to be negligible due to relatively short time for weight measurement.

2.2.4 Drying Characteristics Curves

The drying characteristics curve of the RW drying process of the turmeric were prepared through a graphical representation of time altered measured weight vs time data of the samples being dried for various cases. Each such case referred to a specific combination of water bath temperature and mylar film thickness. Thereby, the drying curves were conveniently presented in terms of the time dependent variation of the moisture ratio (MR), being determined using the expression:

$$MR = \frac{M_t - M_e}{M_0 - M_e} \quad (5)$$

where MR , M_0 , M_t and M_e are the moisture ratio, initial MC, MC at any time and equilibrium moisture content (EMC) respectively (Goyal et al., 2007).

2.2.5 Fitness of Drying Curve Models

Using a set of empirical and non-empirical models, the drying kinetics of sliced turmeric samples were analysed to determine the best fit model. These have been summarized in Table 2.1. Thereby, the best fit model was determined using the combinations of highest R^2 value, lowest residual sum of squares (RSS) and lowest chi-square values (Goyal et al., 2007).

2.2.6 Determination of Moisture Diffusivity and Activation Energy

The moisture removal during RWD has been assumed to follow Fick's second law of diffusion:

$$\frac{\delta M}{\delta t} = D_{diff} \frac{\delta^2 M}{\delta x^2} \quad (6)$$

where M , D_{diff} , and x are the MC, diffusivity and thickness of sample respectively.

Assuming negligible shrinkage and uniform temperature, the consideration of boundary conditions such as $M = M_0$, $0 \leq X < L$ at $t = 0$, $\frac{\partial M}{\partial X} = 0$ at $X = 0$ and $M = 0$ at $X = L$, a solution to the above mentioned partial differential equation can be obtained as Crank equation (Goyal et al., 2007).

$$MR = \frac{8}{\pi^2} \sum_{n=0}^{\infty} \frac{1}{(2n+1)^2} \exp\left[-(2n+1)^2 \frac{\pi^2 Dt}{4L^2}\right] \quad (7)$$

where MR , D and t denote moisture ratio, effective moisture diffusivity and drying time respectively

Neglecting the higher order terms, the above expression can be simplified as:

$$MR = \frac{8}{\pi^2} \exp\left[-\frac{\pi^2 Dt}{4L^2}\right] \quad (8)$$

The logarithmic form of the expression is:

$$\ln MR = \ln \frac{8}{\pi^2} - \frac{\pi^2 Dt}{4L^2} \quad (9)$$

Thus, a graph of $\ln MR$ versus t would provide a straight-line fit with slope and intercept as $-\frac{\pi^2 D}{4L^2}$ and

$\ln \frac{8}{\pi^2}$ respectively.

From the slope, the diffusivity values can be determined for each temperature case. Using diffusivity values determined at various temperatures, Arrhenius equation can be used to determine the activation energy using the expression:

$$D = D_0 \exp\left(-\frac{E_a}{RT}\right) \quad (10)$$

where E_a , R and T are the activation energy, gas constant and temperature respectively (Goyal et al., 2007).

2.2.7 Determination of Nutritional Parameters

For each investigated case, nutritional parameters such as anti-oxidant activity, total phenolic content, total flavonoids content and curcumin content while colour indices analysis were determined using procedures outlined in sections 2.1.5.2, 2.1.5.3, 2.1.5.4, 2.1.5.5 and 2.1.5.6 of the Ph.D. thesis respectively.

2.2.8 Statistical Analysis

The procedure and methodology being adopted for the statistical analysis of the RW dried turmeric powder was similar to that being outlined in section 2.1.6 of the Ph.D. thesis.

2.2.9 Fourier Transformed Infrared Spectroscopic Analysis

The FTIR spectral analysis was conducted for RW dried turmeric powder (Make: Shimadzu, Model: IR Affinity). FTIR spectrometer being operated in transmission mode was deployed for measurements conducted in the resolution range of 400 – 4000 cm^{-1} . For the FTIR analyses, 2 g of turmeric powder sample was mixed with 300 mg of KBr. Thereby, the thoroughly mixed sample was transformed into a pellet using a hydraulic pressure system (Tontul et al., 2018).

2.3 Statistical Design Based Sensitivity and Optimality of Process-Product Characteristics of Refractance Window Dried *Curcuma longa*

Prior to statistical design based experimental investigations, trial and error based approaches were followed to evaluate the optimality of the RWD process parameters such as mylar film thickness and temperature on the evaluated responses such as MC, TPC, TFC, AA, CC and colour indices for the *Curcuma longa* slices systems. Thereby the sensitivity of mylar film thickness was evaluated to be insignificant to influence measured responses. Hence, drying time and temperature were identified to be critical degrees of freedom for the chosen system. In addition, air-velocity was also chosen for statistical design based insights into the optimality of process parameter to achieve desired combinations of responses.

2.3.1 Raw Materials and Chemicals

Sections 2.1.1 and 2.1.2 respectively summarize various raw materials and chemicals being deployed for the conducted investigations.

2.3.2 Sample Preparation

Sections 2.2.2 of this Ph.D. thesis details the sample preparation process to achieve 1 mm slice sample thickness of turmeric rhizomes that were used for RWD.

2.3.3 Optimization of Refractance Window based Drying Process

The RWD experiments were conducted using a lab scale water bath based indigenous batch experimental set-up. For these experimental investigations, the sample and mylar film thickness have been regarded as parameters. These have been set as 1 mm and 250 μm respectively. These choices were based on preliminary trials, conducted experiments (section 2.2) and literature based insights (Akinola et al., 2014; Azizi et al., 2017; Ochoa Martinez et al., 2012). Thereby, the specific combinations of process parameters set were considered based on the parametric data set indicated by the response surface methodology (RSM). These will be discussed in the following section. Thereafter, drying experiments were terminated and the dried samples were grounded to a powder form using a dry portable electric blender and subsequent sieving using an 80-mesh sieve (0.177 mm particle size of the powdered turmeric samples) (Daulay et al., 2019). Thereby, the powdered sieved sample was subjected to nutritional evaluations.

2.3.4 Experimental Design

The statistical design of experiments was based on the Box-Behnken Design (BBD) methodology being applied using Design expert software (Version 7). Thereby, only few experimental data sets were required (Table 2.2). The minimum and maximum values of independent variables were mandatory for the software to generate the design based data sets. To do so, few trials were conducted with lowest and highest possible values of drying time and literature based insights for the temperature and air-velocity (Pua et al., 2010). In this regard, it is important to note that the upper and lower limit of these parameters have to be set such that there is maximum retention of nutritional constituents and no further degradation occurs for the bioactive compounds. The BBD based RSM design involved 15 experimental data sets in which three sets correspond to those being investigated at the central point. The BBD enabled better precision in the centered factor space (Box & Behnken, 1960; Montgomery, 2009). Thus, 3-factors-3-level BBD with three runs at the center point was adopted for the RW drying studies of turmeric slices. Based on preliminary trials, the degrees of freedom water bath temperature (A), and air-velocity (B) and drying time (C) were

altered as 65 – 95 °C, 0.5 – 1 m/s and 75 – 360 min respectively. The measures data of the six responses namely AA, TPC, TFC, MC, CC and colour indices were analysed using a second order polynomial (SOP) model:

$$Y_k = \beta_{k0} + \sum_{i=1}^n \beta_{ki} x_i + \sum_{i=1}^n \beta_{kii} x_i^2 + \sum_{i=1}^{n-1} \sum_{j=i+1}^n \beta_{kij} x_i x_j \quad (11)$$

where, Y_k = response variable with Y_1 = AA (%); Y_2 = TPC (g GAE/g dry sample); Y_3 = TFC (g quercetin/g dry sample), Y_4 = CC (% w/w), Y_5 = MC (%) and Y_6 = Colour indices (L values); x_i represents coded independent variables (x_1 = temperature, x_2 = air-velocity and x_3 = drying time); β_{k0} refers to the correlation coefficient at the center point of the design i.e., point (0, 0, 0) and β_{ki} , β_{kii} and β_{kij} refer to the linear, quadratic and cross-product regression coefficients respectively.

After experimentally determining all response variables values, the software environment was utilized to identify best fit model among alternate models to represent either of the responses (anti-oxidant activity or total phenolic content or total flavonoids content or curcumin content) in terms of specific combination of the independent variables namely temperature, time and air-velocity. The alternate models considered by the software include linear, two function interaction, quadratic and cubic models. Among these, the best model was identified as the model that possessed highest F-value and lowest p-value. Thereafter, for each response variable, statistical analysis was conducted.

During equilibrium studies, it was analysed that the equilibrium time varied significantly with the water bath temperature of the RWD process. Thus, temperature on its own can't be considered as an independent variable in the RSM based design. In other words, the design of experiments can be targeted in two alternate ways i.e by either considering temperature as variable and drying time as response or considering both temperature and drying time as independent variables. Also, the independent variable range with the BBD based RSM design approach were chosen such that the design could identify the optimality of drying time and temperature with the range for the chosen responses. In other words, equilibrium time was not sought in the RSM process that adopted time as an additional independent variable.

Table 2.2: RSM design based data set summary for various combinations of temperature, air-velocity and drying time.

| S. No. | Run | Temperature (°C) | Air-velocity (m/s) | Drying time (min) |
|--------|-----|------------------|--------------------|-------------------|
| 1. | 1 | 65 | 0.50 | 217.5 |
| 2. | 2 | 95 | 0.50 | 217.5 |
| 3. | 3 | 80 | 0.50 | 75 |
| 4. | 4 | 80 | 0.75 | 217.5 |
| 5. | 5 | 95 | 0.75 | 75 |
| 6. | 6 | 80 | 0.75 | 217.5 |
| 7. | 7 | 95 | 0.75 | 360 |
| 8. | 8 | 65 | 0.75 | 75 |
| 9. | 9 | 65 | 0.75 | 360 |
| 10. | 10 | 95 | 1.00 | 217.5 |
| 11. | 11 | 80 | 0.75 | 217.5 |
| 12. | 12 | 80 | 1.00 | 75 |
| 13. | 13 | 80 | 0.50 | 360 |
| 14. | 14 | 80 | 1.00 | 360 |
| 15. | 15 | 65 | 1.00 | 217.5 |

2.3.5 Nutritional Analysis

For each experimental data set, parameters such as MC, AA, TPC, TFC, CC and colour indices were determined using procedures outlined in sections 2.1.5.1, 2.1.5.2, 2.2.5.3, 2.2.5.4, 2.2.5.5 and 2.2.5.6 of the Ph.D. thesis respectively.

2.3.6 Statistical Analysis

The statistical analysis was performed based on 95 % confidence level and total error criteria. Analysis of variance (ANOVA) was adopted to affirm best model fitness in terms of F-value and p-value

associated to each significant and insignificant model terms. 3D response surface plots have been prepared for the graphical analysis of the response variable sensitivities with respect to the variation in any two independent variables. Terms with significant F-value, low p-value and insignificant lack of fit affirm fitness of model coefficients to best represent each response. Further, model adequacy has been affirmed from the appropriate status of R² and adjusted R² values (Pua et al., 2010).

2.3.7 Optimization of Process Variables

The best fit model for each response variable were utilized for numerical optimization. The non-linear programming based optimization modelling environment in the software considered the simultaneous maximization of AA, TPC, TFC, CC and colour indices and minimization of MC responses within the specified boundaries of temperature, time and air-velocity. Thereby, optimal value set was determined for the RWD process (Mondal et al., 2020).

2.3.8 Validation of Optimum Values

The RSM based optimal data set has been validated through a triplicate data set being evaluated experimentally. Thereby, the average response values assured confidence in the value indicated by the RSM. For each response, the standard errors were calculated and the relative variation between the experimentally and modelling based values were evaluated. Thereby, the RSM based optimal data has been validated experimentally (Majdi et al., 2020).

2.4 Fortification of the Optimal Refractance Window Dried *Curcuma longa*

Powder Product

2.4.1 Raw materials, Chemicals and Sample Preparation

Turmeric was procured from market complex, Indian Institute of Technology Guwahati, Kamrup, Assam, India and was packed in a polythene pouch to prevent contamination in due course of its

transportation. Sodium ferric ethylenediaminetetraacetate (NaFeEDTA), folic acid, potassium bromide, enzymes and other chemicals were obtained from Sigma Aldrich India. Subsequently, the procured raw turmeric was washed with tap water to remove surface contaminants and dirt. Thereby, the sample was wiped with tissue paper to remove excess water. Thereafter, the wiped turmeric was peeled. Eventually, using an adjustable slicer, the peeled turmeric was sliced to achieve sliced sample pieces with 1 mm thickness.

2.4.2 Refractance Window Drying

The RWD experiments were conducted with optimal combinations of sample thickness, mylar film thickness, temperature, air velocity and drying time that were being achieved with prior experimental investigations. These have been set as 1 mm, 250 μm , 95 $^{\circ}\text{C}$, 0.75 m/s and 75 min respectively. Thereafter, the dried samples were powdered using a dry portable electric grinder. Eventually, sieved samples through a 80-mesh sieve were obtained that possessed an average particle size of 0.177 mm (Daulay et al., 2019).

2.4.3 Fortification with Sodium Ferric Ethylenediaminetetraacetate and Folic Acid

The relevant prior art for turmeric fortification indicated the following relevant procedures. Tripathi and Patel, (2010) reported 6 mg iron fortification per 100 g of finger millet flour. Similarly, Karn et al., (2014) fortified 100 g of curry powder with 20 mg iron. Further, Berry et al., (2009) mixed dry folic acid with maize flour and achieved 100 – 150 $\mu\text{g/day}$ folic acid on a bio-availability basis. Modupe et al., (2019) reported salt fortification with folic acid to achieve 1 % folic acid of the total salt content. Considering these mentioned quantities of fortification, 100 g of RW dried turmeric powder was mixed with 20 mg NaFeEDTA or 20 mg folic acid to eventually achieve iron and folic acid fortified turmeric powder samples. For both cases, dry mixing using a spatula was followed.

2.4.4 Characterizations of Refractance Window Dried *Curcuma longa* Powder Products

2.4.4.1 Associated Studies

For RW dried turmeric powder, folic acid fortified and NaFeEDTA fortified turmeric powder samples, characterization were addressed in terms of associated parameters such as bulk density, solubility, swelling power, water binding capacity, dispersion time, hygroscopicity and colour. A brief account of adopted procedures has been as follows.

To measure the bulk density of RW dried turmeric powder, folic acid fortified and NaFeEDTA fortified turmeric powder samples bulk density, 5 g of turmeric powder was placed first into a measuring cylinder of 10 mL. The volume occupied by the turmeric powder in the cylinder was recorded and the bulk density was calculated as the ratio of the weight to the volume of the turmeric powder sample (Tontul et al., 2018).

The solubility of the RW dried turmeric powder, folic acid fortified and NaFeEDTA fortified turmeric powder samples was determined by first mixing 1 g of turmeric powder sample with 100 mL distilled water at ambient temperature. The mixing was carried out using a magnetic stirrer (Make: Tarson) being operated for 5 min at 600 rpm. Thereafter, the mixture was centrifuged for 5 min at 3000 G. Thereby, 20 mL of the obtained supernatant was decanted to pre-weighed petri dish and was dried at 70 °C until constant weight of the system was achieved. Subsequently, percentage solubility was determined in terms of the weight difference of the processed petri dish sample and empty petri dish system. Thereby, solubility was evaluated using the expression:

$$\text{Solubility (\%)} = \frac{W_d}{W_s} \times 100 \quad (13)$$

The swelling power as calculated using the following expression.

$$\text{Swelling Power (g/g)} = \frac{W_{sd}}{W_s} \quad (14)$$

In the above expressions, W_s , W_d and W_{sd} are original sample weight, dried residue and dried sediment mass (Chisenga et al., 2019; Tontul et al., 2018).

Using the following method, the water binding capacity (WBC) of RW dried turmeric powder, folic acid fortified and NaFeEDTA fortified turmeric powder samples was measured. Firstly, 5 g of turmeric powder was mixed with 75 mL of distilled water. Thereby, the system was agitated for 1 h at 860 rpm and 20 °C. Thereafter, the sample was centrifuged at 3000 G for 10 min. Subsequently, the supernatant was removed, drained for 10 min and weighed. Subsequently, WBC was evaluated using the expression:

$$\text{WBC (\%)} = \frac{M_w - M_d}{M_d} \times 100 \quad (15)$$

where, M_w and M_d are the wet weight (g) and dry weight basis of the powder (g) (Chen et al., 2017).

The dispersion time was determined through the following procedure. Firstly, 80 mL of distilled water was transferred into a 100 mL beaker and was kept in the ambient environment (27 °C). Thereby, 1 g of the powder sample was placed in a slider that separated the powder and liquid surface. The dispersion time measurement started at the very instance that corresponds to the powder sample and liquid being brought into contact through the quick removal of the slider that separated the powder and liquid. Thereby, the time was measured for the complete spontaneous wetting and immersion of the 1 g powder (Castoldi et al., 2017).

The hygroscopicity of the sample was determined by following the procedure summarized by Tontul et al., 2018. According to the authors, hygroscopicity can be expressed in terms of the moisture mass (g) being absorbed by 100 g of sample during 7 days of storage at 25 °C and 92 % relative humidity. To achieve these conditions, a desiccator with a saturated Na_2SO_4 solution was arranged. Thereby, 1 g of the sample was weighed in a petri dish and was transferred into a desiccator for mentioned time period (7 days). Subsequently, hygroscopicity was determined using the expression:

$$\text{Hygroscopicity(\%)} = \frac{\frac{x}{a_h + W_i}}{1 + \frac{x}{a_h}} \times 100 \quad (16)$$

where x corresponds to the enhancement in powder sample (g), a_h corresponds to the powder sample amount

being used for the measurement (g) and W_i refers to the water content of the powder being exposed to the humid environment (Caparino et al., 2012).

The colour measurement for powder samples was conducted by following the procedure being outlined in section 2.1.5.6 of the Ph.D. thesis.

2.4.4.2 Fourier Transformed Infrared Spectroscopic Analysis

The FTIR spectral analysis was conducted for RW dried turmeric powder, folic acid fortified and NaFeEDTA fortified turmeric powder samples using the procedure outlined in section 2.2.9 of the Ph.D. thesis respectively.

2.4.4.3 Thermal Analysis

The thermal transition properties of RW dried turmeric powder, folic acid fortified and NaFeEDTA fortified turmeric powder samples were determined using a differential scanning calorimeter (Make: Neitzsch, Model: DSC-3500 Sirius,) and a thermo gravimetric analyser (Make: Libra, Model: TG 209). Being calibrated with indium, the instrument considered an empty pan as a reference set. For the measurements, 6 mg sample was kept on to the aluminium DSC and TGA pans and the system was subsequently sealed. Thereby, with a heating rate of 10 °C/min in the calorimeter, the powder samples were scanned in the temperature range of 0 – 600 °C and 0 - 1000 °C for DSC and TGA respectively in cases of RW dried turmeric powder, folic acid fortified and NaFeEDTA fortified turmeric powder (Hasanvand et al., 2015).

2.4.4.4 Particle Size, Poly Dispersity Index, and Zeta Potential

The average particle size, polydispersity index (PDI), and zeta potential of the RW dried turmeric powder, folic acid fortified and NaFeEDTA fortified turmeric powder samples were determined using a

dynamic light scattering (Make: Beckman Coulter, Model: Delsa Nano C) size analyser (Hasanvand et al., 2015).

2.4.4.5 Morphological Characterization

Field emission scanning electron microscopy (Make: Zeiss, Model: Sigma) was deployed for the morphological characterization of the RW dried turmeric powder, folic acid fortified and NaFeEDTA fortified turmeric powder samples. To do so, an appropriately small quantity of turmeric powder was placed in the carbon tape and was coated with a thin layer (< 20 nm) of gold using a sputter coater. The coated samples were investigated under 3 kV (Hasanvand et al., 2015).

2.4.4.6 X-Ray Diffraction Analysis

The crystallographic structural analysis of RW dried turmeric powder, folic acid fortified and NaFeEDTA fortified turmeric powder samples was conducted using X-ray diffractometer (Make: Rigaku Technologies, Model: Smartlab). To do so, the instrument was operated with Cu K-alpha-1 radiation (0.154 nm), 40 kV voltage and 30 mA current. While conducting the analysis, about 20 mg of the sample powder was loaded onto a glass plate for subsequent scanning in the range of 5 to 90 °Bragg angle and 0.02° per second measurement frequency (Hasanvand et al., 2015).

2.4.5 In-vitro Analysis

The in-vitro digestion was conducted for RW dried turmeric powder, folic acid fortified and NaFeEDTA fortified turmeric powder samples. A brief account of the adopted methodology has been outlined in the following sub-sections.

2.4.5.1 Digestion Process

The in-vitro digestion process was conducted to simulate the human digestive system. Thus, a two-step digestion process was followed that referred to the simulated gastric and intestinal digestion processes. For the gastric digestion process, 5 g of dried powder sample was first mixed with 30 mL of 140 mM NaCl and 5 mM KCl solutions. Thereby, 0.5 mL of pepsin solution (11000U/ml) was added for subsequent stirring for 2 h in a shaking water bath kept at 37 °C. During the stirring process, 1 M HCl solution was used to maintain the system at a pH of 2. Thereafter, the sample pH was adjusted to 5 by using 1 M NaHCO₃ solution. Subsequently, the intestinal digestion procedure was followed through the addition of 2.5 mL of pancreatin-bile solution (0.45 g of bile salts and 0.075 g of pancreatin in 37.5 mL of 0.1M NaHCO₃ solution) to the gastric digested mixture. Eventually, 40 µL of 0.3M CaCl₂ was added and the system pH was adjusted to 7.0 using 1M NaOH solution. Thereafter, the system was incubated for 2 h in a shaking water bath (Make: Labtop) at 37 °C. Finally, the digested samples were cooled in ice for 10 min and centrifuged at 5000 rpm for 40 min at 4 °C. The final digested sample was subjected to the curcumin, folic acid and iron content analysis. (Wahengbam et al., 2019).

2.4.5.2 Bio-accessibility of Curcumin

The supernatant of the centrifuged sample after in-vitro digestion was collected and analysed following the method listed at 2.1.5.5.

2.4.5.3 Bio-accessibility of Folic Acid

For folic acid estimation, standard folic acid solution in 0.01 N NaOH was used to prepare a calibration curve, that confirmed a linear fitness plot between the folic acid concentration and the absorbance being measured. Eventually, the absorbance of the digested sample was measured at 284 nm with a UV/Vis spectrophotometer. Thereby, the concentration of the sample was obtained from the calibration curve (Li et al., 2011).

2.4.5.4 Bio-accessibility of NaFeEDTA

After completing in-vitro digestion studies, the NaFeEDTA content in the digested sample was analysed using atomic absorption spectrophotometer (AAS) (Make: Varian, Model: Spectra AA 220 FS). Thereby, the iron content of the sample was determined using a calibration curve being prepared with stock solutions of NaFeEDTA (Contreras-Jiménez et al., 2019).

2.4.6 Statistical Analysis

The procedure and methodology being adopted for the statistical analysis of the RW dried turmeric powder, folic acid fortified and NaFeEDTA fortified turmeric powder samples was similar to that being outlined in section 2.1.6 of the Ph.D. thesis.

2.5 Storage and Shelf-life Studies

2.5.1 Sample Preparation

The storage and shelf-life studies were conducted for RW dried turmeric powder, folic acid fortified and NaFeEDTA fortified turmeric powder samples. The raw materials, chemicals deployed and subsequent sample preparation procedure was similar to those presented in section 2.4.1 of the Ph.D. thesis. The refractance window drying of the prepared intermediate samples was as per the procedure outlined in section 2.4.2 of the thesis. Thereby, fortification procedures with NaFeEDTA and folic acid were followed according to the procedure outlined in section 2.4.3 of the Ph.D. thesis. For the mentioned samples, storage and shelf-life studies were targeted in terms of the sorption characteristics, nutritional and physical variations in the samples during storage period.

2.5.2 Evaluation of Sorption Characteristics

The sorption characteristics were determined for RW dried turmeric powder, folic acid fortified and NaFeEDTA fortified turmeric powder samples. To do so, procedure for sorption characteristics being

followed by Iglesias and Chirife, (1982) were chosen. For all mentioned samples, the adsorption isotherm studies were carried out at 40 °C. To do so, firstly, 20 g of turmeric powder samples were placed in petri plates and were thereby kept in eight distinct desiccators. These desiccators constituted saturated solution of various salts that customized the achievement of relative humidity (RH, 11.2 to 79.9 %) in the environment. Eventually, desired RH condition was met in distinct desiccators. Thereby, they were placed in an incubator (Make: Dass & Co) at 40 °C. To prevent mould growth, a dish containing 5 mL toluene was placed in the desiccators that facilitated an environment with relative humidity over and above 75 %. Thereafter, periodical weight measurement of the samples was conducted until moisture equilibrium (constant weight) was achieved. Thereafter, the samples were analysed for the moisture content. The moisture sorption isotherms were prepared in terms of the equilibrium moisture content and water activity plots. The fitness of the appropriate model for experimentally determined water activity and equilibrium moisture content was ensured through the Guggenheim-Anderson-de Boer (GAB) model being expressed as:

$$M = \frac{M_0 \times C \times K \times a_w}{(1 - K \times a_w)(1 - K \times a_w + C \times K \times a_w)} \quad (17)$$

where, M_0 is the monolayer moisture content (db), M is the EMC (db) and, C and K are constants. In the above expression, parameter (M_0 , C , K) values were determined using non-linear curve fitting module of the Origin Pro 9 software (Kumar and Mishra, 2004).

2.5.3 Shelf-Life Characteristics

For the mentioned RW dried turmeric powder, folic acid fortified and NaFeEDTA fortified turmeric powder samples, zipper pouches (14 cm × 10 cm) were deployed to host 20 g of the powder samples. Thereby, the pouches were kept in a desiccator at 38 ± 1 °C. The desiccator constituted a saturated solution of potassium nitrate to thereby facilitate an environment with 90 % relative humidity. About eight sample pouches for all three cases were kept in each desiccator to thereby ensure that all pouches were maintained at similar environmental condition. Thereby, the moisture content of the sample was determined

at an uniform interval until a moisture content of 8 % was reached. Eventually, the sample shelf life was estimated using the expression:

$$\int d\theta = \frac{W_s}{P^*KA} \int_{X_i}^{X_c} \frac{dX}{RH - a_w} \quad (18)$$

where θ denotes shelf-life (days), W_s refers to weight of dry solids (g), P^* refers to saturated vapour pressure of water at ambient temperature (Pa), K refers to water vapour permeability of the packaging material ($\text{kg/m}^2\text{dayPa}$), A denotes area of the package (m^2), RH refers to relative humidity of the environment in which the package is placed (%), a_w denotes water activity of the powder, X_i refers to initial moisture content (% db) and X_c denotes critical moisture content (% db).

For the packaging material, water vapour permeability K ($\text{kg/m}^2\text{dayPa}$) was determined using the expression:

$$K = \frac{dw/d\theta_p}{A_p P^*} \quad (19)$$

where $dw/d\theta_p$ is the slope of the straight line fit of the plot drawn between time and weight of silica gel kept within the packaging material, A_p is the surface area of the packaging material (m^2), and P^* is saturation vapour pressure of water at the packaging environment temperature (38 ± 1 °C) (Pa) (Jaya and Das, 2005).

2.5.4 Nutritional and Physical Characteristics of Stored Dry *Curcuma longa* Powder Samples

One zipper pouch (20 g) for RW dried turmeric powder, folic acid fortified and NaFeEDTA fortified turmeric powder samples were taken out at regular duration of time for assessment of physical and nutritional characteristics of the stored powder. The storage study was conducted at 38 ± 1 °C and the samples were analysed in terms of the associated nutritional and physical variations of the samples at specified time periods of the samples (0, 9, 17, 24 weeks). The nutritional and physical variations of the powder samples were determined in terms of MC, AA, TPC, TFC, CC, colour indices, bulk density, solubility, swelling power, water binding capacity, dispersion time and hygroscopicity. Relevant

procedures delineated in sections 2.1.5 and 2.4.4.1 for nutritional and physical variations have been duly followed for the mentioned parametric evaluations.

For folic acid estimation, standard folic acid solution in 0.01 N NaOH was used to prepare a calibration curve, that confirmed a linear fitness plot between the folic acid concentration and the absorbance being measured. For the sample whose folic acid needs to be determined, 5 g of sample was taken and mixed with 25 mL of 0.01 N NaOH in a vortex mixer for 2 min. The aqueous solution was then filtered using a Whatman No. 1 filter paper. Eventually, the absorbance of the filtered sample was measured at 284 nm with a UV/VIS spectrophotometer. Thereby, the concentration of the sample was obtained from the calibration curve (Li et al., 2011).

For iron content estimation, 1 g of the sample was converted into ash by incineration at 550 °C for 5 h. Thereafter, the incinerated sample was digested with nitric acid (Baker 69 – 70 %). The digested sample was eventually filtered using a Whatman No. 1 filter paper. The filtrate was then analysed for iron content using an atomic adsorption spectrophotometer. Thereby, the iron content of the sample was determined using a calibration curve being prepared with stock solutions of NaFeEDTA (Contreras-Jiménez et al., 2019).

2.5.5 Sensory Analysis and Nutritional Characteristics of Refractance Window Dried Turmeric Powder Based Golden Milk Product

2.5.5.1 Sensory Analysis

The sensory analysis was conducted for only RW dried turmeric powder samples. This is due to the fact that the fortified samples deployed non-food grade folic acid and iron precursors. Subsequently, sensory analysis was conducted by preparing golden milk samples. The sensory analysis first involved variant turmeric constitution (0.5 g – 2 g in 100 mL of milk (Amul Taaza)) based turmeric fortified milk product preparation. Thereby, the prepared fortified liquid samples were subjected to sensory characterization through a panel of fourteen subjects that possessed adequate knowledge with respect to the

Table 2.3: The 9-point hedonic scale for sensory analysis.

| S. No. | Point | Response |
|--------|-------|--------------------------|
| 1. | 9 | Like extremely |
| 2. | 8 | Like very much |
| 3. | 7 | Like moderately |
| 4. | 6 | Like slightly |
| 5. | 5 | Neither like nor dislike |
| 6. | 4 | Dislike slightly |
| 7. | 3 | Dislike moderately |
| 8. | 2 | Dislike very much |
| 9. | 1 | Dislike extremely |

product characteristics and associated sensory attributes of dairy products. With their expertise in terms of their regular involvement with the sensory evaluation of such products, sensory analysis data was customized using 9-point hedonic scale (Table 2.3) to represent colour and appearance, taste, aroma, mouthfeel, aftertaste, consistency and overall acceptability (Stone and Sidel, 2004).

2.5.5.2 Characterization of Optimal Gold Milk Formulation

For the sensory analysis based optimal gold milk formulation, relevant characterizations were carried out in terms of AA, TPC, TFC and CC. All these experiments were carried out in triplicates and the average data has been reported in the thesis.

2.5.5.2.1 Total Phenolic Content

The total phenolic content was estimated using the FCR method (Tharasena and Lawan, 2014). The sample processing procedure involved the mixing 1 mL of golden milk with 1 mL of prepared FCR reagent in a vortex mixer. After 5 minutes of mixing, 10 mL of 7 % sodium carbonate solution was added to the mixture. After subsequent vortex mixing, the final solution volume was adjusted to 25 mL. Finally, the solution was kept in a dark environment for 2 h at ambient temperature followed with absorbance evaluation

at 750 nm using UV spectrophotometer (UV-2600, Shimadzu, Singapore). Using a calibration chart being prepared with gallic acid, the TPC content of the sample was expressed in terms of mg of GAE/g sample.

2.5.5.2.2 Total Flavonoid Content

AlCl₃ method was adopted to determine the TFC (Tharasena and Lawan, 2014). Firstly, this involved vortex mixing of 1 mL of golden milk with 4 mL distilled water. Thereafter, 0.3 mL of 5 % NaNO₃ solution was added and the mixture was again subjected to vortex mixing for 5 minutes. Eventually, 0.3 mL of 10 % AlCl₃ was added to the mixture for subsequent vortex mixing for 6 minutes. Later, 2 mL of 1M NaOH was added to the solution and the mixture subjected to mixing. The final solution with a volume of 10 mL was kept in a dark environment for 30 minutes at ambient temperature. Thereafter, the mixture absorbance was measured at 517 nm. Using quercetin based calibration chart, the TFC of the sample was determined in terms of mg of quercetin equivalent/g sample.

2.5.5.2.3 Anti-oxidant Activity

The golden milk product was subjected to total antioxidant activity evaluation by using the 1,1-diphenyl-2-picryl hydrazyl (DPPH) assay method (Sutanto et al., 2015). To do so, firstly, 2 mL golden milk was mixed with 2 mL DPPH stock solution (1:1). Simultaneously, a control sample was prepared using distilled water and DPPH stock solution. Thereafter, the samples were incubated at 30 min in a dark environment at room temperature. Eventually, the samples were analysed for their absorbance at 510 nm. Thereby, the % AA was determined using the expression:

$$A.A (\%) = \frac{A_c - A_s}{A_c} \times 100 \quad (20)$$

where A_c and A_s corresponds to the absorbance of control and sample respectively.

2.5.5.2.4 Curcumin Content

The curcumin content of the golden milk was determined by adopting the procedure summarized by Talukdar and Uppaluri, (2021). Firstly, 1 mL of milk was placed in a test tube and was boiled with 10 mg dried turmeric sample. Thereafter, the sample was cooled to room temperature to add 5 mL of 95 % methanol. After subsequent sonication for 10 min, 5 mL of extract was mixed with 95 % methanol to achieve a solution volume of 10 mL. Thereafter, the solution was filtered using Whatman filter paper No. 1. After filtration, 0.4 mL of filtrate was added to 5 mL of 95 % methanol. The absorbance of the samples was measured at 425 nm using a UV-VIS spectrophotometer. Using the measured absorbance of the standard and turmeric processed samples, the curcumin content of the golden milk was determined using a calibration curve in terms of % curcumin content w/w.

2.6. Preparation and Characterization of Lakadong Turmeric Based Golden Milk Product

2.6.1 Raw Materials

Lakadong turmeric (from Meghalaya, India) was purchased from MIHA Trading firm of Guwahati, India.

2.6.2 Sample Preparation

To eliminate surface contamination and dirt, Lakadong raw turmeric rhizomes were washed with tap water. Thereafter, they were wiped with tissue paper to remove excess water. Subsequently, the samples were peeled using an adjustable slicer (Model: VDNSI) to achieve sliced Lakadong turmeric samples with 1 mm thickness.

2.6.3 Tray and Refractance Window Drying

Laboratory scale tray drier (Make: International commercial traders, Kolkata, India) was deployed to dry 1 mm slices at 60 °C for 8 h. For the RWD, the Lakadong turmeric slices were placed on the mylar film and dried at 95 °C for 75 mins. The obtained dried samples from tray and RW drying systems were thoroughly grounded to achieve powdered samples.

2.6.4 Characterizations of Dried Lakadong Turmeric Samples

Physical and chemical characterization were conducted for the Lakadong turmeric tray, RWD and fresh samples. Procedures delineated in sections 2.1.5.1 - 2.1.5.6 were followed to determine characteristics such as MC, AA, TPC, TFC, CC and colour indices for mentioned samples. Also, the procedures summarized in section 2.2.9 have been followed to conduct the FTIR analyses of fresh, tray dried and RW dried Lakadong samples.

2.6.5 Preparation and Characterization of Lakadong Turmeric Based Golden Milk

Among tray dried and RW dried Lakadong turmeric powdered samples, the latter is well known for its superior curcumin content. Thereby, golden milk product was prepared with 100 mL of milk (Amul Taaza) and variant constitution of RW dried Lakadong turmeric powder (0.5 – 2 g). Thereby, sensory analyses were conducted for all prepared golden milk samples by adopting delineated procedure of section 2.5.5.1 of the Ph.D. thesis. Similarly, using methods summarized in section 2.5.5.2, alternate characterizations were carried out for the optimally prepared golden milk product.



Chapter 3:
Techno-economic Efficacy of Refractance
Window Dried *Curcuma longa*



Techno-economic Efficacy of Refractance Window Dried *Curcuma*

longa

In this chapter, the key findings associated with the optimality of oven, tray and refractance window drying methods have been summarized from the perspective of the maximum retention of nutritional parameters. A comparative assessment has been targeted among oven, tray and refractance window drying processes along with the sample variety (slice and paste). The best drying method and sample form for high retention of nutritive properties was found from the conducted studies. Section 3.1 presents a brief introduction to the chapter. Thereby, section 3.2 details upon various drying times of the dried turmeric sample. Section 3.3 delineates on the physical and nutritive analysis of the dried product. The section 3.4 provides a sensitivity analysis of the drying process with respect to the responses and section 3.5 details upon the statistical analysis of the responses. Further section 3.6 mentions the energy consumption for each drying method. Thereby, the subsequent cost estimations to produce the dried turmeric product has been discussed in section 3.7. Finally, literature comparison with relevant prior art and conclusion of the chapter have been presented respectively in sections 3.8 and 3.9.

3.1 Introduction

Drying process significantly influences mass and heat transfer rates of processed foods. The pertinent phenomena are complex functions of the composition, structure, shape and size of the samples. Drying methods to process foods include hot-air oven, convective, tray, freeze, spray, vacuum, microwave and fluidized bed drying methods. Among these, due to longer drying period and lower drying rates, hot-air oven drying is not highly promising to retain taste, color and nutritional content of the product (El-Safy,

2014). On the other hand, tray drying is economical and commonly deployed but suffers from the limitations of product quality. Application of such tray drying is apparent for various leafy and non-leafy vegetables (Mondal et al., 2020a; Mondal et al., 2020b). From processing time perspective, tray drying requires about 2.5 - 5.5 h of high temperature (70 – 50 °C) operation. The drying time increases to about 18 – 24 h for freeze drying at a reduced temperature of 20 °C (Nindo et al., 2003). From the cost perspective, it is well known that vacuum (Drouzas et al., 1999) and freeze (Nindo & Tang, 2007) drying are significantly expensive methods and are not recommended towards scale-up assisted marginal enhancement in process cost to achieve the high volume of the dried products.

To overcome the limitations of the mentioned drying processes such as high processing time, higher cost, scalability and retention of nutritional parameters, a novel drying method such as refractance window drying (RWD) can be targeted. On the other hand, limited investigations have been conducted with respect to the RWD of vegetable resources and their comparative assessment with other conventional processes such as tray drying. Akinola et al., (2016) observed that 3 mm carrot slice could be effectively dried below 10 % (db) moisture content (MC) in 200 min drying time using RWD. Abbasid et al., (2015) inferred that for 2 mm tomato slices, the RWD process provided higher resemblances of nutritional characteristics with respect to those evaluated for the fresh sample.

For the turmeric drying, few oven drying studies have been targeted but not the RWD. A comparative assessment of RWD process in terms of its conceptual cost competitiveness with other horticultural produce drying methods has not been addressed till date. Such investigations would be useful to further enhance the applicability of novel RWD process in the food process industries. Considering all indicated lacunae, the chapter devotes to the RWD characteristics of turmeric samples. Both paste and slice samples have been considered along with a comparative assessment of the oven and tray dried samples. Finally, cost efficacy of RWD process in comparison with oven and tray drying methods has been addressed to indicate upon its economic competence and effectiveness.

3.2 Drying Time

An operating temperature of 60 °C for oven (O) and tray (T) drying process and for 1 mm slice (S) and paste (P) was targeted with the available literature information on the oven and tray drying of turmeric slice. The drying time for oven and tray drying for both 1 mm slice (OS and TS) and 1 mm paste (OP and TP) was obtained through the drying kinetic studies. The RWD time at 60 °C for both slice (RS) and paste (RP) samples were determined from drying kinetics studies. These variations in temperature and sample thickness have also been ensured through preliminary experiments at the chosen combinations of drying time and temperature to ensure upon the achievement of the good combinations of nutritional parameters in relevant cases. In oven and tray drying, time taken to dry 1 mm slices and 1 mm thick paste at 60 °C was 8 h and 4.5 h respectively. Similar experiments were conducted in RWD to dry 1 mm slices and 1 mm thick paste for 7 and 3 h at 60 °C respectively.

3.3 Physical and Nutritional Characteristics Oven, Tray and Refractance Window Dried *Curcuma longa* Samples

3.3.1 Yield and Rehydration Ratio of Refractance Window Dried *Curcuma longa* Samples

The fresh and raw agricultural produces possess higher MC. For long term storage, there is a need to reduce the MC. Henceforth, drying processes are applied. After drying, it is important to achieve a good sample yield for sustainability. Alternate drying processes affirm variant yield values. Hence, it is necessary to identify the best process with the highest yield after drying.

With a MC of 87 – 88 % in fresh turmeric, the yield of turmeric after RWD was about 12 – 13 % (Table 3.1). This is significantly higher with respect to the yield of the samples obtained with oven and tray drying (yield about 8 – 10 %). Drying yields of freeze dryer, spray dryer and refractance window (RW) dryer were reported for pomegranate juice sample. In these studies, the spray drying system allowed the materials to adhere to the wall chamber. Hence, few smaller particles were lost during cyclone operation and entered into the exhaust air. However, doctor blade installed on the RW system was able to scrape off

almost all the dried product. Hence, the RW system based product yield was much higher than the yield obtained with other mentioned drying processes (Baeghbal et al. 2016).

Higher yield is directly correlated with the moisture content of dried turmeric sample. After repetitive experiments, it was found that the yield of turmeric after RWD was 12 — 13 % but was only 8 — 10 % after oven and tray drying. This could be attributed to the fact that RWD is a fast-drying process in comparison to the slower oven and tray drying process. Due to the fast drying RWD process, higher retention of the constituents prompted cellular integrity is possible in the RWD. However, this was not the case for oven and tray drying process. Thus, the cellular degradation of carbohydrates and protein compounds may have occurred in the oven and tray drying process. However, future investigations are required to converge on this hypothesis.

During reconstitution of dehydrated products, the amount and rate of water absorption considerably alters the sensory properties and preparation time of the dried products. Hence, rehydration characteristics of a dried product are often used as a quality index and are opined to very likely indicate physical and chemical changes during drying. This is due to the pertinent role of these processing conditions, sample pretreatment and composition (Jafari et al., 2016).

For 5 g dried turmeric sample, the rehydration rate was analysed to be 1.50, 2.0 and 5.60 (Table 3.1) for oven dried, tray dried and RW dried samples respectively. Thus, drying methods had a significant influence on the rehydration rate. Actually, such variations could be due to the distinction between different types of energy transition from the resource to the product (Abonyi et al., 2002). Relevant prior art also corroborates with the observation that the RW dried sample had higher rates of rehydration ratio index in comparison to the samples obtained with oven and tray drying processes (Nindo and Tang, 2007).

The ability of food products to reconstitute as a slice or diced sample primarily depends on the internal structure of the dried pieces and on damage extent caused to the water holding constituents (e.g., proteins and starch) in due course of drying. For example, in a hot air-drying process, higher drying rate during the initial time frame may cause case hardening of the product surface and hence forth an irreversible loss in the fast reconstitution ability (Nindo et al., 2003). Also, Nindo et al., (2003) inferred that the

Table 3.1: Yield and rehydration ratio data of oven, tray and refractance window dried turmeric powder products.

| S. No. | Characteristics | RWD | Oven | Tray |
|--------|-------------------|-------|------|-------|
| 1. | Yield (%) | 13.36 | 9.01 | 10.58 |
| 2. | Rehydration Ratio | 5.60 | 1.50 | 2.00 |

* All standard deviations for yield and RR were in the range 1 – 1.5 and 0.5 – 0.7 respectively.

Table 3.2: Moisture content data of dried *Curcuma longa* with alternate drying methods.

| S. No. | Drying methods | Temperature (°C) | Drying time (h) | Moisture content (% wb) |
|--------|----------------|------------------|-----------------|-------------------------|
| 1. | Oven Slice | 60 °C | 8 | 3.80 |
| 2. | Oven Paste | 60 °C | 4.5 | 2.00 |
| 3. | Tray Slice | 60 °C | 8 | 2.90 |
| 4. | Tray Paste | 60 °C | 4.5 | 1.20 |
| 5. | RWD Slice | 60 °C | 7 | 4.20 |
| 6. | RWD Paste | 60 °C | 3 | 3.70 |

* All standard deviations were in the range 0.1 – 0.3.

rehydration capacity results were in good agreement with shrinkage data and thereby concluded that a structure with lesser shrinkage had higher capacity to absorb water upon reconstitution. Hence, it can be inferred that the RWD processed sample did not undergo severe quality degradation in comparison to the oven and tray dried samples. Hence, the RWD process delivered the dried product with a higher rehydration ratio index.

3.3.2 Moisture Content

The estimation of MC of the dried sample is important in the context of its shelf life. For a drying process to be successful, a threshold level of less than 8 % MC is necessary in the dried product. This is due to the fact that the sample with a MC of less than 8 % will prohibit microbial contamination and can be henceforth stored for a long period of time (Mondal et al., 2020a).

Table 3.2 summarizes the MC of slice and paste turmeric samples obtained after oven, tray and RW drying. For the fresh turmeric sample, the MC has been evaluated to be significantly high and about 87 – 88 % (wb). Thereby, it confirmed upon the need for a significant reduction in the MC. It can be seen from the table that the MC varied from 2.9 - 4.2 % (wb) and 1.2 - 3.7 % (wb) for 1 mm slice and paste cases respectively and for all drying methods.

For both slice and paste sample, lowest (2.90 and 1.20 % wb) and highest (4.20 and 3.70 % wb) MC was obtained with tray drying and RWD at 60 °C respectively. Compared to tray and oven drying, significantly lower drying time was required for the RWD to achieve an MC value lower than 8 % (wb) to prevent microbial growth. This is due to effective heat transfer for the RWD case being facilitated by the Mylar film as a contact media between the slice/paste sample and hot water bath. In other words, besides convection, conduction played a dominant role to effectively reduce the MC in the sample. Santo et al., (2016) reported similar trends for carrot drying. For several cases, MC for paste has been significantly low comparison with the slice sample. This is due to the disruption of the cellular structure of turmeric during paste preparation that releases bound MC for its effective removal.

3.3.3 Total Phenolic Content

Phenolics constituents are important for the broad spectrum bio-chemical activities such as anti-mutagenic, anti-inflammatory and anti-aging abilities. Numerous studies affirmed direct relationship between the intake of phenolic content and reduction in cardiovascular and carcinogenic risks. Phenolic compounds have been found to be essential for the plants due to their ability to defend against infection and injury.

Fig. 3.1 depicts the total phenolic content (TPC) of turmeric slice and paste samples for various cases. The TPC of fresh turmeric is 81.55 mg GAE/g sample. The TPC enhanced to 125.86, 140.21 and 161.95 mg GAE/g sample for slice and 109.13, 111.30 and 118.69 mg GAE/g sample for paste samples obtained with oven at 60 °C, tray at 60 °C and RWD at 60 °C methods respectively. This enhancement in TPC for all dried samples is due to the significant reduction in MC of the dried samples. Highest and lowest

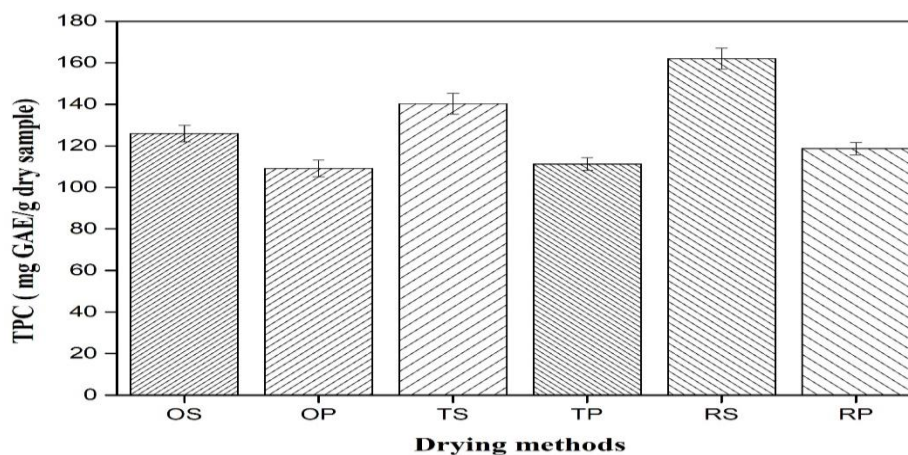


Figure 3.1: Total phenolic content of tray, oven and refractance window dried turmeric slice and paste samples [OS – Oven dried slice; OP – Oven dried paste; TS – Tray dried slice; TP – Tray dried paste; RS – Refractance window dried slice; RP – Refractance window dried paste].

TPC was achieved for the slice and paste samples subjected to RWD at 60 °C and oven drying at 60 °C respectively.

For all cases, TPC in the paste sample was significantly lower than those obtained for the slice samples. This is due to breakage of cellular structure that enhances the release of phenolic compounds and subsequent oxidation for the paste case. Among all cases, the RWD indicated the highest TPC values and this is due to significantly lower drying time. The significantly faster MC loss during RWD has been hypothesized to reduce oxygen partial pressure at the dried sample air interface due to the higher vapour pressure of the evaporated water (Abonyi et al., 2002). Thereby, lower oxidation would impede a reduction in the TPC (Santos et al., 2016). Further, it can be analyzed that RWD at 60 °C is the best, to facilitate higher TPC retention. This is due to faster removal of the MC at this condition.

3.3.4 Total Flavonoid Content

Several research articles confirmed that higher concentration of flavonoids is significantly correlated to the reduced risk of cardiovascular disease. This occurs through an improvement in vascular function and

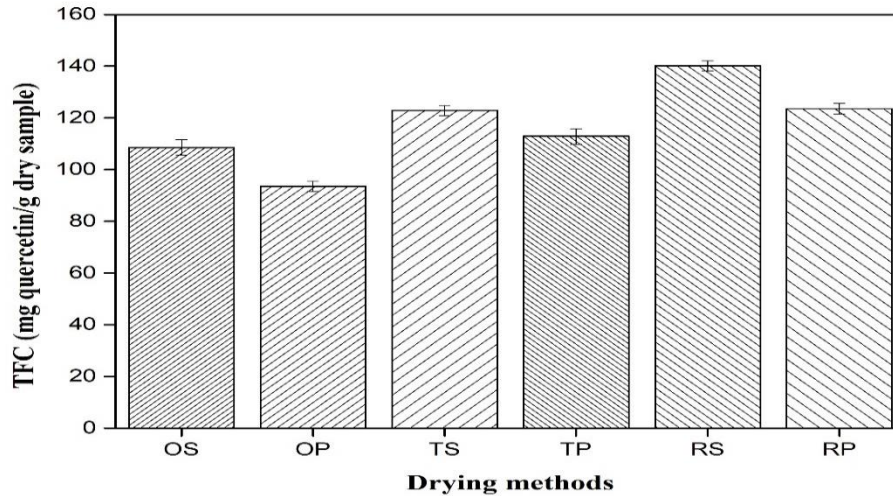


Figure 3.2: Total flavonoids content of tray, oven and refractance window dried turmeric slice and paste samples.

a modulation of inflammation. Moreover, the total flavonoid content (TFC) also protects cell and tissue damage from free radicals through their antioxidant characteristic and potential.

Fig. 3.2 illustrates the TFC of turmeric paste and slice samples obtained after drying with various methods. For the fresh sample, the average TFC has been evaluated as 17.41 mg quercetin/g sample. However, for the dried sample, the TFC can be analyzed to increase significantly to 108.54, 122.90 and 140.16 mg quercetin/g sample for slice case and 93.54, 112.90 and 123.54 mg quercetin/g sample for the paste case and with oven drying at 60 °C, tray drying at 60 °C and RW drying at 60 °C respectively. The enhancement in TFC after drying has been due to a significant reduction in the MC.

For all cases, slice samples possessed higher TFC than the paste samples. This is due to better cellular integrity in slice samples in terms of favouring oxidation of hidden TFC in the cell structure (Hernandez-Santos et al., 2016). The RWD at 60 °C reported highest TFC values for both slice and paste samples. This is due to the fast drying rate in short duration that detracts TFC oxidation and enhance the release of flavonoids from the samples (Rababah et al., 2015). Compared to oven and tray drying, it can be analysed that significant TFC retention can be achieved through RWD hence its promising performance.

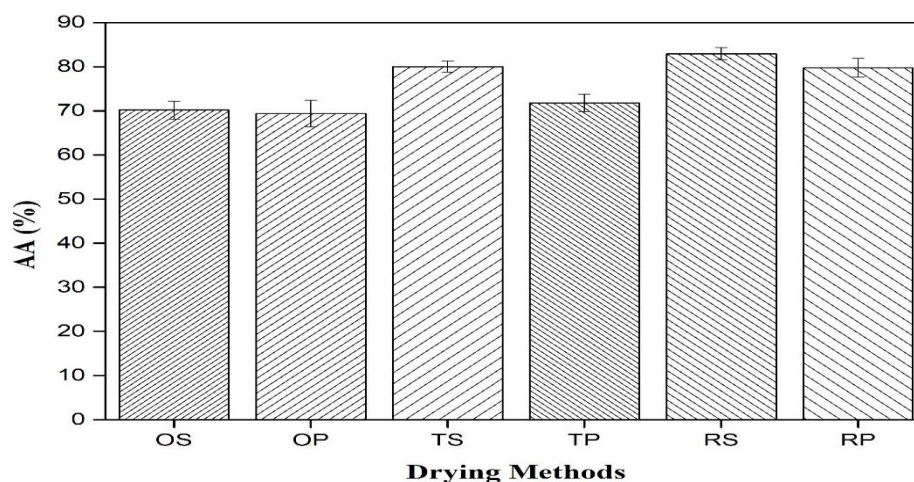


Figure 3.3: Antioxidant activity of tray, oven and refractance window dried turmeric slice and paste samples.

3.3.5 Antioxidant Activity

Oxidative stress is caused due to an imbalance between reactive oxygen species and the anti-oxidative defence systems that can lead to damage in cellular components such as lipids, protein and DNA. This has been conveyed to be a major reason for many diseases such as cancer, cardiovascular diseases, neurodegenerative diseases, rheumatoid arthritis, atherosclerosis and hypertension. Antioxidants inhibit or delay the oxidative process by blocking both the initiation and propagation of oxidizing chain reactions. Hence, antioxidant activity (AA) is an important parameter and its retention in significant quantities is required in the formulation of functional food.

Fig. 3.3 depicts the variations of AA for various drying methods. As shown, the AA varied as 70.17, 80.00 and 82.93 % for slice and 69.38, 71.76 and 79.75 % for paste samples obtained with oven drying at 60 °C, tray drying at 60 °C and RW drying at 60 °C processes respectively.

For fresh turmeric, the average AA value is 27.27 %. Compared to the fresh sample, such enhancement in AA is due to significant loss of moisture content due to drying. Among all cases, the lowest and highest AA have been obtained for samples obtained with oven drying at 60 °C, and RWD at 60 °C respectively.

Compared to paste samples, slice samples had marginally higher AA values. This is due to cellular disruption during grinding that enhanced surface area and reduced the retention of heat sensitive AA compounds in paste. It is well known that prolonged heating at higher temperature allows oxidation of AA compounds. However, RWD facilitates relatively lower temperatures and significantly shorter drying times and these features prompt upon the highest AA values. Thereby, the process conditions effectively reduce the oxidation of phenolic anti-oxidants (Santos et al., 2016). Also, the retention of TFC and TPC enabled high retention of AA content in the dried turmeric sample being obtained with the RWD (Delma and Verma, 2012).

3.3.6 Curcumin Content

With its lipophilic nature, curcumin is a polyphenolic compound that enables yellow colour to the turmeric. Due to the presence of curcumin as an active constituent and other volatile oils the turmeric is widely used as a medicine for the treatment of many ailments. It is vastly used as an anti-inflammatory agent. Therefore, the retention of curcumin is required from both a textural and polyphenolic characteristic. For fresh samples, the curcumin content (CC) was 0.73 % w/w CC. This is due to high MC of the sample. Fig. 3.4 shows significantly higher values of 3.59, 3.63 and 4.21 % w/w CC for slice sample and 2.80, 3.48 and 3.85 % w/w CC for paste samples obtained with oven drying at 60 °C, tray drying at 60 °C and RW drying at 60 °C respectively.

Among all samples, the slice sample achieved with the RWD at 60 °C possessed the highest value of CC of 4.21 % w/w CC. Compared to oven drying, tray drying and RWD, RWD was able to retain high CC. Compared to the quantitative differences in the retention of TPC, TFC and AA in RWD with respect to tray or oven drying, the quantitative difference in the retention of curcumin has been significantly low. This is due to the fact that heat treatment does not significantly influence the constitution of curcumin in the dried turmeric sample (Prathapan et al., 2009).

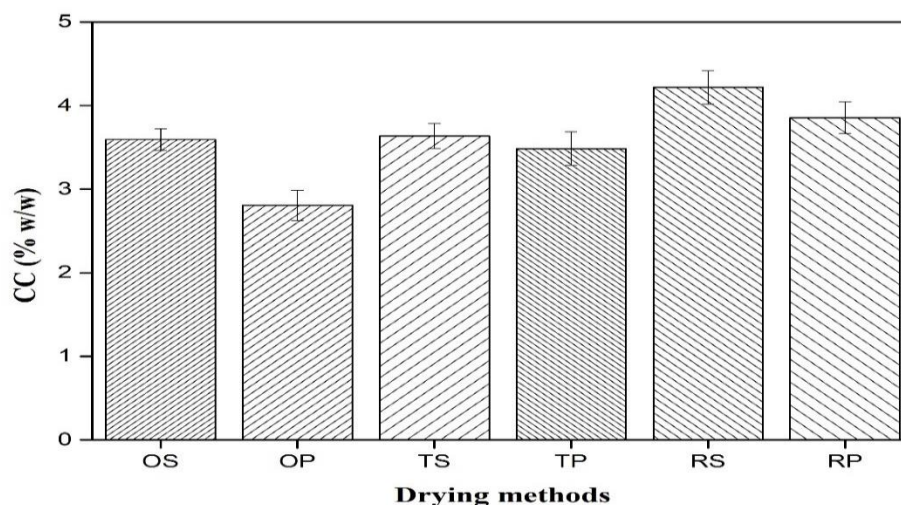


Figure 3.4: Curcumin content of tray, oven and refractance window dried turmeric slice and paste samples.

3.3.7 Colour Indices

The colour of a food product plays a major role in food product quality and as well influences the acceptability traits of the consumers. Also, the colour of a food product can be used as an indicator to predict the alterations in chemical and quality of the product due to the drying process. During colour analysis, the colour parameters are represented by L , a and b values reported by the Data Colour colorimeter. These parameters are co-related to one another. Hence, any alteration in a and b values will also change the L values. Therefore, the alteration in the values of colour indices (L , a and b values) could be used to determine the effect of drying process on the colour characteristics of the processed sample.

Table 3.3 summarizes these colour indices for oven, tray RW drying process and slice/paste samples. The table conveys that the drying process (oven, tray and RW drying) reduced the L parameter (whiteness) in comparison with the fresh sample for all cases. The L , a and b values for fresh sample was 63.67, 43.07 and 75.12 respectively. After drying, these varied as 42.88 – 24.75 (L), 25.65 – 14.36 (a), and 45.27 – 30.17 (b). Such reduction could be due to the formation of brown pigmented substances during drying. The oven and tray drying process affirmed lowest value of lightness than the values achieved with

Table 3.3: Colour indices of oven, tray and refractance window dried turmeric samples.

| S. No. | Sample | Drying process | <i>L</i> | <i>a</i> | <i>b</i> |
|--------|--------|----------------|----------|----------|----------|
| 1. | Slice | Oven | 36.63 | 17.65 | 29.22 |
| | | Tray | 38.57 | 19.98 | 36.72 |
| | | RWD | 42.88 | 25.65 | 45.27 |
| 2. | Paste | Oven | 24.75 | 14.36 | 30.17 |
| | | Tray | 27.12 | 18.12 | 33.90 |
| | | RWD | 32.64 | 21.23 | 38.89 |

* All standard deviations for *L*, *a* and *b* were in the range of 1 – 2, 2 – 3 and 1 – 3 respectively.

the RWD for both paste and slice sample. From the *L*, *a* and *b* values in the table, it can be understood that the RWD gave better results for both paste and slice sample. This may be due to the alterations in the drying process and drying time for each sample. With higher drying time, the oven and tray drying affirmed lower *L*, *a* and *b* values. The highest *L*, *a* and *b* indices were obtained for the RWD of slice. Corresponding lowest values were obtained for the oven dried paste sample. In the sample forms, highest colour values were achieved for the slice sample. This could be due to the cellular integrity of the slice sample that prevented rapid browning of the sample. Thereby, *L*, *a* and *b* indices could be kept without much reduction to the slice sample (El-Safy, 2014).

3.4 Sensitivity Analysis of Alternate Drying Process

The sensitivity analysis refers to the variation of a particular response after conducting drying. During sensitivity analysis, the maximum or minimum value being obtained after drying is important as it infers upon the percentage increase and reduction of the response with regard to the process parameter. In this chapter, the sensitivity analysis has been carried out to analyse the effect of oven, tray and RW drying on slice and paste form of the turmeric samples. The evaluation was based on the reduction of MC in due course of drying process and corresponding enhancement or diminution of responses such as AA, TPC, TFC, CC and colour indices of the samples. The sensitivity under these circumstances refers to the

proportional significance or marginal increase or reduction of nutritional characteristics with alteration in the MC. For example, if an 100 % increase in AA of the sample after drying can be achieved for a corresponding moisture content reduction by 100 %, it could be inferred that there has been negligible influence of the drying process parameters to detriment or increment upon evaluated response. The Table 3.4 (a – c) summarizes the sensitivity based analysis on all the response characteristics. From the table it can be observed that the RWD has higher percentage increase for AA, TPC, TFC and CC and lower percentage reduction for MC and colour content in comparison to tray and oven drying processes.

Table 3.4: Sensitivity characteristics of responses for (a) refractance window (b) tray and (c) oven dried turmeric samples.

| (a) | | | | | |
|--------|--------|----------|--------------|---------------|----------|
| S. No. | Sample | Response | Fresh sample | Max/min value | % change |
| 1. | Slice | MC | 87 | 4.20 | 95.17 |
| | | AA | 27.26 | 82.93 | 204.23 |
| | | TPC | 81.5 | 161.95 | 98.719 |
| | | TFC | 17.41 | 140.16 | 705.06 |
| | | CC | 0.73 | 4.218 | 477.83 |
| | | Colour | 63.67 | 42.88 | 32.65 |
| 2. | Paste | MC | 87 | 3.70 | 95.74 |
| | | AA | 27.26 | 82.93 | 204.23 |
| | | TPC | 81.5 | 118.69 | 45.63 |
| | | TFC | 17.41 | 123.54 | 609.64 |
| | | CC | 0.73 | 3.85 | 428.01 |
| | | Colour | 63.67 | 32.64 | 48.73 |

(b)

| S. No. | Sample | Response | Fresh sample | Max/min value | % change |
|--------|--------|----------|--------------|---------------|----------|
| 1. | Slice | MC | 87 | 2.90 | 96.66 |
| | | AA | 27.26 | 80.00 | 193.47 |
| | | TPC | 81.5 | 140.21 | 72.04 |
| | | TFC | 17.41 | 122.90 | 605.93 |
| | | CC | 0.73 | 3.63 | 398.13 |
| | | Colour | 63.67 | 38.57 | 39.42 |
| | | 2. | Paste | MC | 87 |
| AA | 27.26 | | | 71.76 | 163.27 |
| TPC | 81.5 | | | 111.30 | 36.56 |
| TFC | 17.41 | | | 112.90 | 548.49 |
| CC | 0.73 | | | 3.48 | 377.37 |
| Colour | 63.67 | | | 27.12 | 57.40 |

(c)

| S. No. | Sample | Response | Fresh sample | Max/min value | % change |
|--------|--------|----------|--------------|---------------|----------|
| 1. | Slice | MC | 87 | 3.80 | 95.63 |
| | | AA | 27.26 | 70.17 | 157.43 |
| | | TPC | 81.5 | 125.86 | 54.44 |
| | | TFC | 17.41 | 108.54 | 523.48 |
| | | CC | 0.73 | 3.59 | 392.32 |
| | | Colour | 63.67 | 36.63 | 42.46 |
| | | 2. | Paste | MC | 87 |
| AA | 27.26 | | | 69.38 | 154.52 |
| TPC | 81.5 | | | 109.13 | 33.90 |
| TFC | 17.41 | | | 93.54 | 437.32 |
| CC | 0.73 | | | 2.80 | 284.39 |
| Colour | 63.67 | | | 24.75 | 61.12 |

3.5 Statistical Analysis of Oven, Tray and Refractance Window Drying Process

The one-way analysis of variance (ANOVA) usually confirms that beyond 95 % confidence interval level, no statistical differences exist for all variables. To do so, each dried sample was subjected to a minimum of three trails for all cases. It has been analysed that for all sample responses (AA, TPC, TFC, CC, MC and *L* values of colour indices), high F-values and low p-values have been obtained (Table 3.5 a – 1). These findings affirm upon the greater significance of the conducted experiments.

Table 3.5: The ANOVA based statistical data summary for various cases (a) AA of slice, (b) AA of paste, (c) TFC of slice, (d) TFC of paste, (e) TPC of slice, (f) TPC of paste, (g) CC of slice, (h) CC of paste, (i) MC of slice, (j) MC of paste, (k) colour indices of slice and (l) colour indices of paste samples of *Curcuma longa*.

(a)

| S. No. | | DF | Sum of Squares | Mean Square | F Value | Prob>F |
|--------|-------|----|----------------|-------------|---------|---------|
| 1. | Model | 2 | 269.56 | 134.78 | 33.27 | 0.00056 |
| 2. | Error | 6 | 24.30 | 4.05 | | |
| 3. | Total | 8 | 293.86 | | | |

(b)

| S. No. | | DF | Sum of Squares | Mean Square | F Value | Prob>F |
|--------|-------|----|----------------|-------------|---------|----------|
| 1. | Model | 2 | 174.46 | 87.23 | 14.05 | 0.000544 |
| 2. | Error | 6 | 37.23 | 6.20 | | |
| 3. | Total | 8 | 211.70 | | | |

(c)

| S. No. | | DF | Sum of Squares | Mean Square | F Value | Prob>F |
|--------|-------|----|----------------|-------------|---------|----------|
| 1. | Model | 2 | 1519.94 | 759.97 | 96.34 | 0.000027 |
| 2. | Error | 6 | 47.32 | 7.88 | | |
| 3. | Total | 8 | 1567.26 | | | |

(d)

| S. No. | | DF | Sum of Squares | Mean Square | F Value | Prob>F |
|--------|-------|----|----------------|-------------|---------|---------|
| 1. | Model | 2 | 1361.94 | 680.97 | 133.14 | 0.00001 |
| 2. | Error | 6 | 30.68 | 5.11 | | |
| 3. | Total | 8 | 1392.63 | | | |

(e)

| S. No. | | DF | Sum of Squares | Mean Square | F Value | Prob>F |
|--------|-------|----|----------------|-------------|---------|----------|
| 1. | Model | 2 | 1981.03 | 990.51 | 174.79 | 0.000004 |
| 2. | Error | 6 | 34 | 5.66 | | |
| 3. | Total | 8 | 2015.03 | | | |

(f)

| S. No. | | DF | Sum of Squares | Mean Square | F Value | Prob>F |
|--------|-------|----|----------------|-------------|---------|---------|
| 1. | Model | 2 | 150.71 | 75.35 | 13.29 | 0.00624 |
| 2. | Error | 6 | 34 | 5.66 | | |
| 3. | Total | 8 | 184.71 | | | |

(g)

| S. No. | | DF | Sum of Squares | Mean Square | F Value | Prob>F |
|--------|-------|----|----------------|-------------|---------|---------|
| 1. | Model | 2 | 0.75 | 0.377 | 14.81 | 0.00478 |
| 2. | Error | 6 | 0.152 | 0.025 | | |
| 3. | Total | 8 | 0.906 | | | |

(h)

| S. No. | | DF | Sum of Squares | Mean Square | F Value | Prob>F |
|--------|-------|----|----------------|-------------|---------|---------|
| 1. | Model | 2 | 1.713 | 0.856 | 21.06 | 0.00194 |
| 2. | Error | 6 | 0.244 | 0.040 | | |
| 3. | Total | 8 | 1.957 | | | |

(i)

| S. No. | | DF | Sum of Squares | Mean Square | F Value | Prob>F |
|--------|--------------|----|----------------|-------------|---------|--------|
| 1. | Model | 2 | 2.66 | 1.33 | 33.25 | 0.0005 |
| 2. | Error | 6 | 0.24 | 0.04 | | |
| 3. | Total | 8 | 2.9 | | | |

(j)

| S. No. | | DF | Sum of Squares | Mean Square | F Value | Prob>F |
|--------|--------------|----|----------------|-------------|---------|----------|
| 1. | Model | 2 | 9.62 | 4.81 | 160.33 | 0.000006 |
| 2. | Error | 6 | 0.18 | 0.03 | | |
| 3. | Total | 8 | 9.8 | | | |

(k)

| S. No. | | DF | Sum of Squares | Mean Square | F Value | Prob>F |
|--------|-------|----|----------------|-------------|---------|--------|
| 1. | Model | 2 | 65.26 | 32.63 | 7.16 | 0.025 |
| 2. | Error | 6 | 27.32 | 4.55 | | |
| 3. | Total | 8 | 92.59 | | | |

(l)

| S. No. | | DF | Sum of Squares | Mean Square | F Value | Prob>F |
|--------|-------|----|----------------|-------------|---------|--------|
| 1. | Model | 2 | 98.29 | 49.14 | 9.54 | 0.0136 |
| 2. | Error | 6 | 30.88 | 5.14 | | |
| 3. | Total | 8 | 129.17 | | | |

3.6 Energy Consumption of Drying Processes

An energy estimation study was carried out to evaluate upon the drying method and sample type that demanded minimum energy needs during the drying process duration. Thereby, the parameter was determined for the drying of one batch of turmeric slice and paste for oven, tray and RW dried process respectively. Table 3.6 summarizes the energy consumption in the oven, tray and RW drying process of

Table 3.6: Energy consumption data of oven, tray and refractance window drying of turmeric system.

| S. No. | Sample | Drying process | Energy consumption (kWh/m ²) |
|--------|--------|----------------|---|
| 1. | | Oven | 19.20 |
| 2. | Slice | Tray | 24.00 |
| 3. | | RWD | 14.00 |
| 1. | | Oven | 10.80 |
| 2. | Paste | Tray | 13.50 |
| 3. | | RWD | 6.00 |

slice and paste samples. From the table, it can be seen that the energy consumption for oven, tray and RW drying was higher for the slice sample case in comparison to the paste sample case. This is due to greater energy needs for the former case.

3.7 Comparative Economic Competence

It is imperative to achieve a highly nutritive product drying. Thereby, the product must have high nutritional retention and it must be achievable within low economic cost. Henceforth, to verify the feasibility of oven, tray and RW drying a conceptual cost estimation was carried out. The cost estimation study enabled the identification of drying process with minimum cost.

Fig. 3.5 depicts the processing cost of drying per kg fresh sample and processing cost of drying per kg dried sample for all drying cases. As illustrated, among all processes, lowest annualized processing cost was obtained for the RWD process being operated at 60 °C. For such system, the processing cost was about Rs. 82.61 and Rs. 47.75 per kg of fresh sample for slice and paste cases respectively and Rs. 636.59 and Rs. 367.97 per kg of dried sample product for slice and paste cases respectively. Following this, the oven annualized processing costs have been significantly higher and refer to Rs. 102.84 and Rs. 68.48 per kg of fresh sample for slice and paste cases respectively and Rs. 792.47 and Rs. 527.73 per kg of dried sample product for slice and paste cases respectively.

For the tray drier, the annualized processing costs are the highest and correspond to Rs. 119.58 and Rs. 77.76 per kg fresh sample for slice and paste cases respectively and Rs. 921.45 and Rs. 599.24 per kg dried sample for slice and paste cases respectively. The obtained costs for all processes are significantly higher in comparison with the actual cost of the turmeric sample (about Rs. 120 – 200/- per kg for fresh sample). However, it shall be observed here that the costs indicated in this work are conceptual and for laboratory scale systems. With process scale up, the costs can reduce significantly. However, the cost competence is very likely to remain as it is for the RWD process due to lower electricity costs associated to lesser processing time and simpler process equipment in comparison with the tray and oven drying processes.

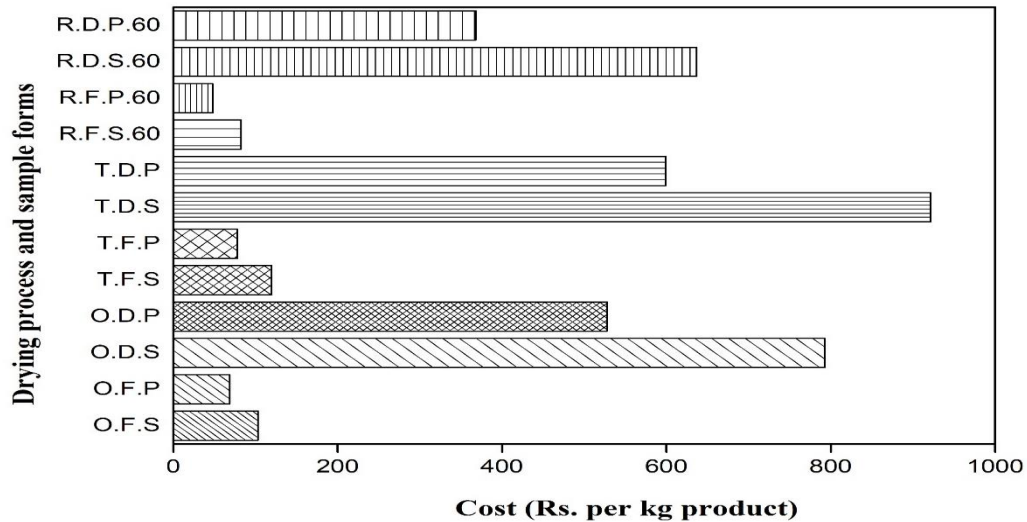
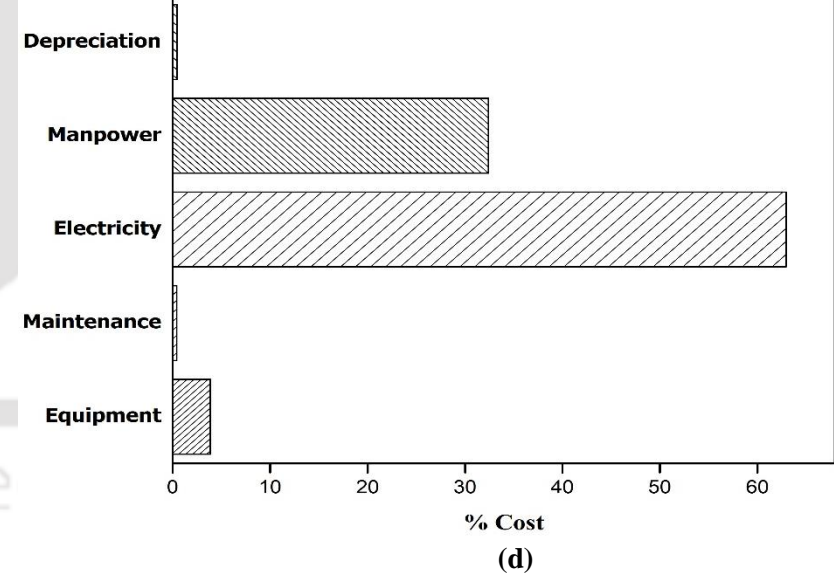
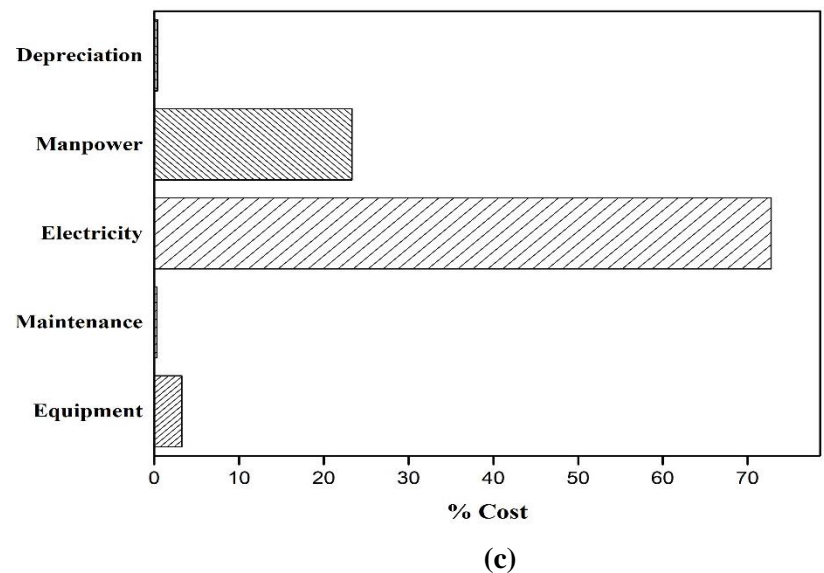
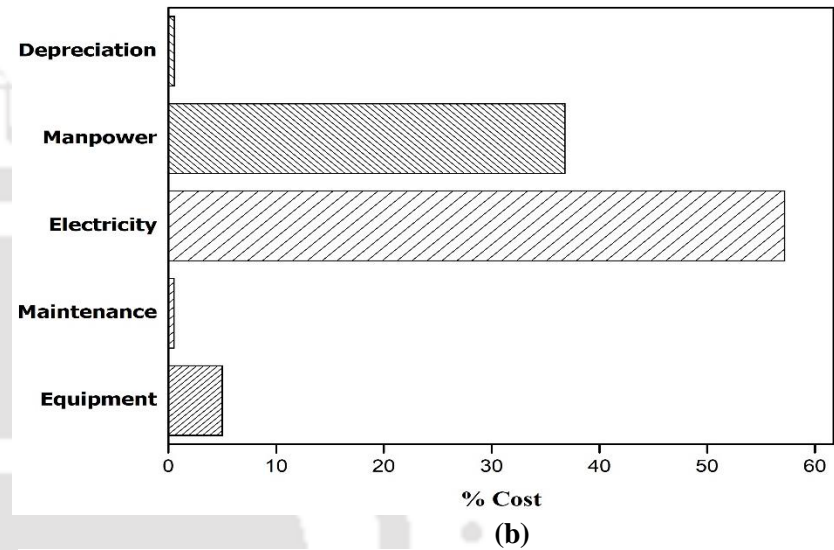
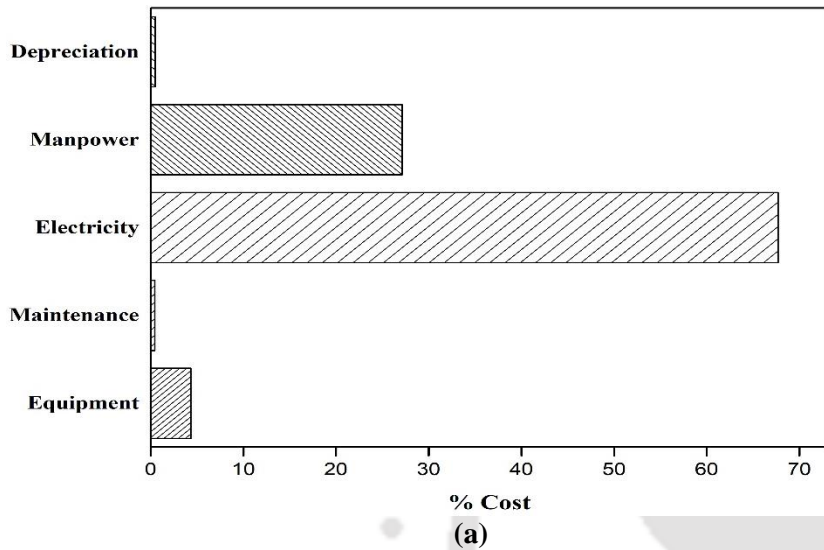


Figure 3.5: Total annualized conceptual cost (Rs per kg of dried sample) of laboratory scale dried turmeric system and with oven, tray and refractance window drying process (R – RWD; T – Tray dryer; O – Oven dryer; D – dried; F – Fresh; S – slice and P – paste).



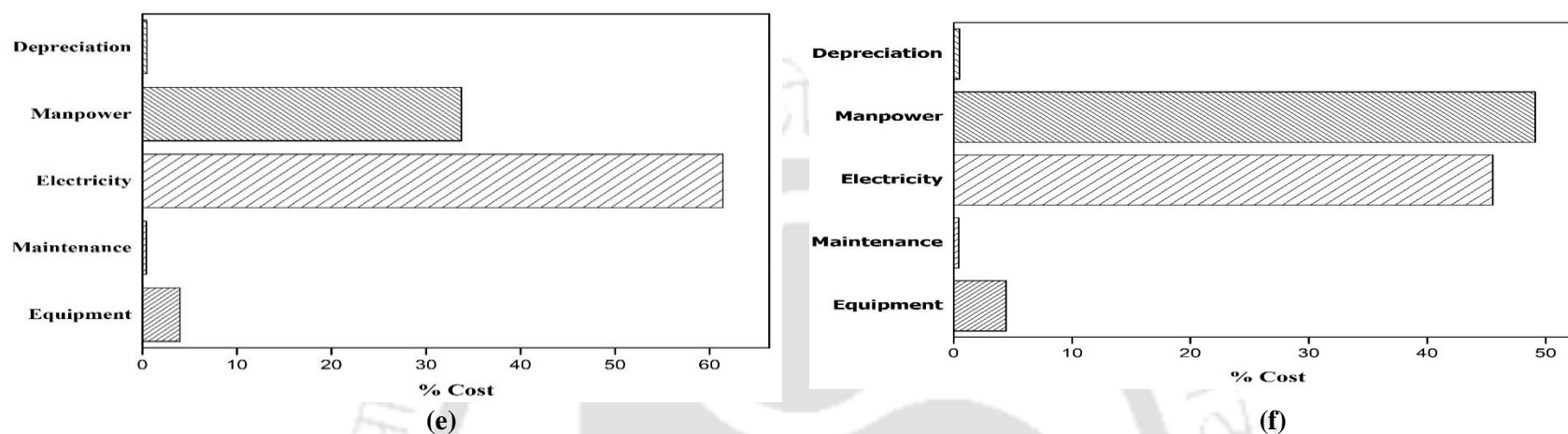


Figure 3.6 (a -f): Bar diagrams depicting percentage contributions of electricity cost, manpower cost, equipment cost, maintenance cost and depreciation cost to the total conceptual annualized cost of alternate drying methods: (a) oven for slice, (b) oven for paste, (c) tray for slice, (d) tray for paste, (e) RW for slice and (f) RW for paste.

Table 3.7: A summary of best data of tray, oven and refractance window dried turmeric products.

| S. No. | Drying Method | Sample Constitution | T (°C) | Time (h) | TFC (mg quercetin/g dry sample) | TPC (mg GAE/ g dry sample) | AA (%) | CUR (%w/w) | MC (%) | Reference |
|--------|---------------|---------------------|--------|----------|---------------------------------|----------------------------|--------|------------|--------|------------------------|
| 1. | RWD | 1 mm slice | 60 | 7 | 140.1 | 161.9 | 82.9 | 4.2 | 4.2 | This Work |
| 2. | Tray | 1 mm slice | 60 | 8 | 122.9 | 140.2 | 80 | 3.6 | 2.9 | |
| 3. | RWD | 1 mm paste | 60 | 3 | 123.5 | 118.6 | 79.7 | 3.8 | 3.7 | |
| 4. | Tray | 1 mm paste | 60 | 4.5 | 112.9 | 111.3 | 71.7 | 3.4 | 1.2 | |
| 5. | Oven | Rhizome | 70 | 21 | - | - | - | 2.97 | - | (Raza et al., 2018) |
| 6. | Oven | Rhizome | 55 | 5 | - | - | - | 4.6 | 8 | (Pradeep et al., 2016) |

The contribution of various costs in alternate drying process systems has been depicted in bar diagrams (Fig. 3.6). As affirmed, for the RWD system, the cost contributions of electricity are lower for the best choice of RWD process (61.43 and 45.54 %) in comparison to those obtained for the oven drying (67.67 and 57.16 %) and tray drying (72.75 and 62.92 %) processes for the slice and paste samples respectively. With respect to the overall costs, corresponding equipment costs are about 3.98 and 4.42 % in the RWD process, 4.30 and 4.99 % in oven drying processes and 3.24 and 3.85 % in tray drying process for both slice and paste samples respectively. Thus, manpower costs and electricity costs dominate the costs of all processes in comparison with the equipment costs. The economic analysis presumes that the RWD can be scaled up and the drying kinetics will be independent of the scaling scheme. However, this is only valid for the case of process scalability considering proportional measures i.e., the dimensions and sample sizes do get scaled up proportionally. Nonetheless, since conceptual cost analysis has been considered, the assumption is justified.

3.8 Literature Comparison

Table 3.7 summarizes the best findings of this work with those available in the literature. The available literature does not elaborate upon the slice and paste sample drying using RWD, oven and tray drying processes. The best findings only correspond to those reported in the literature as far as comparative assessment is concerned and they clearly indicate that the RWD has the edge due to lower processing time and associated costs for the slice and paste turmeric sample drying to reach the lowest MC of 4.20 %. Also, analytical characterizations have also been not reported other than CC in all critical literature findings. Thus, the findings of this work are novel and are expected to serve as a useful guideline to further the commercial application of RWD process for the drying of horticultural and agro-produce.

3.9 Summary

This chapter summarized the findings involved during comparative assessment of tray, oven and RW drying of *Curcuma longa*, and affirmed prominent findings. Firstly, the RWD proved to be significantly

effective to provide better nutritional parameters in a time span of 7 h, as opposed to 8 h for the conventional drying process. Secondly, among all cases, the optimal process and sample combinations have been RWD and turmeric slices. The turmeric slices upon RW drying at 60 °C affirmed 4.20 % (wb); 161.95 mg GAE/g dry sample; 140.16 mg quercetin/g dry sample; 82.93 %; 4.21 % w/w and 42.88 L values for MC, TPC, TFC, AA, CC and colour indices respectively. Thirdly, among paste and slice samples, RWD provided higher retention for both paste and slice in comparison to oven and tray drying process and was the RWD has been the most cost effective process. Fourthly, further investigations are required to evaluate the optimality of sample thickness, drying time and temperature in the range of 0.5 – 2 mm, 0.30 – 2 h and 60 – 95 °C respectively. Finally, the conceptual annualized laboratory scale processing cost of the RWD process (operated at 60 °C) was the lowest among RWD, tray and oven drying processes. This corresponds to Rs. 47.75 and Rs. 367.97 per kg of fresh and dried paste sample respectively. Contrary to this, higher processing costs were obtained for tray and oven drying processes. This is due to greater processing time of these drying systems in comparison with the RWD process system. In summary, promising drying characteristics have been indicated by RWD for the achievement of dried *Curcuma longa* with a better combination of nutrition and other parameters. These findings are expected to be effective to further enhance successful avenues for functional food development with RWD processed *Curcuma longa*.





Chapter 4:
Process and Product Characteristics of
Refractance Window Dried *Curcuma longa*



Process and Product Characteristics of Refractance Window

Dried *Curcuma longa*

In this chapter, the findings with respect to the optimality of alternate drying temperatures and mylar film thickness have been focussed from the perspective of the maximum retention of nutritional parameters. Due to its cost efficacy and better nutritional retention, the refractance window drying method has been chosen as the most relevant drying method. After a brief introduction in section 4.1, section 4.2 focuses on the drying characteristics curve by conducting studies on the effect of temperature and mylar film thickness on the equilibrium drying time. Thereafter, sections 4.3 and 4.4 respectively present the results obtained in drying kinetics studies in terms of fitness of drying models and moisture diffusivity with activation energy respectively. Thereafter, sections 4.5 present the findings associated to the moisture content, total phenolic content, total flavonoids content, anti-oxidant activity, curcumin content and colour indices of the refractance window dried turmeric slice sample. Following this, section 4.6 provides relevant inferences on the optimal final film thickness. Thereafter, section 4.7 elucidates on the effect of drying temperature on the functional groups present in the RW dried turmeric slice samples. Thereby, section 4.8 details on the statistical analysis for all cases. Subsequently, section 4.9 addresses relevant literature comparison. Finally, 4.10 summarizes the key findings of the chapter.

4.1 Introduction

The available literature with respect to efficacy of mylar film thickness for the refractance window drying (RWD) process have been studied by Azizi et al., (2017) for kiwi slices (0.8 - 2.4 mm) in the parametric range of 80 – 100 °C and 100 – 300 µm film thickness. A critical insight into the literature indicates several lacunae. Firstly, nutritional properties have not been studied. This is important from the perspective of growing emphasis towards functional products. Secondly, the work was targeted for kiwi fruit sample and the findings cannot be generalized for the RWD characteristics

of *Curcuma longa*. This is due to the reason that *Curcuma longa* rhizomes and kiwi slices have diverse properties with significant variation in moisture content (MC) (81 – 82 % for kiwi and 87 – 88 % for turmeric) and total soluble solid content (7 % for kiwi and 13 % for turmeric).

In the previous chapter, a comparative assessment of tray, oven, RWD system has been addressed. However, detailed investigations are further required for the RWD characteristics of *Curcuma longa*. This is required from the perspectives of product-process design and operational characteristics. To precisely outline, firstly, there is a need to bench mark the novel drying process to significantly reduce processing time. Secondly, the sensitivity of the process with respect to process parameters needs to be judiciously addressed to achieve the desired product properties. Thus, optimality of mylar film thickness and criticality of drying kinetics are necessary to compare the RWD with other conventional drying methods such as tray and sun drying.

Considering these lacunae, the chapter addresses the RWD characteristics and kinetics of 1 mm turmeric slice sample in the water bath temperature range of 65 – 95 °C. Optimal equilibrium drying time for the sample has been evaluated for variant choice of mylar film thickness (125, 250 and 350 µm). Drying rate, nutritional, drying kinetic characteristics have been targeted to examine the criticality of mylar film thickness. Drying rate characteristics refer to moisture removal rate curves. Nutritional analysis involved determination of anti-oxidant activity (AA), total phenolic content (TPC), total flavonoid content (TFC) and curcumin content (CC). Drying kinetic studies enabled the determination of diffusivity and activation energy associated to the RWD process. Based on the obtained trends in the data, the combinatorial optimality of temperature, time, mylar film thickness have been identified for best combinations of drying rate, kinetic and nutritional characteristics. All experiments were conducted without air circulation so as to serve as a benchmark for future investigations.

4.2 Drying Characteristics Curve

Fig. 4.1 (a – c) depict the influence of drying temperature on moisture ratio (MR) vs drying time curves for 125, 250 and 350 µm mylar film thickness cases. The RWD drying curves for turmeric

exhibited similar trends apparent for other food materials studied till date by adopting RWD and tray drying processes (Hernandez-Santos et al., 2016; Ochoa-Martínez et al., 2012). The pertinent MR trends depict an initial rapid reduction phase followed with slower reduction to eventually reach the equilibrium moisture content of the dried turmeric sample. The rapid initial MR reduction phase has been due to high moisture diffusivity that exists due to the abundant availability of MC on the surface. As drying proceeds, the moisture availability reduces on the sample surface. Due to this effect, moisture migration occurs from the inner portions to the sample surface.

During the course of drying process, there is restriction for the migration of MC from the inner portion to outer surface leading to decrease in moisture diffusivity. This is due to the reason that the drying enables hardening of the sample surface and a crust gets formed in due course of time. Therefore, the moisture migration from the inner portions has been reduced further and this translates into reduced moisture diffusivity. Hence, progressively slower MR values have been apparent in later stages of the drying due to rapid MR reduction in the initial drying phase (Mondal et al., 2020a; Mondal et al., 2020c).

For the 125 μm mylar film case, the MR reduced rapidly from 1 – 0.013, 0.017, 0.010 and 0.017 for a variation in drying time of 180, 120, 75 and 45 minutes and drying temperatures of 65, 75, 85 and 95 $^{\circ}\text{C}$ respectively. These values varied from 1 – 0.019, 0.015, 0.04 and 0.02 for a drying time variation up to 225, 120, 60 and 45 min respectively for the 250 μm mylar film case and 1 – 0.010, 0.017, 0.014 and 0.012 for drying time variation up to 210, 150, 75 and 60 min respectively for 350 μm mylar film case. Thus, greater moisture removal was achieved at higher drying temperature due to enhanced moisture diffusivity. This is due to the reason that at the elevated temperature, greater vapour pressure differences do exist between the turmeric slices and surrounding environment (Mondal et al., 2020b). The critical influence of the mylar film thickness can be further understood from the activation energy trends discussed later in the chapter.

The apparent reduction in MC variation in the later duration of drying time interval (from 210 – 345, 150 – 255, 90 – 150 and 60 – 75 min drying time for 125 μm at 65, 75, 85 and 95 $^{\circ}\text{C}$ respectively and similar values for 250 μm and 350 μm cases) was due to a reduction in moisture transport from

inner portions to the outer sample surface. The equilibrium drying time have been evaluated as 345, 255, 150 and 75 min for drying temperature of 65, 75, 85 and 95 °C and 125 µm case; 360, 270, 165 and 90 min for similar drying temperature values and 250 µm case and 375, 285, 180 and 105 min for same drying temperature and 350 µm case. Thus, it is apparent that equilibrium drying time to a certain extent increased with mylar film thickness (40 %) and decreased with drying temperature (73 %). Due to being an independent analysis of the experimental investigations addressed in this work, no citations are available for such outcomes of the work.

In general, drying time of food materials is strongly influenced with its characteristics such as type of material, composition and structural integrity. The maximum drying time of 90 min mentioned in the article was based on experiments targeted through the methodology of drying kinetics. Similar approach has been reported by Ochoa-Martinez et al., (2012) for the refractance window drying of mango samples. The authors affirmed that for a batch RWD process, the drying time and temperature were about 60 min and 92 °C respectively for a sample thickness of 1 mm. Compared to the mango slices case, the turmeric drying is expected to take longer time due to greater structural integrity and associated difficulty to remove bound moisture content in the sample (Mondal et al., 2020c). Hence, the chosen drying time for the 1 mm turmeric sample case is relevant and is in accordance with the drying time mentioned in the appropriate literature.

4.3 Fitness of Drying Models

Fitness characteristics of alternate drying models to represent evaluated RWD characteristics have been summarized in Table 2.1 of Chapter 2. For all cases (various combinations of drying temperature and mylar film thickness values), among all alternate drying models that were tested to represent pertinent RWD based turmeric drying process characteristics, Singh et al., (2014) model has been inferred to be the best fit model. This is due to highest R^2 (> 0.99) and lowest combination of RSS

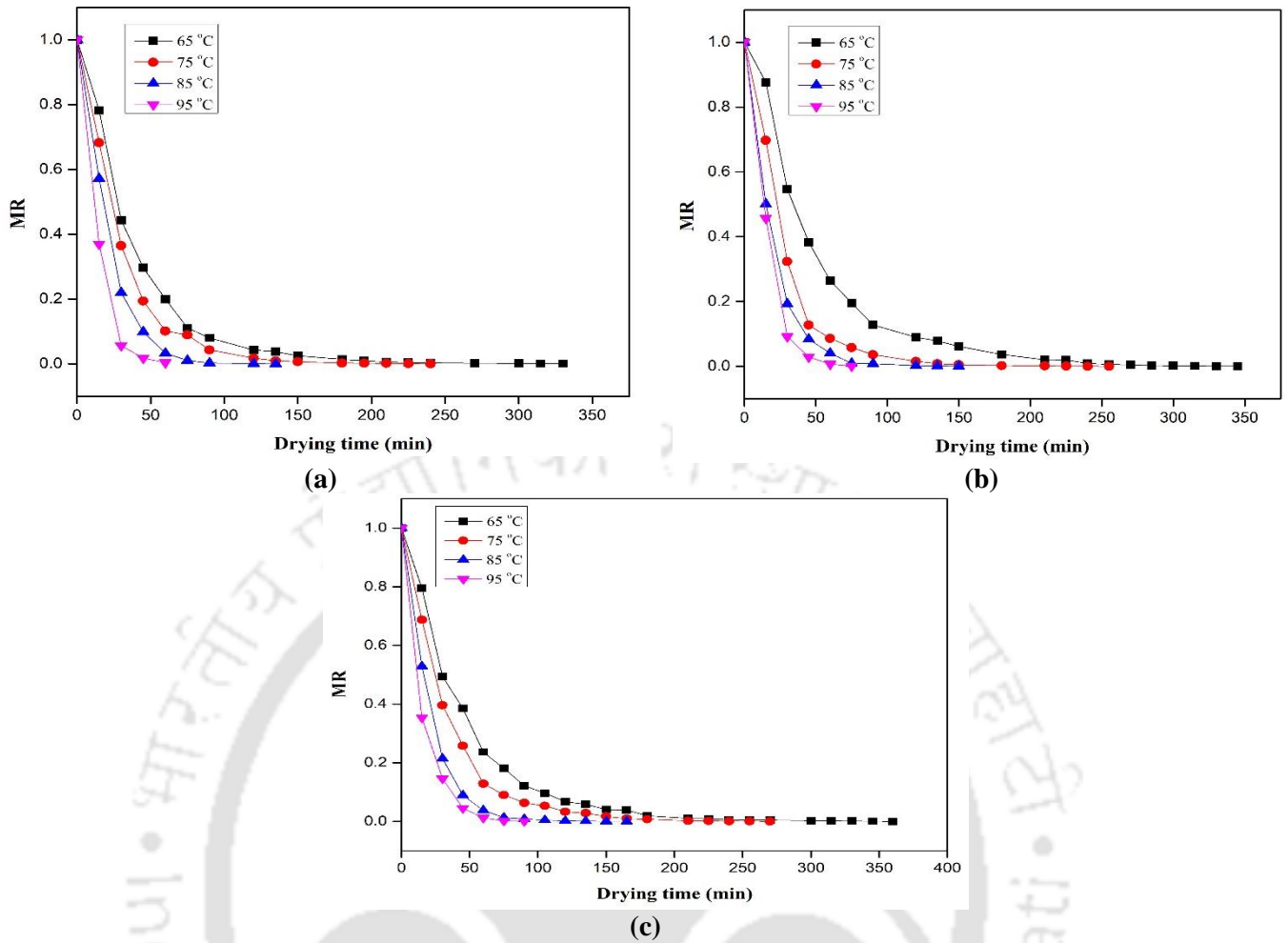


Figure 4.1: Moisture ratio (MR) vs drying time profiles of RWD *Curcuma longa* samples for various mylar film thickness cases (a) 125, (b) 250 and (c) 350 μm .

(0.002 – 0.023) and chi-squared values (0.0002 – 0.001). Table 4.1 presents the model parameters obtained for various cases of the fitted models. The model parameter k varied from 0.020 – 0.059 for a variation in temperature from 65 – 95 °C for 250 μm mylar film thickness case. Further, k varied to a lesser extend (0.020 – 0.070) with mylar film thickness (125 – 350 μm). Similar trends are applicable for parameter ‘ a ’.

The temperature had a positive effect on the drying rate constant. Thus, compared to time, temperature had a predominant influence to enhance moisture removal from the samples. Doymaz, (2005) and Goyal et al., (2007) reported similar drying behaviour trends for plum and okra respectively using tunnel and oven drying approaches.

Table 4.1: Fitness and model parameters of best fit model to represent refractance window drying kinetics.

| S. No. | Film Thickness (μm) | Temp ($^{\circ}\text{C}$) | Models | R^2 | χ^2 | RSS | a | k |
|--------|----------------------------------|-----------------------------|----------------------|-------|----------|-------|----------|-------|
| 1. | 125 | 65 | Singh et al., (2014) | 0.99 | 0.0007 | 0.012 | -0.00002 | 0.025 |
| | | 75 | Singh et al., (2014) | 0.99 | 0.0005 | 0.007 | 0.0003 | 0.033 |
| | | 85 | Singh et al., (2014) | 0.99 | 0.001 | 0.007 | 0.0029 | 0.045 |
| | | 95 | Singh et al., (2014) | 0.99 | 0.001 | 0.003 | 0.0067 | 0.070 |
| 2. | 250 | 65 | Singh et al., (2014) | 0.99 | 0.001 | 0.023 | -0.0004 | 0.020 |
| | | 75 | Singh et al., (2014) | 0.99 | 0.001 | 0.019 | 0.0002 | 0.036 |
| | | 85 | Singh et al., (2014) | 0.99 | 0.0002 | 0.002 | 0.0005 | 0.050 |
| | | 95 | Singh et al., (2014) | 0.99 | 0.001 | 0.007 | 0.007 | 0.059 |
| 3. | 350 | 65 | Singh et al., (2014) | 0.99 | 0.0003 | 0.008 | -0.0001 | 0.022 |
| | | 75 | Singh et al., (2014) | 0.99 | 0.0002 | 0.004 | -0.0002 | 0.030 |
| | | 85 | Singh et al., (2014) | 0.99 | 0.0003 | 0.003 | 0.0006 | 0.048 |
| | | 95 | Singh et al., (2014) | 0.99 | 0.0006 | 0.003 | 0.0004 | 0.060 |

4.4 Moisture Diffusivity and Activation Energy

Using the $\ln MR$ vs drying time plots (depicted in Fig. 4.2), the diffusivity of turmeric in RWD systems has been evaluated along with its activation energy. Table 4.2 summarizes these data. As shown, the moisture diffusivities increased from $3.49 - 15.0 \times 10^{-11} \text{ m}^2/\text{s}$ for a drying temperature variation from $65 - 95 \text{ }^{\circ}\text{C}$ and $250 \mu\text{m}$ thickness case. However, it increased to $3.71 - 15.9 \times 10^{-11} \text{ m}^2/\text{s}$ for $125 \mu\text{m}$ case and reduced to $3.36 - 13.9 \times 10^{-11} \text{ m}^2/\text{s}$ for $350 \mu\text{m}$ case. The moisture diffusivity obtained is in agreement with the value range of $10^{-8} - 10^{-12}$ for food materials (Zogzas et al., 1996). Ochoa-Martinez et al., (2012) reported similar effective moisture diffusivity of $44.0 \times 10^{-11} \text{ m}^2/\text{s}$ for 1 mm thick mango slices using RWD process at $92 \text{ }^{\circ}\text{C}$. The moisture diffusivity enhanced with temperature due to higher temperature facilitating better moisture removal. The activation energy determined from Arrhenius plots (Fig. 4.3) varied between $52.57 - 49.43 \text{ kJ/mol}$ for a mylar film thickness variation from $125 - 350 \mu\text{m}$. These values are found to be in agreement with the value range provided for various food materials ($12 - 110 \text{ kJ/mol}$) (John et al., 2014).

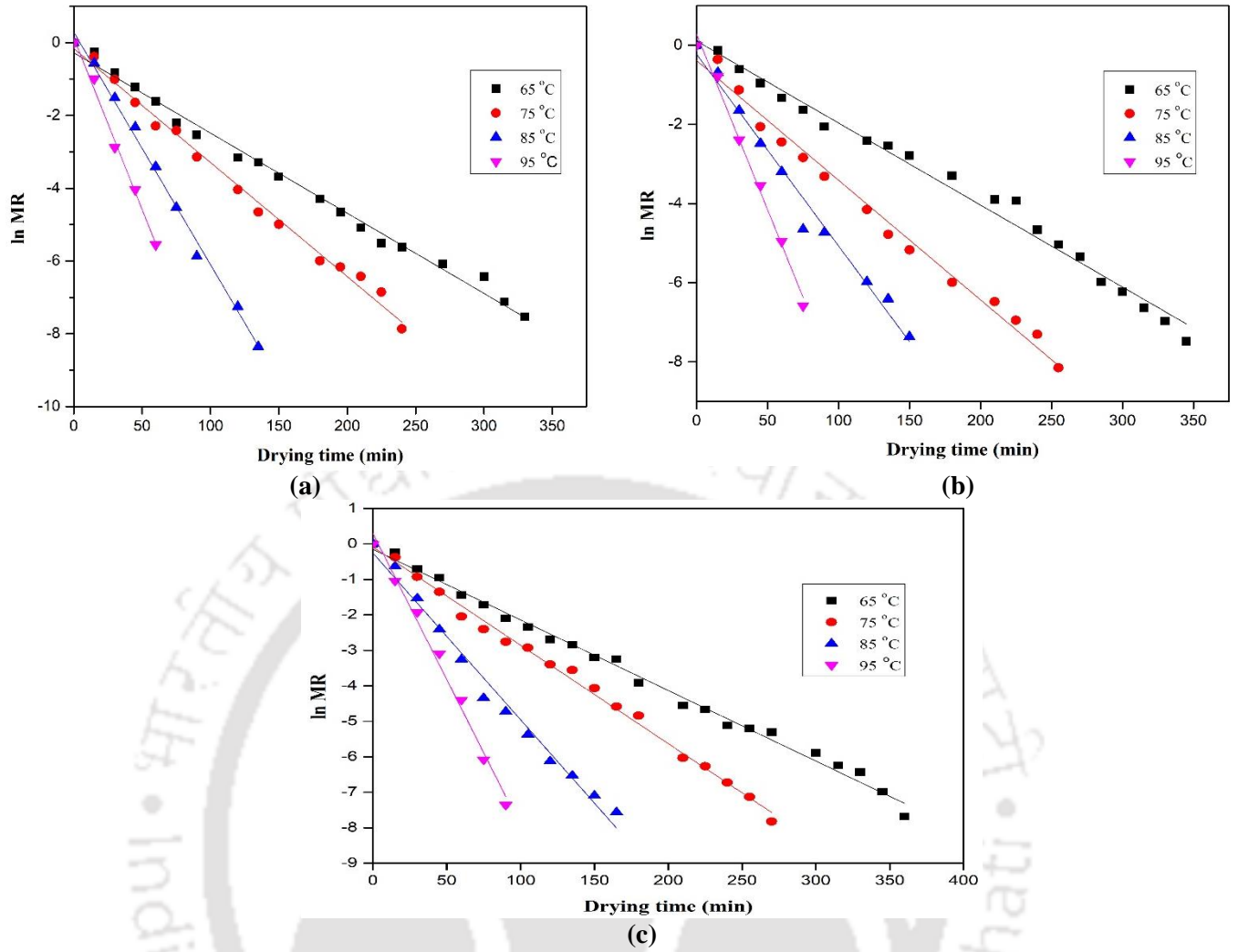


Figure 4.2: Fitness plots of the logarithm of Moisture Ratio versus drying time for various cases of mylar film thickness (a) 125, (b) 250 and (c) 350 μm.

Table 4.2: Moisture diffusivity and activation energy data of refractance window dried *Curcuma longa* for various combinations of temperature and film thickness.

| S. No. | Temp (°C) | 125 μm | | 250 μm | | 350 μm | |
|--------|-----------|---------------------------------|-------------|---------------------------------|-------------|---------------------------------|-------------|
| | | Diffusivity (m ² /s) | Ea (kJ/mol) | Diffusivity (m ² /s) | Ea (kJ/mol) | Diffusivity (m ² /s) | Ea (kJ/mol) |
| 1. | 65 | 3.71 × 10 ⁻¹¹ | | 3.49 × 10 ⁻¹¹ | | 3.36 × 10 ⁻¹¹ | |
| 2. | 75 | 5.29 × 10 ⁻¹¹ | 52.57 | 5.10 × 10 ⁻¹¹ | 49.94 | 4.68 × 10 ⁻¹¹ | 49.43 |
| 3. | 85 | 1.08 × 10 ⁻¹⁰ | | 8.18 × 10 ⁻¹¹ | | 7.94 × 10 ⁻¹¹ | |
| 4. | 95 | 1.59 × 10 ⁻¹⁰ | | 1.50 × 10 ⁻¹⁰ | | 1.39 × 10 ⁻¹⁰ | |

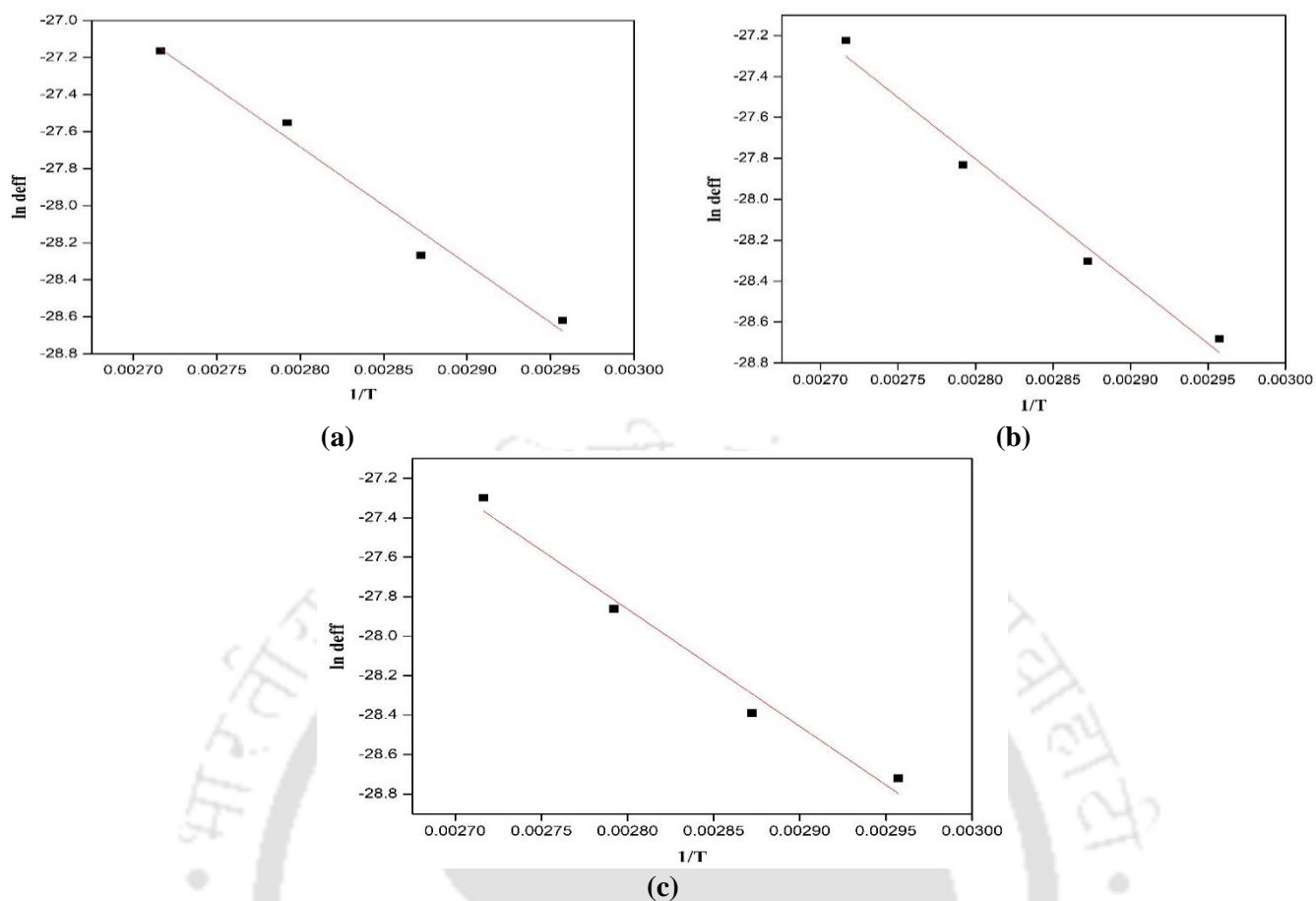


Figure 4.3: Arrhenius plots for moisture Diffusivity estimation for various cases of mylar film thickness (a) 125, (b) 250 and (c) 350 μm .

4.5 Physical and Nutritional Characteristics

4.5.1 Moisture Content

The MC obtained for various samples has been presented in Table 4.3. Fresh turmeric had a MC of 87 – 88 % (wb). Such MC poses a greater challenge to achieve very low MC in the dried samples for effective storage and shelf life. The final MC of the samples varied from 5.63 – 4.28 %, 5.16 – 4.10 % and 5.72 – 4.57 % (wb) for 125, 250 and 350 μm cases respectively. For long term and safe storage of food products, the recommend final moisture content in literature is about 4 – 8 % (Mondal et al., 2019).

Table 4.3: Equilibrium moisture content data of refractance window dried 1 mm thick *Curcuma longa* slices.

| S. No. | Temp (°C) | 125 µm | | 250 µm | | 350 µm | |
|--------|--------------|-------------------------------------|--------|-------------------------------------|-----------|-------------------------------------|-----------|
| | | Equilibrium drying time (min) | MC (%) | Equilibrium drying time (min) | MC (%) | Equilibrium drying time (min) | MC (%) |
| 1. | 65 | 345 | 5.63 | 360 | 5.16 | 375 | 5.72 |
| 2. | 75 | 255 | 5.18 | 270 | 5.09 | 285 | 5.45 |
| 3. | 85 | 150 | 4.74 | 165 | 4.34 | 180 | 4.91 |
| 4. | 95 | 75 | 4.28 | 90 | 4.10 | 105 | 4.57 |

* All standard deviations for equilibrium drying time and MC were in the range of 15 min and 0.1 – 0.3 respectively.

Dried turmeric samples obtained after RWD possessed a moisture content of 4.10 %. The corresponding MC of tray and oven dried turmeric samples were 2.90 and 3.80 % (Table 4.6) respectively. The marginally lower MC values for tray and oven drying systems have been due to significantly longer drying time. In general, agro-horticultural produces such as turmeric can be characterized to have a good shelf life through a reduction of their MC to about 4 – 8 % (Mondal et al. 2019). Since RWD processed turmeric samples possessed 4.10 % MC, the samples can be regarded to have good shelf-life characteristics, which has also been the case of tray and oven dried samples. During RWD of turmeric, the MC underwent marginal reduction for the 250 µm thick mylar sheet case in comparison to that achieved for the 125 µm thick mylar sheet case. This is due to similar drying temperature but enhanced drying time by 15 mins for the 250 µm mylar sheet film thickness case.

The experimental investigations affirmed that lower time was required for the RWD system in conjunction with tray and oven drying systems. This is due to effective radiative heat transfer that served as an additional mode of heat transport through the mylar film system that existed as an interface between the moist samples and hot water bath system.

Similar inferences have been reported in several available prior art. The drying time reduced significantly from 960 minutes (conventional drying) to 75 minutes (RWD) during tomato powder drying at 60 °C (Abul-Fadl & Ghanem, 2010). Similarly, for mango slices, Ochoa-Martinez et al.,

(2012) reported a reduction in drying time from 240 minutes (tray drier at 60 °C) to 60 minutes (RWD at 92 °C) so as to achieve a reduced MC of 5 % (wb). Similarly, Ghanem, (2010) indicated a significant reduction of time in the drying of liquid strawberries using RWD (90 minutes) in comparison with the hot air oven drying process (300 minutes). Also, for the kiwi fruit system, Jafari et al., (2015) as well inferred that lower duration was required to dry the samples in comparison with the oven drying process. The moisture content trends being reported in the article are in good agreement with those reported by Satwase et al., (2013).

4.5.2 Total Phenolic Content

Fig. 4.4 depicts the total phenolic content (TPC) of turmeric slice samples. The TPC of fresh turmeric was found to be 81.52 mg GAE/g sample. The TPC range varied between 163.54 – 187.63 mg GAE/g sample for 125 µm case; between 164.31 – 189.76 mg GAE/g dry sample for 250 µm case; between 163.10 – 186.31 mg GAE/g dry sample in 350 µm case for a variation in water bath temperature from 65 – 95 °C. The highest TPC (189.76 mg GAE/g sample) was obtained at 250 µm for 95 °C. In comparison to the TPC values obtained for 125 and 350 µm cases, TPC values obtained with 250 µm is marginally high. This is due to the heat sensitivity of the TPC constituents in the turmeric samples and optimal retention at moderate heat transfer rates (Hernandez-Santos et al., 2016). It can be inferred that the TPC values did not vary significantly. This is due to similar drying rates for all cases as is evident from diffusivity values summarized in Table 4.2.

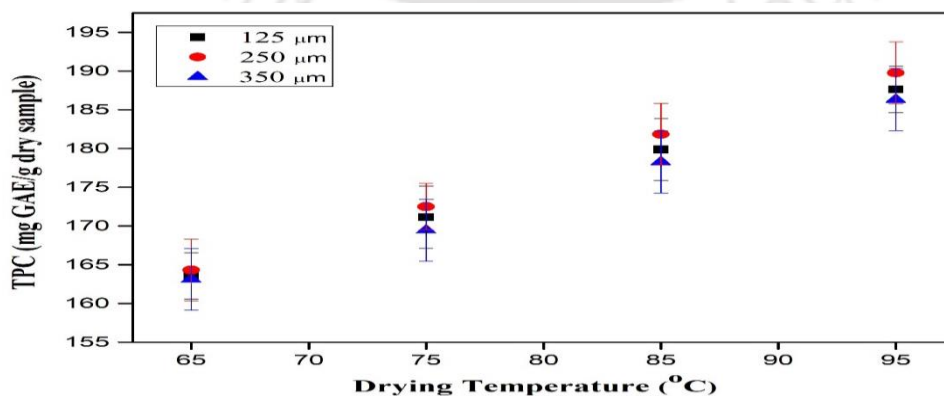


Figure 4.4: Effect of drying temperature and mylar film thickness on the total phenolic content of refractance window dried *Curcuma longa*.

4.5.3 Total Flavonoids Content

The TFC of dried turmeric slice samples are shown in Fig. 4.5. For fresh sample, the average TFC was found to be 17.41 mg quercetin/g sample. However, for dried turmeric slice sample the TFC varied from 140.45 – 157.17 mg quercetin/g sample for 125 μm mylar film case; 142.50 – 160.96 mg quercetin/g sample for 250 μm mylar film case and 138.72 – 155.31 mg quercetin/g sample for 350 μm mylar film case and for water bath temperature variation from 65 – 95 $^{\circ}\text{C}$ water bath temperature respectively. Compared to 125 and 350 μm film thickness, 250 μm samples to an extent had higher TFC values. Also, it can be observed that with higher temperature, TFC can be better retained in the sample. This is due to the reason that with high temperature, the drying time reduced and thereby enabled lower time frame for heat exposure so as to retain heat sensitive constituents. The RWD at 95 $^{\circ}\text{C}$ reported highest TFC values for all film thickness cases. Due to short drying time, the cellular integrity of the slices is not compromised and the hidden TFC in the cell structure is not oxidised (Hernandez-Santos et al., 2016). Thereby, such conditions enable enhanced release of flavonoids from the samples (Rababah et al., 2015).

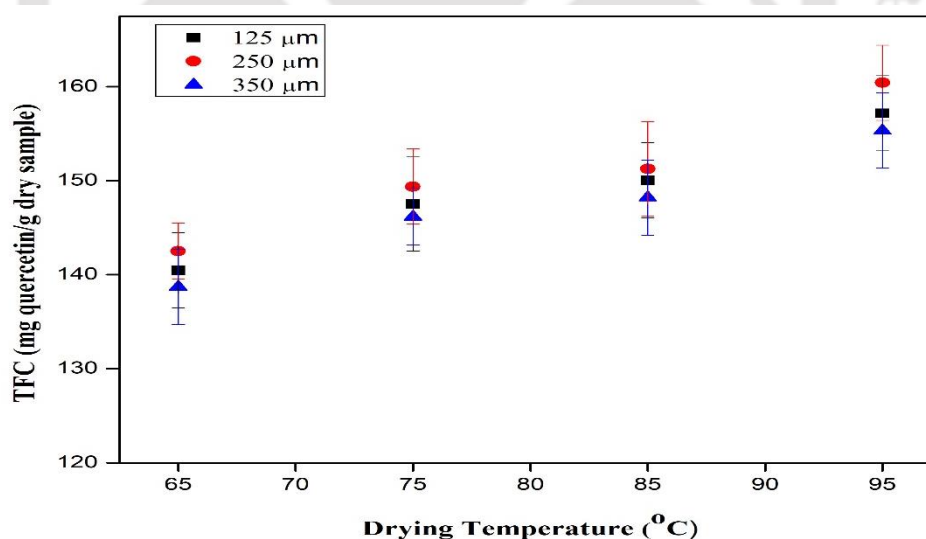


Figure 4.5: Effect of drying temperature and mylar film thickness on the total flavonoids content of refractance window dried *Curcuma longa*.

4.5.4 Antioxidant Activity

For functional food formulation, AA is an important parameter due to its radical scavenging properties. Therefore, it is important to retain higher AA in processed food products. Fig. 4.6 depicts the variations of AA for all film thickness cases (125, 250 and 350 μm) and for the hot water bath temperature range of 65 – 95 $^{\circ}\text{C}$. For such water bath temperature variation, the AA varied from 79.21 – 86.27 % for 125 μm case; 82.10 – 89.10 % for 250 μm case and 77.92 – 85.03 % for 350 μm case.

Compared to the AA of fresh sample (27.27 %), the AA in dried samples were high due to MC reduction upon drying. The highest AA of 89.10 % was obtained for samples dried with RWD at 95 $^{\circ}\text{C}$ in 250 μm . Compared to 125 and 350 μm film thickness, 250 μm based dried samples to an extent had higher AA values. However, for all film cases, the higher temperature indicated better retention of AA. With prolonged heating, anti-oxidant constituents undergo higher oxidation. Since higher temperature enabled shorter heating duration to achieve equilibrium moisture content, higher AA can be retained in such samples. The high retention of TPC and TFC also enables higher retention of AA content in the dried samples (Demla & Verma, 2012).

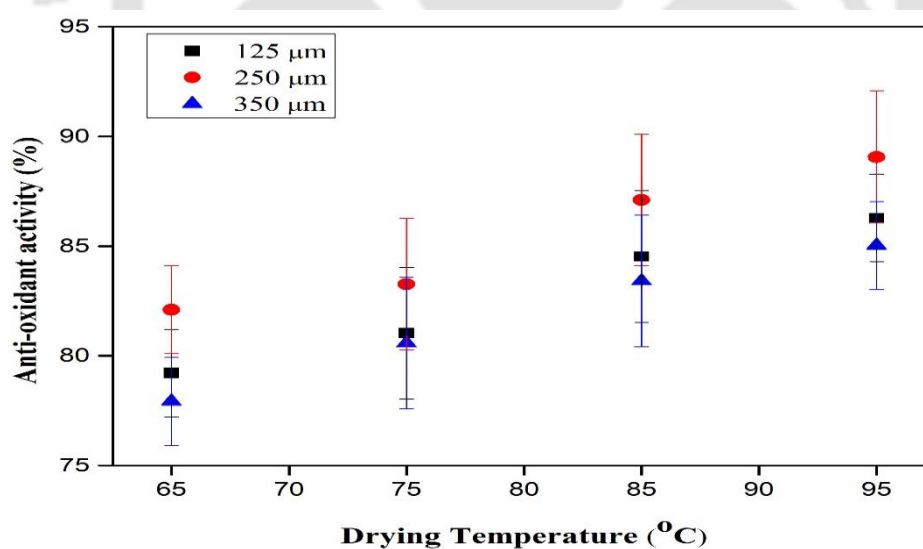


Figure 4.6: Antioxidant activity characteristics of refractance window dried *Curcuma longa*.

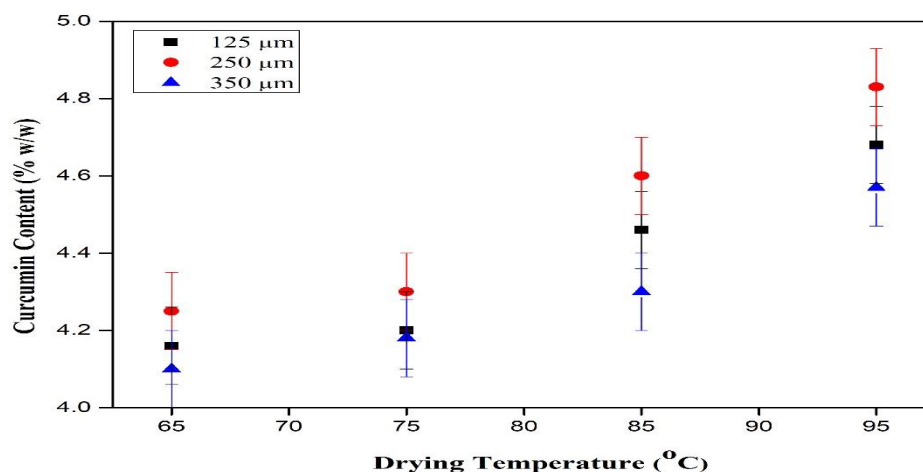


Figure 4.7: Curcumin content variation with drying temperature and mylar film thickness.

4.5.5 Curcumin Content

The yellow colour in turmeric is due to polyphenolic constituent curcumin which is lipophilic in nature (Geethanjali et al., 2016). The CC is 0.73 % w/w CC in the fresh turmeric sample. For the dried sample and for a temperature of 65 – 95 °C, the CC values are higher and vary from 4.16 – 4.68 % w/w CC for 125 μm mylar film case; from 4.25 – 4.83 % w/w CC for 250 μm mylar film case and from 4.10 – 4.57 % w/w CC for 350 μm mylar film case (Fig. 4.7). Among all samples, RWD sample obtained at 95 °C for 250 μm case possessed highest CC of 4.83 % w/w CC. Compared to 125 and 350 μm, the RWD at 250 μm retained marginally high CC. Unlike TPC, TFC and AA, heat treatment does not significantly influence the concentration of curcumin (Prathapan et al., 2009). Hence, the quantitative variation is low for the CC with respect to mylar film thickness and water bath temperature.

4.5.6 Colour Indices

The colour of a product has an important role in the acceptance of the product by the consumers. The *L*, *a* and *b* indices of fresh turmeric is 63.67, 43.07 and 75.12 respectively. After drying in the water bath temperature range of 65 – 95 °C, the colour values reduced significantly. Accordingly, the *L*, *a* and

Table 4.4: Colour index data summary of dried *Curcuma longa* samples at various combinations of drying temperature and mylar film thickness.

| S. No. | Temperature (°C) | 150 μm | | | 250 μm | | | 350 μm | | |
|--------|------------------|-------------------|----------|----------|-------------------|----------|----------|-------------------|----------|----------|
| | | <i>L</i> | <i>a</i> | <i>b</i> | <i>L</i> | <i>a</i> | <i>b</i> | <i>L</i> | <i>a</i> | <i>b</i> |
| 1. | 65 | 42.79 | 22.14 | 46.37 | 43.34 | 22.27 | 47.81 | 42.00 | 20.82 | 46.50 |
| 2. | 75 | 48.32 | 27.30 | 51.03 | 49.23 | 27.96 | 52.35 | 48.85 | 26.13 | 52.10 |
| 3. | 85 | 51.12 | 28.61 | 55.45 | 52.78 | 29.71 | 57.25 | 52.30 | 27.30 | 54.19 |
| 4. | 95 | 55.16 | 30.23 | 60.17 | 56.67 | 31.05 | 62.13 | 55.45 | 29.50 | 61.00 |

* All standard deviations for *L*, *a* and *b* were in the range of 1 – 2, 2 – 3 and 1 – 3 respectively.

b values varied between 42.79 – 55.16, 22.14 – 30.23 and 46.37 – 60.17 for 150 μm , 43.34 – 56.67, 22.27 – 31.05 and 47.81 – 62.13 for 250 μm and 42.00 – 55.45, 20.82 – 29.50 and 46.50 – 61.00 for 350 μm respectively. Thus, with reduced temperature, the colour values reduced significantly. This could be due to the fact that with reduced drying temperature the drying time increased and lead to enhanced heating of the turmeric slice sample. Thereby, it led to the darkening of the slice. The best *L*, *a* and *b* values was obtained for 250 μm at 95 °C. However, significant alteration in the *L*, *a* and *b* values with film thickness has not been achieved (Table 4.4). Since curcumin content plays an important constituent in the colour of turmeric, the *L*, *a* and *b* values for varied mylar film thickness cases did not alter significantly at the same temperature.

4.6 Optimal Mylar Film Thickness

Based on nutritional and other characteristics evaluated for the turmeric sample such as TPC, TFC, AA, CC, MC and colour indices, it can be concluded that the mylar film thickness alteration influenced marginally the nutritional responses. Among all cases, 250 μm mylar film case to an extent indicated better parametric values for the mentioned responses. In this regard, it can be noted that since 125 μm mylar film thickness enabled faster drying rate in comparison with higher mylar film thickness of 250 and 350 μm , the 350 μm mylar film demanded longer drying time duration to achieve equilibrium MC. On the other hand, prolonged exposure to heat is detrimental to retain nutritional characteristics

of the samples (Hernandez-Santos et al., 2016). Therefore, an optimal combination of heating rate and drying time is required to achieve desired combinations of nutritional characteristics.

Based on extensive analysis, the optimal heating rate and drying time correspond to the moderate mylar film thickness of 250 μm using which maximum bioactive constituents and hence their properties can be retained in the dried turmeric sample. Also, moisture removal rate and equilibrium drying time have been moderate for the case. The sensitivity of nutritional parameters with film thickness is not significant in comparison with water bath temperature. For this reason, marginal improvement or deterioration of dried turmeric nutritional characteristics has been achieved for a variation in mylar film thickness.

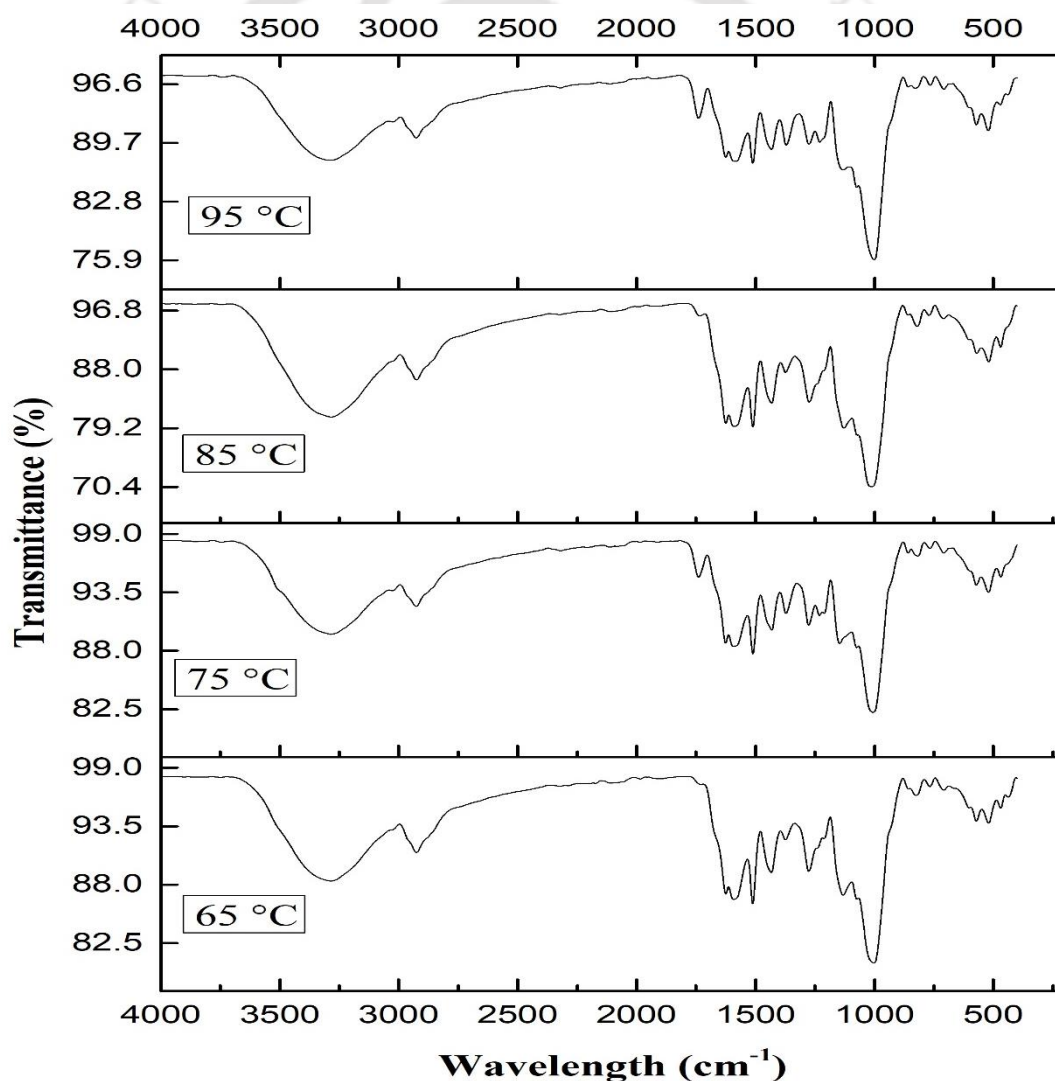


Figure 4.8: The FTIR spectra of dried turmeric powder products obtained at various water bath temperature values.

4.7 Fourier Transformed Infrared Spectroscopic Analysis

The Fourier Transformed Infrared (FTIR) analyses was conducted for sample processed with 250 μm mylar film and at water bath temperature of 65, 75, 85 and 95 $^{\circ}\text{C}$. Thereby, the effect of drying temperature and mylar film thickness have been studied on the functional groups present in the dried turmeric sample to study the effect of drying temperature and time on the functional groups present in turmeric. Fig. 4.8 presents the FTIR spectra and do confirm upon no significant alteration in the peaks of the spectra for all cases. The appearance of the peak in the wavenumber range of 1700-1800 cm^{-1} and for the 75 $^{\circ}\text{C}$ and 95 $^{\circ}\text{C}$ cases could be due to the existence of the carbonyl compound (Nandiyanto et al., 2019). Similar inference has been present in a relevant prior art (Safie et al., 2015). This is due to the RW drying process not inducing any alteration in the chemical constitution of the turmeric sample.

4.8 Statistical Analysis

The statistical assessment was carried out using one-way analysis of variance (ANOVA) and the Tukey model was used for the comparison of the mean values. Table 4.5 (a – r), summarize the findings and affirm that for all cases of drying temperature and mylar film thickness, high F values were obtained. The ANOVA based statistical analysis indicated a lower p-value for AA, TPC, TFC, CC, MC and colour indices. Hence, it can be inferred that the AA, TPC, TFC, CC, MC and colour indices values did not vary significantly and affirmed reasonable acceptability and good confidence of the measured values. Thereby, all findings and trends of the chapter can be confirmed to have good confidence levels.

Table 4.5 ANOVA based statistical analysis data summary for various parameters and mylar film thickness cases: (a) AA, 150 μm , (b) AA, 250 μm , (c) AA, 350 μm , (d) TFC, 150 μm , (e) TFC, 250 μm , (f) TFC, 350 μm , (g) TPC, 150 μm , (h) TPC, 250 μm , (i) TPC, 350 μm , (j) CC, 150 μm , (k) CC, 250 μm (l) CC, 350 μm , (m) MC, 150 μm , (n) MC, 250 μm and (o) MC, 350 μm , (p) colour indices, 150 μm , (q) colour indices, 250 μm and (r) colour indices, 350 μm .

| (a) | | | | | | |
|--------|-------|----|----------------|-------------|---------|---------|
| S. No. | | DF | Sum of Squares | Mean Square | F Value | Prob>F |
| 1. | Model | 3 | 50.5347 | 16.8449 | 2.93 | 0.09956 |
| 2. | Error | 8 | 45.98547 | 5.74818 | | |
| 3. | Total | 11 | 96.52017 | | | |

(b)

| S. No. | | DF | Sum of Squares | Mean Square | F Value | Prob>F |
|--------|-------|----|----------------|-------------|---------|---------|
| 1. | Model | 3 | 455.10 | 151.70 | 6.18 | 0.00877 |
| 2. | Error | 12 | 294.40 | 24.53416 | | |
| 3. | Total | 15 | 749.51 | | | |

(c)

| S. No. | | DF | Sum of Squares | Mean Square | F Value | Prob>F |
|--------|-------|----|----------------|-------------|---------|---------|
| 1. | Model | 3 | 287.02 | 95.67 | 3.62 | 0.04536 |
| 2. | Error | 12 | 316.97 | 26.41 | | |
| 3. | Total | 15 | 603.99 | | | |

(d)

| S. No. | | DF | Sum of Squares | Mean Square | F Value | Prob>F |
|--------|-------|----|----------------|-------------|---------|---------|
| 1. | Model | 3 | 435.76 | 145.25 | 7.80 | 0.00924 |
| 2. | Error | 8 | 148.90 | 18.61 | | |
| 3. | Total | 11 | 584.66 | | | |

(e)

| S. No. | | DF | Sum of Squares | Mean Square | F Value | Prob>F |
|--------|-------|----|----------------|-------------|---------|---------|
| 1. | Model | 3 | 486.15 | 162.05 | 11.25 | 0.00305 |
| 2. | Error | 8 | 115.18 | 14.39 | | |
| 3. | Total | 11 | 601.34 | | | |

(f)

| S. No. | | DF | Sum of Squares | Mean Square | F Value | Prob>F |
|--------|-------|----|----------------|-------------|---------|---------|
| 1. | Model | 3 | 428.21 | 142.73 | 9.74 | 0.00477 |
| 2. | Error | 8 | 117.18 | 14.64 | | |
| 3. | Total | 11 | 545.39 | | | |

(g)

| S. No. | | DF | Sum of Squares | Mean Square | F Value | Prob>F |
|--------|-------|----|----------------|-------------|---------|---------|
| 1. | Model | 3 | 973.73 | 324.57 | 26.19 | 0.00017 |
| 2. | Error | 8 | 99.11 | 12.38 | | |
| 3. | Total | 11 | 1072.84 | | | |

(h)

| S. No. | | DF | Sum of Squares | Mean Square | F Value | Prob>F |
|--------|-------|----|----------------|-------------|---------|--------|
| 1. | Model | 3 | 1133.04 | 377.68 | 30.20 | 0.0001 |
| 2. | Error | 8 | 100.02 | 12.50 | | |
| 3. | Total | 11 | 1233.07 | | | |

(i)

| S. No. | | DF | Sum of Squares | Mean Square | F Value | Prob>F |
|--------|-------|----|----------------|-------------|---------|---------|
| 1. | Model | 3 | 906.13 | 302.04 | 19.59 | 0.00048 |
| 2. | Error | 8 | 123.31 | 15.41 | | |
| 3. | Total | 11 | 1029.44 | | | |

(j)

| S. No. | | DF | Sum of Squares | Mean Square | F Value | Prob>F |
|--------|-------|----|----------------|-------------|---------|---------|
| 1. | Model | 3 | 0.35 | 0.11 | 8.88 | 0.00632 |
| 2. | Error | 8 | 0.10 | 0.01 | | |
| 3. | Total | 11 | 0.46 | | | |

(k)

| S. No. | | DF | Sum of Squares | Mean Square | F Value | Prob>F |
|--------|-------|----|----------------|-------------|---------|---------|
| 1. | Model | 3 | 0.65 | 0.21 | 18.49 | 0.00058 |
| 2. | Error | 8 | 0.09 | 0.01 | | |
| 3. | Total | 11 | 0.74 | | | |

(l)

| S. No. | | DF | Sum of Squares | Mean Square | F Value | Prob>F |
|--------|-------|----|----------------|-------------|---------|---------|
| 1. | Model | 3 | 0.38 | 0.12 | 12.66 | 0.00209 |
| 2. | Error | 8 | 0.08 | 0.01 | | |
| 3. | Total | 11 | 0.46 | | | |

(m)

| S. No. | | DF | Sum of Squares | Mean Square | F Value | Prob>F |
|--------|-------|----|----------------|-------------|---------|---------|
| 1. | Model | 3 | 3.23 | 1.07 | 14.85 | 0.00124 |
| 2. | Error | 8 | 0.58 | 0.07 | | |
| 3. | Total | 11 | 3.82 | | | |

(n)

| S. No. | | DF | Sum of Squares | Mean Square | F Value | Prob>F |
|--------|-------|----|----------------|-------------|---------|---------|
| 1. | Model | 3 | 3.04 | 1.01 | 11.42 | 0.00291 |
| 2. | Error | 8 | 0.71 | 0.08 | | |
| 3. | Total | 11 | 3.76 | | | |

(o)

| S. No. | | DF | Sum of Squares | Mean Square | F Value | Prob>F |
|--------|-------|----|----------------|-------------|---------|--------|
| 1. | Model | 3 | 2.26 | 0.75 | 20.26 | 0.0004 |
| 2. | Error | 8 | 0.29 | 0.03 | | |
| 3. | Total | 11 | 2.56 | | | |

(p)

| S. No. | | DF | Sum of Squares | Mean Square | F Value | Prob>F |
|--------|-------|----|----------------|-------------|---------|---------|
| 1. | Model | 3 | 255.48 | 85.16 | 17.72 | 0.00068 |
| 2. | Error | 8 | 38.44 | 4.80 | | |
| 3. | Total | 11 | 293.93 | | | |

(q)

| S. No. | | DF | Sum of Squares | Mean Square | F Value | Prob>F |
|--------|-------|----|----------------|-------------|---------|---------|
| 1. | Model | 3 | 283.32 | 94.44 | 24.58 | 0.00021 |
| 2. | Error | 8 | 30.73 | 3.84 | | |
| 3. | Total | 11 | 314.05 | | | |

(r)

| S. No. | | DF | Sum of Squares | Mean Square | F Value | Prob>F |
|--------|-------|----|----------------|-------------|---------|---------|
| 1. | Model | 3 | 287.53 | 95.84 | 22.77 | 0.00028 |
| 2. | Error | 8 | 33.67 | 4.20 | | |
| 3. | Total | 11 | 321.20 | | | |

4.9 Literature Comparison

The best obtained data in this work has been compared with relevant literature data and presented in Table 4.6. As presented in the table, the best case in this work corresponds to 90 minutes equilibrium time which is significantly lower than the tray drying and oven drying cases reported in the

literature. Since tray drying was conducted at its best process condition (60 °C), the temperature of the system is significantly lower than that of the RWD being operated at the best process condition of 95 °C. The comparative investigations affirmed that the RWD method can better retain desired nutritional characteristics than the conventional tray drying process.

While tray drying investigations have not been addressed in this work for the turmeric system, the available prior art of Ochoa-Martinez et al., (2012) affirm upon this towards this inference. This is due to faster drying facilitated through the heat conduction enabled in the refractance window drying process. For the kiwi slices system (Azizi et al., 2017), the optimal mylar film thickness refers to 100 µm film thickness. The said film enabled the realization of equilibrium moisture content for a drying time of 40 min. However, for the case of turmeric system, the optimal mylar film thickness and drying time were 250 µm and 90 min respectively. Since kiwi slices and turmeric have variant physical and chemical characteristics, the optimal findings of the two systems cannot be compared with one another. The kiwi system data has been considered due to being the only data available till date with respect to the sensitivities of mylar film thickness for agri and horticultural produce systems. Thus, the reported findings of the article are anticipated to serve as a benchmark data to further enhanced the application of RWD and other improved drying processes for agri and horticultural produces. Compared to the tray and oven drying system, the RWD system enables the realization of a dried product with lower moisture content and nutritional characteristics.

4.10 Summary

Important conclusions can be deduced from the work reported in this article. Firstly, the RWD nutritional characteristics of turmeric are critically influenced with hot water bath temperature but not with mylar film thickness (125 – 350 µm). Secondly, mylar film thickness enhancement increased equilibrium drying time by about 40 % (from 75 to 105 minute) due to the reduction in conductive heat transfer rates at 95 °C. However, temperature had a potential impact in reducing equilibrium drying temperature by 73 % for the mylar film thickness case of 250 µm. Thirdly, drying kinetics data indicated best fitness with Singh et al., (2014) model with diffusivity varying from 3.71×10^{-11} to

$1.39 \times 10^{-10} \text{ m}^2/\text{s}$ and activation energy marginally varying from 52.57 – 49.43 kJ for variant mylar film thickness. Fourthly, the optimal combinations of temperature (95 °C) and mylar film thickness (250 μm) enabled a significant reduction in equilibrium time to 90 min and marginally better retention of nutritional characteristics. Finally, the FTIR analyses inferred that no significant change occurred in the RW dried turmeric slice due to drying at different bath temperatures. Dried turmeric samples with better retention of nutritional characteristics are anticipated to provide useful edge in fortified products such as golden milk and turmeric tablets. Thereby, processes such as RWD can be targeted for mentioned commercial applications.

Table 4.6: Comparative summary of optimal process-product characteristics of *Curcuma longa* dried samples.

| S. No. | Drying Method | Sample | Film thickness | T (°C) | Time (min) | Optimal Parameters | | | | | Ref |
|--------|---------------|---------------------|-------------------|--------|------------|---------------------------------|----------------------------|--------|-----------|--------|----------------------|
| | | | | | | TFC (mg quercetin/g dry sample) | TPC (mg GAE/ g dry sample) | AA (%) | CC (%w/w) | MC (%) | |
| 1. | RWD | Turmeric (1 mm) | 250 μm | 95 | 90 | 160.9 | 189.7 | 89.1 | 4.83 | 4.10 | This work |
| 2. | Tray | Turmeric (1 mm) | - | 60 | 480 | 122.9 | 140.2 | 80.0 | 3.60 | 2.90 | |
| 3. | RWD | Kiwi (1.6 mm slice) | 100 μm | 90 | 40 | - | - | - | - | - | Azizi et al., (2016) |





Chapter 5:
Response Surface Methodology Based
Optimality of RWD Process Parameters



Response Surface Methodology Based Optimality of RWD Process Parameters

In this chapter, the findings based on the response surface methodology based refractance window drying of turmeric slices have been addressed. Water bath temperature, drying time and air velocity have been targeted for the Box-Behnken design based experiments and optimization. After a brief introduction in section 5.1, sections 5.2 and 5.3 respectively delineate on the control data set and Box-Behnken design based experimental data set. Thereafter, sections 5.4, 5.5 and 5.6 respectively present the influence of drying time, water bath temperature and air velocity on the responses of the dried sample namely MC, TPC, TFC, CC, AA and colour indices. Thereby, section 5.7 and 5.8 delineate on the optimal data set based on experimental design and best fit model selection. Following this, section 5.9 summarizes results obtained from variance analysis for MC, TPC, TFC, CC, AA and colour indices. The following sections 5.10 and 5.11 elaborate on response surface analysis and numerical optimization and experimental validation of optimal data. Finally, Section 5.13 and 5.14 present literature comparison with prior art and a summary of the chapter.

5.1 Introduction

Response surface methodology (RSM) based process parametric optimality has been investigated for several food processing and engineering systems such as extraction and fermentation. Wang et al., (2012) targeted the RSM based optimization of polysaccharides production from *Panax japonicas* rhizomes. The authors considered carotenoids as response variables and attempted to maximize the extraction of polysaccharides through the evaluation of the anti-oxidant activity (AA) of the polysaccharides. Considering the ratio of ethanol to ammonium sulfate, temperature, pH value and time of extraction as process parameters, Lui et al., (2015) followed Box-Behnken design (BBD) approach and designed 27 runs (with 24 factorial experiments and 3 runs at the center point) for the

maximization of anthocyanin extraction from purple sweet potatoes. Based on analysis of variance (ANOVA) findings, the authors inferred that the second order polynomial equation was the best among alternate models. Finally, model validation was targeted for the optimal solution obtained from numerical optimization. It was found that the experimentally measured values were very close to RSM based numerically optimized. Sumic et al., (2016) carried out optimization of shiitake mushroom using vacuum dryer to obtain samples with good shelf life. The authors inferred that 57.1 °C and 100 mbar are the optimal process parameter values. In another study, Bazaria and Kumar, (2018) investigated the RSM based optimality of beetroot juice powder production using spray drying technology. BBD was followed for the realization of the optimality of inlet air temperature (160 – 180 °C), maltodextrin addition rate (5 – 15 %) and feed flow rate (400 – 600 mL/h). Moghaddam et al., (2019) investigated the influence of spray drying process parameters such as air temperature (100 – 140 °C), maltodextrin concentration (30 – 60 %), and aspiration rate (30 – 50 %). Using RSM, the authors optimized the physio-chemical properties of sour cherry powder (bulk density, hygroscopicity, moisture content, and water-soluble index). Thereby, process parametric optimality was achieved.

Till date, the parametric optimality of refractance window drying (RWD) process considered trial and error-based optimization for alternate produces such as carrot, onion etc. (Akinola et al., 2009; Hernandez et al., 2016). In such several investigations, alternate processes and their parametric criticality (such as temperature and sample size) have been considered for any agro-horticultural produce. The RSM based optimization of process parameters such as water bath temperature, drying time and air-velocity have not been targeted for the RWD of any agri-horticultural produce. Such investigations can provide useful insights with respect to the associated sensitivities. Thereby, mathematical modelling expressions can be developed for the optimal design and operation of large scale RWD systems for the cost effective drying of turmeric produce.

Considering these lacunae, the chapter addresses the RSM based RWD characteristics of turmeric slices in the water bath temperature, drying time and air-velocity range of 65 – 95 °C, 75 – 360 min, and 0.5 – 1 m/s respectively. These parametric ranges have been set based on preliminary trials. For all cases, mylar film and sample thickness of 250 µm and 1 mm respectively have been chosen based on similar preliminary trials. Desired response characteristics include anti-oxidant activity (AA),

total phenolic content (TPC), total flavonoids content (TFC), moisture content (MC), colour indices and curcumin content (CC). Thereby, targeting the maximum of all mentioned product characteristics, the RSM based process parametric optimality was targeted in terms of the optimality of drying temperature, time and air-velocity. Finally, the optimal process product characteristics were validated.

5.2 Control Data Set

The triplicate experiments were performed to visualize the comparative criticality of the findings of this work. These refer to a case corresponding to stagnant RWD operation (impeller free system), 95 °C water bath temperature and 90 min drying time. For the case, the average characteristics have been obtained as 4.10 %, 89.100 %, 189.76 mg GAE/g sample, 160.96 mg quercetin/g sample, 4.83 % w/w and 56.67, 31.05 and 62.13 (*L*, *a* and *b* values) for MC, AA, TPC, TFC, CC and colour indices respectively.

5.3 Box Behken Design Based Experimental Design

The overall RSM based methodology involves the hierarchy of experimental design using BBD, identification of best fit model based on statistical analysis such as ANOVA and parametric optimization and validation through numerical optimization of the best fit model (Sun et al., 2010). Table 5.1 summarizes the data associated to variant degrees of freedom combinations indicated by the BBD based RSM design. Thereby, the responses have been determined based on triplicate runs. Mean and standard deviation values have been also determined and presented in Table 5.1.

5.4 Effect of Drying Temperature on Responses

Table 5.1 affirms that the sample AA enhanced significantly with drying temperature. For water bath temperature range of 65 – 95 °C and for 75 min drying time duration, the AA enhanced from 82 to 90 %. However, for the 95 °C bath temperature case, prolonged exposure (upto 360 min) ensured reduction in AA. Further, corresponding variations at the center point (at 80 °C) have not been significant.

For the TPC case and for 75 min drying time, the TPC enhanced from 90 to 190 mg GAE/g sample for the water bath temperature range of 65 – 95 °C. However, the TPC reduced for the case of prolonged time duration (360 min) at 95 °C but not for the 65 °C based water bath temperature (121 to 165 mg GAE/g sample).

The TFC values have been analyzed to enhance with increasing water bath temperature (56 to 160 mg quercetin/g sample for 65 – 95 °C). Corresponding variation for prolonged drying (360 min) were from 142 - 99 mg quercetin/g sample for a variation in bath temperature range of 65 – 95 °C. On the other hand, for 75 min drying duration, the corresponding variation has been from 56 – 160 mg quercetin/g sample.

Similar trends exist for CC variation with drying temperature. The highest CC (4.81 % w/w) has been obtained for 95 °C and 75 min case. The enhancement in AA, TPC, TFC and CC is due to enhanced dry sample matter content with temperature. This is due to the greater removal of moisture per gram of sample at higher temperature that fostered faster drying rates (Mondal et al., 2020a).

The MC varied with mentioned water bath temperature range from 3.50 – 20.20 %. At 95 °C, the MC was lowest with a value of 3.50 % for a drying time of 360 min. However, at a drying temperature 65 °C, the highest MC of 20.20 % was obtained for 75 min.

In case of colour, the *L* values ranged from 35.25 – 59.82 for 65 – 95 °C. For prolonged duration of 360 min and in the water bath temperature range 65 – 95 °C, the *L* values decreased (43.34 – 35.25). For all temperature cases, the *L* values were high (56.67 – 59.82) for lower drying duration of 75 min. The *L* values for 80 °C was 57.45 at 75 min. The *L* values further decreased to 39.21 for 360 min duration at 80 °C.

5.5 Effect of Drying Time on Responses

Drying time significantly influenced all response variable characteristics. For all cases, the optimal retention of response characteristics was indicated by the best drying time corresponding to the chosen drying temperature (Table 5.1). These refer to 360 and 75 min for water bath temperatures of 65 and 95 °C respectively. The lowest (59 %) and highest (90 %) AA were obtained for the cases of 75

min and for 65 and 95 °C respectively. Further, longer duration (360 min) at 95 °C and shorter duration (75 min) at 65 °C enabled a significant reduction in the AA characteristics

For the TPC case, the best drying duration refer to 75 min and 360 min for 95 and 65 °C cases respectively. Corresponding TPC variation has been from 90 to 190 mg GAE/ g sample. Further, the response reduced from 190 – 121 mg GAE/ g sample for prolonged duration (from 75 – 360 min) at 95 °C but not at 65 °C (from 90 – 165 mg GAE/ g sample).

For the TFC cases, 95 °C and 75 min duration affirmed the best TFC value. However, the TFC varied from 99 to 142 mg quercetin/g sample for a time duration of 360 min. Also, the TFC varied from 56 to 160 mg quercetin/g sample for 75 min time duration and for enhanced drying temperature (65 – 95 °C). The corresponding variations in AA, TPC and TFC have been due to associated sensitivities of volatile constituents with respect to the drying time and temperature. In other words, longer drying periods at higher temperature is detrimental to the retention of these response variables.

For the curcumin case, similar trends have been analyzed. For drying duration range of 75 – 360 min, the response varied from 4.80 to 4.20 % w/w and for temperature variation from 65 – 95 °C. However, the variable sensitivity has been comparably insignificant with respect to the trends prevalent for AA, TPC and TFC. This is due to significant contribution of non-volatile constituents towards curcumin (Mondal et al., 2020b).

For all cases, the moisture content reduced with drying time. The lowest moisture content for 65, 80 and 95 °C cases were 5.16, 4.50 and 3.50 % and for 360, 217.5 and 360 min drying time respectively. At 95 °C, the desired required moisture content value below the 8 % for the inhibition of microbial growth was achieved at 75 min duration. However, it was 217.5 and 360 min for 80 and 65 °C cases respectively.

With increasing drying time, the *L* values reduced for all cases. The highest and lowest *L* values were obtained for 75 and 360 min respectively and water bath temperature for all cases (65 – 95 °C). For a drying period of 360 and 75 min, the highest *L* values was obtained at 65 °C and the lowest was obtained at 95 °C. At 65 °C, the *L* values increased with time. However, at 95 °C, it decreased with time. At 80 °C, the *L* values increased with time upto 217.5 min and eventually decreased upto 360 min.

Table 5.1: Box-Behken Design based data summary of refractance window dried *Curcuma longa* system.

| Run | Temperature (°C) | Air-velocity (m/s) | Drying time (min) | AA (%) | TPC (mg GAE/g dry sample) | TFC (mg quercetin dry sample) | CC (%w/w) | MC (%) | Colour (L values) |
|-----|------------------|--------------------|-------------------|--------|---------------------------|-------------------------------|-----------|--------|-------------------|
| 1 | 65 | 0.50 | 217.5 | 64 | 105 | 90 | 3.46 | 14.00 | 49.73 |
| 2 | 95 | 0.50 | 217.5 | 79 | 131 | 119 | 4.78 | 4.19 | 40.56 |
| 3 | 80 | 0.50 | 75 | 65 | 110 | 84 | 2.86 | 11.61 | 57.45 |
| 4 | 80 | 0.75 | 217.5 | 84 | 174 | 151 | 4.43 | 4.50 | 51.82 |
| 5 | 95 | 0.75 | 75 | 90 | 190 | 160 | 4.84 | 4.00 | 55.70 |
| 6 | 80 | 0.75 | 217.5 | 85 | 177 | 150 | 4.40 | 4.50 | 51.56 |
| 7 | 95 | 0.75 | 360 | 77 | 121 | 99 | 4.26 | 3.50 | 35.25 |
| 8 | 65 | 0.75 | 75 | 59 | 90 | 56 | 2.11 | 20.20 | 59.82 |
| 9 | 65 | 0.75 | 360 | 82 | 165 | 142 | 4.28 | 5.63 | 43.74 |
| 10 | 95 | 1.00 | 217.5 | 80 | 133 | 120 | 4.77 | 3.70 | 40.12 |
| 11 | 80 | 0.75 | 217.5 | 83 | 176 | 149 | 4.39 | 4.56 | 50.01 |
| 12 | 80 | 1.00 | 75 | 67 | 112 | 89 | 3.15 | 11.10 | 57.15 |
| 13 | 80 | 0.50 | 360 | 71 | 118 | 103 | 3.98 | 4.31 | 39.75 |
| 14 | 80 | 1.00 | 360 | 70 | 119 | 101 | 3.76 | 4.25 | 39.21 |
| 15 | 65 | 1.00 | 217.5 | 66 | 108 | 93 | 3.55 | 13.80 | 49.16 |

* All standard deviations for AA, TPC, TFC, CC, MC and Colour indices were in the range 1 – 2, 3 – 4, 3 – 5, 0.1 – 0.2, 0.25 – 0.5 and 1 – 2 respectively.

5.6 Effect of Air-Velocity on Responses

The data summarized in Table 5.1 affirms that the intermediate air-velocity value of 0.75 m/s favoured best response characteristics. Other independent variable values for all cases can be analyzed to be 95 °C (high) and 75 min (low). For the AA case, maximum AA has been obtained as 90 %, which is higher than the corresponding values obtained at lower and higher velocity (64 – 80 % for variation in air-velocity from 0.5 – 1 m/s). On the other hand, for the long drying duration of 360 min and 95 °C case, the AA values reduced to 77 %. Further, the center point AA values at 80 °C have been lower.

For the TPC case at 95 °C, the TPC varied from 131 – 190 mg GAE/g sample with a variation in air-velocity from 0.5 – 1 m/s. The highest TPC value of 190 mg GAE/g sample has been obtained for a corresponding air-velocity of 0.75 m/s.

For the TFC case, similar trends exist. For the lower drying time of 75 min, the TFC varied from 56 – 160 mg quercetin/g sample for a variation in air-velocity from 0.5 – 1 m/s. The maximum value of TFC was obtained for the intermediate air-velocity of 0.75 m/s.

Similar trends exist for the case of CC variation with air-velocity. The maximum CC has been obtained as 4.80 % w/w for the intermediate air-velocity of 0.75 m/s. In summary, compared to AA, TPC and TFC, the CC sensitivity with air-velocity has not been significant. This is due to the dominant role of non-volatile constituents towards curcumin but not AA, TPC and TFC (Mondal et al., 2020c; Prathapan et al., 2009).

For the MC case at 95 °C, the MC varied from 3.5 – 4.1 % with a variation in air-velocity from 0.5 – 1 m/s. The highest MC value of 20.20 % was obtained for 0.75 m/s at 65 °C. However, the lowest was 3.50 % for 360 min at 95 °C. For all cases of air velocity, the MC reduced with increasing drying time.

For the colour indices case, highest and lowest *L* values were obtained at 65 and 95 °C and at 0.75 m/s but with a drying time of 75 min and 360 min respectively. At 80 °C, almost similar results were obtained for 0.50 and 1 m/s air-velocity cases and at respective drying time of 75 and 360 min.

5.7 Optimal Data Set based on Experimental Design

Based on experimental findings summarized in Table 5.1, the best set of RSM based experimental data set correspond to 95 °C, 75 min and 0.75 m/s for water bath temperature, drying duration and air-velocity respectively.

5.8 Model Selection

Among alternate multiple variable regression models, the best fit model has been identified based on the combination of high F-value and low p-value. The relevant findings have been summarized in Table 5.2 – 5.7. Among alternate models, the most appropriate model has been the quadratic model with adequate F value (all being high with 181.13, 435.61, 755.62, 1120.76, 425.36 and 79.07 for AA, TPC, TFC, CC, MC and colour indices respectively) and p-values (all being low) for all cases. Thus, non-linear models are applicable for all responses in terms of the mentioned independent variables.

5.9 Variance Analysis

The variance analysis findings of AA, TPC, TFC, CC, MC and colour indices have been summarized in Table 5.2 – 5.7. A detailed account of ANOVA has been presented in the following sub-sections.

5.9.1 Anti-oxidant Activity

For the most appropriate (quadratic) model representing AA, the R^2 and adj R^2 were found to be 0.996 and 0.991 respectively. These indicate appropriate model adequacy. The F-value, p-value and lack of fit were 181.13, <0.0001 and 0.58 (insignificant) respectively. All ANOVA parameters (F-values of 504.17, 2.67 and 60.17 and p-values of <0.0001, 0.1634 and 0.0006 for temperature, air-velocity and time respectively) can be analyzed to affirm best fitness of the quadratic model. In the most appropriate (quadratic) model, all terms had adequate competence (F-value and p-value being high and low respectively). Accordingly, the AA can be mathematical presented as:

$$AA = 84 + 6.87A + 0.50B + 2.38C - 0.25AB - 9AC - 0.75BC - 1.50A^2 - 10.25B^2 - 5.50C^2$$

where A, B and C are temperature, air-velocity and drying time respectively.

In the above equation, all linear terms (temperature, air-velocity and time) had a contributory role towards the response. However, the higher order terms for all degrees of freedom had a detrimental influence on the AA.

5.9.2 Total Phenolic Content

For the TPC analysis, the ANOVA parametric are similar to those indicated for AA. For the most appropriate (quadratic) model, high F-value (435.61) and low p-value (<0.0001) have been obtained. With a lack of fit value of 1.96, the model fitness is appropriate. For the regressed parameters, the F-value have been obtained as 388.54, 2.17 and 14.97 for temperature, air-velocity and time respectively. Corresponding p-values have been obtained to be <0.0001, 0.2005 and 0.0118. Also, very good combinations of R² (0.998) and adj R² (0.996) were obtained. These are in good agreement with the obtained F and p values. Accordingly, the TPC response characteristics can be mathematically represented as:

$$TPC = 175.67 + 13.37A + 1B + 2.63C - 0.25AB - 36AC - 0.25BC - 14.83A^2 - 41.58B^2 - 19.33C^2$$

where A, B and C are temperature, air-velocity and drying time respectively.

In the above equation, all linear terms (temperature, air-velocity and time) had a contributory role towards the response. However, the higher order terms for all degrees of freedom had a detrimental influence on the TPC.

5.9.3 Total Flavonoid Content

Also, for the TFC case, ANOVA parameters trends are similar to those obtained for AA and TPC. For, the best fit quadratic model F, p and lack of fit values have been obtained as 755.62, <0.0001 and 2.58 (insignificant) respectively. All these confirms good corresponding F-values for linear terms viz. temperature, air-velocity and time have been obtained as 877.50, 3.14 and 201.03 respectively. For all such cases, corresponding p-value were obtained as <0.0001, 0.1365 and <0.0001. The R² and adj

R^2 values have been obtained as 0.999 and 0.997 respectively and affirmed good fitness. Accordingly, best fit TFC model can be expressed as:

$$TFC = 150 + 14.62A + 0.88B + 7C - 0.5AB - 36.75AC - 1.75BC - 12.25A^2 - 32.25B^2 - 23.50C^2$$

where A, B and C are temperature, air-velocity and drying time respectively.

In the above equation, all linear terms (temperature, air-velocity and time) had a contributory role towards the response. However, the higher order terms for all degrees of freedom had a detrimental influence on the TFC.

5.9.4 Curcumin Content

For the best fit quadratic model, corresponding ANOVA parameters indicate good fitness (0.999 R^2 , 0.998 adj R^2 , insignificant lack of fit (2.91), 690.29 F-value and <0.0001 p-value). Also, all terms in the most appropriate (quadratic) model can be inferred to be promising (appropriate value of F (high) and p (low)). Thereby, their fitness can be as well confirmed. For the best fit parameter values (temperature, air-velocity and time), the corresponding F-values have been obtained as 2481.62, 2.03 and 992.41. Corresponding p-values have been obtained as <0.0001, 0.2139 and <0.0001. For the CC, the best fit model can be expressed as:

$$CC = 4.41 + 0.66A + 0.019B + 0.42C - 0.025AB - 0.69AC - 0.13BC + 0.084A^2 - 0.35B^2 - 0.62C^2$$

where A, B and C are temperature, air-velocity and drying time respectively.

In the above equation, it is apparent that the linear terms viz. temperature, air-velocity and time had a contributory role on response. On the other hand, the few higher order terms had a negative influence. Among quadratic terms while temperature had a positive effect, air-velocity and time had a negative effect.

5.9.5 Moisture Content

For the case of MC and for the best fit quadratic model, corresponding ANOVA parameters indicate good fitness (0.998 R^2 , 0.996 adj R^2 , insignificant lack of fit (3.99), 425.36 F-value and <0.0001 p-value). Also, all terms in the most appropriate (quadratic) model can be inferred to be promising

(appropriate value of F (high) and p (low)). Thereby, their fitness can be as well confirmed. For the best fit parameter values (temperature, air-velocity and time), the corresponding F-values have been obtained as 1866.25, 2.05 and 1089.67. Corresponding p-values have been obtained as <0.0001, 0.2110 and <0.0001. For the MC, the best fit model can be expressed as:

$$MC = 4.64 - 4.78A - 0.16B - 3.65C - 0.072AB + 3.52AC + 0.11BC + 2.40A^2 + 1.89B^2 + 1.30C^2$$

where A, B and C are temperature, air-velocity and drying time respectively.

In the above equation, it is apparent that the linear terms viz. temperature, air-velocity and time had a negative role on response. Also, except interaction of temperature and air-velocity all higher order terms had a positive influence on the MC.

5.9.6 Colour Indices

For the *L* values of the dried sample colour and for the best fit quadratic model, corresponding ANOVA parameters indicate good fitness (0.993 R^2 , 0.980 adj R^2 , insignificant lack of fit (1.40), 79.07 F-value and <0.0001 p-value). Also, all terms in the most appropriate (quadratic) model can be inferred to be promising (appropriate value of F (high) and p (low)). Thereby, good model fitness is confirmed. For the best fit parameter values (temperature, air-velocity and time), the corresponding F-values have been obtained as 99.98, 0.36 and 548.22. Corresponding p-values have been obtained as 0.0002, 0.0574 and <0.0001. For the colour indices, the best fit model can be expressed as:

$$Colour = 51.13 - 3.85A - 0.23B - 9.02C + 0.032AB - 1.09AC - 0.060BC - 3.00A^2 - 3.24B^2 + 0.50C^2$$

where A, B and C are temperature, air-velocity and drying time respectively.

In the above equation, it is apparent that the linear terms viz. temperature, air-velocity and time had a negative role on the response. On the other hand, while higher order terms of temperature, drying time and air-velocity had a negative influence, the time and interaction between temperature and air-velocity had a positive influence.

5.10 Response Surface Analysis

The response surface plots (Fig. 5.1 – 5.6) confirm that for all responses namely AA, TPC, TFC, CC, MC and colour indices had significant influence of drying temperature and time and marginal influence of air-velocity have been apparent. With the exception of MC and colour indices, all cases namely AA, TPC, TFC and CC, the linear terms of all degrees of freedom are contributory and indicate enhanced characteristics with a corresponding enhancement. However, for the AA, TPC and TFC cases, all higher order terms had a detrimental influence. Also, for the CC, MC and colour indices case, mixed influence exists. Also, the higher bath temperature of 95 °C fostered faster drying to thereby facilitate higher values of heat transfer rates, moisture diffusivity and hence moisture removal. All these translated into a higher retention of the response variables. The plots affirmed that 95 °C drying temperature (high), 75 min drying time (low) and 0.75 m/s air-velocity (intermediate) favoured best responses for all evaluated characteristics. Also, the response plots affirm that prolong exposure to higher temperature is detrimental for all responses.

5.11 Numerical Optimization and Validation

The numerical optimality was targeted using Design Expert 7.0 software. AA, TPC, TFC, CC, and colour indices were simultaneously maximized while MC was minimized as the sole objective function. The independent variables were constrained to their corresponding lower and higher values. Thereby, the optimal process parametric and response characteristics refers to 95 °C, 75 min and 0.76 m/s and 90.52 % AA, 188.22 mg GAE/g sample TPC, 158.65 mg quercetin/g sample TFC, 4.80 % w/w, 3.67 % MC and 54.87 L values respectively. For the best data set, triplicate experiments have been conducted. The standard deviation for numerically optimized response refers to 0.5 – 1.2, 1.5 – 2.16, 0.79 – 2.41, 0.02 – 0.04, 0.2 – 0.4, and 2.1 – 3.0 for AA, TPC, TFC, CC, MC and colour indices respectively. With low standard deviation values (< 5 %), good fitness values can be ensured for the developed model. Hence, the optimal values have been validated.

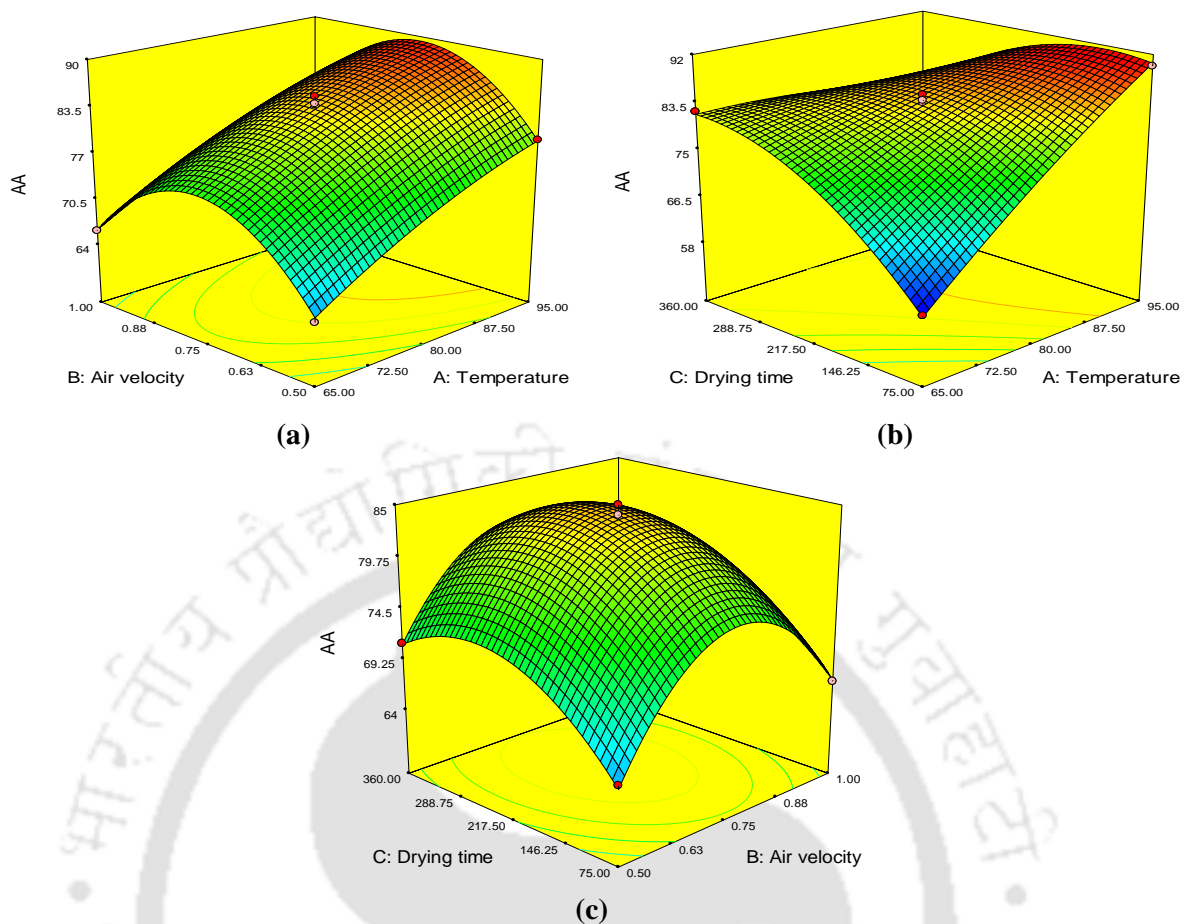


Figure 5.1: Response surface plot of anti-oxidant activity of refractance window dried turmeric samples and for various binary combinations (a) temperature and air velocity, (b) temperature and drying time and (c) air velocity and drying time.

Table 5.2: ANOVA data summary of best fit model to represent anti-oxidant activity of BBD-RSM based RWD of *Curcuma longa*.

| S. No. | | F-values | p-values | |
|--------|--------------------|----------|----------|-----------------|
| 1. | Model | 181.13 | <0.0001 | Significant |
| 2. | A-Temperature | 504.17 | <0.0001 | |
| 3. | B-Air-velocity | 2.67 | 0.1634 | |
| 4. | C-Drying time | 60.17 | 0.0006 | |
| 5. | AB | 0.33 | 0.5887 | |
| 6. | AC | 432.00 | <0.0001 | |
| 7. | BC | 3.00 | 0.1438 | |
| 8. | A ² | 11.08 | 0.0208 | |
| 9. | B ² | 517.23 | <0.0001 | |
| 10. | C ² | 148.92 | <0.0001 | |
| 11. | Lack of Fit | 0.58 | 0.6812 | Not significant |
| 12. | R ² | 0.996 | | |
| 13. | Adj R ² | 0.991 | | |

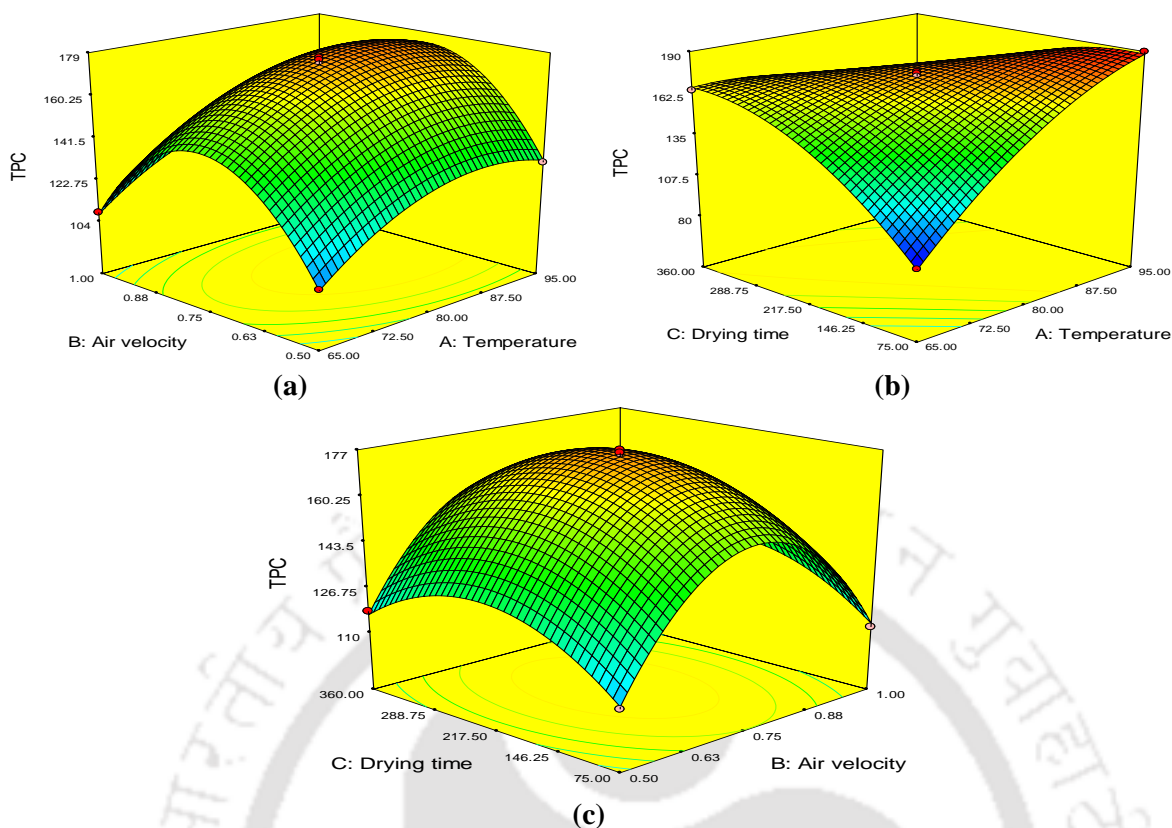


Figure 5.2: Response surface plot of total phenolic content of refractance window dried turmeric samples and for various binary combinations (a) temperature and air velocity, (b) temperature and drying time and (c) air velocity and drying time.

Table 5.3: ANOVA data summary of best fit model to represent total phenolic content of BBD-RSM based RWD of *Curcuma longa*.

| S. No. | | F-values | p-values | |
|--------|--------------------|----------|----------|-----------------|
| 1. | Model | 435.61 | <0.0001 | Significant |
| 2. | A-Temperature | 388.54 | <0.0001 | |
| 3. | B-Air-velocity | 2.17 | 0.2005 | |
| 4. | C-Drying time | 14.97 | 0.0118 | |
| 5. | AB | 0.068 | 0.8048 | |
| 6. | AC | 1407.42 | <0.0001 | |
| 7. | BC | 0.068 | 0.8048 | |
| 8. | A ² | 220.56 | <0.0001 | |
| 9. | B ² | 1733.39 | <0.0001 | |
| 10. | C ² | 374.69 | <0.0001 | |
| 11. | Lack of Fit | 1.96 | 0.3549 | Not significant |
| 12. | R ² | 0.998 | | |
| 13. | Adj R ² | 0.996 | | |

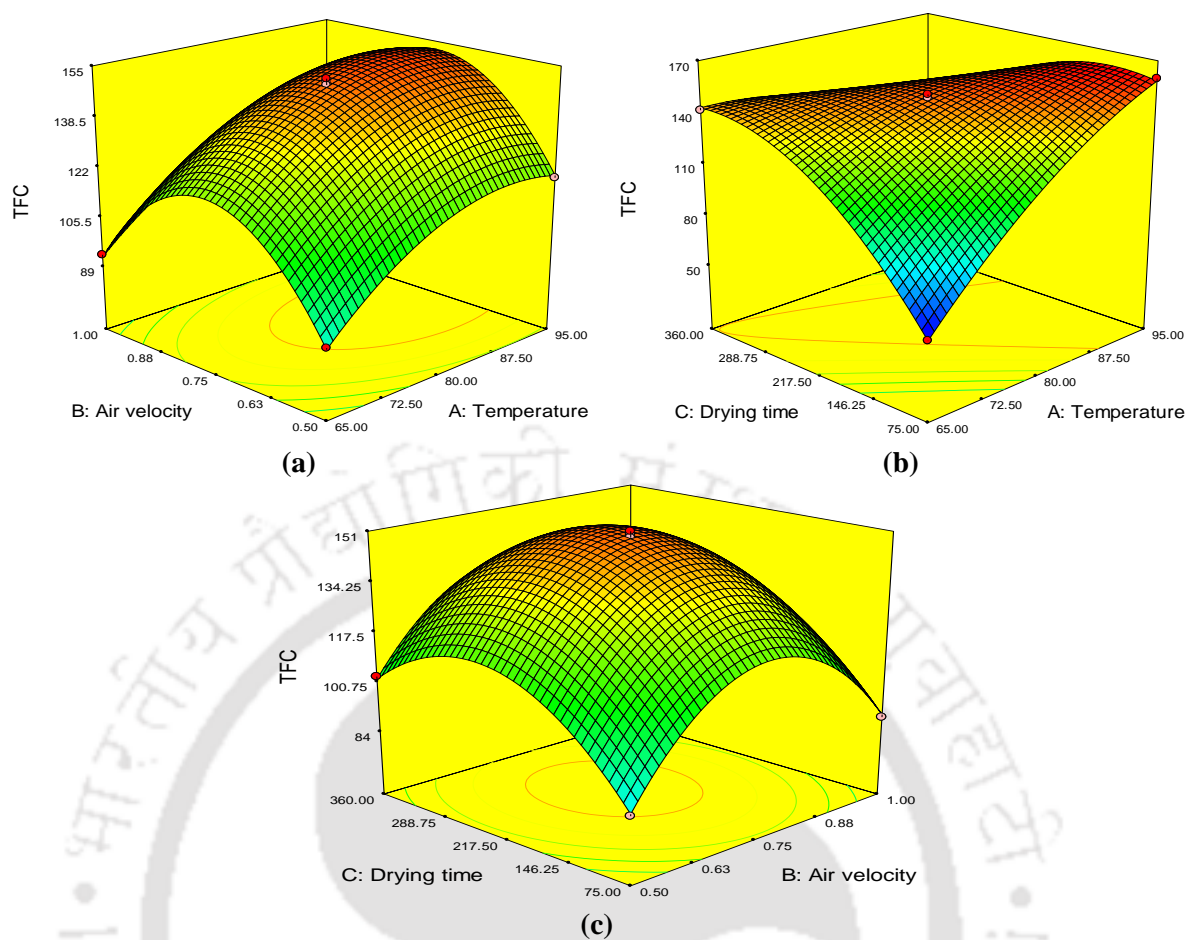


Figure 5.3: Response surface plot of total flavonoid content of refractance window dried turmeric samples and for various binary combinations (a) temperature and air velocity, (b) temperature and drying time and (c) air velocity and drying time.

Table 5.4: ANOVA data summary of best fit model to represent total flavonoid content of BBD-RSM based RWD of *Curcuma longa*.

| S. No. | | F-values | p-values | |
|--------|--------------------|----------|----------|-----------------|
| 1. | Model | 755.62 | <0.0001 | Significant |
| 2. | A-Temperature | 877.53 | <0.0001 | |
| 3. | B-Air-velocity | 3.14 | 0.1365 | |
| 4. | C-Drying time | 201.03 | <0.0001 | |
| 5. | AB | 0.51 | 0.5060 | |
| 6. | AC | 2770.38 | <0.0001 | |
| 7. | BC | 6.28 | 0.0541 | |
| 8. | A ² | 284.14 | <0.0001 | |
| 9. | B ² | 1969.35 | <0.0001 | |
| 10. | C ² | 1045.68 | <0.0001 | |
| 11. | Lack of Fit | 2.58 | 0.2913 | Not significant |
| 12. | R ² | 0.999 | | |
| 13. | Adj R ² | 0.997 | | |

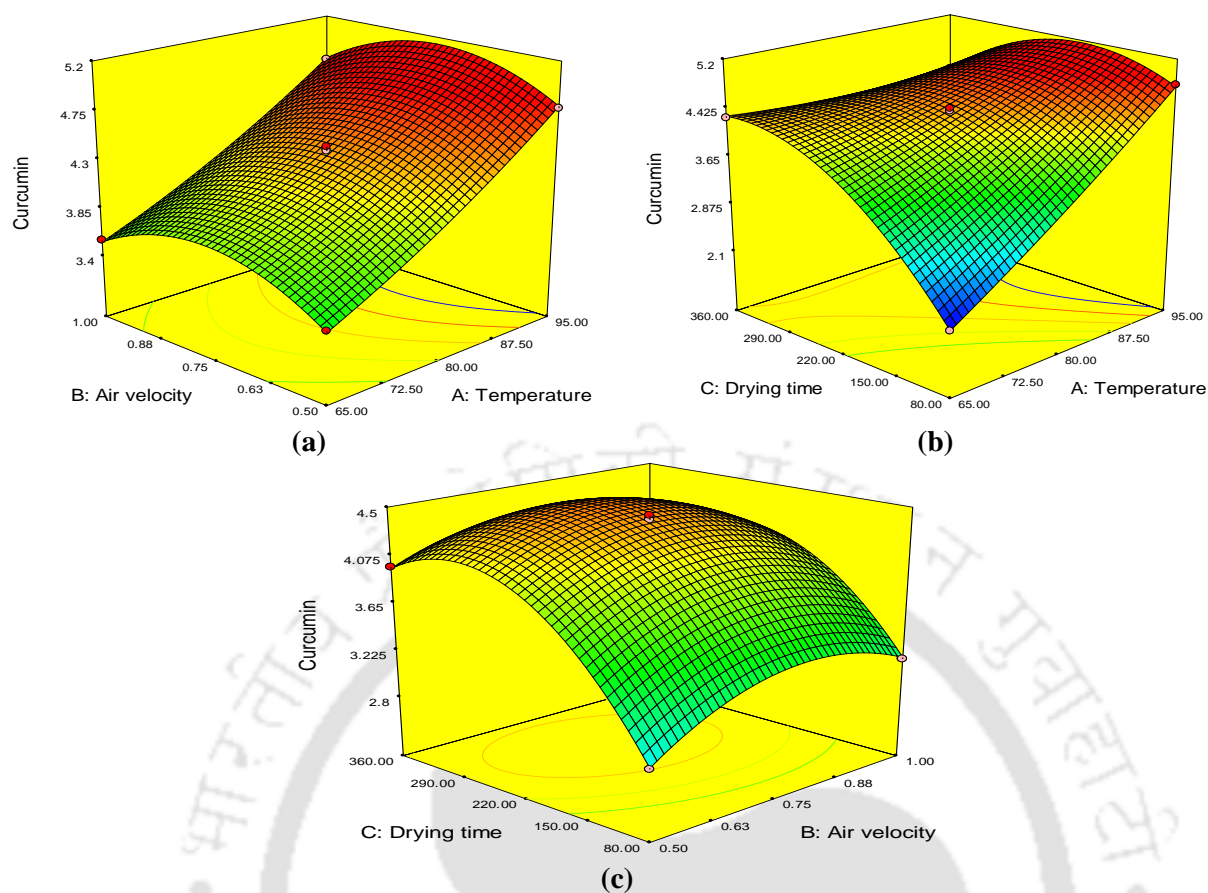


Figure 5.4: Response surface plot of curcumin content of refractance window dried turmeric samples and for various binary combinations (a) temperature and air velocity, (b) temperature and drying time and (c) air velocity and drying time.

Table 5.5: ANOVA data summary of best fit model to represent curcumin content of BBD-RSM based RWD of *Curcuma longa*.

| S. No. | | F-values | p-values | |
|--------|--------------------|----------|----------|-----------------|
| 1. | Model | 690.29 | <0.0001 | Significant |
| 2. | A-Temperature | 2481.62 | <0.0001 | |
| 3. | B-Air-velocity | 2.03 | 0.2139 | |
| 4. | C-Drying time | 992.41 | <0.0001 | |
| 5. | AB | 1.80 | 0.2373 | |
| 6. | AC | 1361.79 | <0.0001 | |
| 7. | BC | 46.84 | 0.0010 | |
| 8. | A ² | 18.84 | 0.0034 | |
| 9. | B ² | 327.35 | <0.0001 | |
| 10. | C ² | 1016.83 | <0.0001 | |
| 11. | Lack of Fit | 2.91 | 0.1813 | Not significant |
| 12. | R ² | 0.999 | | |
| 13. | Adj R ² | 0.998 | | |

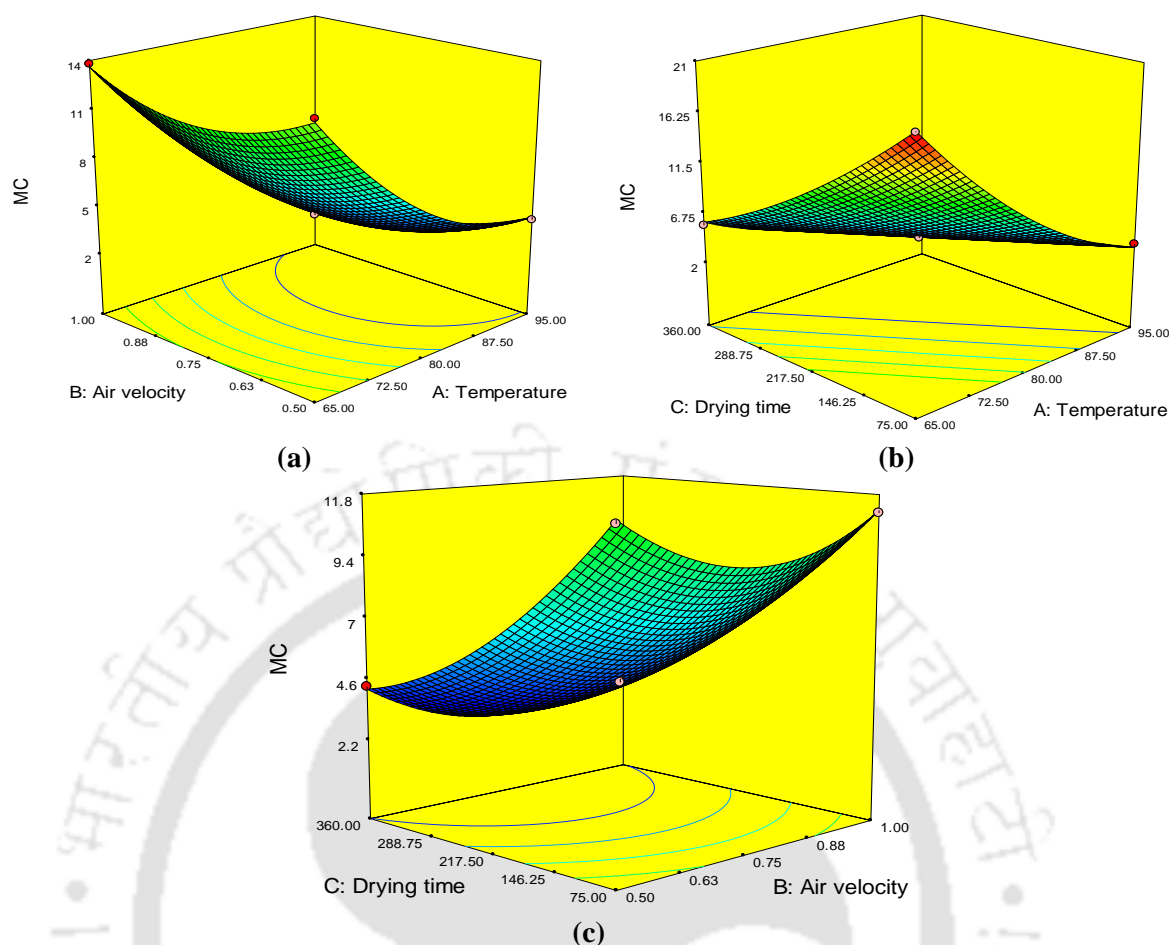


Figure 5.5: Response surface plot of moisture content of refractance window dried turmeric samples and for various binary combinations (a) temperature and air velocity, (b) temperature and drying time and (c) air velocity and drying time

Table 5.6: ANOVA data summary of best fit model to represent moisture content of BBD-RSM based RWD of *Curcuma longa*.

| S. No. | | F-values | p-values | |
|--------|--------------------|----------|----------|-----------------|
| 1. | Model | 425.36 | <0.0001 | Significant |
| 2. | A-Temperature | 1866.25 | <0.0001 | |
| 3. | B-Air-velocity | 2.05 | 0.2110 | |
| 4. | C-Drying time | 1089.67 | <0.0001 | |
| 5. | AB | 0.21 | 0.6626 | |
| 6. | AC | 505.30 | <0.0001 | |
| 7. | BC | 0.52 | 0.5044 | |
| 8. | A ² | 217.22 | <0.0001 | |
| 9. | B ² | 134.01 | <0.0001 | |
| 10. | C ² | 63.26 | 0.0005 | |
| 11. | Lack of Fit | 3.99 | 0.2067 | Not significant |
| 12. | R ² | 0.998 | | |
| 13. | Adj R ² | 0.996 | | |

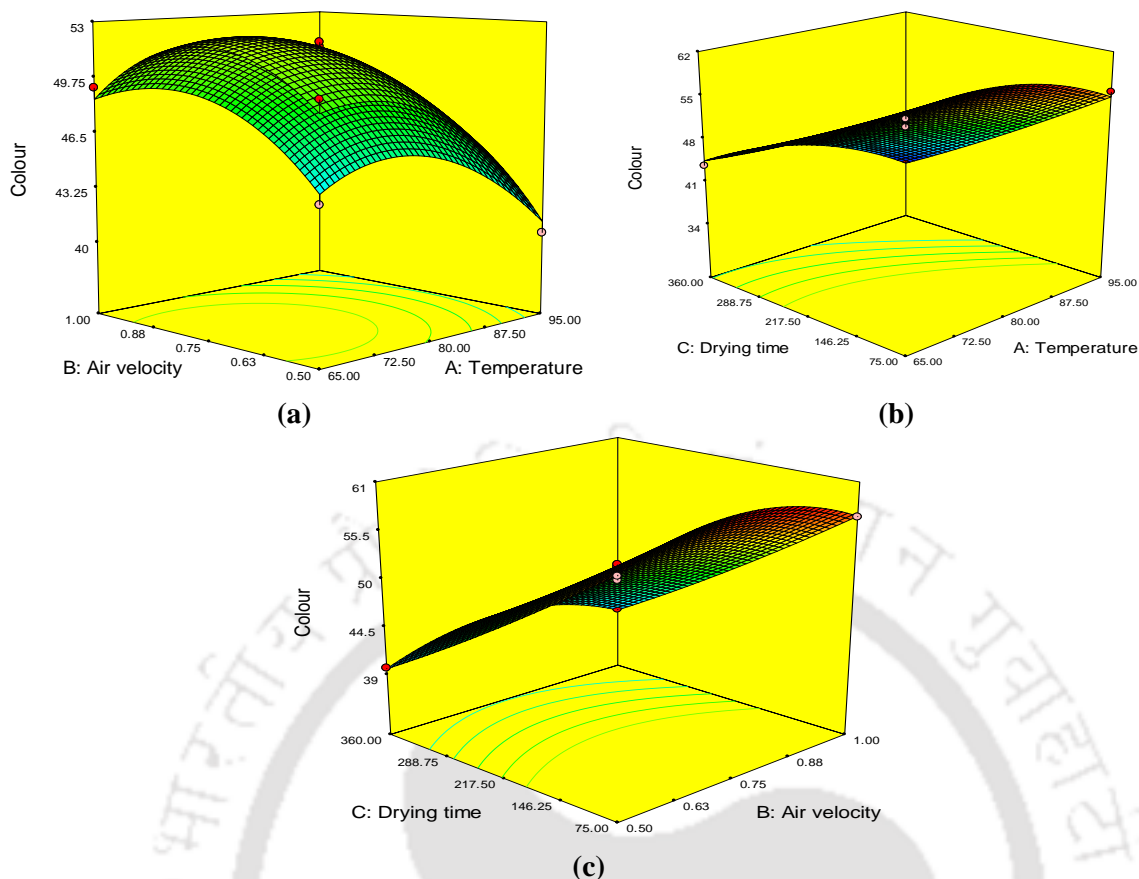


Figure 5.6: Response surface plot of colour indices of refractance window dried turmeric samples and for various binary combinations (a) temperature and air velocity, (b) temperature and drying time and (c) air velocity and drying time.

Table 5.7: ANOVA data summary of best fit model to represent colour indices of BBD-RSM based RWD of *Curcuma longa*.

| S. No. | | F-values | p-values | |
|--------|--------------------|----------|----------|-----------------|
| 1. | Model | 79.07 | <0.0001 | Significant |
| 2. | A-Temperature | 99.98 | 0.0002 | |
| 3. | B-Air-velocity | 0.36 | 0.0574 | |
| 4. | C-Drying time | 548.22 | <0.0001 | |
| 5. | AB | 0.00035 | 0.9547 | |
| 6. | AC | 4.02 | <0.1013 | |
| 7. | BC | 0.012 | 0.9166 | |
| 8. | A ² | 27.98 | 0.0032 | |
| 9. | B ² | 32.59 | 0.0023 | |
| 10. | C ² | 0.77 | 0.4205 | |
| 11. | Lack of Fit | 1.40 | 0.4424 | Not significant |
| 12. | R ² | 0.99 | | |
| 13. | Adj R ² | 0.98 | | |

5.12 Comparison of Experimental and Statistical Design based Optimal Data Set

Based on control studies and RSM based experimental and numerical optimization studies, the best data sets have been summarized in Table 5.8. Compared to the control study, much variation in the nutritional characteristics have not been obtained for both RSM based experimental and numerical optimization approaches. Thus, air-velocity can be concluded to be an insensitive independent variable to influence the optimal characteristics of batch RWD based turmeric samples. A reduced drying time was obtained in experimental and numerical optimization based drying time (75 min as opposed to 90 min) confirms that the control experiment parameters were chosen appropriately. In summary, RSM based experimental data sheet is sufficient to identify best data set for the RWD processed turmeric samples and further numerical optimization is not required.

5.13 Literature Comparison

The best obtained data in this work has been compared to the best literature reported data. These have been summarized in Table 5.9. The best experimental findings of the work refer to 95 °C, water bath temperature, 75 min drying time and 0.75 m/s air velocity. Corresponding values for the control cases are 89.10 % (AA), 189.76 mg GAE/g sample (TPC), 160.96 mg quercetin/g sample (TFC), 4.83 % w/w (CC), 4.10 % (MC) and 56.67 (*L* values) and for the experimental investigation 90 % (AA), 190 mg GAE/g sample (TPC), 160 mg quercetin/g sample (TFC), 4.84 % w/w (CC), 4 % (MC) and 55.70 (*L* values). Since no literature data is available for the optimality of turmeric slice sample, other relevant data have been considered namely CCD based vacuum drying of mushroom slice, (Sumic et al., 2016), BBD based convective drying of sweet potato flour (Ahmed et al., 2011) and CCD based drum drying of jackfruit (Pua et al., 2009). Since, the input and output parameter for all literature cases have been not relevant, they cannot be related to the finding of this work. However, the BBD based total phenolic content optimality for the case of sweet potato flour in Ahmed et al., (2011) affirmed

Table 5.8: Control, RSM based experimental and RSM based numerically optimized data summary of *Curcuma longa* samples.

| S. No. | | Optimized temp (°C) | Optimize d time (Min) | Air velocity (m/s) | AA (%) | TPC (mg GAE/g dry sample) | TFC (mg quercetin/g dry sample) | CC (% w/w) | MC % | Colour |
|--------|------------------------|---------------------|-----------------------|--------------------|--------|---------------------------|---------------------------------|------------|------|--------|
| 1. | Control | 95 | 90 | - | 89.10 | 189.76 | 160.96 | 4.83 | 4.10 | 56.67 |
| 2. | RSM based experimental | 95 | 75 | 0.75 | 90.00 | 190.00 | 160.00 | 4.84 | 4.00 | 55.70 |
| 3. | RSM based optimized | 95 | 75 | 0.76 | 90.52 | 188.22 | 158.65 | 4.80 | 3.67 | 54.87 |

Table 5.9: Literature and best results data summary for RSM design based RWD of *Curcuma longa*.

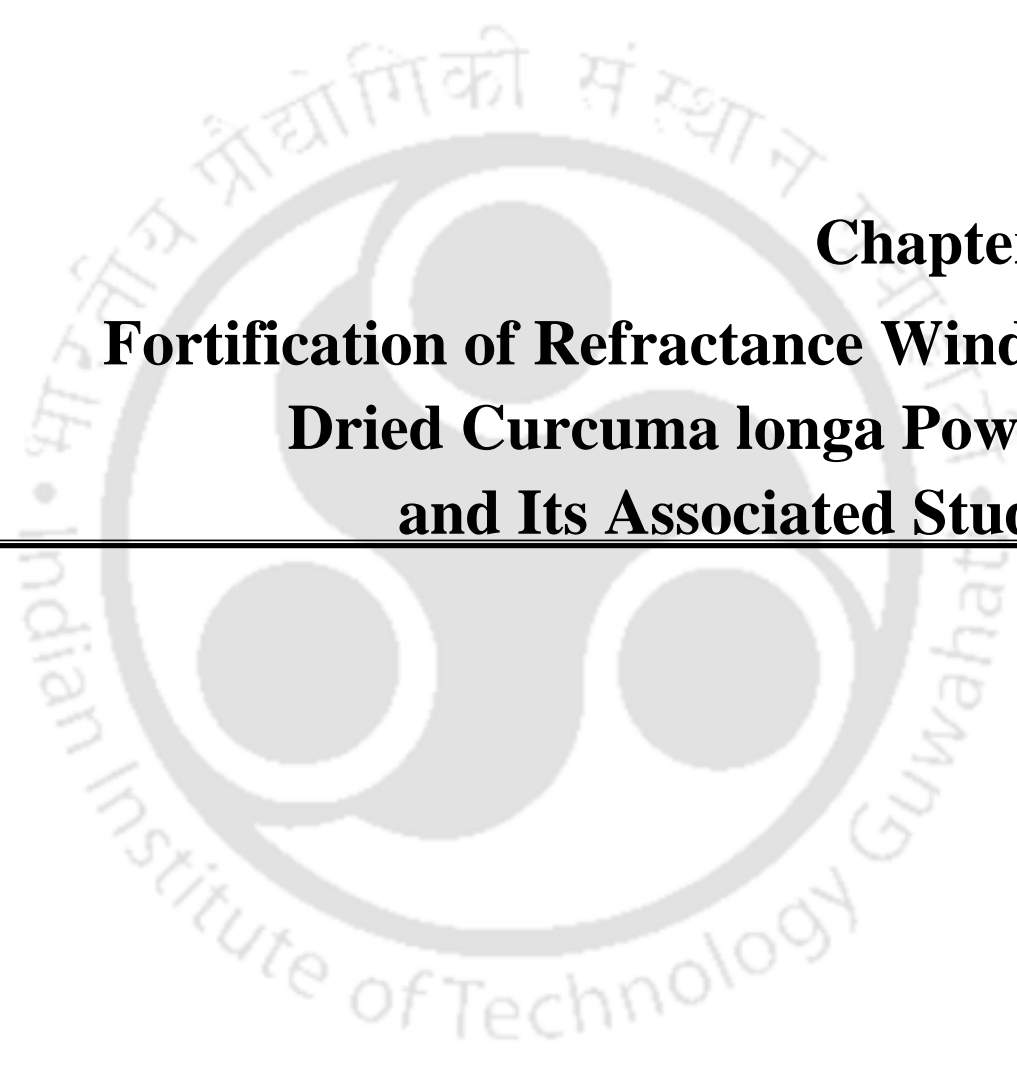
| S. No. | System | Drying Method | Parameters | Independent variables | Dependent variables | Best findings | References |
|--------|--------------------|-------------------|---|---|--|--|----------------------|
| 1. | Turmeric slices | RWD | 1 mm slice thickness | 65 – 95 °C 0.5 – 1 m ² /s 75 – 360 min | MC, AA, TPC, TFC, CC, colour | 4.84 % 90 %, 190 mg GAE/g dry sample, 160 mg quercetin/g dry sample 4.84 % w/w, 55.70 (L values) | This work |
| 2. | Mushroom slices | Vacuum | | 46 – 74 °C 20 – 580 mbar | Total solids 65- 90 % water activity 0.32 – 0.76 | 57.1 °C and 100 mbar 85.40 % total solids 0.615 a _w value | Sumic et al., (2016) |
| 3. | Sweet potato flour | Convection drying | | 55 – 65 °C 1 – 3 w/v citric acid 1 – 3 soaking time | Anthocyanin 15 – 40 mg/100g TPC 50 – 70 mg/g | For anthocyanin 62.91°C, 1.38%, 2.53 min For TPC 60.94°C, 1.04% and 2.24 min Anthocyanin 19.78 mg/100 g TPC 61.55 mg/g. | Ahmed et al., (2011) |
| 4. | Jackfruit | Drum drying | Drum clearance: 0.001 inch Pool level: 10 cm | Steam Pressure: 300-400 kPa Rotational speed: 1-3 rpm | 5.6 – 7.9 % moisture content 0.01 – 0.04 water activity | Steam pressure:336 kPa Rotation speed 1.2 rpm M.C: 6.767% Water activity:0.24 | Pua et al., (2009) |

60.94 °C drying temperature, 1.04 % citric acid w/v and 2.24 min soaking time with response as 61.55 mg. The optimal TPC altered as 50 – 70 mg/g. As a tuber, the optimal data of RWD of turmeric had marginally the same results for both experimental and numerical optimization studies.

5.14 Summary

Important conclusions can be deduced from the findings reported in this chapter. Firstly, the RWD nutritional characteristics of turmeric have been significantly influenced with water bath temperature and drying time but not with air-velocity. Secondly, higher temperature and shorter drying time increased the retention of nutritional characteristics. Thirdly, the most appropriate model for all evaluated characteristics is the quadratic model. Finally, the optimal data set for RSM was found to be 95 °C bath temperature, 75 min drying time and 0.76 m/s air-velocity for optimal response characteristics of 90.52 % (AA), 188.22 mg GAE/g dry sample (TPC), 158.65 mg quercetin/g dry sample (TFC), 4.80 % w/w (CC), 3.67 % (MC) and 54.87 *L* values (colour indices). In summary, RWD turmeric samples can be characterized with better retention of nutritional characteristics. Thereby, the experimental data based RW dried turmeric samples can be deployed to provide needful quality assurance in downstream fortified products such as golden milk and turmeric tablets.





Chapter 6:
Fortification of Refractance Window
Dried Curcuma longa Powder
and Its Associated Studies



Fortification of Refractance Window Dried *Curcuma longa* Powder and Its Associated Studies

In this chapter, findings related to the fortification and associated studies of the turmeric powder, folic acid fortified refractance window dried turmeric powder and NaFeEDTA fortified RW dried turmeric powder have been delineated. The section 6.1 provides relevant introduction to this chapter. In section 6.2, physical characteristics such as bulk density, swelling power, solubility, dispersion time, hygroscopicity, water binding capacity and colour indices have been discussed. Section 6.3 delineate on the statistical analysis of the results obtained in the previous section. Section 6.4 and 6.5 detail upon the FTIR and TGA analysis respectively. The section 6.6 elaborates on the characterization results achieved with the DSC. Following this, the XRD analysis results have been presented in section 6.7. The FESEM and particle size distribution characterization were discussed in sections 6.8 and 6.9 respectively. The in-vitro digestion findings have been presented in section 6.10. thereafter, section 6.11 details upon literature comparison followed by a chapter summary in section 6.12.

6.1 Introduction

In the previous chapter, the process parameters were optimized for the refractance window (RW) dried turmeric powder. This chapter details upon the characterization-based findings of RW dried turmeric powder and its comparison with folic acid fortified RW dried turmeric and sodium ferric ethylenediaminetetraacetate (NaFeEDTA) fortified turmeric powder.

The influence of fortificants on the mineral fortified dried products is often targeted through associated studies. Karn et al., (2018) fortified Nepalese curry powder with alternate iron compounds. Alam et al., (2010) fortified whole wheat flour with a fortificants premix of ferrous sulfate, ethylenediamine tetraacetic acid (EDTA) and folic acid (20:20:1.5 ppm). Modupe et al., (2019) fortified

salt with folic acid, iron and iodine. Rebellato et al., (2006) fortified whole wheat flour with various iron compounds. Berry et al., (2009) fortified flour with folic acid. Jahan et al., (2001) fortified chickpea seeds and flour using ferrous sulfate hepta-hydrate, ferrous sulfate mono-hydrate and NaFeEDTA fortificants. Similarly, Tripathi et al., (2012) deployed finger millet and sorghum flours as double fortifications vehicles with ferrous fumarate, zinc stearate and EDTA fortificants.

Till date, the parametric optimality of refractance window drying (RWD) process were targeted for vegetables such as carrot, onion etc. (Akinola et al., 2009; Hernandez Santos et al., 2016). These vegetables powders were not studied for fortification and for a comparison of associated characteristics. In such investigations, physical characteristics (solubility, swelling time, hygroscopicity, dispersion time, water binding capacity, bulk density and colour) are often target along with instrumental characteristics such as thermogravimetric analyzer (TGA), differential scanning calorimetry (DSC), field-emission scanning electron microscopy (FESEM), X-ray diffraction (XRD), fourier transform infrared (FTIR), and particle size distribution for the fortified and unfortified dried powder products. Till date no study has been devoted to the turmeric powder product system. Such investigations can provide useful insights to the role of fortification in altering these properties.

Considering the above cited lacunae, this chapter addresses the characterization of RW dried turmeric powder, folic acid fortified RW dried turmeric powder and NaFeEDTA fortified RW dried turmeric powder.

6.2 Physical Characteristics

6.2.1 Bulk Density

Bulk density is an important characteristic for storage, transportation and packaging of the powder products. Hence, it was assessed for various dried samples. Bulk density of the RWD processed turmeric powder were obtained as 0.62 g/mL. Incidentally, the turmeric powder fortified with folic acid and NaFeEDTA possessed bulk density in the range of 0.64 – 0.65 g/mL (Table 6.1). Thus, it can be

Table 6.1: Bulk density data of unfortified and fortified turmeric powder products.

| S. No. | Samples | Bulk density (g/ml) |
|--------|----------------------|---------------------|
| 1. | Unfortified | 0.62 |
| 2. | Folic acid fortified | 0.65 |
| 3. | NaFeEDTA fortified | 0.64 |

* All standard deviations were in the range 0.05 – 0.1.

stated that the density did not vary with the addition of fortificants, and the trends were comparable to those reported in the relevant prior art.

In a previous study, the bulk density of the yoghurt powders produced with freeze drying were analyzed to vary from 0.29 - 0.42 gm/L and these were dependent upon the initial solid content (Tontul et al., 2018). Comparatively, these values have been approximately one third of the values obtained for the RWD sample. Bulk density of the yoghurt powders produced by RW drying were analyzed to vary from 0.52 - 0.59 g/mL, respectively. The lower bulk density of the yoghurt powder sample produced with freeze drying could be possibly due to the porous nature of the sample. The RW dried sample possessed a lesser packed structural morphology. This is due to the grainy shape of the particles. The bulk density of the sample was high for the RWD sample.

Constant and falling rate drying mechanism occur sequentially during drying of food sample. Thus, during constant drying phase, higher drying temperature translates into higher initial drying rates. Thereafter, the drying rate gets controlled due to moisture diffusion from the internal portion of the sample to its surface. Deploying higher temperatures during drying can form a hard and moisture resistant crust at the surface that eventually prevents further loss of moisture. The formation of such a crust generally results in higher bulk density. In summary, the higher bulk density of the RW dried samples is possibly due to the formation of moisture resistant crust (Tontul et al., 2018).

6.2.2 Hygroscopicity

Hygroscopicity, like solubility, is a very important parameter of dehydrated products and has a definite role in influencing their shelf-life characteristics. Hygroscopicity is defined as the moisture absorption ability of a powder from an environment with high relative humidity. It is generally related

Table 6.2: Hygroscopicity data of unfortified and fortified turmeric powder products.

| S. No. | Sample | Hygroscopicity (%) |
|--------|----------------------|--------------------|
| 1. | Unfortified | 8.70 |
| 2. | Folic acid fortified | 8.80 |
| 3. | NaFeEDTA fortified | 8.50 |

* All standard deviations were in the range 0.1 – 0.3.

to the porosity of the powder or amorphous glassy state of the sugars that exist in the tested food sample. Lower hygroscopicity is desired to achieve chemical and microbiological stability in due course of the long-term storage of a food sample.

For RW processed turmeric, NaFeEDTA and folic acid fortified RW processed turmeric powder samples, lower hygroscopicity values have been obtained as 8.70 %, 8.80 % and 8.50 % respectively (Table 6.2). Hence, addition of fortificants did not significantly alter the hygroscopicity property of the turmeric powder sample. The low hygroscopicity could be due to the formation of dense structures that tend to reduce the intake of water into the cells. The formation of dense structures is due to the fast drying during RWD process (Minjares-Fuentes et al., 2016, Tontul et al., 2018) Also, lower hygroscopicity of turmeric is due to its lower sugar content. The fortificants namely folic acid and NaFeEDTA were also stable and had lower affinity to water vapour. Thus, the fortified turmeric samples powder also possessed lower hygroscopicity.

In the literature, similar results have been reported for RW and freeze dried yoghurt sample. The reported values were lower than those obtained for the food powders and could be due to the lower sugar content in the yoghurt (Tontul et al., 2018). For mango powder samples, no significant difference existed in the hygroscopicity of RW dried (18.0 ± 0.36 %) and freeze dried (18.0 ± 0.19 %) sample (Caparino et al., 2012). Hence, the efficacy of the RWD process was found to be equivalent to the freeze drying method for the evaluated parameter in case of mango powder.

6.2.3 Solubility and Swelling Power

Solubility is an important parameter for instant food products. Solubility has been the most reliable criterion for the assessment of a powder product behaviour in aqueous solution.

Table 6.3: Solubility and swelling power data of unfortified and fortified turmeric powder products.

| S. No. | Samples | Solubility (%) | Swelling power (g/g) |
|--------|----------------------|----------------|----------------------|
| 1. | Unfortified | 29.00 | 1.80 |
| 2. | Folic acid fortified | 30.00 | 2.00 |
| 3. | NaFeEDTA fortified | 28.00 | 1.90 |

* All standard deviations for solubility and swelling power were in the range 2 – 3 and 0.1 – 0.2 respectively.

This parameter is attained after the powder undergoes the sequential dissolution steps of sinkability, dispersibility and wettability (Caparino et al., 2012).

For RW dried turmeric, folic acid fortified RW dried turmeric and NaFeEDTA fortified RW dried turmeric, the solubility was about 29, 30 and 28 % respectively (Table 6.3). Thus, a good solubility was achieved and this is promising from product acceptability perspective (Chen et al., 2020). Corresponding swelling power values were 1.8, 2.0 and 1.9 g/g respectively (Table 6.3). It can be seen that the fortificants did not significantly alter the product solubility and swelling power. The literature confirmed that the solubility and swelling power of RW dried powders were similar to freeze dried powder sample and were lower than that of spray and drum dried powders. This is due to the mild processing temperature for both RW and freeze-drying methods (Caparino et al., 2012).

In the relevant prior art, the authors reported that solubility of mango puree for RWD and freeze drying were similar but lower than the values obtained for the samples produced with spray and drum dried processes. One possible reason for this is that the mango puree cell structure does not get disrupted during drying (Castoldi et al., 2015).

6.2.4 Dispersion Time

For all evaluated powders namely, RW processed turmeric, folic acid fortified and NaFeEDTA fortified RW dried turmeric powders, the dispersion time was lower than 20 s (Table 6.4). The relatively short times of powder dispersion confirm good wettability characteristics of the tested samples. The dispersion time gets detrimentally influenced with other parameters such as particle size and density. The literature hypothesized that larger particles possess higher wettability than finer particles. This is often confirmed with the ease to immerse a powdered mass in due course of pouring it on to water

Table 6.4: Dispersion time data of unfortified and fortified turmeric powder products.

| S. No. | Sample | Dispersion time (s) |
|--------|----------------------|---------------------|
| 1. | Unfortified | 20.00 |
| 2. | Folic acid fortified | 17.00 |
| 3. | NaFeEDTA fortified | 19.00 |

* All standard deviations were in the range 2 – 3.

surface. Due to greater surface area of larger but not finer particles, wettability of larger particles is higher and thereby translates into lower dispersion time (Castoldi et al., 2015).

Wettability is the ability of a powdered sample to penetrate into a liquid through the action of capillary forces. It is certainly influenced with composition, morphology, size, geometry and porosity of the powder particles. Lower dispersion time is often desired for a powder product (Seth et al., 2017).

In the present study, the average dispersion time of the powder samples obtained with the RWD altered between 17 – 20 s. The low dispersion time and hence better wettability of the powder samples produced by RW drying could be due to the higher density of the samples. Also, the addition of fortificants did not significantly alter the dispersion time of the samples.

Lower dispersion time values have been obtained for the RWD dried turmeric powder samples. The higher wettability of such samples could be also due to higher drying temperature. However, it can as well be inferred that the phenomenon of hard crust formation due to faster drying translate into the higher wettability of the samples (Tontul et al., 2018). In a related prior art of RW dried yogurt powder, the dispersion time of the RW dried yoghurt powder was 21.5 s which is significantly lower than the value obtained for freeze dried yogurt samples (71 s). This is due to the higher density of RW dried yogurt samples (Tontul et al., 2018).

Table 6.5: Water binding capacity data of unfortified and fortified turmeric powder products.

| S. No. | Sample | Water binding capacity (%) |
|--------|----------------------|----------------------------|
| 1. | Unfortified | 66.00 |
| 2. | Folic acid fortified | 65.00 |
| 3. | NaFeEDTA fortified | 67.00 |

* All standard deviations were in the range 2 – 3.

6.2.5 Water Binding Capacity

The water binding capacity is an important technical property and is related to the hydration capacity of the food samples that have rich constitution of protein and/or fiber content. Such products primary aim towards the regulation of the viscosity of the food product (Duarte-Correa et al., 2021). The water binding capacity of RW dried turmeric, folic acid and NaFeEDTA fortified RW dried turmeric samples were high and were 66, 65 and 67 % respectively (Table 6.5). Thus, the fortificants did not critically alter the water binding capacity of the turmeric samples. According to a relevant literature (Kuttigounder et al., 2011), higher drying temperature ensured higher water binding capacity of the dried samples. Hence, the RW dried turmeric sample possessed higher water binding capacity due to the drying at high temperature that eventually fostered the onset of pasting or gelatinization.

6.2.6 Colour Indices

As a major determinant of product quality, the food colour affects consumer preferences and is often used as an indicator to predict the chemical and quality changes due to thermal processing. From a consumer acceptance perspective, the colour is an important attribute of the dried product. Colour parameters are often represented by L , a and b values. Thus, any alteration in a and b values during drying is always accompanied with a simultaneous change in the L value (El-Safy, 2014). Hence colour measurement parameters (L , a and b) can be used to evaluate upon the pertinent influence of drying temperature and fortification on the colour quality of turmeric powder.

The colour parameters L , a , and b of turmeric powder, folic acid fortified and NaFeEDTA fortified RW dried turmeric have been summarized in Table 6.6. These have also been compared with the L , a , and b values of fresh turmeric. It can be observed that the L , a , and b values of RWD dried turmeric powder did change significantly with respect to corresponding values of the fresh turmeric sample. The values for L , a , and b were all positive and thereby respectively confirmed on the lightness, redness and yellowness for both fresh and dried turmeric samples.

The L parameter reduced from 63.67 (fresh sample) to 56.67, 55.70 and 56.10 for turmeric powder, folic acid fortified and NaFeEDTA fortified RW dried turmeric samples. Such a reduction in

Table 6.6: *L*, *a*, and *b* values of unfortified and fortified RW dried turmeric powder samples.

| S. No. | Samples | <i>L</i> | <i>a</i> | <i>b</i> |
|--------|----------------------|----------|----------|----------|
| 1. | Fresh | 63.67 | 43.07 | 75.12 |
| 2. | Unfortified | 56.67 | 31.05 | 62.13 |
| 3. | Folic acid fortified | 55.70 | 30.10 | 61.80 |
| 4. | NaFeEDTA fortified | 56.10 | 30.80 | 61.20 |

* All standard deviations for *L*, *a* and *b* were in the range of 1 – 2, 2 – 3 and 1 – 3 respectively.

lightness has been attributed to the surface dryness or loss of moisture due to the drying at 95 °C. The measurement trends also confirmed upon the browning of the sample. Another reason is that the non-enzymatic browning (or Maillard reaction) occurs at relatively high drying temperatures. The reduction in *a* (redness) and *b* (yellowness) to 31.05 – 30.80 and 62.13 – 61.20 from 43.07 and 75.12 respectively also corroborates with the reasoning associated to heat treatment (Pradeep et al., 2016). Also, it can be observed from the table that the addition of fortificants did not alter the colour of the samples. In other words, the added fortificants did not alter the high stability characteristics of the dried sample and did not foster chemical reactions associated to the sample degradation.

6.3 Statistical Assessment of Physical Properties

A statistical analysis was carried out using one way analysis of variance (ANOVA) and the model Tukey was used for mean comparison. From the Table 6.7 (a – g) and for all cases of physical characterizations, high F values were obtained. The ANOVA based statistical analysis indicated a low p value for all cases. Hence, it can be inferred that the experimental values did not vary significantly and thereby confirmed reasonable acceptability of the values. Thereby, the average values reported in the chapter do instil good confidence levels in the obtained data.

Table 6.7: Statistical analysis data of dried turmeric products (**a**) solubility, (**b**) swelling power, (**c**) hygroscopicity, (**d**) dispersion time, (**e**) water binding capacity, (**f**) bulk density and (**g**) colour indices.

(a)

| S. No. | | DF | Sum of Squares | Mean Square | F Value | Prob>F |
|--------|-------|----|----------------|-------------|---------|---------|
| 1. | Model | 2 | 18.66 | 9.33 | 1.68 | 0.02634 |
| 2. | Error | 6 | 33.33 | 5.55 | | |
| 3. | Total | 8 | 52 | | | |

(b)

| S. No. | | DF | Sum of Squares | Mean Square | F Value | Prob>F |
|--------|-------|----|----------------|-------------|---------|--------|
| 1. | Model | 2 | 0.175 | 0.087 | 2.54 | 0.0158 |
| 2. | Error | 6 | 0.206 | 0.034 | | |
| 3. | Total | 8 | 0.382 | | | |

(c)

| S. No. | | DF | Sum of Squares | Mean Square | F Value | Prob>F |
|--------|-------|----|----------------|-------------|---------|---------|
| 1. | Model | 2 | 0.086 | 0.043 | 1.5 | 0.02963 |
| 2. | Error | 6 | 0.173 | 0.028 | | |
| 3. | Total | 8 | 0.26 | | | |

(d)

| S. No. | | DF | Sum of Squares | Mean Square | F Value | Prob>F |
|--------|-------|----|----------------|-------------|---------|---------|
| 1. | Model | 2 | 6.00 | 3 | 1.26 | 0.05120 |
| 2. | Error | 6 | 24.00 | 4 | | |
| 3. | Total | 8 | 30.00 | | | |

(e)

| S. No. | | DF | Sum of Squares | Mean Square | F Value | Prob>F |
|--------|-------|----|----------------|-------------|---------|---------|
| 1. | Model | 2 | 7.12 | 3.56 | 1.32 | 0.06183 |
| 2. | Error | 6 | 25.18 | 4.68 | | |
| 3. | Total | 8 | 32.17 | | | |

(f)

| S. No. | | DF | Sum of Squares | Mean Square | F Value | Prob>F |
|--------|-------|----|----------------|-------------|---------|--------|
| 1. | Model | 2 | 0.00142 | 0.00071 | 2.909 | 0.0131 |
| 2. | Error | 6 | 0.00147 | 0.00024 | | |
| 3. | Total | 8 | 0.00289 | | | |

(g)

| S. No. | | DF | Sum of Squares | Mean Square | F Value | Prob>F |
|--------|-------|----|----------------|-------------|---------|--------|
| 1. | Model | 2 | 4.046 | 2.023 | 0.681 | 0.541 |
| 2. | Error | 6 | 17.823 | 2.970 | | |
| 3. | Total | 8 | 21.869 | | | |

6.4 Fourier Transform Infrared Analysis

The Fourier Transform Infrared (FTIR) analysis was conducted for RW dried, folic acid fortified and NaFeEDTA fortified RW dried turmeric powder samples (Fig. 6.1). Such studies will enable useful insights into pertinent functional groups and the influence of fortification on the group shifts. The obtained FTIR spectra of RW dried turmeric matched with that reported previously in earlier investigation (Safie et al., 2015). In the RW dried turmeric powder samples' FTIR spectra, the broad band obtained at 3424 cm^{-1} was because of the stretching vibration of the free hydroxyl-group of phenol ($-\text{OH}$). The band at 2933 cm^{-1} was due to the $\text{sp}^2\text{ C-H}$ bond stretching. The combination of carbonyl bond (C=O) with two aromatic rings was seen at 1622 cm^{-1} . The bands at 1378 and 1027 cm^{-1} were due to the presence of curcumin component (C-O stretch of phenyl alkyl ether). The band at 600 cm^{-1} corresponded to ortho (1,2) and O-CH_3 bond at the aromatic ring.

In the folic acid fortified RW dried turmeric powder sample spectra, the broad band obtained at 3421 cm^{-1} was due to the stretching vibration of the free hydroxyl-group of phenol ($-\text{OH}$). Similar assignment was for the band at 3427 cm^{-1} in the spectra of NaFeEDTA fortified RW dried turmeric powder. The sharp band seen at 2926 cm^{-1} and 2934 cm^{-1} for folic acid fortified RW dried turmeric powder sample and NaFeEDTA fortified RW dried turmeric powder sample respectively was because of the $\text{sp}^2\text{ C-H}$ bond stretching. At 1644 and 1635 cm^{-1} , the conjugation of carbonyl bond (C=O) with two aromatic rings was evident for the folic acid fortified RW dried turmeric powder sample and NaFeEDTA fortified RW dried turmeric powder sample respectively.

Also, the bands for folic acid fortified RW dried turmeric powder sample at 1386 and 1381 cm^{-1} respectively and 1066 and 1107 cm^{-1} for the NaFeEDTA fortified RW dried turmeric powder sample respectively were due to the C-O stretch of phenyl alkyl ether. This assignment confirmed the presence

of curcumin component in the folic acid fortified and NaFeEDTA fortified RW dried turmeric samples. For folic acid fortified RW dried turmeric powder sample and NaFeEDTA fortified RW dried turmeric powder sample, the band at 608 cm^{-1} and 622 cm^{-1} respectively was due to ortho (1,2) and O-CH₃ bond at the aromatic ring. In the folic acid fortified turmeric powder sample spectra, a minor shift was apparent. Similar trends also existed for the NaFeEDTA fortified turmeric sample. These observations infer upon the stable nature of the added fortificants, and their minimal interaction with the dried turmeric powder and without any chemical reaction. Thus, with no chemical reaction, the turmeric powder did not undergo any denaturation and this has been evident in the minimal shift in the wavelength numbers of the functional groups. Thus, in summary, the FTIR spectral bands affirmed similar bands and with minimal peak shifts.

The FTIR spectra have been comparable with the reported FTIR spectra for the curcumin samples (Safie et al., 2015). Also, a relevant prior art addresses FTIR analysis of curcumin in the freeze dried, hot-air dried and sun-dried turmeric samples (Chumroenphat et al., 2021). The peaks obtained in this prior art have been in good agreement with those obtained in this work. However, marginal peak shifts did occur and this is due to the combined effect of alterations in the drying process and fortifications.

6.5 Thermogravimetric Analysis

Thermogravimetric analyzer (TGA) techniques continuously measure the mass of a sample being subjected to either heating or cooling at a controlled rate or being kept at a particular temperature for a period of time. Thermogravimetry has been useful to monitor processes that involve mass change in a food material due to drying, liberation of gases and absorption of moisture. To mimic various types of processing and storage conditions being provided to a food sample, TGA measurements are carried out under a specific environment (such as controlled pressures or atmospheres). Depending on the specific physicochemical processes that occur during the controlled thermal effect, the sample mass may either increase or decrease with temperature or time. Due to evaporation of volatile components and various chemical reactions that liberate gases, heating often leads to a reduction in mass. The ability

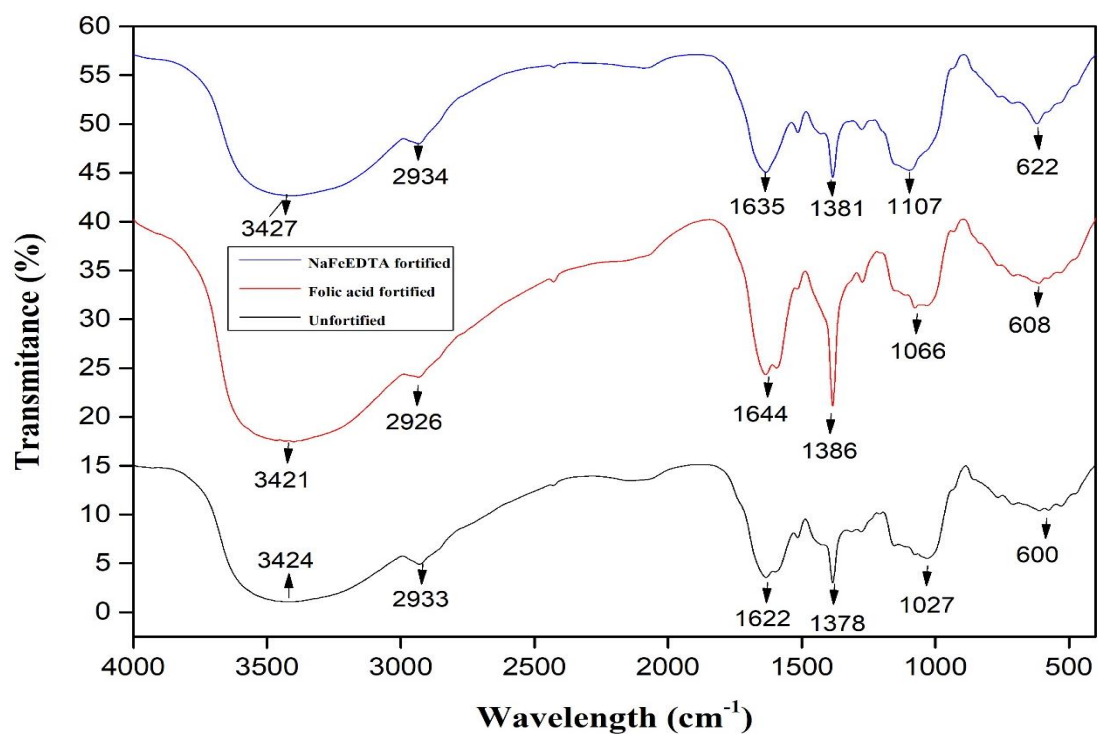


Figure 6.1: FTIR spectra of unfortified and fortified turmeric powder product samples.

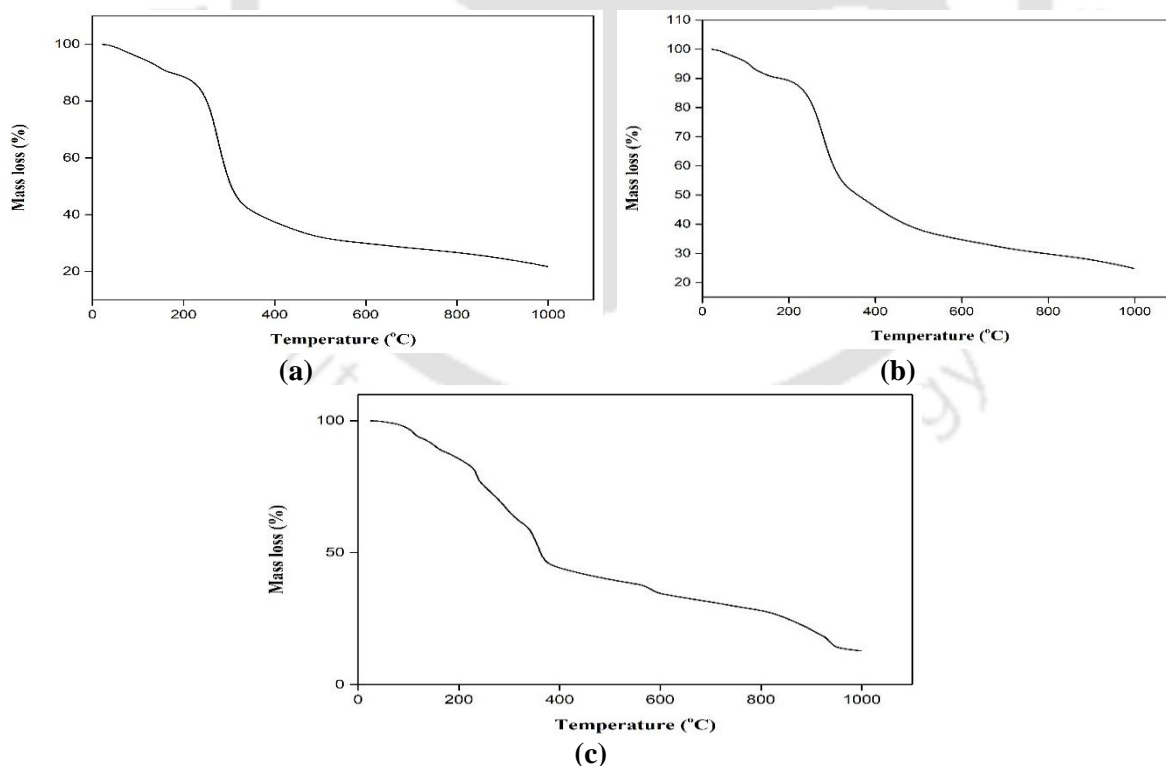


Figure 6.2: Mass loss vs temperature plots for (a) refractance window (RW) dried, (b) folic acid fortified RW dried and (c) NaFeEDTA fortified RW dried turmeric powder samples.

to carefully control the temperature, pressure and composition of the gasses surrounding a sample is extremely valuable for food scientists, as it allows them to model processes such as drying, cooking and uptake of moisture during storage. Through the TGA, the temperature changes of processes can be set to gain mentioned insights in the food product characteristics.

Fig. 6.2 (a – c) depict the TGA mass loss curve as a function of temperature. The initial temperature of the mass loss was approximately 192.93 °C for the RW dried turmeric sample, 196.42 °C for folic acid fortified RW dried turmeric sample and 207.10 and 567.48 °C for the NaFeEDTA fortified RW dried turmeric sample. Thus, below these temperature values, the respective samples did not undergo degradation and corresponding mass losses in the thermogram were due to moisture loss. For all cases, the thermal decomposition of curcumin occurred below 800 °C.

Both turmeric and folic acid fortified turmeric sample exhibited a single stage decomposition. However, the addition of NaFeEDTA led to a multi stage (two) decomposition in the NaFeEDTA fortified turmeric sample (mass loss at 207.10 °C and 567.48 °C). Among these, the latter is due to the presence of NaFeEDTA in the sample. For all cases, 100 % mass loss was observed and this confirmed complete decomposition of turmeric and curcumin at the elevated temperature (Delgado et al., 2016).

6.6 Differential Scanning Calorimetry

Differential Scanning Calorimetry (DSC) involves a thermal analysis for the evaluation of the variant physical properties of a sample with temperature and time. In other words, the measuring equipment attempts to quantify temperature and heat dependent material transformations. In due course of temperature alteration, the DSC allows to measure heat quantity being excessively radiated or absorbed by the sample due to temperature difference between the sample and reference material. Thereby, DSC serves as a very powerful technique to evaluate material properties such as glass transition temperature, melting, crystallization, specific heat capacity, oxidation behaviour, and thermal stability.

Fig. 6.3 (a – c) depict the thermograms obtained from the DSC analysis of RW dried turmeric powder, folic acid fortified and NaFeEDTA fortified RW dried turmeric samples. For the RW dried

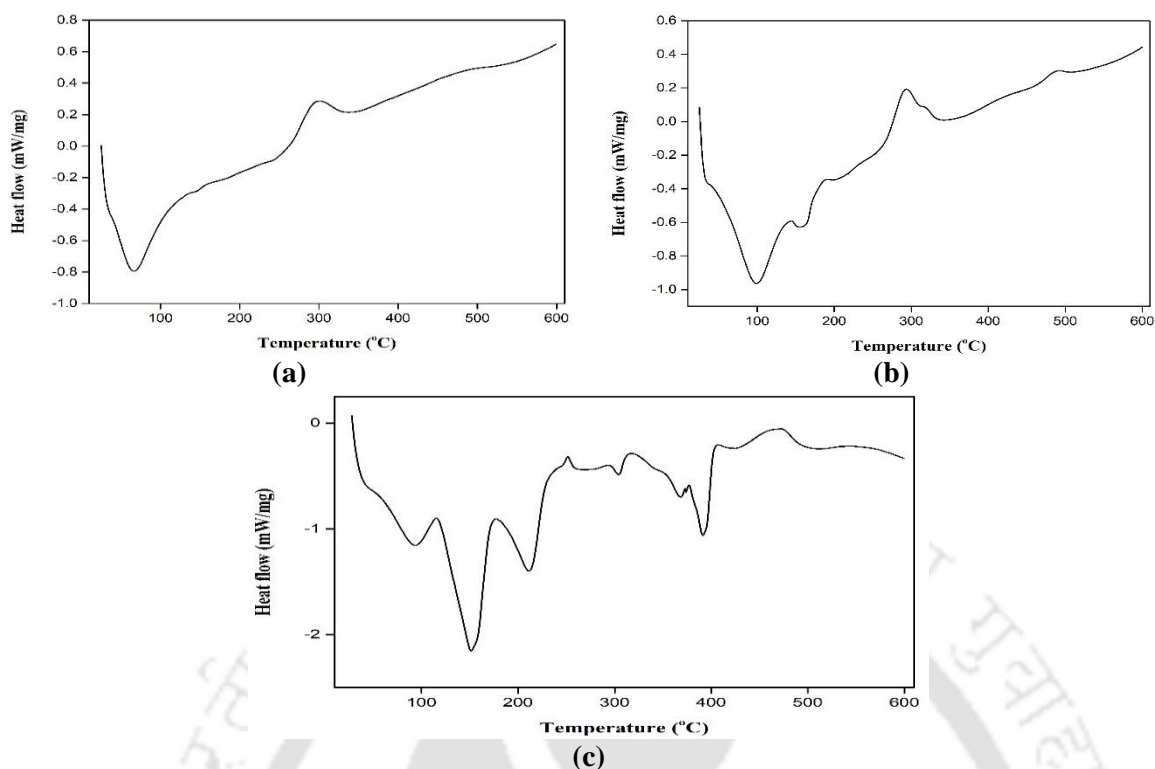


Figure 6.3: Heat flow vs temperature plots for (a) refractance window (RW) dried, (b) folic acid fortified RW dried and (c) NaFeEDTA fortified RW dried turmeric powder samples.

turmeric powder sample, the endothermic peak and exothermic peak were observed at 66.67 °C and 300.65 °C respectively. For the case of folic acid fortified turmeric powder sample, the endothermic peaks can be observed at 99.15 and 157.46 °C. Corresponding exothermic peaks were at 294.01 and 490.00 °C. For the NaFeEDTA fortified turmeric powder sample, the endothermic peaks could be observed at 93.26, 151.02, 211.37, 304.29, 368.49 and 391.44 °C and exothermic peaks at 251.12 and 475.41 °C.

The corresponding thermograms depicted in Fig. 6.3 (a – c) infer that the glass transition temperature (T_g) was 66.67, 99.15 and 93.26 °C for RW dried turmeric powder, folic acid fortified and NaFeEDTA fortified RW dried turmeric powder samples respectively. Pawar et al., (2012) reported a T_g value of 69.4 °C for the amorphous turmeric sample. Thus, the DSC of RW dried turmeric powder sample was similar to that obtained by Pawar et al., (2012), However, the T_g altered for folic acid fortified RW dried turmeric and NaFeEDTA fortified RW dried turmeric powder samples in comparison to RW dried turmeric powder. This is due to the reason that the T_g value of RW dried turmeric powders

was given for a single homogenous compound and the Tg value obtained for folic acid fortified RW dried turmeric powder and NaFeEDTA fortified RW dried turmeric powder samples were for a heterogenous mixture. The Tg value for folic acid and NaFeEDTA is 155 and 100.20 °C respectively (Neves et al., 2019, Branton and Jana, 2017). The Tg value of the sample in the literature altered due to the addition of different compounds (Jadhav et al., 2009). Therefore, the Tg altered for folic acid fortified RW dried turmeric and NaFeEDTA fortified RW dried turmeric powder samples due to addition of folic acid and NaFeEDTA powder. In a relevant prior art (Delgado et al., 2016), an endothermic peak was observed at 178.07 °C for turmeric extract coated with maltodextrin. This referred to a desolvation or loss of volatiles. Laczkowski and Sousdaleff, (2013) also obtained endothermic peaks at variant temperatures for the turmeric sample encapsulated with maltodextrin.

6.7 X-Ray Diffraction

As a non-destructive analysing method, the X-ray diffraction (XRD) is often deployed to confirm upon the crystalline–amorphous state of dried powdered products. In general, crystalline material exhibits a series of sharp peaks. These peaks are in contrast to the broad background pattern obtained for the amorphous products (Minjares-Fuentes et al., 2017).

The X-ray diffraction patterns of RW dried turmeric powder, folic acid fortified and NaFeEDTA fortified RW dried turmeric powder samples have been illustrated respectively in Fig. 6.4 (a – c). In Fig. 6.4 a, sharp peaks do not exist for the RW dried turmeric powder sample. However, peaks have been obtained at 17.14, 19 and 22° with intensities of 5215, 4844 and 5079 count per second respectively. The peak with maximum intensity has been obtained at 17.14°. Similarly, the XRD of folic acid fortified RW dried turmeric powder sample (Fig. 6.4 b) indicated weak intensities. For the sample, at 11, 13.2 and 17°, the peaks intensity values were 5136, 4901 and 5850 count per second respectively. Highest intensity has been at 17° and with 5850 count per count. For both cases, the maximum intensity was obtained at 17.14°. These observations are in agreement with those inferred in a relevant prior art (Kuttigounder et al., 2011). The reported XRD referred to a maximum intensity of 5312 cycles per second at 17°.

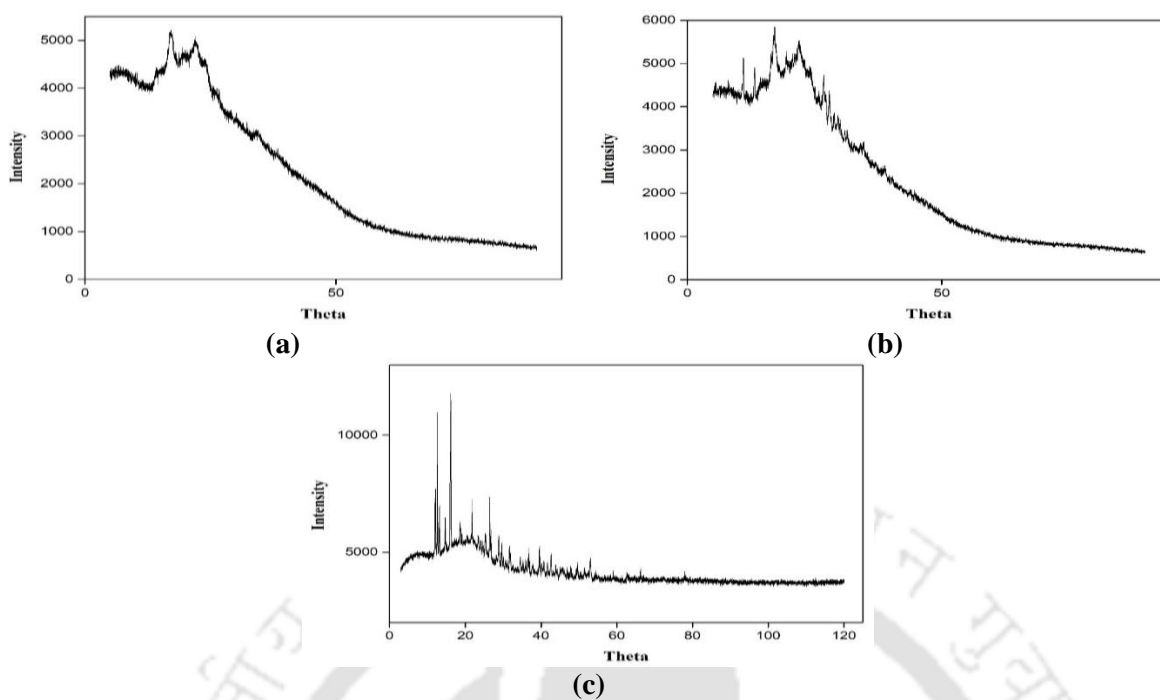


Figure 6.4: XRD spectral diagrams of (a) refractance window (RW) dried, (b) folic acid fortified RW dried and (c) NaFeEDTA fortified RW dried turmeric powder samples.

On the other hand, the X-ray diffraction pattern (Fig. 6.4 c) of NaFeEDTA fortified RW dried turmeric powder varied significantly in comparison to the XRD patterns of RW dried turmeric and folic acid fortified RW dried turmeric powder samples. For the case, sharp peaks at 12.64, 16.14, 21.72 and 26.44° with highest intensities of 10984, 11786, 7243, and 7355 count per second have been observed. The peak with maximum intensity has been achieved at 16.14°. Similar findings have been reported for ferrous sulphate at 18.3° (Branton and Jana, 2017).

The RW dried turmeric and folic acid fortified RW dried turmeric powder samples clearly exhibited amorphous characteristics and did not confirm upon any crystalline peaks. However, the NaFeEDTA fortified turmeric powder sample illustrated sharp peaks and hence its crystalline nature. During RWD, rapid drying of turmeric slices took place and thereby fostered the production of amorphous metastable compounds in dried products. Thus, it can be hypothesized that for crystallization, the time has not been sufficient. However, for the NaFeEDTA fortified RW dried turmeric case, the inorganic NaFeEDTA addition in the crystal form did contribute to the crystalline

state of the product. This was not the case for the folic acid fortified turmeric powder sample due to the amorphous nature of the folic acid. The X-ray diffraction patterns of RW dried turmeric powder and folic acid fortified RW dried turmeric powder samples exhibit a series of thick and intense lines and these are indicative of their amorphous structures. On the contrary, NaFeEDTA fortified turmeric powder sample exhibited a series of thin and intense lines, and hence its crystalline structure. These findings are also in agreement with the inferences deduced in a relevant prior art (Minjares-Fuentes et al., 2017, Caparino et al., 2012).

6.8 Field-Emission Scanning Electron Microscopy

The field-emission scanning electron microscopy (FESEM) has been considered to be an emerging technique for the assessment of surface morphology characteristics of powder samples and their elemental composition. With the advantage of accelerated voltage integration with the energy-dispersive spectroscopy (EDS), the method has a definite advantage to analyse smaller dimensions of the sample. In contrast to other techniques, FESEM offers high quality, low-voltage images with relatively minimal electrical charging of the sample (Sasikumar et al., 2020). Thereby, morphological investigation can infer on the possible structural changes due to the addition of folic acid and NaFeEDTA in the RW dried turmeric powder.

Fig. 6.5 (a) depicts the structural morphology of RW dried turmeric powder. Fig. 6.5 (b – c) illustrate the structural morphology of folic acid fortified RW dried turmeric and NaFeEDTA fortified RW dried turmeric powder samples. In this regard, it shall be noted that the RWD process does influence various physical properties such as texture, size, shape, and apparent density of the powder samples (Lakshmi et al., 2018). The RWD processed samples appeared rough. This may be due to the grinding process. Further, the roughness and non-homogeneity of RW dried turmeric, folic acid fortified and NaFeEDTA fortified RW dried turmeric powder samples have been also attributed to the partial starch gelatinization and consequent possible retrogradation during the RWD. In this regard, it shall be noted that the RW dried powder samples constitute starch along with other constituents such as protein, crude fiber and fat. While these can be inferred, they could not be ascertained (Kuttigounder et al., 2011).

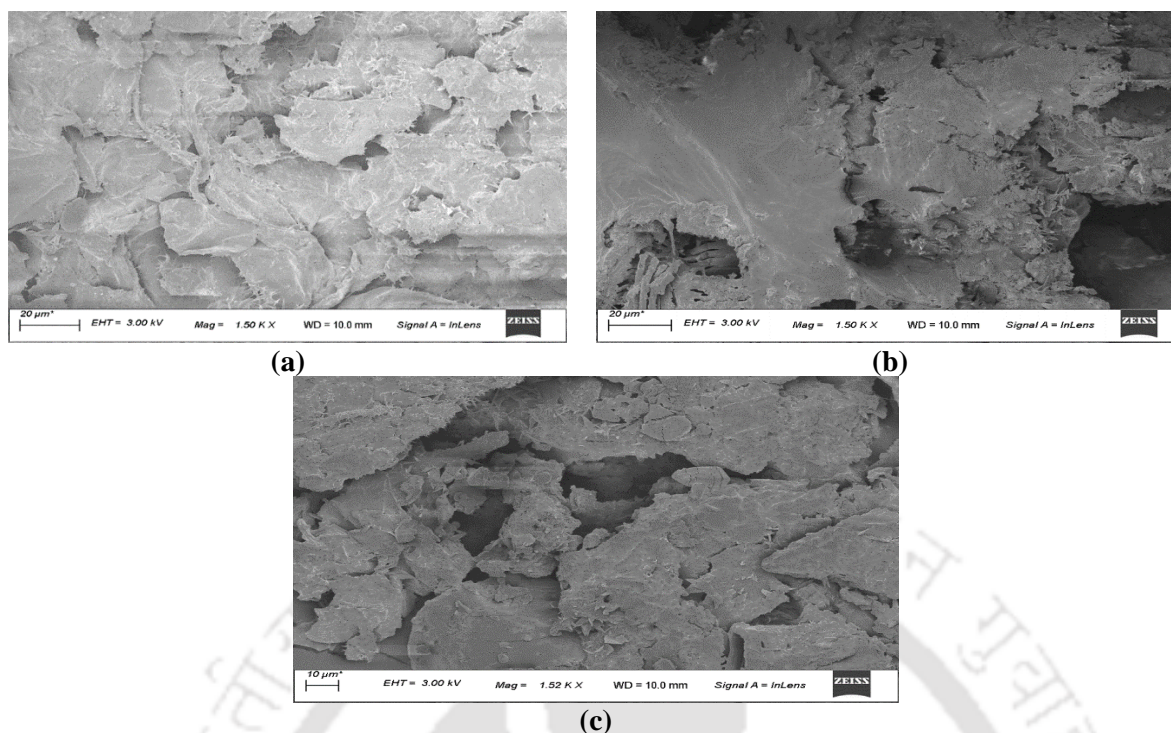


Figure 6.5: Scanning electron microscope image of (a) RW dried, (b) folic acid fortified RW dried and (c) NaFeEDTA fortified RW dried turmeric powder products.

The porous particle surface being observed in all sample morphologies has been due to the melting of starch granules in due course of the disruption in the protein-starch matrix. The findings were in good agreement with those inferred in a relevant prior art (Lakshmi et al., 2018). The obtained microstructure was similar to the morphology of turmeric powder reported in a recent study with respect to the biorefinery of the turmeric waste (Sharma et al., 2021). From the depicted images, it can be concluded that the addition of folic acid and NaFeEDTA did not alter the morphology of the RW dried turmeric powder. This is due to non-degradation of the sample after fortification. To a certain extent, smoother structure has been observed in folic acid fortified RW dried turmeric and NaFeEDTA fortified RW dried turmeric powder samples. Such structures did not adhere with the turmeric powder system. The smooth structure of folic acid could not be significantly distinguished from that of the turmeric powder. This is due to the amorphous nature of both folic acid and turmeric powder system. While the NaFeEDTA powder could be distinguished from the turmeric powder in terms of morphology, the same has been ascertained to the crystalline nature of the NaFeEDTA compound.

6.9 Particle Size Distribution, Polydispersity Index and Zeta Potential of Turmeric Powder Products

Particle size distribution studies enable the determination of the size, frequency and size range of particles in samples such as granulate, suspension or emulsion. Also, polydispersity index (PDI) can be used as a measure of the broadness of molecular weight distribution i.e. measure of the heterogeneity of a sample based on size. The larger the polydispersity index of a sample, the broader is its molecular weight. Zeta potential is the charge that exists at the interface of a solid surface and a liquid medium.

Dynamic light scattering technique was deployed to determine the particle size distributions, polydispersity index and zeta potential of RW dried turmeric, folic acid fortified RW dried turmeric and NaFeEDTA fortified RW dried turmeric powder samples. Table 6.8 summarizes the mean particle size, polydispersity index and zeta potential of RW dried turmeric, folic acid fortified RW dried turmeric and NaFeEDTA fortified RW dried turmeric powder samples.

The average mean diameter of RW dried turmeric, folic acid fortified RW dried turmeric and NaFeEDTA fortified RW dried turmeric powder samples were about 1599, 1560 and 1543 nm respectively. This conveys that the particle size of the samples has been in the micro range. Furthermore, corresponding polydispersity index values were 0.29, 0.27 and 0.25. These values affirmed excellent particle distribution and homogenous particle mean diameters. Thereby, the values confirmed that the fine powders can be explored for their significantly higher water-binding characteristic and foster higher quality of the products.

It is well known that the smaller particle size distribution translates into higher stability of the particles. This is due to Brownian motion (Mason et al., 2006). All samples possessed a polydispersity index value lower than 0.40 and thereby confirmed upon the pertinent narrow size distribution (Hasanvand et al., 2015). The addition of folic acid and NaFeEDTA did not significantly alter physical characteristics of the turmeric powder. Hence, the particle size distribution and polydispersity index of folic acid fortified RW dried turmeric and NaFeEDTA fortified RW dried turmeric powder were almost similar to that of the RW dried turmeric powder. Moreover, these fine samples can be used as a nutritional constituent for therapeutic purposes. Hence, the fine powder samples can be considered as a

Table 6.8: Particle size, polydispersity index and zeta potential data summary for unfortified and fortified refractance window dried turmeric powder samples.

| S. No. | Sample | Particle size (nm) | PDI | Zeta potential |
|--------|----------------------|--------------------|------|----------------|
| 1. | Unfortified | 1599 | 0.29 | -24.50 |
| 2. | Folic acid fortified | 1560 | 0.27 | -27.50 |
| 3. | NaFeEDTA fortified | 1543 | 0.25 | -25.60 |

* All standard deviations for particle size, PDI and zeta potential were in the range of 18 – 35, 0.05 – 0.02 and 1.2 – 2.1 respectively.

key ingredient in functional food (El-Sayed et al., 2020). The corresponding zeta potential values were -24.50, -27.50 and -25.60. The zeta potential for all the samples were negative. High absolute values of zeta potential usually refer to higher repulsion force of particles and emulsion stability. However, the obtained zeta potential values have been lower and negative in nature. Thereby, the samples have been concluded to precipitate after mixing (Hasanvand et al., 2015).

6.10 In-vitro Digestion based for the Bio-accessibility of Curcumin, Folic Acid and NaFeEDTA in Turmeric Powder Products

Food digestion is a complex process with many involved factors. During human digestion, ingested foods are transformed into necessary nutrients for the sustainable existence of human being through cellular energy, growth and cell repair mechanism. Food digestion implies two main processes that occur simultaneously: (i) mechanical transformation, whereby larger pieces of food get broken down into smaller pieces and (ii) enzymatic transformation, whereby several different enzymes break down macromolecules into smaller molecules that can be absorbed into the human serum. A know how of the physicochemical changes that occur in foods during the digestion process and various affecting factors such as nutrient bio-accessibility (amount of a compound being released from the matrix and thereby solubilized into the water phase for subsequent absorption in the systematic circulation through the gut wall), bioavailability (total amount of a compound being released and absorbed into the human serum for subsequent delivery into various body tissues) and digestibility (the fraction of food

components that eventually gets transformed into potentially accessible matter) are desired characteristics of functional foods.

The efficacy of newly developed foods in terms of the above-mentioned parameters is very much dependent on the availability of digestion models. These models accurately simulate the complex physicochemical and physiological events that occur in the human gastrointestinal tract. In-vivo feeding methods, using animals or human being as models, are highly competent to analyse the pertinent complex multistage processes in biological system and their adaptability and efficacy for the developed foods. However, these are technically difficult, costly, and limited by ethical issues when potentially harmful substances are involved. Consequently, there is a real need to use in-vitro models that closely mimic the physiological processes that occur during human digestion. Such models do consider several factors such as the occurrence and concentration of digestive enzymes, pH values in gastric and intestinal phases, digestion time and salt concentrations as primary. Such models should be flexible, accurate, and reproducible. As of now, in vitro digestion models provide a useful alternative to animal and human models and thereby assist to target the rapid screening of food ingredients (Lucas-González et al., 2018).

Bio-accessibility of Curcumin

An in-vitro study of curcumin was carried out to evaluate the bio-accessibility of curcumin after digestion and thereby compare it with the available curcumin content in the sample. Thus, it was found that of the 4.80 % w/w curcumin content of RW dried turmeric, 3.10 % w/w curcumin content has been available for bio-accessibility. According to Park et al., (2019) most curcuminoids are released after incubation in the simulated intestinal fluid. Simulated intestinal fluid is a mixture of pancreatin and bile salts. Bile salts could alter the interface environment and henceforth facilitate good activity of lipase present in pancreatin for the better release of curcumin (Sari et al., 2015). Such an increase in the release would possibly foster the formation of mixed micelles that assist curcumin solubilization (Park et al., 2019).

Bio-accessibility of Folic Acid

The RW dried turmeric powder may also constitute folic acid as its key ingredient. The bio-accessibility of constituent folic acid in the RW dried turmeric powder was insignificant. However, for the fortified folic acid RW dried turmeric powder sample it was 9.77 mg/ 100 g.

Bio-accessibility of Iron Content

The constituent iron content in the RWD processed turmeric powder sample was 41.7 mg/100 g. The bio-accessible iron content of the unfortified turmeric powder was 0.27 mg/100 g (nearly 5 %). After 20 mg NaFeEDTA addition to 100 g turmeric powder sample, the bio-accessible iron content enhanced to 12.74 mg/100 g. Such an enhancement in the bio-accessibility of iron is due to the easy availability of the iron content after fortification.

Recommended Daily Allowance and Possible Ways for Improving the Bio-accessibility of Iron Content

The Recommended Daily Allowance (RDA) for folic acid is 400 µg/day for both men and women but 600 µg/day for pregnant women (Bailey, 2000.) Also, the RDA of iron varies with age of the person and as 15 - 18 mg/day for women, 27 mg/day for pregnant women, 11 – 8 mg/day for men and 8 mg/day for senior citizen (Nair and Iyenger, 2009). In the present study, it was found that the bio-accessibility folic acid was in excess constitution to that being inferred from the RDA. However, for iron, the bio-accessible available amount was sufficient as per the RDA of men and senior citizen but not for women and pregnant woman. From literature review, it was difficult to conclude upon a precise amount of NaFeEDTA and folic acid for fortification. Therefore, a higher quantity of 20 mg was considered. Moreover, the same amount of NaFeEDTA and folic acid was taken for fortification as it assists in the effective comparison of the influence of NaFeEDTA and folic acid on the fortification characteristics of the turmeric powder. Thus, the study ensured a preliminary assessment of the bio-accessibility of folic acid and iron content. These were obtained as 9.77 mg/100 g and 12.74 mg/100 g.

However, further study of bio-accessibility is required to analyze the required percentage as per RDA specifications.

The presence of tannin and phytic acid hinders the bioavailability of iron in food (Luo et al., 2010). Therefore, various technologies such as micro- and nanoencapsulation are often deployed to increase the bio-availability of the constituents (Darwish et al., 2021). Micro- and nanoencapsulation enable the deposition of the minerals into a matrix. Such an encapsulation scheme enhances the retention time, stability and prevent oxidation. Thus, controlled release of the minerals ensured improved bioavailability of the minerals (Subroto et al., 2022). Iron encapsulation is often used for baby food and powdered drink mixes. Liposomes and fatty acid ester encapsulation method was used by Abbasi and Azari, (2011) for the micro-encapsulation of iron in milk. Gutierrez et al., (2016) used niosomes for the iron coating in yoghurt iron fortification. The bioavailability of iron enhanced four-fold due to the nano-encapsulation of iron with solid lipid nanoparticles (Hosny et al., 2015). Also, iron absorption enhanced due to the presence of ascorbic acid (vitamin C) (Shubham et al., 2020). This facilitates the reduction of ferric ions (Fe^{3+}) to ferrous ions (Fe^{2+}) for the easy absorption of iron into the body (Aride et al., 2010). Similar methods can be adopted for the enhanced bio-availability in the fortified samples.

6.11 Literature Comparison

Various physical characterizations were carried out for RW dried based products. A comparative assessment was undertaken with products such as yoghurt, tomato, aloe-vera and mango (Table 6.9). As conveyed in the table, the RW dried turmeric powder showed good physical characteristics and these are evident in the relevant prior art. The results for wettability, solubility, hygroscopicity, bulk density agreed with those reported in the previous studies. In the investigation with yoghurt, aloe vera, mango and tomato by Tontul et al., (2018), Minjares-Fuentes et al., (2016), Caparino et al., (2012) and Castoldi et al., (2015) respectively the obtained results showed that RWD facilitated better wettability, solubility, hygroscopicity bulk density and colour results. Minjares-Fuentes et al., (2016) and Caparino et al., (2012) inferred that RWD gave similar results to that obtained by freeze

drying but better than those obtained with spray and drum drying. The RW dried turmeric powder, folic acid fortified RW dried turmeric powder and NaFeEDTA fortified RW dried turmeric powder had high

Table 6.9: Comparative data summary of fortification and associated characteristics of dried turmeric products.

| S. No. | Drying method | Sample | Parameters | Key findings | References |
|--------|--|--------------|---|---|--|
| 1. | RWD | Turmeric | Wettability, Solubility, Hygroscopicity Bulk density | Wettability: 20 s Solubility: 29 % Hygroscopicity: 8.70 % Bulk density: 0.62 g/ml ³ | This study |
| 2. | RWD | Yoghurt | Wettability, Solubility, Hygroscopicity Bulk density | Wettability: 35 s, Solubility: 78 %, Hygroscopicity: 10 % Bulk density: 17.1 g/ml ³ | Tontul et al., (2018) |
| 3. | RWD, Freeze drying, Spray drying | Aloe vera | Solubility Hygroscopicity | Freeze drying and RWD shown same results but better than spray drying | Minjares- Fuentes et al., (2017) |
| 4. | RWD, Drum drying Freeze drying | Mango | Colour Bulk density Solubility Hygroscopicity | Freeze drying and RWD yielded similar results but better than drum drying | Caparino et al., (2012) |
| 5. | RWD | Tomato | Colour Wettability Solubility | Colour: 53.44 Wettability: 9 s Solubility: 87 % | Castoldi et al., (2015) |

wettability, solubility, and colour results. This was in agreement with the results obtained by Tontul et al., (2018) and Castoldi et al., (2015) for yoghurt and tomato respectively. Low hygroscopicity was obtained for all cases of RW dried turmeric powder, folic acid fortified RW dried turmeric powder and NaFeEDTA fortified RW dried turmeric powder. These are in corroboration with the low hygroscopicity values for aloe vera and mango being reported by Minjares-Fuentes et al., (2016) and Caparino et al., (2012) respectively. In case of colour, high colour retention was observed for RW dried turmeric powder, folic acid fortified RW dried turmeric powder and NaFeEDTA fortified RW dried turmeric powder. These are similar to those inferred by Castoldi et al., (2015) for the tomato sample.

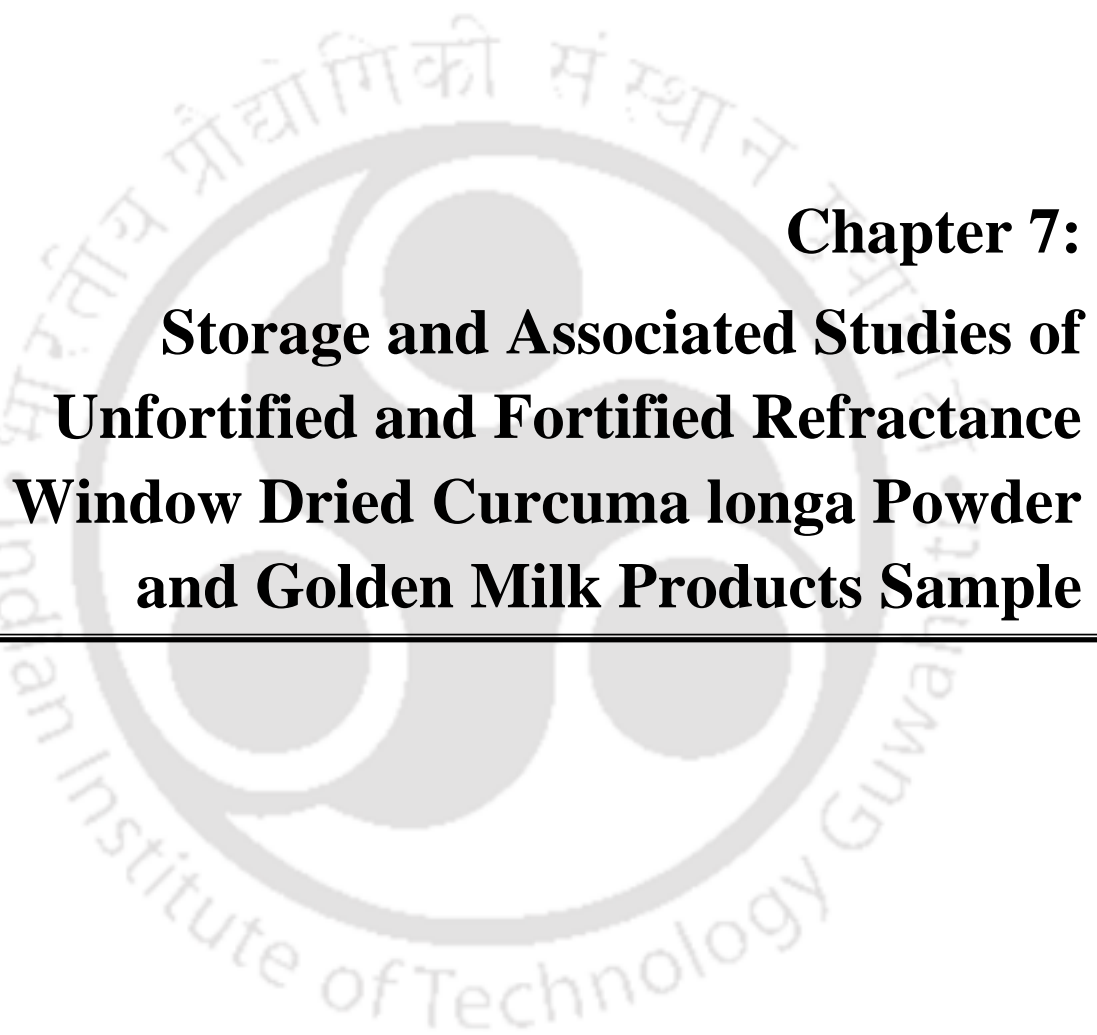
6.12 Summary

The important outcomes of the conducted investigations can be listed as follows.

Firstly, bulk density of RW dried turmeric powder, folic acid fortified turmeric powder and NaFeEDTA fortified turmeric powder did not vary significantly. Secondly, the hygroscopicity of RW dried turmeric powder, folic acid fortified turmeric powder and NaFeEDTA fortified turmeric powder were lower and similar to those obtained with freeze drying process. Thirdly, the solubility and swelling power of RW dried turmeric powder, folic acid fortified turmeric powder and NaFeEDTA fortified turmeric powder were good and do match with the results obtained with freeze drying methods. Fourthly, the dispersion time was less and hence the RW dried turmeric powder, folic acid fortified turmeric powder and NaFeEDTA fortified turmeric powder have better wettability. Fifthly, the water binding capacity for RW dried turmeric powder, folic acid fortified turmeric powder and NaFeEDTA fortified turmeric powder were high. Sixthly, the colour indices of RW dried turmeric powder, folic acid fortified turmeric powder and NaFeEDTA fortified turmeric powder were almost the same. Seventhly, the FTIR spectra has similar pattern for the RW dried turmeric powder, folic acid fortified turmeric powder and NaFeEDTA fortified turmeric powder samples. This affirmed that no major shift in the functional groups occurred due to addition of folic acid and NaFeEDTA. Eighthly, in comparison to RW dried turmeric powder and folic acid fortified turmeric powder, the TGA of NaFeEDTA fortified turmeric powder affirmed extra peak. This confirmed upon the presence of NaFeEDTA and its inorganic nature. Ninthly, the RW dried turmeric powder and folic acid fortified turmeric powder showed similar trends in DSC due to being organic and amorphous in nature while NaFeEDTA fortified turmeric powder showed marginally crystalline nature due to the existence of NaFeEDTA in the sample. Tenthly, XRD results have been the same for RW dried turmeric powder and folic acid fortified turmeric powder but were different for NaFeEDTA fortified turmeric powder due to the presence of NaFeEDTA. Eleventhly, the images of the RW dried turmeric powder, folic acid fortified turmeric powder and NaFeEDTA fortified turmeric powder were similar. Twelfthly, the particle size distribution was same for all cases. Thirteenthly, the in-vitro digestion analysis conducted affirms that there has been a significant increase in the bioavailability of folic acid and NaFeEDTA content after fortification. From this chapter, it could be concluded that the addition of folic acid and NaFeEDTA to the RW dried turmeric powder did not change the physical characteristics and constitution of the native RW dried turmeric powder. This is due to the stability of folic acid and NaFeEDTA compounds being added to

the RW dried turmeric. Thereby, they did not interact with the RW dried turmeric powder and caused physical changes to the powder. However, NaFeEDTA has been detected through TGA, DSC, FESEM and XRD analysis. This is due to its inorganic and crystalline nature.





Chapter 7:

Storage and Associated Studies of Unfortified and Fortified Refractance Window Dried Curcuma longa Powder and Golden Milk Products Sample



Storage and Associated Studies of Unfortified and Fortified Refractance Window Dried *Curcuma longa* Powder and Golden Milk Products

In this chapter, the refractance window dried turmeric powder, folic acid fortified refractance window dried turmeric powder and NaFeEDTA fortified refractance window dried turmeric powder has been studied for moisture isotherm, sensory analysis and storage study. Section 7.1 provides a brief introduction to the chapter objectives. In sections 7.2 and 7.3, the sample preparation and moisture isotherm study have been respectively discussed. Thereafter, section 7.4 elaborates on the permeability of the packaging material. Section 7.5 details upon the shelf-life of the samples. In section 7.6, sensory analysis of the turmeric powder has been discussed. Section 7.7 delineates upon the nutritional properties of the optimal golden milk formulation. Section 7.8 discusses the storage study of the refractance window dried turmeric powder, folic acid fortified refractance window dried turmeric powder and NaFeEDTA fortified refractance window dried turmeric powder along with the study of the physical characteristic like bulk density, swelling power, solubility, dispersion time, hygroscopicity, water binding capacity, moisture content and colour and chemical characteristics like anti-oxidant activity, total phenolic content, total flavonoid content, curcumin content, folic acid and iron content. Section 7.9 shows the statistical analysis of the results obtained in the previous section. The section 7.10 does a literature comparison followed by the summary of the chapter in section 7.11.

7.1 Introduction

In this chapter, the refractance window (RW) dried turmeric powder, folic acid fortified and sodium ferric ethylenediaminetetraacetate (NaFeEDTA) fortified turmeric powder samples were targeted for their moisture isotherm characteristics and shelf-life study. The chapter also details upon

the sensory analysis, nutritional characteristic of the optimal golden milk for refrigerated and unrefrigerated golden milk and their storage characteristics.

In the related prior art, similar investigation was addressed for mango, carrot and garlic and by Jaya and Das, (2005), Singh et al., (2003) and Moreno et al., (2006) respectively. Misra and Kulshrestha, (2003) studied potato flour samples stored at room and refrigerated conditions. Moisture isotherm studies of freeze dried, vacuum shelf dried and flow dried onion powder were conducted by Debnath et al., (2002). Lahari et al., (2020) conducted a storage study of cured and non-cured turmeric rhizome. Using gunny bag, plastic bag, cloth bag and polyethylene terephthalate (PET) jar, Sidhu et al., (2013) conducted a storage study of turmeric rhizome and turmeric powder samples. A curcumin fortified whey based beverage was developed by Ankitha et al., (2018) and lassi enhancement through curcumin supplementation with β -cyclodextrin was studied by Maurya et al., (2020). Meanwhile milk analogs namely coconut, cashew, almond, and oat milks samples were loaded with curcumin by Zheng et al., (2021).

Till date, RW dried turmeric powder, folic acid fortified and NaFeEDTA fortified turmeric powder samples have not been studied for moisture isotherm, shelf life and storage study along with other associated characteristics and in a comparative framework. Considering the above cited lacunae, this article addresses the isotherm and storage study of RW dried turmeric powder, folic acid fortified RW dried turmeric powder and NaFeEDTA fortified RW dried turmeric powder.

7.2 Powder Sample Preparation

The RW dried turmeric, folic acid fortified RW dried turmeric and NaFeEDTA fortified RW dried turmeric powder samples (20 g) were stored in petri plates were kept in desiccators containing appropriate salt solutions. Thereby, these systems were stored in an incubator at 40 °C. At uniform time intervals, the sorption studies were assessed till equilibrium moisture content were reached for the stored samples.

Table 7.1: GAB model fitness parameters for refractance window (RW) dried, folic acid fortified RW dried and NaFeEDTA fortified RW dried turmeric powder samples.

| S. No. | Samples | C | K | Mo | R ² |
|--------|----------------------|-------|-------|-------|----------------|
| 1. | Unfortified | 0.476 | 0.996 | 0.358 | 0.99 |
| 2. | Folic acid fortified | 0.650 | 0.920 | 0.271 | 0.99 |
| 3. | NaFeEDTA fortified | 0.630 | 0.931 | 0.280 | 0.98 |

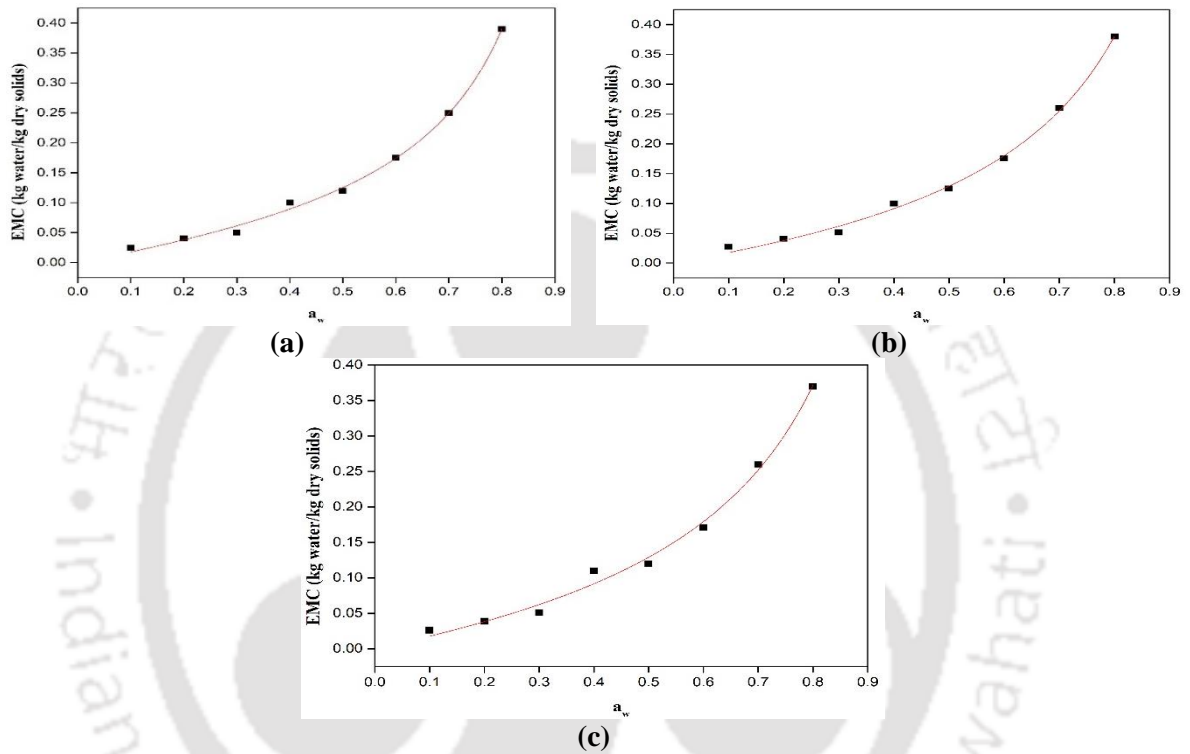


Figure 7.1: Moisture content alteration and water activity for (a) refractance window (RW) dried (b) folic acid fortified RW dried and (c) NaFeEDTA fortified RW dried turmeric powder samples.

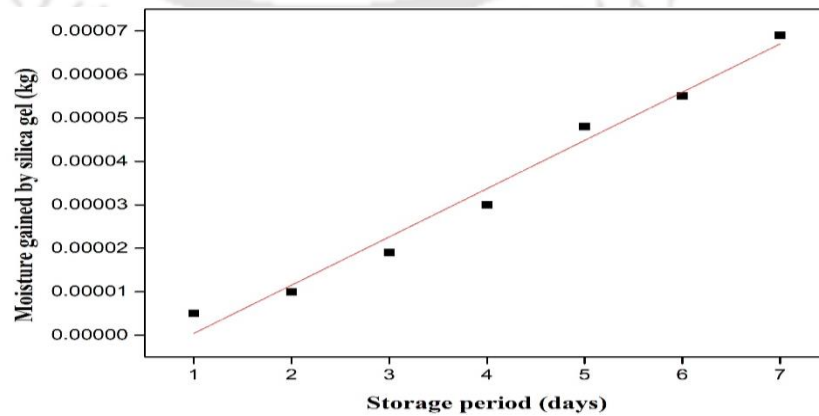


Figure 7.2: Permeability plot for moisture gain by silica gel with time.

7.3 Sorption Characteristics of Powder Products

The moisture sorption behaviour of RW dried turmeric powder, folic acid fortified RW dried turmeric and NaFeEDTA fortified RW dried turmeric samples were studied at 40 °C. The obtained sorption isotherms revealed that the equilibrium moisture content increased with an increase in water activity at a constant temperature (Fig. 7.1). This is a characteristic feature of amorphous materials with richer constitution of hydrophilic components. The observed behaviour has been attributed to the hydrophilic nature of carbohydrates and constituent protein content in the RWD dried powder samples.

Table 7.1 summarizes the analysis results for GAB model to represent the experimentally measured sorption data. The table presents C , K , M_0 , R^2 values. This is in agreement with the inferences of Goula et al., (2005) and Sormoli and Langrish, (2019) for the sorption isotherms of tomato pulp powder and orange juice powder respectively. Thereby, GAB sorption model could best represent the pertinent adsorption behaviour of the RW dried turmeric, folic acid fortified and NaFeEDTA fortified turmeric RW dried powder samples.

7.4 Permeability of Packaging Material

Fig. 7.2 depicts the time dependent cumulative moisture gain with time by silica gel in zipper pound at 38 ± 2 °C and 90 % RH. Thereby, the slope of the straight line fit (dw/dqp) was 0.000011 kg/day. Using Eq. 19, the water vapour permeability K of the zipper pouch for a surface area value of 0.014 m² and p^* value of 6980.5 Pa (saturated vapor pressure of water at 38 °C), was obtained as 1.12×10^{-7} kg/m²dayPa (Jaya and Das, 2005).

7.5 Shelf-Life Assessment

Using Eq. 18, the shelf-life parameters of RW dried turmeric powder, folic acid fortified and NaFeEDTA fortified RW dried turmeric powder samples packed in zipper pouches (38 ± 1 °C) were determined. The water vapour permeability of zipper pouches was calculated using Eq. 19 and was found to be 1.12×10^{-7} kg/m²dayPa, respectively. The initial moisture content (X_i) of RW dried turmeric

powder, folic acid fortified and NaFeEDTA fortified RW dried turmeric powder samples were same for all cases at 4.27 % (db) respectively. The critical moisture content (X_c) was taken as 8.60 % (db) for all cases.

At a storage temperature of 38 °C, for RW dried turmeric powder, folic acid fortified RW dried turmeric and NaFeEDTA fortified RW dried turmeric powder samples the water activity values corresponding to critical moisture content were 0.49, 0.49 and 0.48 respectively. Considering the zipper pouch surface area as 0.014 m² for all cases, total solid weight (W_s) as 0.01918, 0.01938 and 0.01938 kg for RW dried turmeric powder, folic acid fortified RW dried turmeric and NaFeEDTA fortified RW dried turmeric powder samples respectively. The saturated vapour pressure as 6980.5 Pa, Eq. 18, has been used to determine the shelf life of the samples (Seth et al., 2018). Corresponding values were 184, 187 and 183 days for RW dried turmeric powder, folic acid fortified RW dried turmeric and NaFeEDTA fortified RW dried turmeric powder samples respectively in the zipper pouch system.

7.6 Sensory Assessment of Powder Product

As a simple marketing rule, if consumers do not like the appearance, flavour or texture of a food product, they would not buy it. Therefore, the overall sensory experience of a product is crucial for its commercial success. Specific protocols and methods have been developed to estimate and quantify consumers' sensory experiences, Thereby, risk associated to the non-acceptable of a food product can be reduced through a new area of scientific relevance known as sensory descriptive analysis or sensory descriptive evaluation. Appropriate sensory evaluation allows a very useful understanding of the key attributes that helps the commercial success of food products (Sirangelo et al., 2019).

The RW dried turmeric powder has been evaluated for its sensory characteristics. However, the folic acid fortified and NaFeEDTA fortified turmeric powder samples were not analysed for their sensory characteristics due to the non-food grade status of the deployed folic acid and NaFeEDTA fortificants. Nelson and Trout, (1964) provided a vivid account of the associated research methodology. The sensory analysis was conducted with a panel of judges and in terms of the scores provided for

colour and appearance, taste, aroma, mouthfeel, aftertaste, consistency and overall acceptability (Hingne et al. 2020). A hedonic scale with a maximum score of 9 was considered (Table 2.3).

7.6.1 Colour and Appearance

The colour and appearance of a product is a prime entity for its formal acceptance or rejection during sensory analysis. The aesthetic quality of a food product is dominantly influenced by its colour and thereby provides visual inputs for flavour identification. The findings summarized in Fig. 7.3 (a – e) confirm upon the fact that the turmeric constitution in the milk system did significantly alter the colour and appearance scores. In general, either 1 or 1.5 g turmeric powder per 100 mL of milk have been inferred to be acceptable in terms of colour and appearance scores that varied between 8 – 9.0 in a 9-point hedonic scale. Thus, the acceptability level of the parameter has been within the moderate liking to like extremely response level. However, compared to all other samples, colour acceptability score was significantly highest (9.0) for the turmeric milk system with 1 g of turmeric powder. Also, the lowest score of 6.0 was obtained for the milk system with 0.5 g of powder. Moreover, a significant reduction in colour acceptability level has been apparent (6.5 score) for an enhanced turmeric constitution of 2 g per 100 mL milk.

7.6.2 Aroma

After colour and appearance, aroma has significance in the sensory evaluation of a sample and thereby critically influences the formal acceptance or rejection of a sample by a consumer. The smell or aroma of a product influences the olfactory glands of consumers and thereby enhances the desire of the consumers to taste a product. Thus, a product with good aroma attracts a consumer for tasting it and bad or strong aroma hinders the formal acceptance of the food product. The aroma scores for the product have been summarized in Fig. 7.3 (a – e). From Fig. 7.3 (a – e), it can be confirmed that the turmeric constitution in the milk system did significantly alter the aroma scores. In general, either 1 or 1.5 gm of turmeric powder have been acceptable with an aroma score of about 7 – 8.0 in 9-point hedonic scale. Thus, the acceptability level is within moderately liked and liked very much status of the responses.

However, the aroma acceptability has been significantly highest (8.0) for the 1 g turmeric milk constitution in comparison to the other constitution. Also, significantly low score (6.5) was recorded for 0.5 g of turmeric powder and the lowest (5.0) was obtained for 2 g turmeric powder in 100 mL milk. This conveys that at its lowest level, the turmeric has not been able to impart good aroma to the milk system and at its highest level, the aroma is strong such that the milk system (2 g turmeric powder in 100 mL milk) has non-acceptable status for its good consumption.

7.6.3 Taste

Despite having good appearance and colour properties, a food product is usually accepted for its good taste property. Further, the taste of a product is contributed by its smell. Fig. 7.3 (a – e) presents relevant illustrations to infer that the addition of turmeric powder in altered constitutions did significantly influence the taste property of the turmeric milk samples. Compared to the control (milk) sample (6.0), higher taste score was obtained for other samples. These were 6.5, 8.0, 7.0 and 5.0 for 0.5, 1, 1.5, and 2 g turmeric in 100 mL of milk respectively. The highest taste score of 8.0 was achieved for the sample constituting 1 g turmeric in 100 mL of milk. Any further increase in turmeric constitution reduced the flavour score.

Similar inferences have been presented by Jothylingam and Pugazhenthii, (2013) for flavoured milk samples prepared with aloe vera pulp. The author reported optimal constitution of 5 % pulp and further reduction in flavour score due to bitterness for enhanced constitution of 7 % pulp. Moreover, Srikanth et al., (2017) also concluded that beyond 10 % constitution of aloe vera in peda reduced its flavour score. These observations are corroborative to the pertinent trend of reduced flavour score due to enhanced constitution of turmeric in the milk. Thus, in general, the results do indicate that the turmeric milk samples with 0.5 to 1.5 g turmeric constitution have been within the acceptability response levels of liked slightly to liked very much. Thus, the optimal flavour-based constitution of 1 g turmeric in 100 mL milk can enhance the medicinal benefits associated to its consumption.

7.6.4 Aftertaste

The aftertaste is an attribute that lingers in the palate of the mouth after a sample's ingestion into the mouth. The aftertaste of the product is an important sensory attribute and thereby reflects upon its acceptability of the product by the consumers. The average sensory score for the aftertaste of the turmeric flavoured milk has been depicted in Fig. 7.3 (a – e). It is evident from the results that the addition of 1 g and 2 g turmeric powder received significantly highest (8.0) and lowest (5.5) aftertaste score in the milk product system. This may be due to strong taste of the turmeric for 2 g constitution in 100 mL milk. Moreover, the aftertaste score for the turmeric powder flavoured milk has been better with respect to the control case. This is due to the enhanced flavour of turmeric milk. Thus, milk fortification with turmeric powder is promising to enhance aftertaste attribute of the product.

7.6.5 Consistency

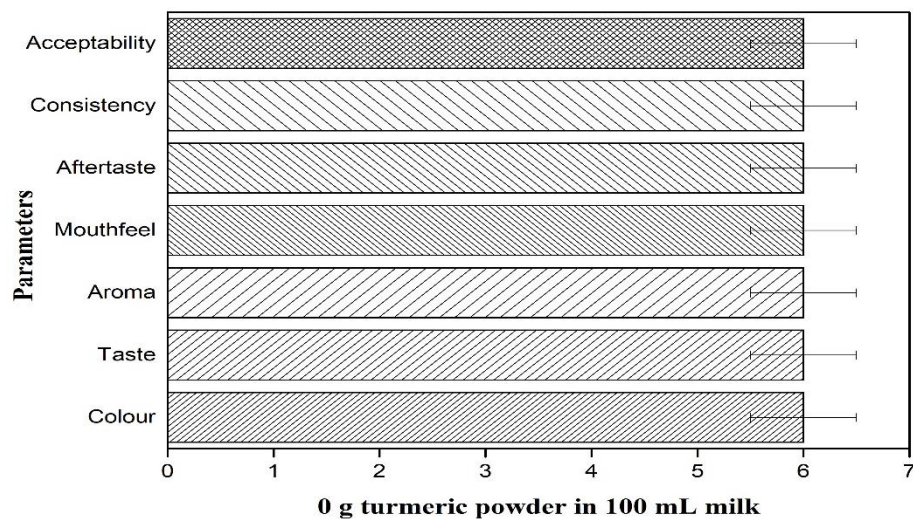
Following taste and aftertaste attributes, the consistency of a product is an important sensory attribute to infer upon its degree of acceptability. The influence of turmeric constitution on the consistency score of flavoured milk has been depicted in Fig. 7.3 (a – e). The consistency score of 1 g turmeric constitution in the flavoured milk affirmed the highest consistency score (7.5) followed with lowest value for the 2 g turmeric constitution (5.5). The probable reason for the 2 g case affirming lower consistency could be due to more constitution of turmeric powder in the milk that fostered the precipitation of the additional turmeric powder. Incidentally, the consistency score for the 0.5 and 1.5 g turmeric powder constitution in the milk (100 mL) indicated higher score than the value obtained for the control case. These were 7.0 and 6.5 respectively. Thus, fortification of milk with turmeric powder enhanced the acceptable consistency of mentioned product. This inference has been supported with the findings of Mudgil et al., (2016). The authors reported reduced consistency score beyond 10 % aloe vera fortification in buttermilk (consistency score reduced from 8.1 at 10 % aloe vera juice to 7.7 at 15 % aloe vera juice and 7.6 at 20 % aloe vera juice).

7.6.6 Mouthfeel

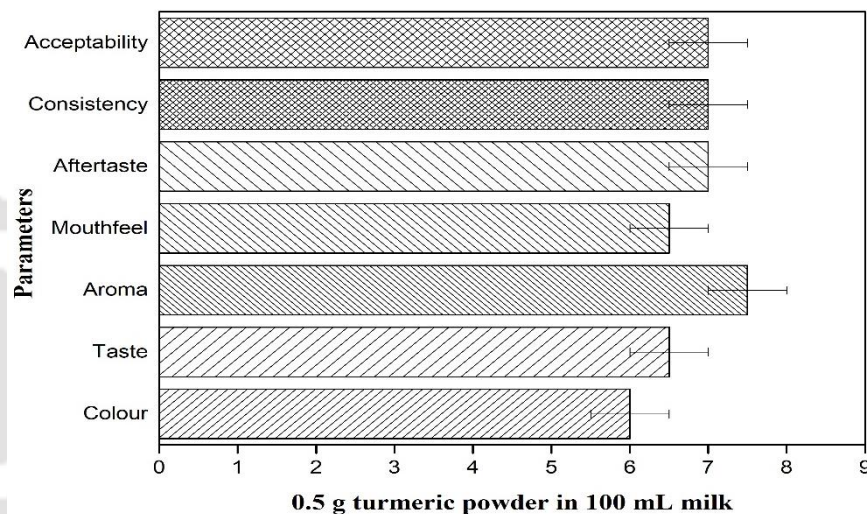
The mouthfeel of the product is an important sensory attribute and refers to an important attribute of the product acceptability. The mouthfeel score of the flavoured milk product for various turmeric constitution cases have been depicted in Fig. 7.3 (a – e). Thus, it is evident that the addition of 1 g turmeric powder constitution received the highest (8.5) and 2 g constitution received the lowest (5.5) mouthfeel score for the flavoured milk product. Moreover, the mouthfeel score with the inclusion of turmeric powder has been higher than that of the control sample. This is due to the enhanced flavour of turmeric milk. Thus, fortification of milk with turmeric powder is promising to impart acceptable mouthfeel attribute to the turmeric flavoured milk product.

7.6.7 Overall Acceptability

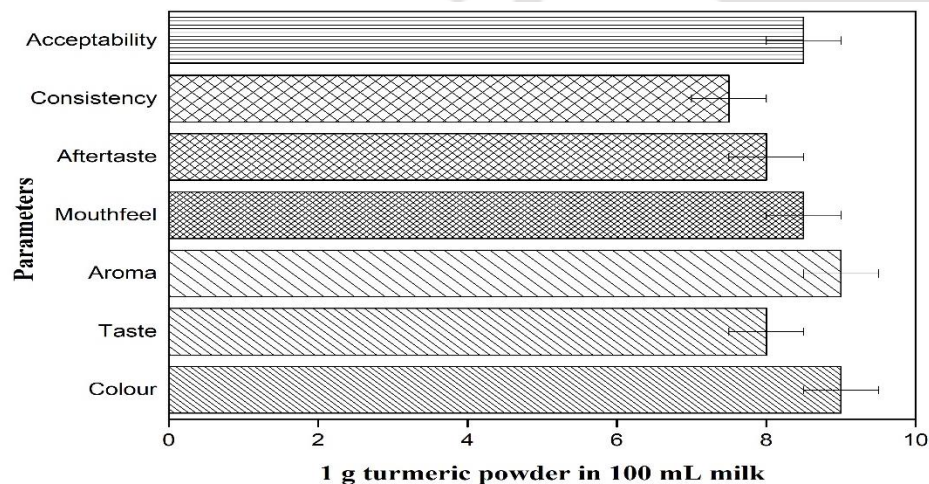
The overall acceptability parameter has a final say in the acceptability of a product by the consumer. The overall acceptability scores of flavoured milk with variant turmeric powder constitution have been presented in Fig. 7.3 (a – e). It can be observed that the variant turmeric powder constitution significantly altered the overall acceptability score of the product. Moreover, the overall product acceptability score reduced for an increase in turmeric powder constitution beyond 1 g in 100 mL milk. The highest (8.5) and lowest (5) acceptability score was obtained for the milk with 1 g and 2 gm constitution of turmeric powder respectively. The reduction in overall acceptability score with higher turmeric powder constitution could be due to the strong flavour of the turmeric. In summary, the results affirmed that the blending of turmeric with milk did not promote any adverse effect on the overall acceptability of the product as the optimally flavoured milk product has been ranked to be above liked very much response i.e. extremely good. On the basis of the results obtained during sensory analysis trials, among all constitutions of turmeric powder, 1 g turmeric powder in 100 mL of milk has been the best for flavoured milk product development.



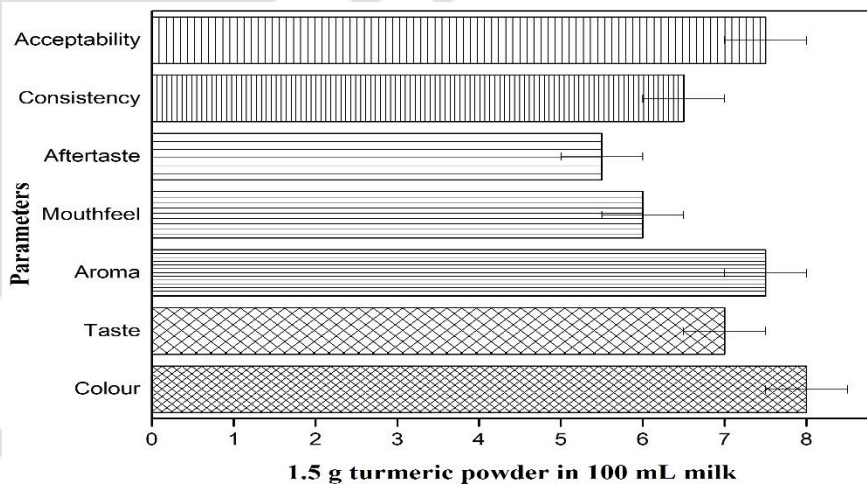
(a)



(b)



(c)



(d)

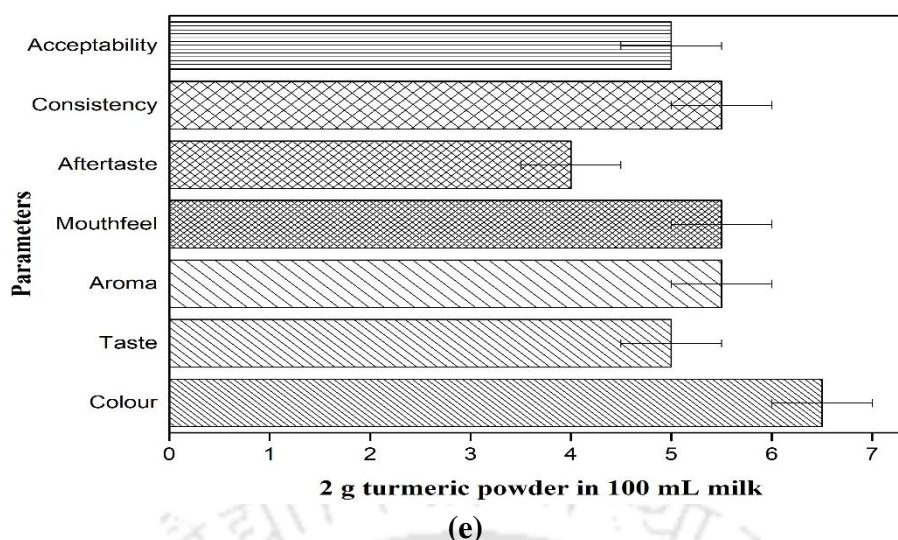


Figure 7.3: Bar chart depicting sensory characteristics of refractance window dried turmeric powder based milk products for varied turmeric constitution cases (a) 0 g, (b) 0.5 g, (c) 1 g, (d) 1.5 g and (e) 2 g in 100 mL milk.

7.7 Nutritional Properties of the Optimal Golden Milk Formulation

As an overall conclusion of the sensory analysis, the milk system that received the highest overall acceptability score was chosen as the optimal product for nutritional analysis in terms of total phenolic content (TPC), total flavonoids content (TFC), anti-oxidant activity (AA) and curcumin content (CC). These studies were carried out for two cases namely unrefrigerated sample and refrigerated sample for the evaluation of the viability period of the golden milk product.

Golden milk samples were prepared for these studies by mixing 1 g RW dried turmeric powder in 100 mL of milk. The obtained product was then divided into two parts. With the first part, immediate analysis was targeted for nutritional analysis and the sample was eventually stored at room temperature. The second part was kept in refrigerated condition for further analysis.

Unrefrigerated Conditions

The results obtained for TPC, TFC, CC and AA on day 0 (0 hr) for RW dried turmeric powder, folic acid fortified and NaFeEDTA fortified RW dried turmeric powder samples have been summarized in Table 7.2 (a). The TPC, TFC, AA and CC values for RW dried turmeric powder were 876.21 mg

Table 7.2: A summary of nutritional characteristic of (a) unrefrigerated golden milk product at 24 hr and (b) refrigerated golden milk products at 24 and 48 hr.

(a)

| S. No. | Hr | Samples | AA (%) | TPC (mg GAE/100 mL) | TFC (mg quercetin/100 mL) | CC (% w/w) |
|--------|----|----------------------|--------|---------------------|---------------------------|------------|
| 1. | 0 | Unfortified | 24.50 | 876.21 | 784.61 | 4.20 |
| 2. | | Folic acid fortified | 23.50 | 878.01 | 788.97 | 4.21 |
| 3. | | NaFeEDTA fortified | 24.10 | 870.87 | 787.49 | 4.19 |

(b)

| S. No | Hr | Samples | AA (%) | TPC (mg GAE/100 mL) | TFC (mg quercetin/100 mL) | CC (% w/w) |
|-------|----|----------------------|--------|---------------------|---------------------------|------------|
| 1. | 24 | Unfortified | 22.50 | 830.14 | 710.76 | 4.14 |
| 2. | | Folic acid fortified | 22.80 | 825.51 | 716.87 | 4.16 |
| 3. | | NaFeEDTA fortified | 22.68 | 829.84 | 717.54 | 4.14 |
| 1. | 48 | Unfortified | 20.90 | 784.29 | 650.71 | 4.10 |
| 2. | | Folic acid fortified | 20.12 | 786.31 | 646.15 | 4.10 |
| 3. | | NaFeEDTA fortified | 21.00 | 781.62 | 649.25 | 4.11 |

* All standard deviations for AA, TPC, TFC and CC were in the range of 0.5 – 0.8, 3 – 5, 2 – 5 and 0.01 – 0.02 respectively.

GAE/100 mL, 784.61 mg quercetin/100 mL, 24.50 % and 4.20 % w/w respectively. Also, similar results were obtained for folic acid fortified and NaFeEDTA fortified RW dried turmeric powder samples. This could be due to the folic acid and NaFeEDTA fortification not adversely affecting the nutrition components of the turmeric powder. However, nutritional analysis could not be carried out for the second day as the milk stored under unrefrigerated condition got spoiled through the curdling effect.

Refrigerated Conditions

The results obtained for day 0 have been similar to those being obtained for the unrefrigerated condition. Further, the sample analysis on day 1 (24 hr) indicated TPC, TFC, AA and CC as 830.14 mg GAE/100 mL, 710.76 mg quercetin/100 mL, 22.50 % and 4.14 % w/w respectively for the unfortified sample in Table 7.2 (b). On day 2 (48 h), the unfortified sample analysis affirmed 784.29 mg GAE/100

mL TPC, 650.71 mg quercetin/100 mL TFC, 20.90 % AA and 4.10 % w/w CC. Similar values were obtained for the folic acid fortified and NaFeEDTA fortified RW dried turmeric powder. Comparatively, except for the CC, these values were significantly lower than the corresponding values obtained for the day 1 (24 h) case. Hence, a significant reduction in the TPC, TFC and AA values was apparent for the day 2 (48 h) sample of the refrigerated golden milk case. However, no such analysis could be carried out on day 3 (72 hr) as the milk got spoiled even for refrigeration case.

7.8 Storage Assessment based Nutritional and Physical Characteristics of Powder Products

Food products undergo dynamic alteration with respect to moisture gain or loss from the product. Such alterations do exist until the system reaches a state of thermodynamic equilibrium with the surrounding environment (Labuza & Hyman, 1998). Thereby, the migratory role of water has a potential influence in the moisture content of food products over a period of time. Thus, the shelf-life and storage properties of a food product may get detrimentally influenced and altered in terms of physical and nutritional attributes. Hence, studies are required to analyse these aspects through storage studies (Saha et al., 2020). In this work, the storage studies of RW dried turmeric powder, folic acid fortified RW dried turmeric powder and NaFeEDTA fortified RW dried turmeric powder samples have been conducted by storing them in a desiccator at 38 ± 1 °C for 180 days. During this period, alterations in the physicochemical properties of the samples have been evaluated at periodic intervals.

7.8.1 Moisture Content

The MC is one of the most important factors in due course of the evaluation of the quality and stability of the RW dried turmeric powder, folic acid fortified RW dried turmeric powder and NaFeEDTA fortified RW dried turmeric powder samples. Thus, low MC of the samples during storage affirms a decelerated and reduce rate of several degradation reactions and associated microbial growth (Ho et al., 2019). It is well known that the powder samples with high MC are susceptible to quality

deterioration at higher temperature. This is due to the hydrolysis of oil and phospholipids followed by an increase in the sample acidity (Saha et al., 2020). Various associated studies for the storage of

Table 7.3 (a): Time dependent moisture content (%) data of stored refractance window dried turmeric powder products.

| S. No. | Sample | 0 weeks | 9 weeks | 17 weeks | 24 weeks |
|--------|----------------------|---------|---------|----------|----------|
| 1. | Unfortified | 4.00 | 4.90 | 6.10 | 7.81 |
| 2. | Folic acid fortified | 4.00 | 4.95 | 6.10 | 7.80 |
| 3. | NaFeEDTA fortified | 4.00 | 4.91 | 6.30 | 7.83 |

* All standard deviations were in the range 0.1 – 0.3.

Table 7.3 (b): Time dependent anti-oxidant activity (%) data of stored refractance window dried turmeric powder products.

| S. No. | Sample | 0 weeks | 9 weeks | 17 weeks | 24 weeks |
|--------|----------------------|---------|---------|----------|----------|
| 1. | Unfortified | 90.00 | 88.60 | 85.40 | 84.80 |
| 2. | Folic acid fortified | 89.91 | 88.70 | 85.90 | 84.00 |
| 3. | NaFeEDTA fortified | 89.97 | 87.40 | 85.10 | 84.50 |

* All standard deviations were in the range 0.5 – 1.0.

powdered food products confirmed that the optimal MC of dried powders shall be in the range of 4 – 8 % for good storage stability (Teijeiro et al., 2018). During storage study, the MC of all samples gradually increased with time (Table 7.3 a). Thereby, the powders may undergo transformations (caking) with time, and may also get modified due to pertinent long-term effect of the environmental and mechanical conditions (Zafar et al., 2017). However, for all tested samples, no caking was observed till 180 days of storage under ambient conditions (Saha et al., 2020).

7.8.2 Anti-oxidant Activity

Using the DPPH method, the initial AA values of the RW dried turmeric powder, folic acid fortified RW dried turmeric powder and NaFeEDTA fortified RW dried turmeric powder samples have

been obtained without any variation and as 90.00 – 89.91 % (Table 7.3 b). After accelerated storage, the AA reduced to about 84.80 – 84.00 %. The AA values of RW dried turmeric powder, folic acid fortified RW dried turmeric powder and NaFeEDTA fortified RW dried turmeric powder samples did

Table 7.3 (c): Time dependent total phenolic content (mg GAE/g sample) data of stored refractance window dried turmeric powder products.

| S. No. | Sample | 0 weeks | 9 weeks | 17 weeks | 24 weeks |
|--------|----------------------|---------|---------|----------|----------|
| 1. | Unfortified | 189.76 | 180.00 | 175.23 | 167.01 |
| 2. | Folic acid fortified | 190.00 | 179.12 | 176.31 | 165.00 |
| 3. | NaFeEDTA fortified | 189.01 | 181.15 | 173.47 | 166.25 |

* All standard deviations were in the range 2 – 4.

Table 7.3 (d): Time dependent total flavonoids content (mg quercetin/g sample) data of stored refractance window dried turmeric powder products.

| S. No. | Sample | 0 weeks | 9 weeks | 17 weeks | 24 weeks |
|--------|----------------------|---------|---------|----------|----------|
| 1. | Unfortified | 160.00 | 153.00 | 145.00 | 139.00 |
| 2. | Folic acid fortified | 159.10 | 152.00 | 147.00 | 138.00 |
| 3. | NaFeEDTA fortified | 161.02 | 151.00 | 146.00 | 140.00 |

* All standard deviations were in the range 2 – 5.

not reduce significantly with time. Hence, the pertinent losses have been insignificant. The storage temperature did not alter the AA reduction trend with time. This is probably due to the AA attribute not being related to a single or several similar compounds but to a class of compounds that exhibited synergy in terms of their respective antioxidant activities (de Carvalho et al., 2020). Thus, the AA indicated greater synergy of various relevant constituents in terms of their effective stability with time.

7.8.3 Total Phenolic Content

The variation in TPC with time for the tested samples has been presented in Table 7.3 (c). A marginal reduction in TPC was apparent for RW dried turmeric powder, folic acid fortified RW dried turmeric powder and NaFeEDTA fortified RW dried turmeric powder samples in due course of the storage period of 180 days. The initial total phenolic content of unfortified and fortified turmeric

powders was 190.00 – 189.01 mg GAE/100 g. After 180 days storage, the TPC reduced to 167.01 – 165.00 mg GAE/100 g. Such a reduction in the phenolic content was due to the oxidation of phenolic compounds along with the activation of oxidative enzymes such as polyphenoloxidase. Similar results

Table 7.3 (e): Time dependent curcumin content (% w/w) data of stored refractance window dried turmeric powder products.

| S. No. | Sample | 0 weeks | 9 weeks | 17 weeks | 24 weeks |
|--------|----------------------|---------|---------|----------|----------|
| 1. | Unfortified | 4.84 | 4.75 | 4.70 | 4.65 |
| 2. | Folic acid fortified | 4.80 | 4.76 | 4.71 | 4.67 |
| 3. | NaFeEDTA fortified | 4.83 | 4.77 | 4.72 | 4.68 |

* All standard deviations were in the range 0.01 – 0.03.

have been reported by Henriquez et al., (2013) for apple peel powder sample at 4, 10, and 25 °C and Udomkun et al., (2016) for papaya powder samples at 30 °C.

7.8.4 Total Flavonoid Content

The initial TFC of RW dried turmeric powder, folic acid fortified RW dried turmeric powder and NaFeEDTA fortified RW dried turmeric powder samples were 161.02 – 159.10 mg/100 g. In due course of accelerated storage, the TFC of the samples reduced to 140.00 – 138.00 mg quercetin/100 g (Table 7.3 d). Thus, the trends have been apparently similar to those observed for the TPC. Such a synergy has been due to the flavonoids also being major phenolic compounds. Hence, the TFC correlated and exhibited similar pattern to reported TPC alteration during storage period of a relevant sample (Del Toro Sanchez et al., 2015). In the relevant literature, Razmkhah et al., (2013) reported that the microencapsulated kenaf seed oil under accelerated storage conditions indicated a reduced TFC in due course of the storage. Similarly, Mrmosanin et al., (2013) also reported a rapid degradation of TFC at higher temperature condition.

7.8.5 Curcumin Content

The yellow colour in the turmeric is due to the polyphenolic constituent curcumin that possesses lipophilic characteristics. In the fresh turmeric sample, the CC was 0.73 % w/w. For RW dried turmeric

powder, folic acid fortified RW dried turmeric powder and NaFeEDTA fortified RW dried turmeric powder samples, the curcumin content was 4.84 – 4.80 % w/w (Table 7.3 e). After 180 days' storage period, the curcumin content alterations have been insignificant (4.68 – 4.65 % w/w). This is due to the

Table 7.3 (f): Time dependent alteration of colour indices of refractance window dried turmeric powder products.

| S. No. | Sample | 0 weeks | | | 9 weeks | | | 17 weeks | | | 24 weeks | | |
|--------|----------------------|----------|----------|----------|----------|----------|----------|----------|----------|----------|----------|----------|----------|
| | | <i>L</i> | <i>a</i> | <i>b</i> | <i>L</i> | <i>a</i> | <i>b</i> | <i>L</i> | <i>a</i> | <i>b</i> | <i>L</i> | <i>a</i> | <i>b</i> |
| 1. | Unfortified | 56.6 | 31.0 | 62.1 | 55.5 | 29.8 | 60.2 | 53.0 | 26.8 | 57.2 | 51.8 | 23.0 | 53.2 |
| | | 7 | 5 | 3 | 2 | 0 | 1 | 2 | 5 | 1 | 7 | 2 | 1 |
| 2. | Folic acid fortified | 55.7 | 30.1 | 61.8 | 54.9 | 29.5 | 60.5 | 52.3 | 26.0 | 56.2 | 51.8 | 23.1 | 53.9 |
| | | 0 | 0 | 0 | 8 | 0 | 0 | 7 | 0 | 5 | 1 | 0 | 5 |
| 3. | NaFeEDTA fortified | 56.1 | 30.8 | 61.2 | 54.8 | 29.2 | 60.3 | 53.3 | 26.5 | 57.1 | 52.3 | 23.4 | 53.4 |
| | | 0 | 0 | 0 | 5 | 3 | 7 | 8 | 4 | 4 | 1 | 1 | 3 |

*All standard deviations for *L*, *a* and *b* were in the range of 1 – 2, 2 – 3 and 1 – 3 respectively.

Table 7.3 (g): Time dependent alteration of folic acid content (mg/100 g sample) of refractance window dried turmeric powder products.

| S. No. | Sample | 0 weeks | 9 weeks | 17 weeks | 24 weeks |
|--------|----------------------|---------|---------|----------|----------|
| 1. | Folic acid fortified | 20.00 | 19.65 | 19.01 | 18.74. |

* All standard deviations were in the range 0.5 – 1.0.

stability of curcumin in due course of accelerated storage of the RW dried turmeric powder, folic acid fortified RW dried turmeric powder and NaFeEDTA fortified RW dried turmeric powder samples.

7.8.6 Colour Indices

For RW dried turmeric powder, folic acid fortified RW dried turmeric powder and NaFeEDTA fortified RW dried turmeric powder samples, the variation of colour parameters in due course of storage period have been summarized in Table 7.3 (f). The results confirmed that, *L* value and whiteness attribute of tested samples remain almost unaltered during storage, However, a marginal reduction in *a* and *b* value can be especially observed during the storage. Thus, storage period marginally influenced the reduction of *a* and *b* values. In this regard, it is known that while *b* values indicate yellowness (+)

or blueness (-), the *a* values infer upon redness (+) or greenness (-) of the tested samples. Also, the curcumin content critically contributes to the turmeric colour. This is due to the pigmented curcumin compounds that imparts its yellow. Hence, good curcumin stability in dried turmeric led to good colour stability of the product.

7.8.7 Folic Acid

The folic acid content variation in the folic acid fortified turmeric samples during accelerated storage condition have been summarized in Table 7.3 (g). For a storage time variation upto 180 days, the total folic acid content reduced from 20.00 – 18.74 mg/100 g sample of turmeric powder. Such a marginal reduction in folic acid content has been due to the stable nature of the folic acid used for fortifications (Pua et al., 2008). Also, the physio-chemical characteristics of the RW dried turmeric powder did not alter significantly due to folic acid fortification.

7.8.8 Iron Content

For the NaFeEDTA fortified RW dried turmeric sample, the variation in iron content during storage condition has been summarized in Table 7.3 (h). With time, an insignificant reduction in iron content has been apparent. The iron content ranged from 20.00 – 19.41 mg/100 g sample. Such an insignificant reduction has been due to the stability of the turmeric powder and NaFeEDTA powder that did not customize a significant interaction. Also, the inorganic and stable nature of the iron powder fostered a minimum loss of iron content with time in the NaFeEDTA fortified turmeric sample.

7.8.9 Bulk Density

The bulk of the turmeric powder samples increased with storage time (Table 7.3 i). This was due to the moisture gain of the powder samples. A similar enhancement in the bulk densities with increasing MC during storage has been reported by Chauhan and Patil, (2013) for mango milk powder system. An increase in bulk density may also be attributed to enhanced cohesiveness between powder particles. This is due to the enhanced absorption of moisture during storage period.

Table 7.3 (h): Time dependent alteration of iron content (mg/100 g sample) of refractance window dried turmeric powder products.

| S. No. | Sample | 0 weeks | 9 weeks | 17 weeks | 24 weeks |
|--------|--------------------|---------|---------|----------|----------|
| 1. | NaFeEDTA fortified | 20.00 | 19.91 | 19.72 | 19.41 |

* All standard deviations were in the range 0.1 – 0.3.

Table 7.3 (i): Time dependent alteration of bulk density (g/mL) of refractance window dried turmeric powder products.

| S. No. | Sample | 0 weeks | 9 weeks | 17 weeks | 24 weeks |
|--------|----------------------|---------|---------|----------|----------|
| 1. | Unfortified | 0.62 | 0.64 | 0.67 | 0.69 |
| 2. | Folic acid fortified | 0.65 | 0.66 | 0.68 | 0.70 |
| 3. | NaFeEDTA fortified | 0.64 | 0.65 | 0.67 | 0.69 |

* All standard deviations were in the range 0.01 – 0.05.

Table 7.3 (j): Time dependent alteration of solubility (%) of refractance window dried turmeric powder products.

| S. No. | Sample | 0 weeks | 9 weeks | 17 weeks | 24 weeks |
|--------|----------------------|---------|---------|----------|----------|
| 1. | Unfortified | 29.00 | 29.00 | 27.00 | 26.00 |
| 2. | Folic acid fortified | 30.00 | 30.00 | 28.00 | 27.00 |
| 3. | NaFeEDTA fortified | 28.00 | 28.00 | 26.00 | 25.00 |

* All standard deviations were in the range 1.00 – 3.00.

7.8.10 Solubility

The alterations in the solubility of RW dried turmeric powder, folic acid fortified RW dried turmeric powder and NaFeEDTA fortified RW dried turmeric powder samples during storage at various RH levels have been presented in Table 7.3 (j). After RW drying, the turmeric powders possessed a solubility of 30 – 29.00 and almost underwent complete dissolution in water. However, with time the solubility of all tested sample did not alter upto the 9th week. Thereafter, the parameter reduced marginally to 25 – 26.00 at the end of the storage period (week 24). These findings confirmed that the solubility of all samples was influenced by the relative humidity conditions of the storage environment and higher RH level contributed to marginal solubility loss (Ho et al., 2019). Such insignificant

solubility loss was due to minor enhancement in moisture content that has adverse influence on the solubility.

7.8.11 Swelling Power

The swelling power of RW dried turmeric powder, folic acid fortified RW dried turmeric powder and NaFeEDTA fortified RW dried turmeric powder samples varied marginally during the duration of the storage period (Table 7.3 k). During the initial storage period, the swelling power remained fairly constant at 1.8 g/g (upto week 9). However, after 9th week, a marginal reduction in swelling power was noticed. Such parametric reduction did not significantly increase with time during the later stages of the storage. Thus, for the maximum storage period of 180 days, the reduction in swelling power was not significant (value of 1.5 g/g). The lower reduction in swelling power with storage period has been due to the stable nature of the turmeric powder. Since all tested samples did not significantly absorb moisture content, their stability remained intact in terms of good swelling power in due course of the total time period of the storage study.

Table 7.3 (k): Time dependent alteration of swelling power (g/g) of refractance window dried turmeric powder products.

| S. No. | Sample | 0 weeks | 9 weeks | 17 weeks | 24 weeks |
|--------|----------------------|---------|---------|----------|----------|
| 1. | Unfortified | 1.80 | 1.80 | 1.70 | 1.50 |
| 2. | Folic acid fortified | 2.00 | 2.00 | 1.90 | 1.70 |
| 3. | NaFeEDTA fortified | 1.90 | 1.90 | 1.80 | 1.60 |

* All standard deviations were in the range 0.1 – 0.3.

Table 7.3 (l): Time dependent alteration of water binding capacity (%) of refractance window dried turmeric powder products.

| S. No. | Sample | 0 weeks | 9 weeks | 17 weeks | 24 weeks |
|--------|----------------------|---------|---------|----------|----------|
| 1. | Unfortified | 66.00 | 66.00 | 64.00 | 61.00 |
| 2. | Folic acid fortified | 65.00 | 65.00 | 63.00 | 59.00 |
| 3. | NaFeEDTA fortified | 67.00 | 67.00 | 65.00 | 60.00 |

* All standard deviations were in the range 1.00 – 2.00.

7.8.12 Water Binding Capacity

The water binding capacity of RW dried turmeric powder, folic acid fortified RW dried turmeric powder and NaFeEDTA fortified RW dried turmeric powder samples has been summarized in Table 7.3 (l). The table infers that with storage time, a marginal reduction in water binding capacity occurred. However, the reduction has been insignificant. Upto week 9, the water binding capacity remained unchanged (66 %). However, after week 17, the reduction was apparent but marginal. This may be due to the stable nature of the tested samples that absorbed lesser moisture during storage. Hence, high water binding capacity could be retained even at longer storage period conditions.

7.8.13 Dispersion Time

The reconstitution properties of powdered food products ensured upon its acceptability in the consumer's market. Dispersion has been one of the most important reconstitution properties. It is defined as the pace at which the powder dissolves upon reconstitution in water. In other words, any

Table 7.3 (m): Time dependent alteration of dispersion time (s) of refractance window dried turmeric powder products.

| S. No. | Sample | 0 weeks | 9 weeks | 17 weeks | 24 weeks |
|--------|----------------------|---------|---------|----------|----------|
| 1. | Unfortified | 20 | 22 | 26 | 30 |
| 2. | Folic acid fortified | 17 | 21 | 25 | 29 |
| 3. | NaFeEDTA fortified | 19 | 23 | 28 | 31 |

* All standard deviations were in the range 1.00 – 2.00.

Table 7.3 (n): Time dependent alteration of hygroscopicity (g/100 g) of refractance window dried turmeric powder products.

| S. No. | Sample | 0 weeks | 9 weeks | 17 weeks | 24 weeks |
|--------|----------------------|---------|---------|----------|----------|
| 1. | Unfortified | 8.70 | 8.60 | 8.40 | 8.10 |
| 2. | Folic acid fortified | 8.80 | 8.60 | 8.50 | 8.00 |
| 3. | NaFeEDTA fortified | 8.50 | 8.40 | 8.30 | 8.00 |

* All standard deviations were in the range 0.1 – 0.2.

powder material can be considered to be of best quality upon the immediate dissolution of all its particles in water and without any lumps. The dispersion time of the RW dried turmeric powder, folic acid fortified RW dried turmeric powder and NaFeEDTA fortified RW dried turmeric powder samples varied between 20 – 17 s (Table 7.3 m). With storage time, the dispersion time considerably increased and this has been attributed to moisture content enhancement in the powder that prompted enhanced cohesiveness of the particles and henceforth greater tendency to form lumps in water (Goula & Adamopoulos, 2005). Also, insignificant differences have been recorded in the dispersibility of RW dried turmeric powder, folic acid fortified RW dried turmeric powder and NaFeEDTA fortified RW dried turmeric powder samples after second week. Thereby, good confidence levels have been ensured and the findings have been in corroboration with relevant prior art (Saha et al., 2020).

7.8.14 Hygroscopicity

Table 7.3 (n) summarizes the findings associated to the hygroscopicity of RW dried turmeric powder, folic acid fortified RW dried turmeric powder and NaFeEDTA fortified RW dried turmeric powder samples being subjected to accelerated storage conditions upto 180 days' time duration. The initial hygroscopicity of RW dried turmeric powder, folic acid fortified RW dried turmeric powder and NaFeEDTA fortified RW dried turmeric powder samples were 8.80 – 8.70 g/100g. Thereafter, the parameter reduced to 8.10 – 8.00 g/100 g in due course of accelerated storage conditions. This has been probably due to the enhanced moisture content in the powder that eventually led to further reduction in the absorbed water (Tan et al., 2021).

7.9 Statistical analysis of data associated to storage studies

A statistical analysis was carried out using one-way analysis of variance (ANOVA) and the model Tukey was used for mean comparison. For all cases of storage studies, high F value have been obtained (Table 7.4). The ANOVA based statistical analysis indicated a low p value for all cases. Hence, it can be inferred that the experimental values did not vary significantly indicating reasonable

acceptability of the values. Thereby, the findings instilled confidence in the average values reported in the article.

Table 7.4: ANOVA based statistical analysis data for various cases **(a)** moisture content, **(b)** anti-oxidant activity, **(c)** total phenolic content, **(d)** total flavonoids content, **(e)** curcumin content, **(f)** colour indices, **(g)** iron content, **(h)** folic acid, **(i)** bulk density, **(j)** solubility, **(k)** swelling power, **(l)** water binding capacity, **(m)** dispersion time and **(n)** hygroscopicity.

(a)

| S. No. | Samples | F values | Prob>F |
|--------|-------------|----------|-----------|
| 1. | Unfortified | 2170.78 | < 0.00005 |
| 2. | Folic acid | 7092.41 | < 0.00005 |
| 3. | NaFeEDTA | 2237.33 | < 0.00005 |

(b)

| S. No. | Samples | F values | Prob>F |
|--------|-------------|----------|-----------|
| 1. | Unfortified | 15.41 | 0.00198 |
| 2. | Folic acid | 17.20 | < 0.00005 |
| 3. | NaFeEDTA | 8.11 | 0.0082 |

(c)

| S. No. | Samples | F values | Prob>F |
|--------|-------------|----------|-----------|
| 1. | Unfortified | 90.52 | < 0.00005 |
| 2. | Folic acid | 269.00 | < 0.00005 |
| 3. | NaFeEDTA | 186.93 | < 0.00005 |

(d)

| S. No. | Samples | F values | Prob>F |
|--------|-------------|----------|-----------|
| 1. | Unfortified | 187.62 | < 0.00005 |
| 2. | Folic acid | 233.00 | < 0.00005 |
| 3. | NaFeEDTA | 162.25 | < 0.00005 |

(e)

| S. No. | Samples | F values | Prob>F |
|--------|-------------|----------|-----------|
| 1. | Unfortified | 99.12 | < 0.00005 |
| 2. | Folic acid | 102.75 | < 0.00005 |
| 3. | NaFeEDTA | 100.47 | < 0.00005 |

(f)

| S. No. | Samples | F values | Prob>F |
|--------|-------------|----------|-----------|
| 1. | Unfortified | 21.10 | < 0.00005 |
| 2. | Folic acid | 14.00 | 0.00151 |
| 3. | NaFeEDTA | 21.00 | < 0.00005 |

(g)

| S. No. | Samples | F values | Prob>F |
|--------|----------|----------|-----------|
| 1. | NaFeEDTA | 329.14 | < 0.00005 |

(h)

| S. No. | Samples | F values | Prob>F |
|--------|------------|----------|-----------|
| 1. | Folic acid | 477.07 | < 0.00005 |

(i)

| S. No. | Samples | F values | Prob>F |
|--------|-------------|----------|-----------|
| 1. | Unfortified | 29.00 | < 0.00005 |
| 2. | Folic acid | 14.75 | 0.0012 |
| 3. | NaFeEDTA | 6.31 | 0.0166 |

(j)

| S. No. | Samples | F values | Prob>F |
|--------|-------------|----------|--------|
| 1. | Unfortified | 6.97 | 0.0823 |
| 2. | Folic acid | 7.15 | 0.0017 |
| 3. | NaFeEDTA | 6.75 | 0.0139 |

(k)

| S. No. | Samples | F values | Prob>F |
|--------|-------------|----------|--------|
| 1. | Unfortified | 7.00 | 0.0728 |
| 2. | Folic acid | 7.78 | 0.0985 |
| 3. | NaFeEDTA | 6.00 | 0.0191 |

(l)

| S. No. | Samples | F values | Prob>F |
|--------|-------------|----------|-----------|
| 1. | Unfortified | 16.75 | < 0.00005 |
| 2. | Folic acid | 24.00 | < 0.00005 |
| 3. | NaFeEDTA | 32.75 | < 0.00005 |

(m)

| S. No. | Samples | F values | Prob>F |
|--------|-------------|----------|-----------|
| 1. | Unfortified | 70.75 | < 0.00005 |
| 2. | Folic acid | 92.75 | < 0.00005 |
| 3. | NaFeEDTA | 97.00 | < 0.00005 |

(n)

| S. No. | Samples | F values | Prob>F |
|--------|-------------|----------|-----------|
| 1. | Unfortified | 21.00 | < 0.00005 |
| 2. | Folic acid | 34.75 | < 0.00005 |
| 3. | NaFeEDTA | 14.00 | 0.00151 |

7. 10 Literature Comparison

In prior literature, few studies have been addressed for the storage characteristics of turmeric powder and turmeric fortified liquid product. A comparative summary of products such as tray dried potato flour and freeze-dried garlic powder has been presented along with the RW dried turmeric powder, folic acid fortified and NaFeEDTA fortified turmeric powder in Table 7.5 (a). In the following table (Table 7.5 b), a comparative summary has been presented for golden milk, turmeric fortified whey beverage and lassi product systems. While for turmeric in a period of 180 days, the moisture content altered in the lower range of 4.10 – 7.80 % in this work, it altered in the higher range of 5.60 – 17 % for the tray dried potato flour Misra and Kulshrestha, (2003). Moreno et al., (2006) studied the antioxidant activity of freeze-dried garlic powder for 14 days. The results obtained confirmed no alteration in antioxidant activity. However, a marginal alteration has been apparent for RW dried turmeric powder and for a period of 6 months. Among turmeric fortified liquid products, the literature confirmed that the anti-oxidant activity increased to 60.56 % for a whey beverage upon curcumin addition (Ankitha et al., 2018). Also, direct mixing of beta-cyclodextrin facilitated 50 – 90 % curcumin retention in curcumin fortified lassi product (Maurya et al., 2020). Incidentally, the golden milk product of this work possessed an antioxidant activity 24.50 % and a curcumin retention of 4.20 % w/w and affirmed very good desired characteristics. Also, the storage studies affirmed 1 and 2 days for unrefrigerated and refrigerated golden milk product in this work with good retention of nutritional

properties. These findings do convey that golden milk product needs to be stored in powder format and through the spray drying process.

7.11 Summary

As listed below, a summary of the findings are as follows:

Firstly, sorption characteristics confirmed the GAB model as the best fit model for fortified and unfortified turmeric sample. Secondly, the shelf-life of all powders were good and were about 180 days. Thirdly, 1 g of turmeric powder in 100 mL milk has been the best. However, the storage period of unrefrigerated and refrigerated golden milk product has been about 1 and 2 days respectively. Finally, storage characteristics and physical properties such as colour, bulk density, solubility, swelling power, water binding capacity, dispersion time and hygroscopicity did not change significantly and were almost similar for RW dried turmeric powder, folic acid fortified turmeric powder and NaFeEDTA fortified turmeric powder. However, among nutritional properties other than curcumin content marginal reduction in AA, TPC and TFC have been observed for all fortified and unfortified turmeric samples.

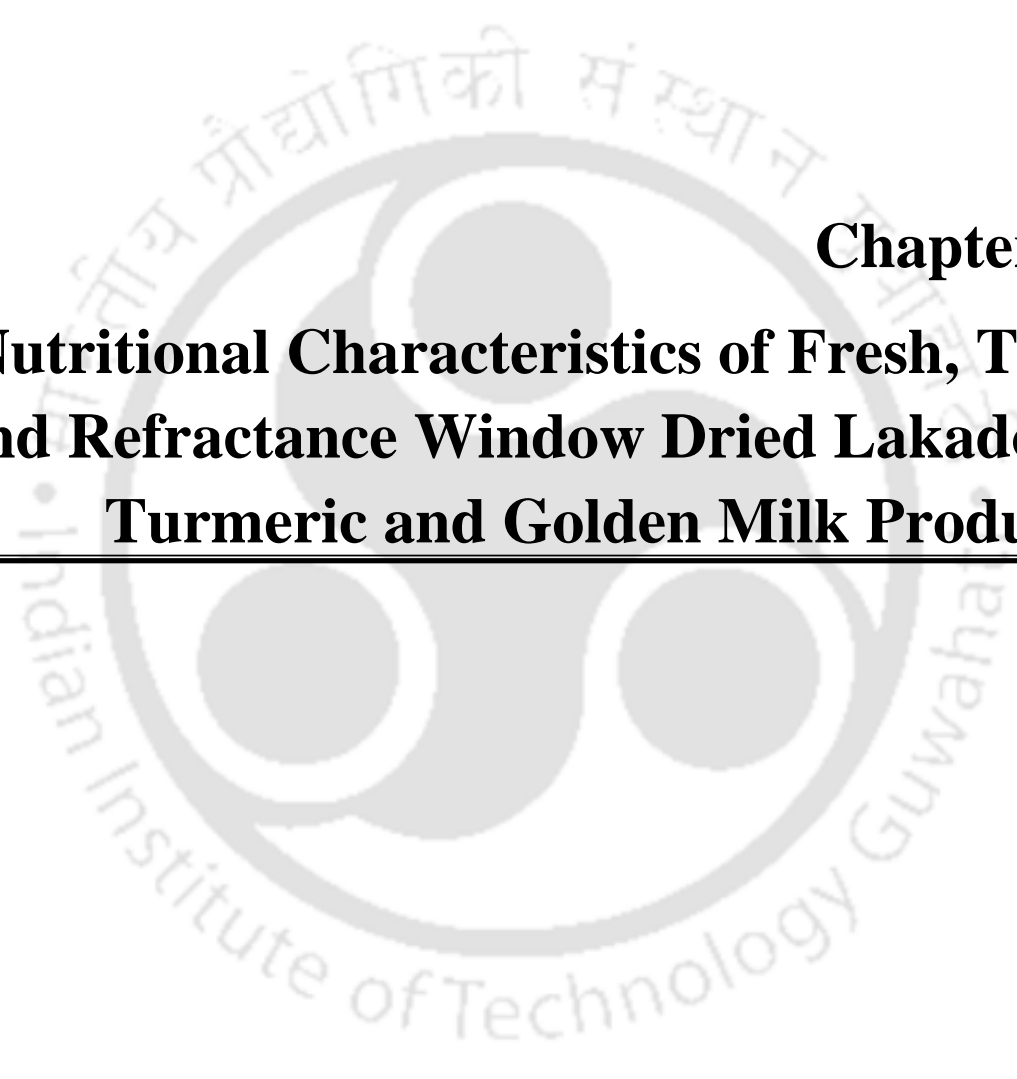
Table 7.5 (a): A summary of storage data of various dried products and turmeric powder products.

| S. No. | System | Parameters | Independent variables | Dependent variables | Methodology | Best findings | References |
|--------|-----------------|---------------------------------|-----------------------|-----------------------------------|--------------------|---|-------------------------------|
| 1. | Turmeric powder | RWD 95°C | 6 months | Physical characteristics | | MC: 4.0 - 7.9 % AA: 90.00 – 71 % TPC: 190.00 – 155 mg GAE/100 g TFC: 160 – 132 mg quercetin/100 g CC: 4.84 – 4.60 % w/w | This work |
| 2. | Potato flour | Tray dryer 55°C for 24 h | 3 – 6 months | Moisture content ascorbic acid | Proximate analysis | 5.6 – 17 % moisture content; 34 – 12 mg/g ascorbic acid | Misra and Kulshrestha, (2003) |
| 3. | Garlic powder | Freeze dried Stored at 30 °C | 0 – 14 days | Anti-oxidant activity | ORAC assay | No change in anti-oxidant activity | Moreno et al., (2006) |

Table 7.5 (b): Data summary of best findings of fortified liquid food products.

| S. No | System | Fortification Method | Independent variables | Dependent variables | Best findings | References |
|-------|---------------|--|--|-----------------------|--|------------------------|
| 1. | Turmeric milk | Direct mixing | Folic acid and iron | AA, TPC, TFC and CC | AA: 24.50 % TPC: 876.21 mg GAE/100 g, TFC: 784.61 mg quercetin/100 g CC: 4.20 % w/w | This work |
| 2. | Whey beverage | Curcumin extract added into 50 ml whey | Curcumin extract and commercial curcumin | Anti-oxidant activity | Curcumin extract 60.56 % AA, Whey beverage 43.51 % AA Storage 14 days | Ankitha et al., (2018) |
| 3. | Lassi | Direct mixing | Beta-cyclodextrin | Curcumin retention | 50 to 90 % curcumin retention Storage 20 days | Maurya et al., (2020) |





Chapter 8:
**Nutritional Characteristics of Fresh, Tray
and Refractance Window Dried Lakadong
Turmeric and Golden Milk Products**



Nutritional Characteristics of Fresh, Tray and Refractance Window Dried Lakadong Turmeric and Golden Milk Products

In this chapter, Lakadong turmeric was chosen for a comparative assessment upon its properties with the refractance window dried turmeric powder properties discussed in the previous chapters. Relevant results of fresh, tray dried and refractance window dried Lakadong turmeric and its comparative performance have been delineated in terms of the maximum retention of nutritional parameters. After a brief introduction in section 8.1, sections 8.2, 8.3, 8.4, 8.5, 8.6 and 8.7 respectively presents findings associated to the moisture content, total phenolic content, total flavonoids content, anti-oxidant activity, curcumin content and colour indices of the refractance window dried Lakadong turmeric slice sample respectively. Thereafter, section 8.8 details upon the FTIR spectral findings. Section 8.9 details upon the sensory scores of Lakadong turmeric based golden milk formulation. The best golden milk constitution has been further studied for total phenolic content, total flavonoids content, anti-oxidant activity and curcumin content. These findings have been summarized in section 8.10 of this chapter.

8.1 Introduction

In this chapter, the finding related to the studies on refractance window (RW) dried Lakadong turmeric variety have been presented in conjunction with those delineated previously for the local turmeric variety. For this purpose, firstly, the fresh Lakadong turmeric was analyzed along with tray dried (60 °C) and RW dried (95 °C) Lakadong turmeric powder sample. The analysis was conducted in terms of desired product characteristics such as moisture content (MC), total phenolic content (TPC), total flavonoid content (TFC), anti-oxidant activity (AA), curcumin content (CC), colour indices and Fourier transform infrared spectroscopy (FTIR). Thereafter a sensory study was carried out to converge upon the best constitution of Lakadong turmeric in the golden milk product system. Needful

Table 8.1: Moisture content (%) data of fresh, tray dried and refractance window dried Lakadong turmeric slice samples.

| S. No. | Sample form | Drying time | Lakadong variety | Local variety |
|--------|-------------|-------------|------------------|---------------|
| 1. | Fresh | - | 86.00 | 87.00 |
| 2. | Tray dried | 8.0 h | 4.60 | 2.90 |
| 3. | RW dried | 1.5 h | 5.50 | 4.00 |

* All standard deviations were in the range 0.1 – 0.2.

characterization of the best constituted golden milk product was addressed in terms of its nutritional characteristics.

8.2 Moisture Content of Turmeric Powder Product

The MC of fresh Lakadong turmeric is 86 %. This high MC of the sample necessitates upon appropriate drying technologies for the minimal time duration based MC reduction in the Lakadong turmeric sample. Table 8.1 presents the variations in MC of fresh, tray dried (60 °C) and RW dried (95 °C) turmeric slices. The MC for 1 mm thick tray dried and RW dried samples were 4.60 and 5.50 % respectively.

The Table 8.1 depicts minimal variation of MC among corresponding samples of turmeric variety. Also, the time required has been significantly lower for the refractance window drying (RWD) (1.5 h) in comparison with tray drying (8 h). Thus, less time is required for the RWD to achieved the desired MC of less than 8 % for good shelf life (Mondal et al., 2019). The RWD required significantly lower drying time due to the transfer of heat between moist turmeric slice and mylar film is through the conduction mode being facilitated through the contact area of the slice and the heated mylar film. Thereby, in addition to convection, conduction and radiation have been effective modes for the water molecules transport in the material and hence efficient moisture evaporation (Talukdar and Uppaluri, 2020).

Table 8.2: Total phenolic content (mg GAE/g sample) data of fresh, tray dried and refractance window dried Lakadong turmeric slice samples.

| S. No. | Sample form | Lakadong variety | Local variety |
|--------|-------------|------------------|---------------|
| 1. | Fresh | 90.74 | 81.52 |
| 2. | Tray dried | 156.81 | 140.21 |
| 3. | RW dried | 207.60 | 189.76 |

* All standard deviations were in the range 2 – 3.

8.3 Total Phenolic Content of Turmeric Powder Product

Tubers and vegetables possess high TPC. The TPC of fresh, tray dried and RW dried Lakadong turmeric slices have been presented in Table 8.2 along with the corresponding values of the local turmeric variety. The TPC for fresh Lakadong turmeric slice has been 90.74 mg GAE/g sample. However, for tray dried and RW dried Lakadong turmeric, it has been elevated to 156.81 and 207.60 mg GAE/g sample respectively. The loss of MC in the dried sample lead to the enhancement in the increase concentration of TPC in dried Lakadong turmeric sample.

From the Table 8.2, it can be observed that the highest TPC has been for the RW dried Lakadong turmeric sample. Also, the Lakadong turmeric possessed marginally higher TPC than that of local turmeric variety and with the RWD process. The high retention of total phenolic compound in slice sample has been due to the RWD technique that facilitates very fast heating of the wet sample. Thereby, enhanced release of phenolic compounds that are bound to the cellular matrix of dried material has been customized. In the RWD, the loss of moisture is very fast during initial drying time phase. Thereby, the partial pressure of oxygen near dried sample gets reduced due to higher vapour pressure being created at the local environment due to moisture evaporation. Such a condition impedes oxidation of TPC causing compounds that are more readily available due to the rapid heating process. Thereby, phenolic compounds do get retained after the RWD process (Hernandez Santos et al., 2016).

Table 8.3: Total flavonoid content (mg quercetin/g sample) data of fresh, tray dried and refractance window dried Lakadong turmeric slice samples.

| S. No. | Sample form | Lakadong variety | Local variety |
|--------|-------------|------------------|---------------|
| 1. | Fresh | 30.19 | 17.41 |
| 2. | Tray dried | 139.28 | 122.90 |
| 3. | RW dried | 173.84 | 160.96 |

* All standard deviations were in the range 4 – 5.

8.4 Total Flavonoid Content of Turmeric Powder Product

Table 8.3 depicts the retention of TFC in Lakadong and local variety turmeric slice along with tray and RW drying methods. The TFC for tray and RW dried Lakadong turmeric slices have been 139.28 and 173.84 mg quercetin/g sample respectively. However, for the fresh sample it was 30.19 mg quercetin/g sample. For the local turmeric variety, the TFC values have been marginally less and were 17.41 (fresh), 122.90 (tray dried) and 160.96 (RW dried) mg quercetin/g sample. After both drying processes, due to MC loss, a significant enhancement in TFC has been recorded. The RWD method has been able to retain higher TFC in both varieties. This is due to the fast drying rate that allows quick heating of the sample and in a short duration. Higher temperature and short drying time together reduce the oxidation of flavonoids and thereby enhance the release of flavonoids from the material. This eventually allows higher TFC in the sample (Rababah et al., 2015).

8.5 Anti-oxidant Activity of Turmeric Powder Product

An important parameter to assess upon the quality of the food product is its AA. The higher the retention of anti-oxidant, the higher is the quality of the food product. Table 8.4 summarizes relevant data and infers that for Lakadong sliced turmeric variety, the AA values have been 29.10, 83.47 and 92.50 % for fresh, tray dried and RW dried respectively. Corresponding values for local variety have been 27.27, 80.00 and 89.10 %. Thus, the drying process significantly enhanced the AA of the turmeric. Among all samples the Lakadong turmeric sample obtained through the RWD at 95 °C provided highest AA. Also, the corresponding alteration between tray and RW dried samples have not been significant.

Table 8.4: Anti-oxidant activity (%) data of fresh, tray dried and refractance window dried Lakadong turmeric slice samples.

| S. No. | Sample form | Lakadong variety | Local variety |
|--------|-------------|------------------|---------------|
| 1. | Fresh | 29.10 | 27.27 |
| 2. | Tray dried | 83.47 | 80.00 |
| 3. | RW dried | 92.50 | 89.10 |

* All standard deviations were in the range 1 – 3.

The heated air during tray drying exposed the dried material to undergo oxidation. This led to a reduction in AA. However, during RWD, the deterioration of anti-oxidant components got reduced due to the process enabling moisture removal at a very fast rate and in less time. Thereby, the process prevented the oxidation of phenolic anti-oxidant from the dried material due to the combination of higher temperature and lesser drying time (Hernandez Santos et al., 2016). Also, higher flavonoid and phenolic content as well enhance the anti-oxidant activity of the dried material (Delma and Verma, 2012).

8.6 Curcumin Content of Turmeric Powder Product

Curcumin is a polyphenolic compound and possesses lipophilic nature. Thereby, it imparts colour to the turmeric. The CC in Lakadong sliced turmeric sample for fresh, tray dried, RW dried cases have been 1.01, 5.78 and 7.10 % w/w respectively (Table 8.5). Corresponding values for the local variety the CC have been 0.73, 3.63 and 4.83 % w/w. For the same quantity of the dried material, the drying process enhanced the percentage of CC in the dried sample in comparison with the fresh sample. From the data, it is apparent that the RW dried Lakadong slice sample possessed highest CC. Also, for all cases, the CC in Lakadong variety has been significantly high. The difference in the quantitative retention of CC through the RWD has been significantly higher in comparison to the quantitative differences for the TPC, TFC and AA. Thus, the RWD process turmeric slice offers best CC for product development.

Table 8.5: Curcumin content (% w/w) data of fresh, tray dried and refractance window dried Lakadong turmeric slice samples.

| S. No. | Sample form | Lakadong variety | Local variety |
|--------|-------------|------------------|---------------|
| 1. | Fresh | 1.01 | 0.73 |
| 2. | Tray dried | 5.78 | 3.63 |
| 3. | RW dried | 7.10 | 4.83 |

* All standard deviations were in the range 0.1 – 0.2.

Table 8.6: Colour indices data of fresh, tray dried and refractance window dried Lakadong turmeric slice samples.

| S. No. | Sample form | Lakadong | | | Local | | |
|--------|-------------|----------|----------|----------|----------|----------|----------|
| | | <i>L</i> | <i>a</i> | <i>b</i> | <i>L</i> | <i>a</i> | <i>b</i> |
| 1. | Fresh | 67.02 | 49.12 | 77.83 | 63.67 | 43.07 | 75.12 |
| 2. | Tray dried | 44.12 | 23.25 | 50.62 | 38.57 | 19.98 | 36.72 |
| 3. | RW dried | 59.15 | 35.58 | 69.74 | 56.67 | 31.05 | 62.13 |

* All standard deviations for *L*, *a* and *b* were in the range of 1 – 2, 2 – 3 and 1 – 3 respectively.

8.7 Colour Indices of Turmeric Powder Product

Colour is an important parameter in the characterization of a food product. The higher the colour factor of a product, the greater is its acceptability by consumer. The fresh Lakadong turmeric possessed *L*, *a* and *b* parameters values of 67.02, 49.12 and 77.83 respectively. Corresponding values for the local variety have been 63.67, 43.07 and 75.12.

With *L*, *a* and *b* parameter values as 59.15, 35.58 and 69.74 respectively, the highest colour retention has been achieved with the RWD process (Table 8.6). The table, also affirmed that the Lakadong variety of turmeric possessed marginally better colour indices than that of the local variety. Such high colour characteristics of the sample could be due to the presence of high CC in the Lakadong variety in comparison to the local turmeric variety. Also, among RWD and tray drying, the RWD gave better colour retention in the dried turmeric sample.

8.8 Statistical Analysis

Tables 8.7 (a – f) presents one-way analysis of variance (ANOVA) results for various cases. The one-way ANOVA affirmed that beyond 95 % confidence interval level, no statistical differences existed for all cases. To do so, each dried sample was subjected to a minimum of three trails for all cases. It has been analysed that for all sample responses (MC, AA, TPC, TFC, CC and *L* values of colour indices), high F-values and low p-values (<0.0001) have been obtained. These findings affirm upon the greater significance of the conducted experiments.

Table 8.7: ANOVA based statistical data for various cases of Lakadong turmeric samples (a) moisture content, (b) total phenolic content, (c) total flavonoid content, (d) anti-oxidant activity, (e) curcumin content and (f) colour index (*L* values).

(a)

| S. No. | | DF | Sum of Squares | Mean Square | F Value | Prob>F |
|--------|-------|----|----------------|-------------|----------|-----------|
| 1. | Model | 2 | 13738.62 | 6869.31 | 12389.54 | < 0.00005 |
| 2. | Error | 6 | 3.32 | 4.05111 | | |
| 3. | Total | 8 | 13741.95 | | | |

(b)

| S. No. | | DF | Sum of Squares | Mean Square | F Value | Prob>F |
|--------|-------|----|----------------|-------------|---------|-----------|
| 1. | Model | 2 | 20466.83 | 10233.41 | 4166.58 | < 0.00005 |
| 2. | Error | 6 | 14.73 | 2.45 | | |
| 3. | Total | 8 | 20481.56 | | | |

(c)

| S. No. | | DF | Sum of Squares | Mean Square | F Value | Prob>F |
|--------|-------|----|----------------|-------------|---------|-----------|
| 1. | Model | 2 | 33726.23 | 16864.61 | 5438.64 | < 0.00005 |
| 2. | Error | 6 | 18.60 | 3.10 | | |
| 3. | Total | 8 | 33747.84 | | | |

(d)

| S. No. | | DF | Sum of Squares | Mean Square | F Value | Prob>F |
|--------|-------|----|----------------|-------------|---------|-----------|
| 1. | Model | 2 | 6824.51 | 3412.25 | 1046.74 | < 0.00005 |
| 2. | Error | 6 | 19.55 | 3.25 | | |
| 3. | Total | 8 | 6844.07 | | | |

(e)

| S. No. | | DF | Sum of Squares | Mean Square | F Value | Prob>F |
|--------|-------|----|----------------|-------------|---------|-----------|
| 1. | Model | 2 | 55.21 | 27.60 | 1951.72 | < 0.00005 |
| 2. | Error | 6 | 0.084 | 0.01 | | |
| 3. | Total | 8 | 55.29 | | | |

(f)

| S. No. | | DF | Sum of Squares | Mean Square | F Value | Prob>F |
|--------|-------|----|----------------|-------------|---------|-----------|
| 1. | Model | 2 | 566.22 | 283.11 | 38.60 | < 0.00005 |
| 2. | Error | 6 | 44 | 7.33 | | |
| 3. | Total | 8 | 610.22 | | | |

8.9 Fourier Transform Infrared Spectroscopy Analysis

The FTIR analyses has been carried out to assess upon the functional group alterations during the tray and RW drying of Lakadong samples. Fig. 8.1 depicts the FTIR spectra for the fresh, tray dried and RW dried Lakadong turmeric samples along with the tray dried and RW dried local turmeric samples. The Fig 8.1 confirmed that there have not been significant alterations in the peaks for various functional groups. The obtained FTIR spectra of Lakadong turmeric matched with that of the local variety turmeric and with those reported previously in the relevant prior art (Safie et al., 2015). With the effective removal of moisture content, the bioactive compounds became prominent due to the application of heat based drying. Such an effect affirmed the activation of the bioactive compounds. Due to this effect, peak shift occurred in the wavenumber range of 1000 to 1500 cm^{-1} in the dried turmeric sample. However, this was not the case for the fresh sample case (Nandiyanto et al., 2019).

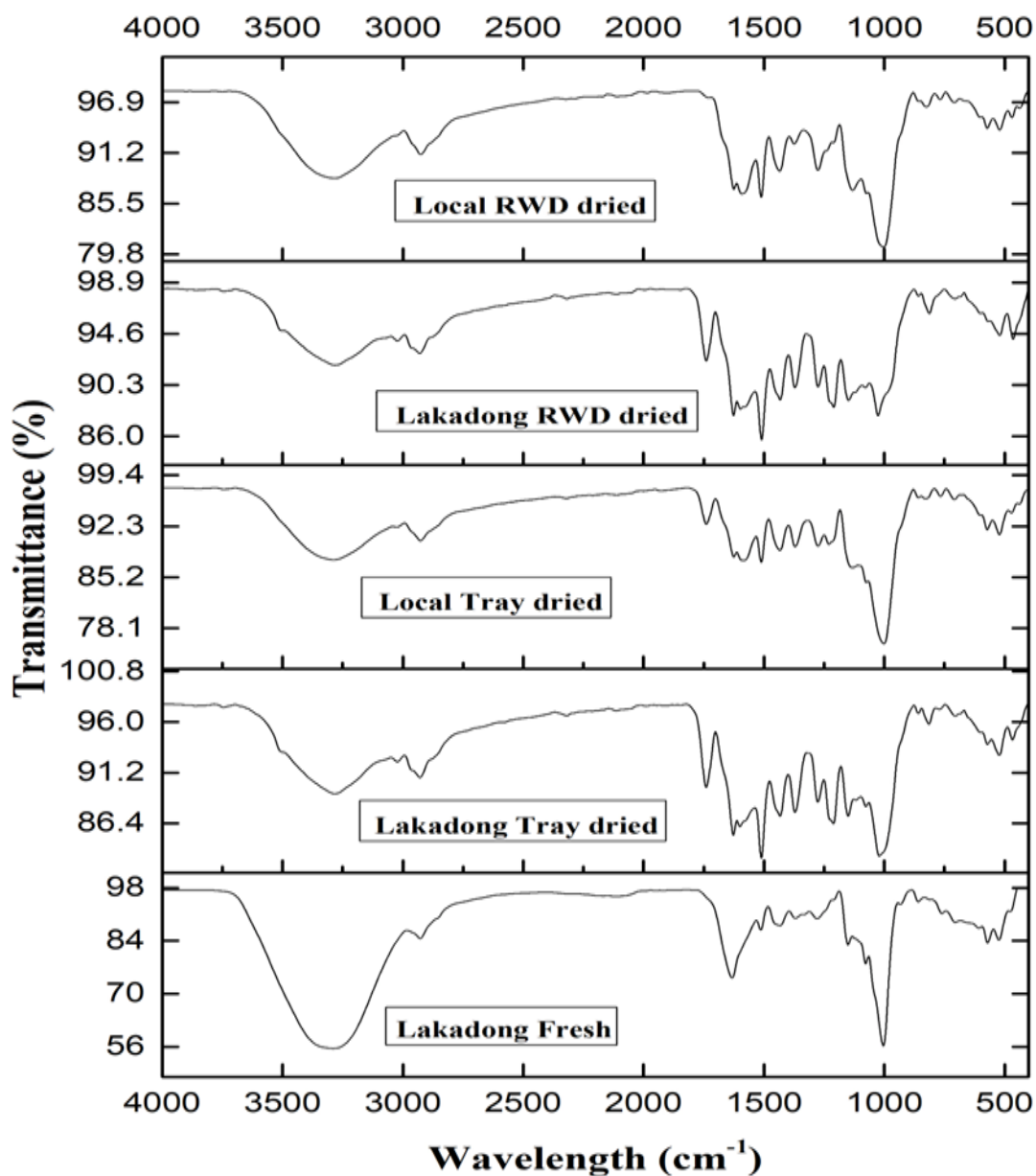


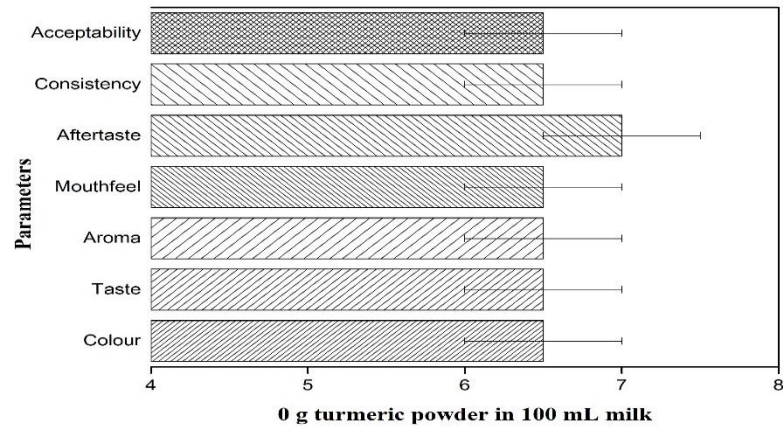
Figure 8.1: FTIR spectra of fresh, tray and refractance window dried Lakadong turmeric powder samples.

8.10 Sensory Data of Turmeric based Golden Milk Product

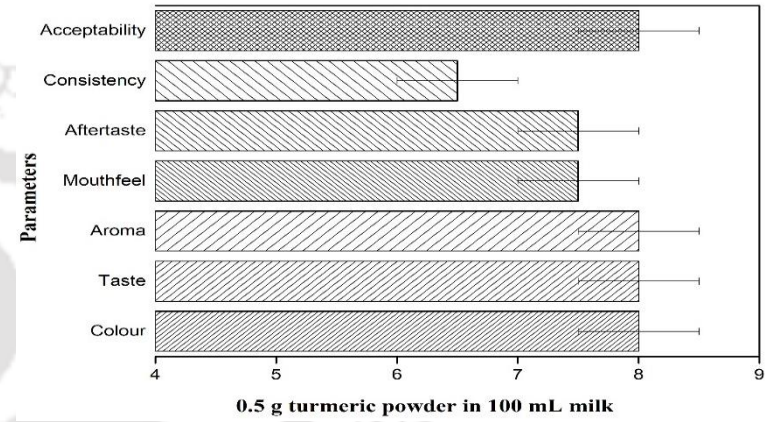
From the product characterizations, it can be analysed that the RW dried Lakadong sample possessed better characterization indices in comparison with the tray dried sample. Hence, a sensory study was carried out for RW dried Lakadong turmeric powder and was compared with the sensory data reported earlier for the local variety. During sensory study, 0, 0.50, 1.0, 1.50 and 2.0 g of RW dried

Lakadong turmeric sample were mixed with 100 mL of milk (Amul Taaza toned milk). Thereby, the sensory study was conducted for the evaluation of parameters such as colour and appearance, taste, aroma, mouthfeel, aftertaste, consistency and overall acceptability with a 9 point hedonic scale (Hingne et al., 2020). Fig. 8.2 illustrated plots for the sensory scores of the Lakadong turmeric based golden milk product.

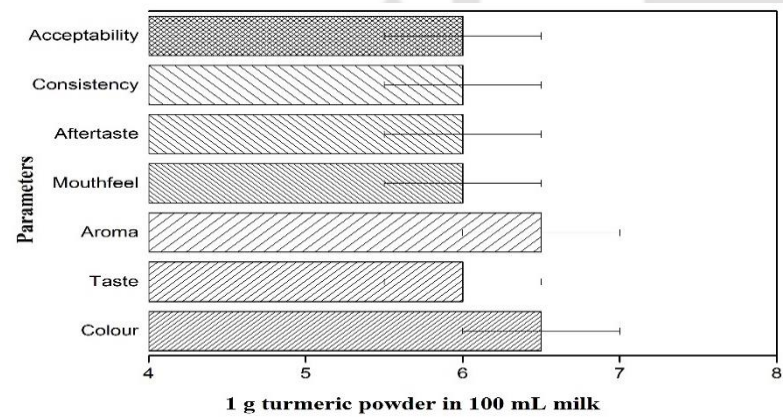
From the Fig. 8.2 (a – e) it can be seen that the among all RW dried the samples 2.0 g Lakadong turmeric powder in 100 mL milk was the least acceptable and the 0.50 g turmeric in 100 mL was the most acceptable. The scores for all the parameters were high for 0.50 g of the turmeric in 100 mL milk case. These were 8 for colour and appearance, taste, aroma and overall acceptability, 7.5 for mouthfeel and aftertaste and 6.5 for consistency. The consistency altered for all cases with a score of 5 – 6.5. The score of colour and appearance, taste, aroma, mouthfeel, aftertaste and overall acceptability varied significantly with the concentration of turmeric powder in 100 mL milk. In comparison to the results obtained for the local variety of turmeric and for RW dried samples that inferred 1.0 g turmeric to be best and 2.0 g turmeric to be the least acceptable in golden milk formulation, the Lakadong turmeric based golden milk had lower content (0.50 g) for the best sensory scores. This is due to higher curcumin content in the RW dried Lakadong turmeric powder in comparison to the RW dried local variety of the turmeric powder.



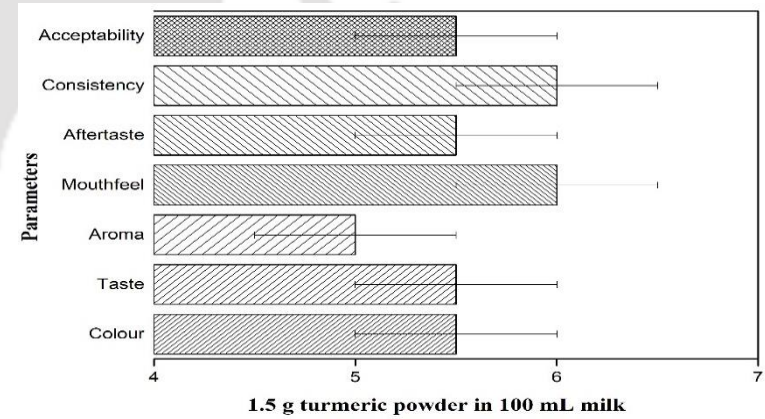
(a)



(b)



(c)



(d)

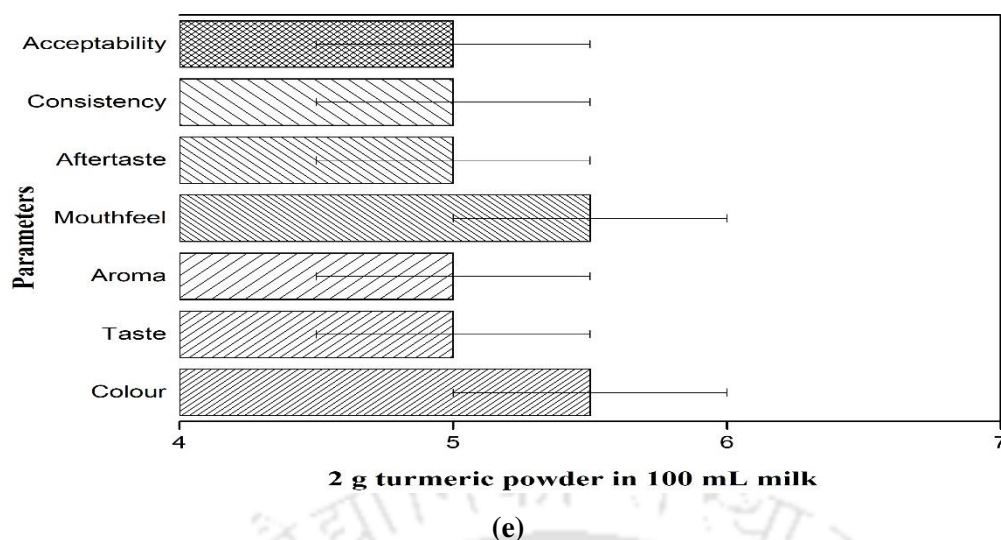


Figure 8.2: Bar charts depicting the sensory characteristics of refractance window dried Lakadong turmeric powder based golden milk products.

8.11 Nutritional Characteristics of Lakadong Turmeric Based Golden Milk

Product

From the sensory study, it was inferred that 0.50 g turmeric powder in 100 mL milk has been the most accepted golden milk formulation. Hence, such optimality formulated milk product has been evaluated for its TPC, TFC, AA and CC. The obtained results were 921.74 mg GAE/100 mL sample, 843.75 mg quercetin/100 mL sample, 28.70 % and 6.28 % w/w for TPC, TFC, AA and CC respectively. Corresponding values for the local variety based best formulation were 876.21 mg GAE/100 mL sample, 784.61 mg quercetin/100 mL sample, 24.50 % and 4.20 % w/w curcumin content for TPC, TFC, AA and CC respectively. From the results, it can be seen that except for CC, the Lakadong variety based formulation had marginally better results than that of the local variety based formulation. This could be due to the marginally better result obtained that were achieved for the Lakadong turmeric case in comparison with the local turmeric case and with the RWD for both cases.

8.12 Summary

This chapter provided very useful insights with respect to the tray and RW dried Lakadong turmeric samples. Firstly, the MC has been similar (86 – 87 %) for both Lakadong and local turmeric varieties. Secondly, the AA, TFC and TPC have been moderately higher for the Lakadong variety in comparison with the local variety and for the RWD. Thirdly, the CC has been significantly higher and best for the Lakadong variety (7.10 % w/w) but not the local variety (4.83 % w/w) and with the RWD. Fourthly, the colour indices of Lakadong variety have been marginally higher and this is due to its higher CC. Fifthly, among all cases the RWD performed better than the tray drying process. Sixthly, the FTIR analysis confirmed that there has not been any significant alteration in the peaks of the prominent functional group for both turmeric varieties and after tray and RW drying. Seventhly, the RW dried Lakadong turmeric powder at its constitution of 0.50 g in 100 mL milk provided best sensory scores. Finally, among Lakadong and local turmeric based milk products, the former possessed marginally higher TPC, TFC, AA but high CC and can henceforth recommended as the best choice. However, cost of the Lakadong variety needs to be assessed to make the golden milk formulation as a competitive product.





Chapter 9:
Conclusions and Future Work



Conclusions and Future Work

In this chapter, section 9.1 details upon all the important conclusions of the conducted research work for the fulfillment of the thesis objectives. Section 9.2 provides possible directions for the future work in the chosen field of research.

9.1 Conclusions

The experimental investigations have been effective to address and resolve all set objectives of the thesis. Thereby, a brief account of these have been presented in the following sub-sections.

9.1.1 Techno-economic Efficacy of Refractance Window Dried *Curcuma longa*

- Among, tray, oven and RW drying processes, the RWD process provided higher nutritional retention in turmeric (*Curcuma longa*) in a short span of drying time (1 h).
- Among slice and paste sample of the turmeric, the slice sample possessed higher nutritional properties.
- The RWD at 60 °C enabled 4.20 % wb moisture content, 161.95 mg GAE/g sample TPC, 140.16 mg quercetin/g sample TFC, 82.93 % AA, 42.88 (*L* values) colour index and 4.21 % w/w CC in the RW dried turmeric sample.
- Sensitivity analysis of the experiments affirmed that the RWD process had higher percentage alteration with respect to AA, TPC, TFC, CC and colour indices.
- Among, tray, oven and RW drying processes, least energy consumption was found in the RWD process.

- Among, tray, oven and RW drying processes, the conceptual annualized laboratory scale processing cost of the RWD process (operated at 60 °C) has been lowest. These were Rs. 636.59 and Rs. 367.97 per kg of dried sample product for slice and paste cases respectively

9.1.2 Process and Product Characteristics of Refractance Window Dried *Curcuma longa*

- Among various process parameters, the drying characteristics were critically influenced with the water bath temperature and drying time but not with the mylar film thickness.
- To represent drying kinetics, the Singh et al., (2014) model has been the best fitted model. Corresponding diffusivity altered from 3.71×10^{-11} to 1.39×10^{-10} m²/s for a bath temperature variation from 65 – 95 °C. Incidentally, activation energy altered marginally from 52.57 – 49.43 kJ.
- For the turmeric, the best nutritional properties have been achieved at a water bath temperature of 95 °C and at all mylar film thickness values.
- The optimal mylar film thickness (250 µm) provided marginally better results in comparison with those obtained with 150 and 350 µm mylar film thickness values.
- The turmeric powder prepared with the water bath temperature range of 65 – 95 °C did not indicate shift or alteration of the functional group related peaks in the FTIR spectra.

9.1.3 Response Surface Methodology Based Optimality of Refractance Window Drying Process Parameters

- Water bath temperature and drying time but air velocity critically influenced the nutritional characteristics of the RW dried turmeric samples.
- With high F and low p values, the quadratic model has been the most appropriate model to represent all responses namely AA, TPC, TFC, CC, MC and colour indices of the RW dried samples.

- The optimal numerical data set for the RSM has been affirmed to be 95 °C bath temperature, 75 min drying time and 0.76 m/s air-velocity, 90.52 % (AA), 188.22 mg GAE/g sample (TPC), 158.65 mg quercetin/g sample (TFC), 4.80 % w/w (CC), 3.67 % MC and 54.87 (*L* values) colour index.

9.1.4 Fortification of Refractance Window Dried Turmeric Powder and its Associated Studies

- Fortification with 20 mg folic acid/100 g turmeric and 20 mg NaFeEDTA/100 g turmeric has been effective to provide promising characteristics in the fortified turmeric sample.
- For all cases namely unfortified sample and the fortified turmeric samples, the bulk density did not vary significantly.
- For all cases, the solubility, swelling power and water binding capacity have been good.
- The hygroscopicity and dispersion time has been low for fortified and unfortified turmeric powder samples.
- The colour indices of RW dried turmeric, folic acid fortified and NaFeEDTA fortified turmeric powder were almost similar and no shift has been observed for the functional groups during FTIR analysis.
- The particle size distribution and FESEM images have been similar for the fortified and unfortified turmeric powder samples.
- The TGA, DSC and XRD analyses inferred that the RW dried turmeric powder and folic acid fortified turmeric powder exhibited similar trends due to fortificants being organic and amorphous in nature. However, the NaFeEDTA fortified turmeric powder sample exhibited crystalline nature due to the crystallized iron in the NaFeEDTA form.
- The in-vitro digestion analysis affirmed that upon fortification, the bio-availability of folic acid and NaFeEDTA content increased in the respective samples.

9.1.5 Storage and Associated Studies of Unfortified and Fortified *Curcuma longa* Powder and Golden Milk Products Samples

- The GAB model has been the best fitted model to represent moisture isotherms (R^2 value of 0.99) of unfortified and fortified turmeric powder sample with the packaging material permeability of 1.12×10^{-7} kg/m²dayPa, the shelf life of RW dried turmeric powder, folic acid fortified and NaFeEDTA fortified turmeric powder samples have been 184, 187 and 183 days respectively.
- From sensory analysis, 1 g turmeric powder in 100 mL milk has been inferred to be the best constitution for the golden milk product.
- The TPC, TFC, AA and CC of golden milk product have been 876.21 mg GAE/100 mL, 784.61 mg quercetin/100 mL 24.50 % antioxidant activity and 4.20 % w/w curcumin content respectively. The presence of folic acid and NaFeEDTA did not alter the nutritional characteristics of the golden milk product. Also, based on nutritional characteristics, it can be concluded that the unrefrigerated golden milk was viable for 1 day and refrigerated golden milk was viable for 2 days.
- For a storage period of 6 months in zipper pouch packaging material various physical properties such as colour, bulk density, solubility, swelling power, water binding capacity, dispersion time and hygroscopicity have been almost similar for the RW dried turmeric powder, folic acid fortified turmeric powder and NaFeEDTA fortified turmeric powder sample. However, excepting the CC, the nutritional properties namely AA, TPC and TFC reduced during the later stage of the storage tenure. This is due to the reason that the CC is more stable in conjunction with other mentioned responses.

9.1.6 Nutritional Characteristic of Fresh, Tray and Refractance Window dried Lakadong Turmeric Powder and Golden Milk Product

- The RW dried Lakadong turmeric slices possessed better nutritional characteristics in comparison to the tray dried sample, Thereby, the sample possessed 207.60 mg GAE/g

sample (TPC), 173.84 mg quercetin/g sample (TFC), 94.50 % (AA) and 7.10 % w/w (CC).

Also, the MC has been 5.50 % and the colour index has been 59.50 (*L value*). These values have been higher than those obtained for the local turmeric variety based RW dried powder.

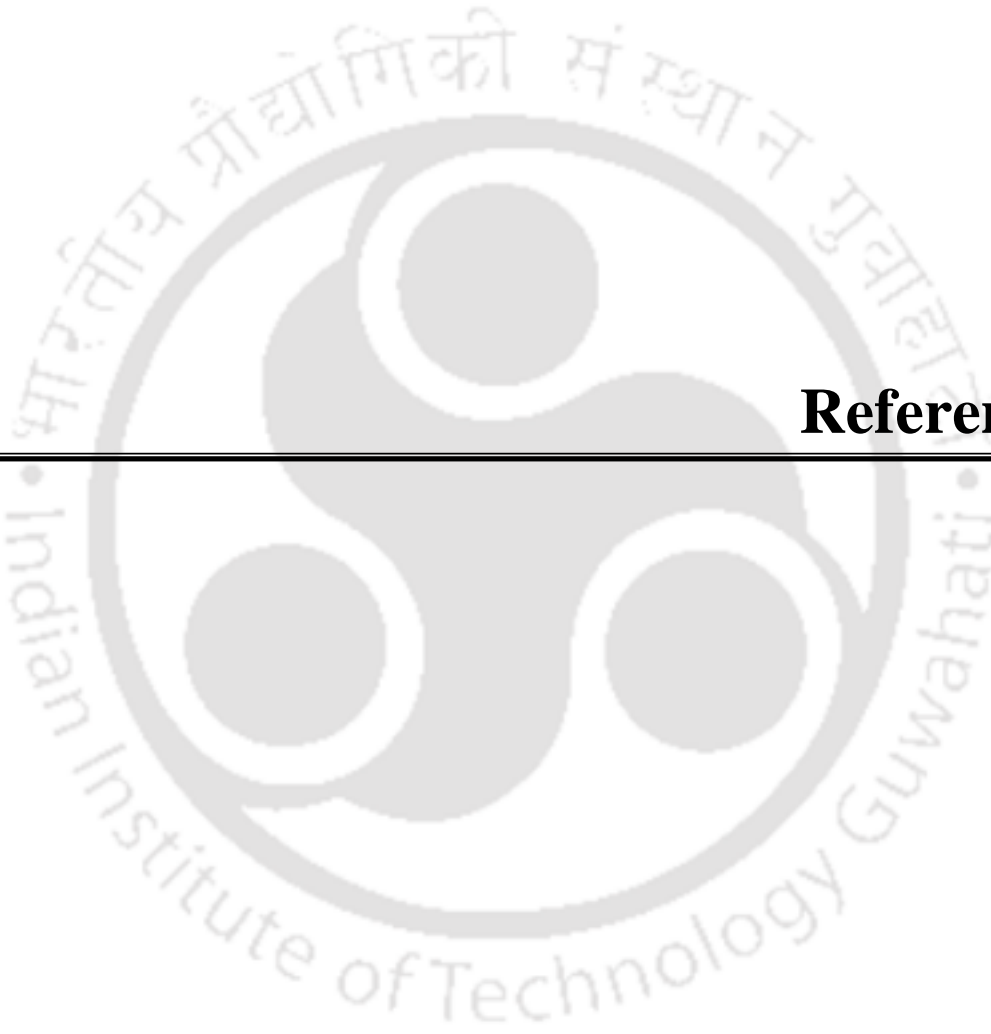
- The FTIR analyses have been similar for both tray and RW dried turmeric powder sample and do not involve a shift in the functional groups.
- Sensory analysis affirmed that 0.50 g of Lakadong turmeric powder in 100 mL milk has been the optimal formulation.
- The TPC, TFC, AA and CC of Lakadong turmeric based golden milk are 921.74 mg GAE/mL sample (TPC), 843.75 mg quercetin/mL sample (TFC), 28.70 % (AA) and 6.28 % w/w (CC). All these have been higher than those obtained for the local turmeric variety.

9.2 Future Research Directions

Based on RWD process based product development approach, the following can be listed for possible exploration towards further research in the future.

- Fortification research with alternate minerals and micronutrients such as zinc, calcium, vitamin A etc. and relevant associated studies.
- Storage study with low-cost golden milk product packaging technology.
- Continuous process scheme can be developed, targeted and optimized for the large scale RWD of turmeric samples and in pilot scale mode.
- RWD in conjunction with microwave drying can be studied to further reduce the drying time to 30 min duration in the entire hybrid process scheme.
- The RWD of sliced leafy and non-leafy vegetables samples can be targeted along with formulation research in ready to eat/cook products.
- Other liquid product systems such as purees, milk slurry products with RW dried turmeric such as soups, cookies and chips can be targeted for nutritional, sensory and storage characteristics.





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Publications



1. Publications

- a. Talukdar, P. & Uppaluri, R. V. Process and Product Characteristics of Refractance Window Dried *Curcuma longa* (2021). *Journal of Food Science*, 86 (2), pp. 443-453.
- b. Talukdar, P. & Uppaluri, R. V. Techno-economic Efficacy of Refractance Window Dried *Curcuma Longa* (2023). *Agro and Food Processing Technologies*, *Nature – Springer*, pp. 81 – 98.

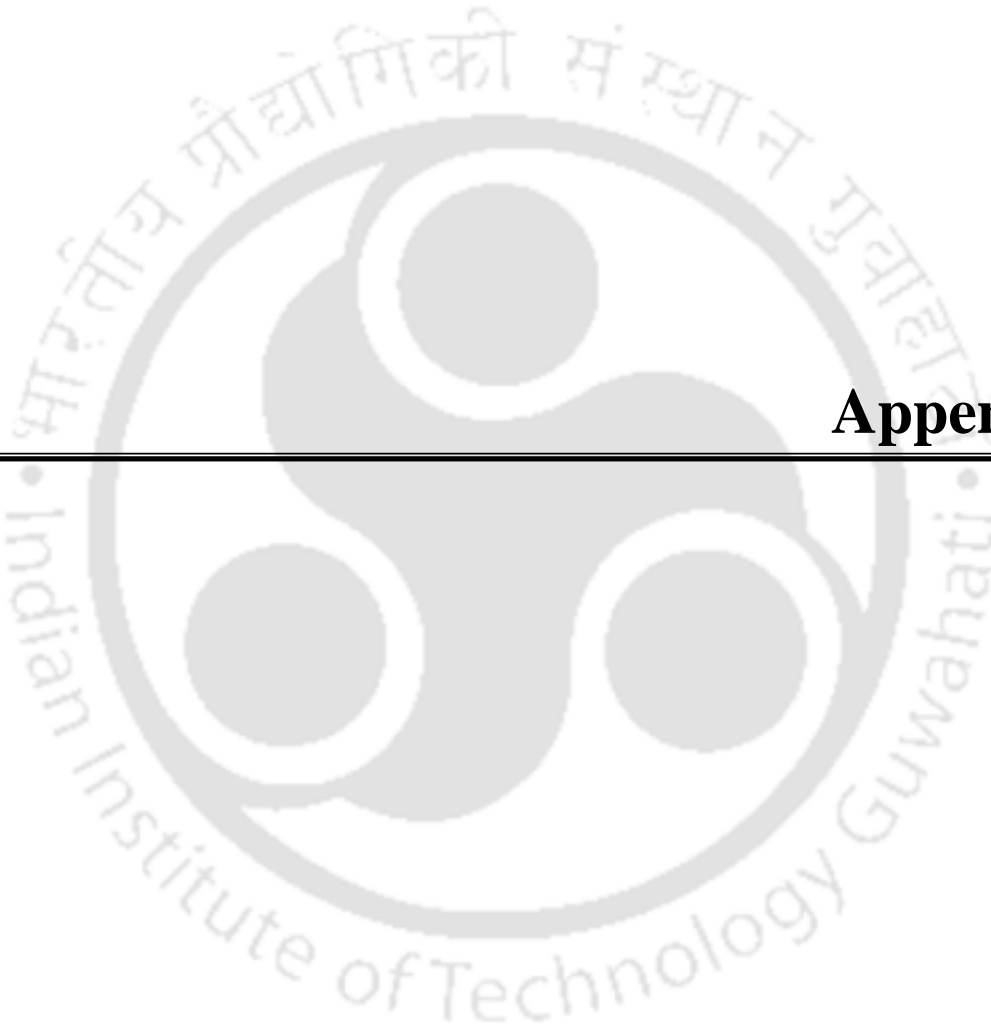
2. Articles Submitted

- a. Talukdar, P. & Uppaluri, R. V. Sensitivity analysis and optimality of process-product characteristics of refractance window dried *Curcuma longa*. *Applied Research*.

3. Articles to be submitted

- a. Talukdar, P. & Uppaluri, R. V. Fortification of RW Dried Turmeric Powder and Its Associated Studies
- b. Talukdar, P. & Uppaluri, R. V. Storage, Nutritional and Sensory Characteristics of RW dried and Fortified Turmeric Powder and Golden Milk Products
- c. Talukdar, P. & Uppaluri, R. V Nutritional Characteristic of Fresh, Tray and RW dried Lakadong Turmeric and Golden Milk Product System





Appendix



Appendix A: Calibration Curves for Determination of Total Phenolic Content, Total Flavonoid Content, Curcumin Content, Folic Acid Content

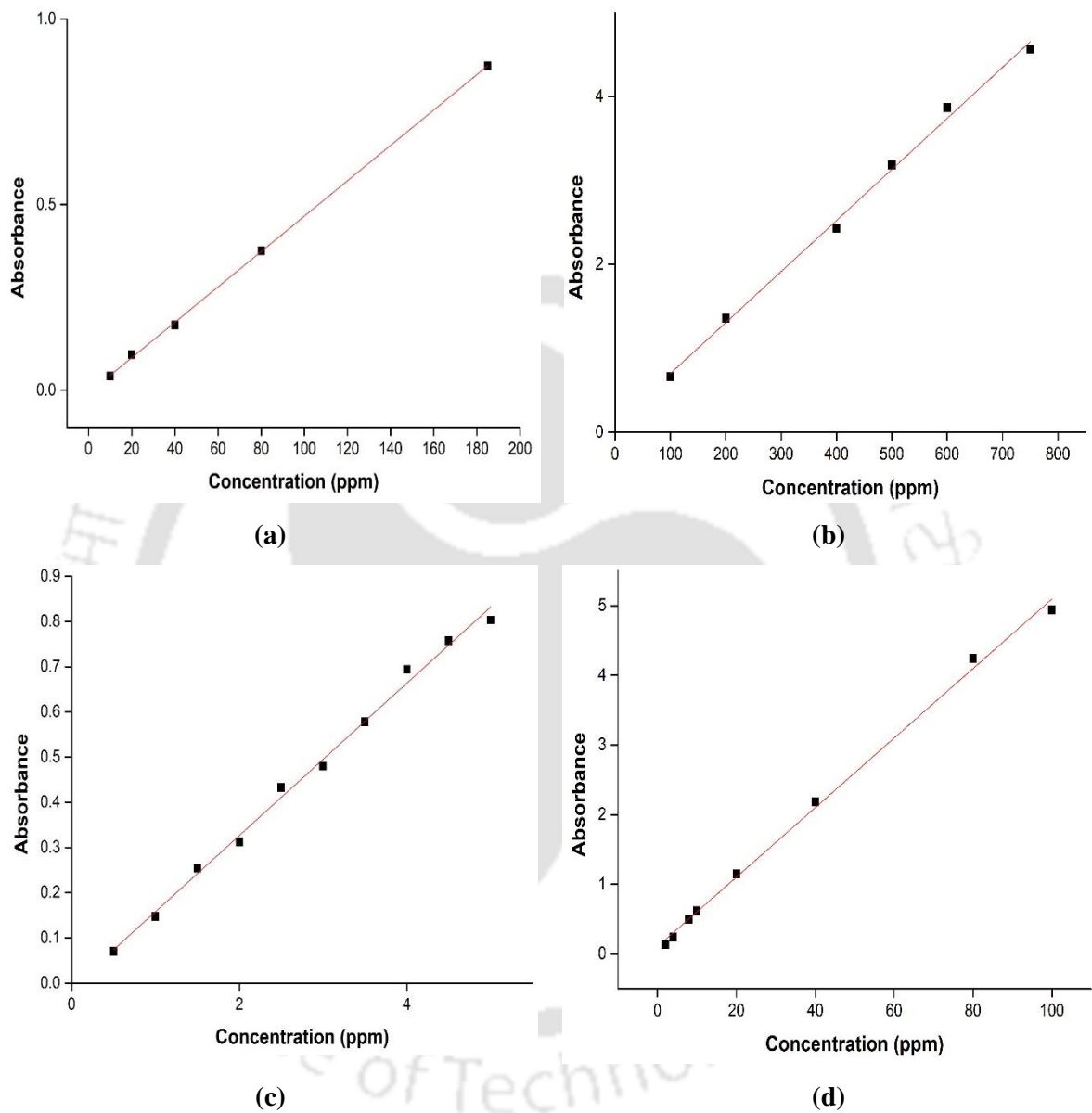


Fig A1: Calibration curves for determination of (a) TPC, (b) TFC (c) CC and (d) Folic acid using UV-Spectrophotometer

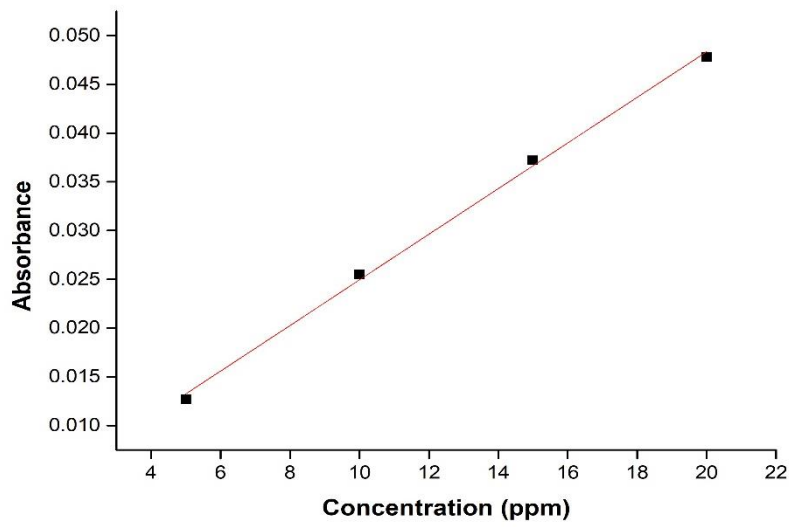


Fig A2: Calibration curves for determination of iron using AAS.



Appendix B: Summary of Conceptual Cost and Economic Assessment Model for Laboratory Scale Turmeric Oven, Tray and Refractance Window Drying Methods

A) Parameters

EQUIPMENT COST

| Dryer type | Hot air oven | Tray dryer | Refractance window drying process |
|------------------------|--------------|------------|-----------------------------------|
| Cost of equipment (Rs) | 40,000 | 35,000 | 29510 |
| Life span (years) | 10 | 10 | 10 |
| Personal Interest (%) | 10.5 | 10.5 | 10.5 |
| Loan for year (years) | 5 | 5 | 5 |

| | Slicer | Grinder |
|-------------------|--------|---------|
| Cost of equipment | 1799 | 2399 |
| Life span | 10 | 10 |

MAINTENANCE COST 10 % of equipment cost

DEPRECIATION COST 10 % of process cost

MANPOWER COST

Amount/month (Rs) 12,000

ELECTRICITY COST

Power tariff (Rs) 7.25

Working days/year 300

Drying method Hot air drying Tray drying Refractance window drying (RWD)

Power (kW) 2.4 3 2
RWD at 60 °C

Sample form Slice Paste Slice Paste Slice Paste

Time taken/batch 8 4.5 8 4.5 7 3

| | | | | | | |
|-----------------------------|---------|------|---|------|---|-----|
| Batch/day | 2 | 3 | 2 | 3 | 2 | 4 |
| Amount of sample/batch (kg) | 2 | 2 | 2 | 2 | 2 | 2 |
| Normalization factor | 7 | 4.67 | 7 | 4.67 | 7 | 3.5 |
| | Grinder | | | | | |
| Power (kW) | 0.5 | | | | | |
| Time taken/batch (h) | 0.16 | | | | | |

B) Cost model expressions

a. Manpower cost

$$C_{mp} = \text{Per day manpower cost} \times \text{Total number of working days}$$

where, C_{mp} is the Total Manpower Cost

b. Equipment cost

$$C_{eq} = \frac{\text{Equipment price}}{\text{Lifespan of the equipment} \times \text{Annual amount of sample}}$$

where, C_{eq} is the Total Equipment Cost

c. Electricity cost

$$C_{ec} = \text{Equipment power rating} \times \text{Hours of equipment used} \times \text{electricity tariff}$$

where, C_{ec} is the Total Electricity Cost

d. Total cost

$$C_{tc} = \text{Manpower cost} + \text{Equipment cost} + \text{Electricity cost} + \text{Maintenance cost} + \text{Depreciation cost}$$

where, C_{tc} is the Total Cost

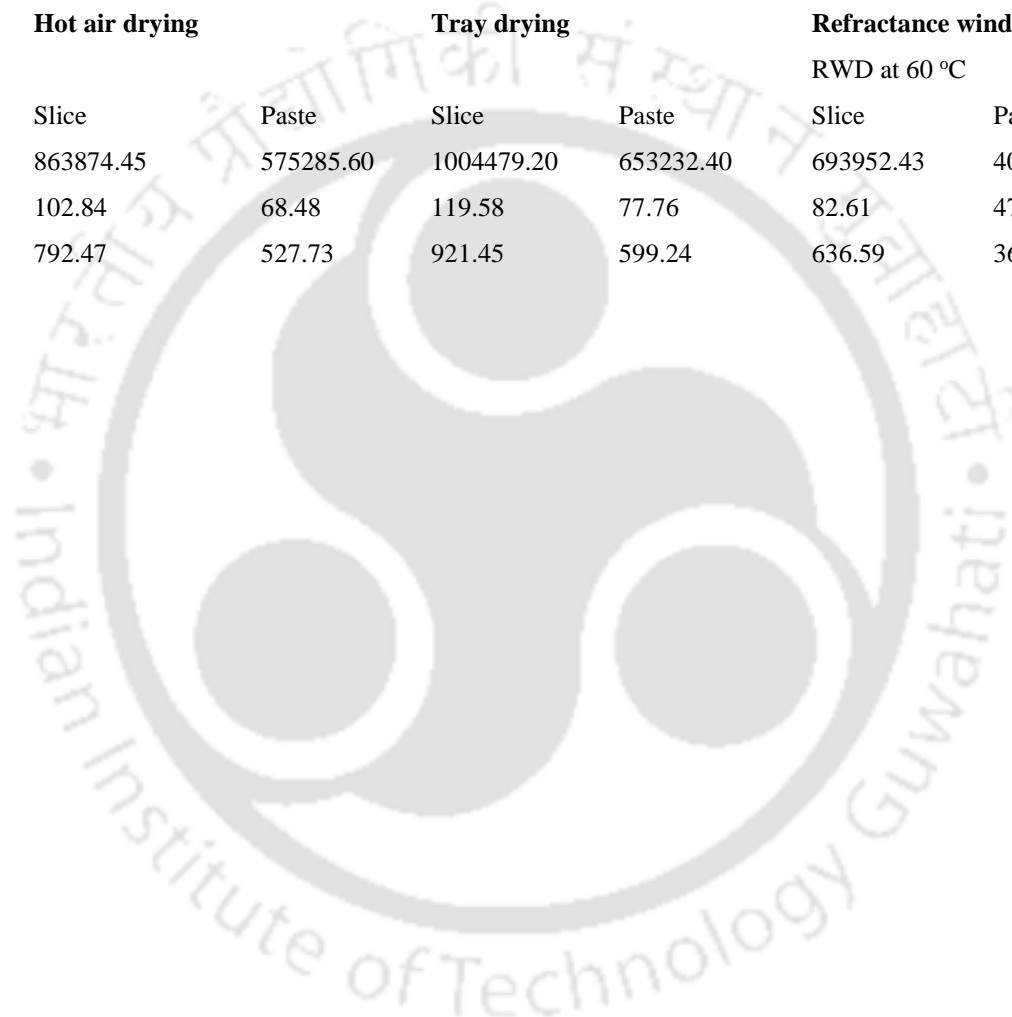
C) Other Parameters

- All processes normalized to 1090 kg of fresh turmeric produced processed in one year.
- Equipment sizing considered using Guthrie's correlation with an exponent factor of 0.65.

- Manpower escalation costs considered with Guthrie's correlation and exponent factor of 0.25.

D) Total annualized costs of laboratory dryer systems (for 1090 kg of fresh turmeric processed per year)

| Drying method | Hot air drying | | Tray drying | | Refractance window drying (RWD) | |
|---------------------------|----------------|-----------|-------------|-----------|---------------------------------|-----------|
| | Slice | Paste | Slice | Paste | RWD at 60 °C | |
| Sample form | | | Slice | Paste | Slice | Paste |
| Total cost (Rs) | 863874.45 | 575285.60 | 1004479.20 | 653232.40 | 693952.43 | 401125.92 |
| Cost/kg fresh sample (Rs) | 102.84 | 68.48 | 119.58 | 77.76 | 82.61 | 47.75 |
| Cost/kg dried sample (Rs) | 792.47 | 527.73 | 921.45 | 599.24 | 636.59 | 367.97 |





Appendix C: Images of Equipment and Samples used in this Ph.D. Studies along with the Products Obtained



Oven dryer



Tray dryer



RWD dryer



Mylar film



Fresh slice



Fresh paste



Sample in mylar film



Dried sample in mylar film



Oven dried slice



Oven dried paste



Tray dried slice



Tray dried paste



RW dried slice



RW dried paste



Fresh local turmeric



Fresh lakadong turmeric



Local turmeric powder



Lakadong turmeric powder



Local turmeric sensory assessment



Lakadong turmeric sensory assessment



Appendix D: Sample Calculation for the Evaluation of Various Parameters

In this section a sample calculation has been presented for the determination of various parameters of RW dried turmeric powders samples.

a) **Yield:** Weight of fresh sample taken (W_f): 9.80 g; Weight of RW dried sample (W_s): 1.31 g

Using equation (12), % Yield = $(1.31/9.80) \times 100 = 13.36 \%$

b) **Rehydration Ratio:** Weight of RW dried sample taken (W_d): 1.5 g; Weight of rehydrated sample (W_r): 8.40 g

Rehydration Ratio = $(8.40/1.50) = 5.60$

c) **Moisture Content:** Weight of fresh sample taken (W_f): 9.80 g; Weight of RW dried sample (W_s): 1.31 g

Using equation (1), % MC = $(9.80 - 1.31/9.80) \times 100 = 86.63 \%$

d) **Anti-oxidant Activity:** Absorbance of control sample (A_c): 0.385; Absorbance of experimental sample (A_s): 0.064

Using equation (2), % AA = $\{(0.385 - 0.064)/0.385\} \times 100 = 82.93 \%$

e) **Total Phenolic Content:** Value of slope (m): 0.0046; Absorbance of experimental sample: 0.745

TPC = $(0.745/0.0046) = 161.95 \text{ mg GAE/g dry samples}$

f) **Total Flavonoid Content:** Value of slope (m): 0.0060; Absorbance of experimental sample: 0.869

TPC = $(0.869/0.0060) = 144.83 \text{ mg quercetin/g dry samples}$

g) **Curcumin Content:** Value of slope (m): 0.165; Absorbance of experimental sample: 0.696

TPC = $(0.696/0.165) = 4.21 \%$ w/w

h) Sensitivity analysis: AA value of dried sample (%): 82.93; AA value of fresh sample (%): 27.26

Using Equation (3), % change = $\{(82.93 - 27.26)/27.26\} = 204.23 \%$ increase

i) **Energy Consumption:** Power value of water bath (kW): 2; Time taken for RW drying (h): 7

Using equation (4), Energy consumption = $2 \times 7 = 14 \text{ kW/h}$

j) **Moisture Diffusivity:** Thickness of sample (2L): 1 mm; $\pi = 3.14$; slope = - 0.0199

$$D \text{ (kJ/mol)} = - \{(-0.0199) \times 4 \times (0.05)^2\} / (3.14)^2 \times 60 \times 10^{-6} = 3.36 \times 10^{-11} \text{ m}^2/\text{s}$$

k) **Activation Energy:** Universal gas constant: 8.314 J/mol K; slope = - 5945.50

$$E_a \text{ (kJ/mol)} = - \{(-5945.50) \times 8.314\} / 1000 = 49.43$$

l) **Solubility (%):** Weight of sample taken (W_s): 0.5 g; Weight of sample residue (W_d): 0.145 g

$$\text{Using Equation (13), } (0.145/0.5) \times 100 = 29 \%$$

m) **Swelling power (g/g):** Weight of dried sediment (W_{sd}): 0.9 g; Weight of sample taken (W_s): 0.5 g

$$\text{Using Equation (14), } (0.9/0.5) = 1.8 \text{ g/g}$$

n) **Water Binding Capacity (%):** Weight of wet sample (M_w): 8.3 g; Weight of dried sample (M_d): 5 g

$$\text{Using Equation (15), } \{(8.3 - 5)/5\} \times 100 = 66 \%$$

n) **Hygroscopicity (%):** Enhancement in powder sample (x): 2.20 g; Powder sample amount being used for the measurement (a_h): 1 g; Water content of the powder being exposed to the humid environment (% wb): 4.2 %

$$\text{Using Equation (16), } \{(3.56 \times 2.56)/1.042\} \times 100 = 8.74 \%$$

