

**Formulation and Characterization of Leafy and
Non-Leafy Vegetables Based Mix Soup
Formulations**

Thesis submitted in partial fulfillment of the
requirements for the degree of

DOCTOR OF PHILOSOPHY

by

Imdadul Hoque Mondal



**School of Agro and Rural Technology
Indian Institute of Technology Guwahati
Guwahati–781039, India
March, 2021**

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Guwahati – 781039, India**

March, 2021





Dedicated

to

my parents and my family

members





Centre for Rural Technology
Indian Institute of Technology Guwahati
Guwahati – 781039, Assam, India

CERTIFICATE

It is certified that the work contained in this thesis entitled “**Formulation and Characterization of Leafy and Non-Leafy Vegetables Based Mix Soup Formulations**” submitted by **Mr. Imdadul Hoque Mondal** for the award of the degree of Doctor of Philosophy has been carried out in the School of Agro and Rural Technology, Indian Institute of Technology Guwahati under our supervision. To the best of our knowledge, the Ph.D. thesis work has not been submitted elsewhere for the award of a degree or diploma.

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DISCLAIMER

The experimental, characterization and optimality data presented in the Ph.D. thesis was performed by me and reported after due verification. To the best of our knowledge, the work summarized in this Ph.D. thesis has not been submitted elsewhere for the award of a degree or diploma.

(Mr. Imdadul Hoque Mondal)



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Abstract

To encourage translational and value added product development of endemic and underutilized horticultural produces, current Ph.D. thesis is targets upon a robust experimental and design methodology to formulate and characterize leafy and non-leafy vegetable based mix soup recipes with high nutritional content in terms of proximate, vitamin C and antioxidant activity characteristics. The targeted leafy and non-leafy vegetables in the thesis includes Kolmou (*Ipomoea aquatica* Forssk.), Pui (*Basella alba*) and Jatialao sak (*Lagenaria siceraria* leaves) and Kolphul (*Musa balbisiana* Colla blossom), Kaskal (*Musa splendida*), green Komora (*Benincasa hispida*) and Posola (*Musa balbisiana* Colla pseudostem), respectively.

After ensuring a critical discussion with respect to available prior art, lacunae, objectives and thesis organization in Chapter 1, the adopted overall research methodology has been presented in Chapter 2 of the thesis. Thereafter, Chapter 3 addresses the comparative efficacy of tray and oven drying processes towards the chosen leafy vegetables in terms of the nutritional characteristics of the dried vegetable samples. Chapters 4 and 5 targets upon the optimization of process-product characteristics of the chosen leafy and non-leafy vegetables, respectively. Both trial and error and statistical design approaches have been addressed in these chapters. Thereafter, Chapters 6 and 7 address the findings associated to the optimal leafy and non-leafy vegetable mix constitution based on nutritional optimization and sensory analysis. Finally, Chapter 8 summarizes the subjective and objective conclusions of the Ph.D. thesis and possible directions for future research.

Further details with respect to the above mentioned chapters of the thesis are as follows. The first chapter targets upon efficacy of intermittent air flow assisted tray drying process and oven drying

process for the evaluation of nutritional characteristics of the dried chosen vegetables. For this purpose, the alternate drying process parameters were kept constant (60 °C and 4 h for chosen leafy vegetables and 60 °C and 10 h for chosen non-leafy vegetables). The process optimization was based on superior combinations of mentioned nutritional characteristics. Accordingly, tray drying process has been evaluated to be superior among tray and oven drying processes.

A critical summary of the adopted research methodology in Chapters 4 and 5 is as follows. The preliminary process parametric optimization of drying temperature and time was targeted for the simultaneous minimization of moisture content and maximization of vitamin C and antioxidant activity as response variables. Thereafter, for the optimal choice set, mentioned proximate characteristics have been evaluated. Also, the trial and error methodology targeted drying kinetics studies so as to determine best fit kinetic model, moisture diffusivity and activation energy. The statistical design based parametric optimization was addressed through the analysis of variance, best fit model identification and subsequent numerical optimization approaches. The best fit response expressions to determine nutritional sensitivity with drying temperature and time have been further deployed to converge upon the optimal soup formulations.

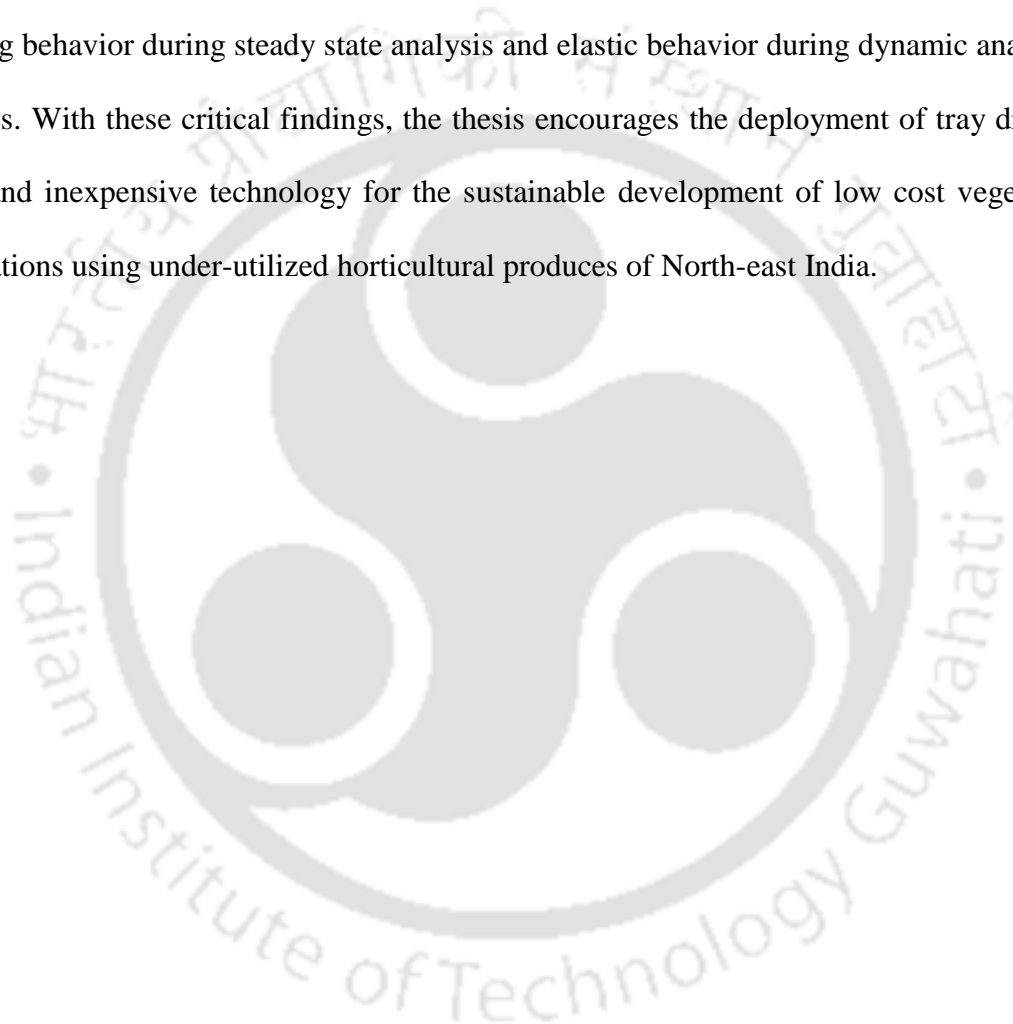
The tray drying process-product characteristics have been evaluated in the range of drying temperature from 50 – 70 °C and drying time of 90 – 300 min, 150 – 390 min, 240 – 420 min, 390 – 720 min, 240 – 450 min, 360 – 630 min and 240 – 420 min for Kolmou, Jatilao, Pui, Kolphul, Kaskal, Komora and Posola, respectively to yield variant combinations of optimal responses and parameters. Thereby, the corresponding responses (moisture content, Vitamin c and antioxidant activity) have been evaluated to vary as 76.11 – 2.87%, 10.39 – 55.33 mg/100g and 18.39 – 88.69% for Kolmou; 46.88 – 3.28%, 34.07 – 112.29 mg/100g and 30.31 – 87.28% for Jatilao; 55.28 – 3.82%, 53.65 – 129.25 mg/100g, and 6.76 – 24.59% for Pui; 46.71 – 3.27%,

32.54 – 86.39 mg/100g and 43.49 – 86.39 % for Kolphul; 27.36 – 2.17%, 52.39 – 89.17 mg/100g and 50.74 – 72.28% for Kaskal; 45.18 – 3.85%, 118.65 – 301.52 mg/100g and 4.92 – 22.94% for Komora; 30.45 – 2.03%, 24.38 – 51.78 mg/100g and 12.07 – 25.29% for Posola. The optimal process-product characteristics have been obtained for drying temperature, time, moisture content, vitamin C and antioxidant activity as 61.28 °C, 236.9 min, 2.87%, 57.02 mg/100g and 91.36% for Kolmou; 61.62 °C, 292 min, 3.28%, 113.36 mg/100g and 89.37% for Jatilao; 63.90 °C, 342.19 min, 4.37%, 130.98 mg/100g and 24.99% for Pui; 61.82 °C, 611.52 min, 3.98%, 87.69 mg/100g and 89.04% for Kolphul; 58.66 °C, 400.31 min, 5.09%, 90.73 mg/100g and 72.34% for Kaskal; 62.77 °C, 530.83 min, 5.09%, 310.40 mg/100g and 22.67% for Komora and 57.59 °C, 389.42 min, 2.84%, 53.38 mg/100g and 25.72% for Posola.

A critical summary of the adopted research methodology in Chapters 6 and 7 is as follows. The methodology involved a robust mathematical model (for nutritionally best mix identification) that supplemented a trial and error based overall approach to evaluate the sensory characteristics. The mathematical model was built based on the response model equations being identified in Chapters 4 and 5 of the Ph.D. thesis. This non-linear programming model was solved using MS-Excel solver with an objective function being set as the simultaneous maximization of vitamin C content and antioxidant content and minimization of moisture content. Following this, a trial and error based sensory analysis was addressed for mathematically optimized vegetable only soup formulations and individual vegetable based control soup formulations. Further, sensory optimization was also carried out for variant cases of dry soup mix to corn flour ratio. Thereafter, the best soup formulation was investigated for rheological characterization.

The optimal leafy and non-leafy vegetable soup formulations refer to individual drying based optimal constitution of leafy vegetables (10:74.95:15.05 for Kolmou, Jatilao and Pui,

respectively) and non-leafy vegetables (21.58:25:43.42:10 for Kolphul, Kaskal, Komora and Posola, respectively). Further, the optimal dry vegetable mix to corn flour ratio was obtained as 40 : 60. The reconstituted soup mix using 20g : 400 mL ratio for dry soup mix : water indicated an overall sensory score of 8.4/9 and 8.6/9 for the leafy and non-leafy vegetable mix soup formulations, respectively. Also, the rheological analysis of both soups affirmed pseudo-plastic shear thinning behavior during steady state analysis and elastic behavior during dynamic analysis for both cases. With these critical findings, the thesis encourages the deployment of tray drying as a viable and inexpensive technology for the sustainable development of low cost vegetable soup formulations using under-utilized horticultural produces of North-east India.



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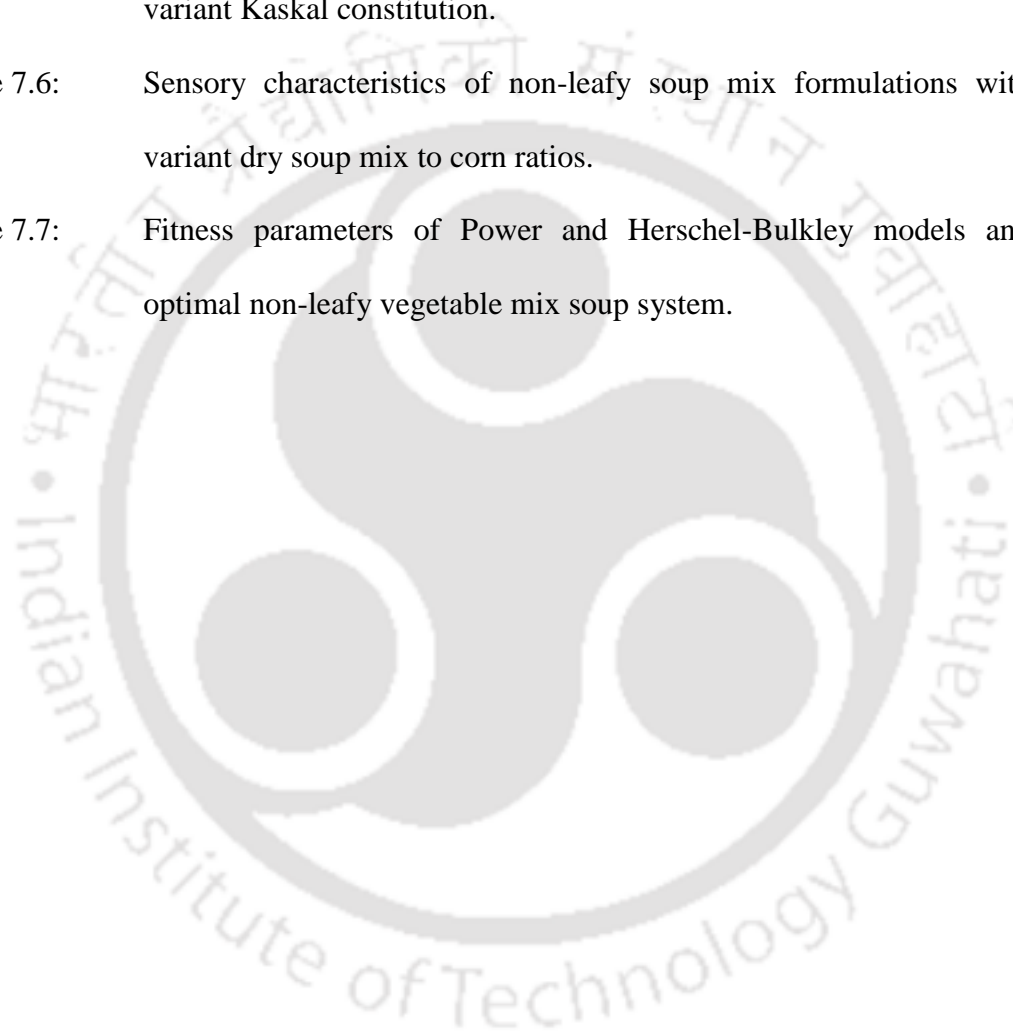
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Abbreviation

MT	Million tonnes
NE	North-East
GAE	Gallic acid equivalents
QE	Quercetin equivalents
DPPH	2,2-Diphenyl-1-Picrylhydrazyl
DCPIP	2, 6-dichlorophenol indophenol sodium salt
AOAC	Association of Official Analytical Chemists
MR	Moisture ratio
RSS	Residual sum of squares
CCD	Central Composite Design
RSM	Response Surface Methodology
ANOVA	Analysis of Variance
C.V.	Coefficient of variation
NLP	Non-linear programming
GRG	Generalized reducing gradient method

Nomenclature

F_M	Leafy vegetable mix based soup formulation
F_K, F_J, F_P	Control leafy vegetable soup formulations being prepared with Kolmou, Jatilao and Pui respectively

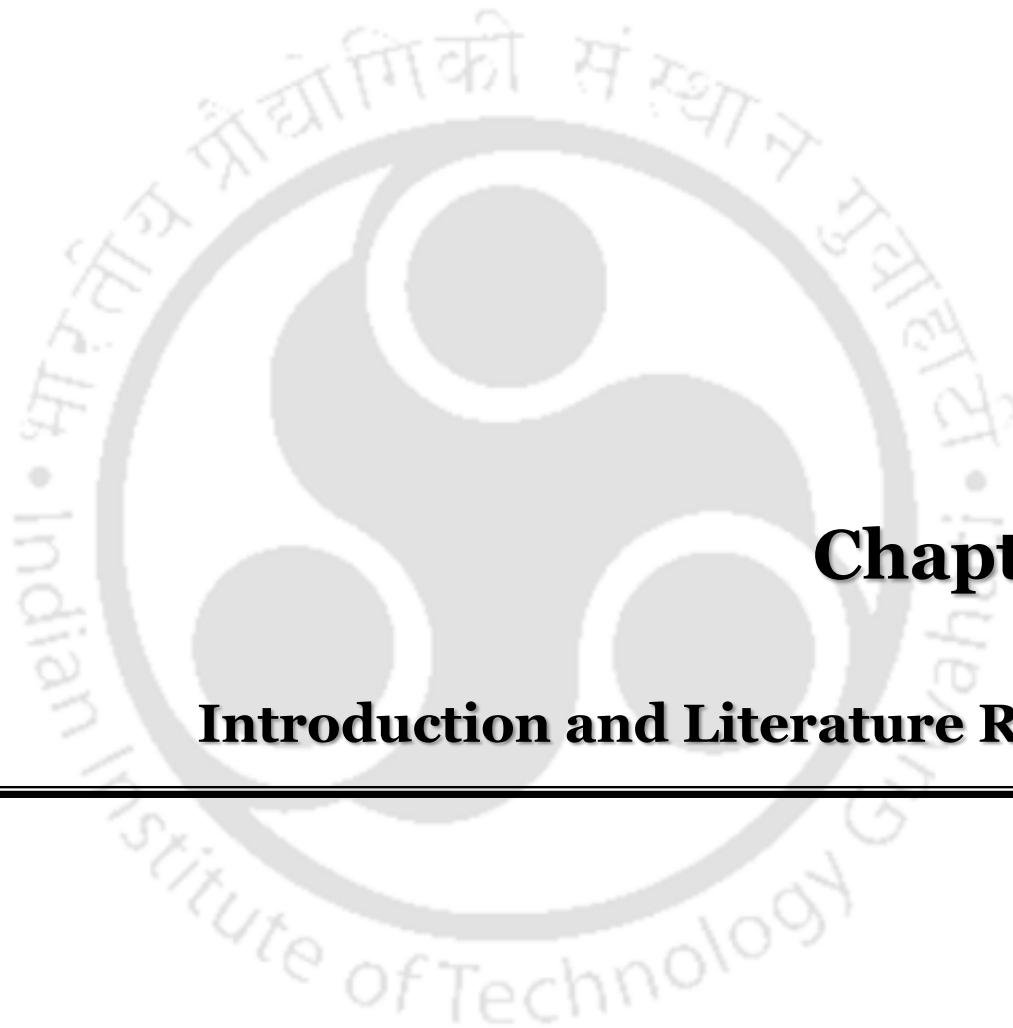
$F_1 - F_4$	Mixed vegetable soup formulations with variant dry leafy vegetables to corn flour ratios (20:80 – 50:50)
F_A, F_B, F_C, F_D	Control leafy vegetable soup formulations being prepared with Kolphul, Kaskal, Komora and Posola respectively
$F_{M1} - F_{M4}$	Optimal non-leafy vegetable mix soup formulations achieved with MS-Excel solver
$F_5 - F_8$	Mixed vegetable soup formulations with variant dry non-leafy vegetables to corn flour ratios (20:80 – 50:50)

Notations

W_r	Raw/fresh sample weight (g)
W_s	Oven/tray dried sample weight (g)
W_d	Sample weight measured after drying at 105 °C for overnight (g)
W_a	Weight of ash (g)
W_l	Weight loss in due course of ignition (g)
W_f	Weight of fat (g)
A_s, A_{std}	Absorbance of the diluted and blank samples respectively
V_1, V_2	Volume of ascorbic acid and sample extract consumed (mL)
AA	Antioxidant activity
A_c	Absorbance of control sample
M, M_0, M_t, M_e	Moisture content, Initial moisture content, moisture content at any time and equilibrium moisture content (%) respectively
a, b, c, k, M_0, n	Drying model parameters

D_{diff}, D	General moisture diffusivity and effective moisture diffusivity (m^2/s)
x, L	Sample thickness and half of sample thickness respectively (mm)
E_a	Activation energy (kJ/mol)
A, B	RSM based independent variables: drying temperature and time respectively
T_k, T_j, T_p	Drying temperature ($^{\circ}C$) corresponding to Kolmou, Jatilao and Pui respectively
T_a, T_b, T_c, T_d	Drying temperature ($^{\circ}C$) corresponding to Kolphul, Kaskal, Komora and Posola respectively
t_k, t_j, t_p	Drying time (min) corresponding to Kolmou, Jatilao and Pui respectively
t_a, t_b, t_c, t_d	Drying time (min) corresponding to Kolphul, Kaskal, Komora and Posola respectively
E, F, G	vitamin C (mg/100g), antioxidant activity (%) and moisture content (%) respectively for Excel solver based mixed soup formulation
k, j, p	Kolmou, Jatilao and Pui respectively
a, b, c, d	Kolphul, Kaskal, Komora and Posola respectively
w_1, w_2, w_3	Weight factors
w_k, w_j, w_p	% Weight of dried Kolmou, Jatilao and Pui respectively
w_a, w_b, w_c, w_d	% Weight of dried Kolphul, Kaskal, Komora and Posola respectively
G'	Storage modulus (Pa)
G''	Loss modulus (Pa)





Chapter 1:

Introduction and Literature Review



Introduction and Literature Review

In this chapter, the first section (section 1.1) provides a brief overview of horticultural produces across the globe followed with emphasis towards vegetable production sector. Following this, the section devotes towards the prominence of underutilized leafy and non-leafy vegetables in the horticultural sector from nutritional perspectives. Thereafter, the section devotes towards the need for the processing of such abundant horticultural produces by adopting relevant low cost food processing based preservation techniques and product development methodologies associated to the development of ready to cook vegetable soup products. In the following section (section 1.2), the available prior-art with respect to three primary areas of research namely optimization of alternate drying methods are being investigated for the preservation of vegetable produces; optimization of process parameters and ready to cook vegetable soup product formulation and characterization. Thereafter, section 1.3 of 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 1.4. Finally, a brief account of the thesis organization has been presented in section 1.5 of the chapter.

1.1 Preamble

1.1.1 Significance of Horticultural Produces

Primarily, horticulture involves the cultivation and production of three major crop groups namely fruits, vegetables and flowers. Among these, the former two dominate the entire sector in terms of the volume and business value of the produces. Due to being important elements of human food consumption system, fruits and vegetables are well known sources for the desired combinations of various macro and micro-nutrients and thereby meet the nutritional and physiological needs of sound human health. Besides such fulfillment of basic human needs, both vegetable and fruit produces ensure steady income for the farm community and foster a sustainable livelihood environment. Rapid advancement in science and technology enabled the sector to adopt low cost preservation and processing techniques for the sustained economic and nutritional benefit to the consumers through value added product development and improvised shelf life strategies.

In the recent past, the horticultural sector grew faster than any other crop (Weinberger and Lumpkin 2005). With an abundant produce of 676.9 MT (million tonnes) and 1159 MT, respectively, the fruit and vegetable produces dominated the horticultural sector in 2014. Among these, major global contribution has been from China (23.4%) and India (17.3%) (**Fig. 1.1**) with respect to the total world vegetable produce (Indian horticulture database 2013–14, National Horticulture Board, Ministry of Agriculture, Government of India). During 2017-2018, the horticultural produce of India was reported to be about 311.71 MT. Thereby, within a span of 13 years (2004 – 2005 to 2017 – 2018), the country's vegetable produce enhanced from 101.2 –

184.4 MT (Horticultural statistics 2018, Ministry of Agriculture & Farmers' Welfare, Government of India).

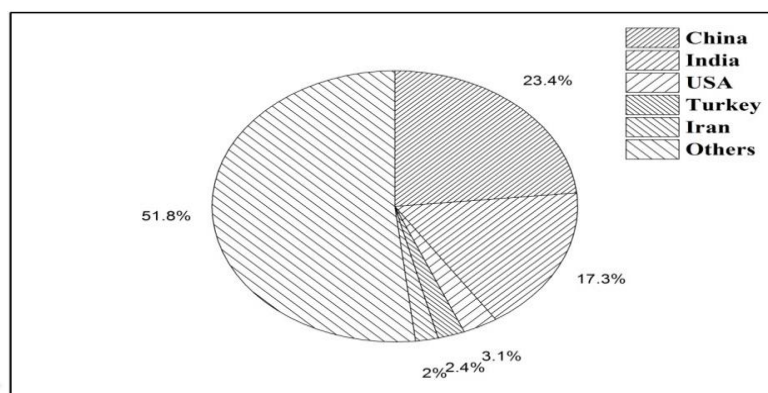


Fig. 1.1: Pie-chart representing production volume of vegetable produces in various countries.

(Courtesy: Indian horticulture database 2013–14, National Horticulture Board, Ministry of Agriculture, Government of India)

Being a rich and variegated source of minerals, vitamins and phytochemicals, the dominant sub-sector of the vegetable produces in the horticultural sector is very important from the perspective of nutritional replenishment and associated physiological health factors of human beings. Broadly, the farm produced vegetables can be categorized into leafy and non-leafy vegetables. In general, the leafy vegetables refer to those in which the leaves of the vegetable are edible for human consumption. The widely available leafy vegetables of North-East (NE) India include mustard leaves, spinach, fenugreek leaves, drumstick leaves, bottle gourd leaves, Indian spinach, water spinach etc. On the other hand, the non-leafy vegetables refer to those in which other portions of the plant are edible. In the NE region of India, the non-leafy vegetables that are widely produced include banana blossom, bottle gourd, ash gourd, banana pseudo-stem, Indian bean, pea, beet etc. Apart from these, a significant proportion of wild vegetables have not been explored from the perspective of value added product development. Thus, they are restricted to

particular regions of the world and despite having good nutritional characteristics, their wider utility is yet to be explored with rapid advances in low cost food processing and preservation technologies.

The current Ph.D. thesis emphasis is towards the utility of underutilized vegetables in the development of value added food products such as low cost vegetable soups. The thesis devotes towards the value addition of many underutilized vegetables which despite being enriched with nutritional and medicinal constituents are ignored even from the perspective of the sustainable income of the farmers that cultivate them. According to Food loss and waste (FLW) index report (2021), household food waste estimates at 50 kg/capita/year in India. Underutilized and lesser known vegetables contribute a significant proportion to the vegetable basket of a country. Among these, a significant proportion of such vegetables such as Kolmou (*Ipomoea aquatica* Forssk.), Pui (*Basella alba*) and Jatialao sak (*Lagenaria siceraria* leaves) and Kolphul (*Musa balbisiana* Colla blossom), Kaskal (*Musa splendida*), green Komora (*Benincasa hispida*) and Posola (*Musa balbisiana* Colla pseudostem) are being wasted annually in India. Being a habitat of water bodies, Kolmou is neglected due to the reason that the harvesting of Kolmou is a tedious job in terms of picking it from the perennial plant. On the other hand, consumer often prefers using only the immature Pui leaves in their cuisines, to thereby drive the wastage of a significant proportion of the vegetable. Posola and Kolphul, by-products of banana tree is often discarded and disposed in agricultural fields and thereby promotes microbial growth and thereby promotes various environmental and health hazards. On the other hand, due to saturation of consumption against their huge production of Kaskal and Komora, a significant proportion of them are wasted every year. Apart from this, the perishable nature of these produces drives deterioration and thereby unwanted wastage of the seasonal vegetables. Such waste can be minimized through

sustainable development goal 12.3 (SDG 12.3). It aims to minimize food loss at retail and consumer level and to reduce the loss across the supply chain. Thereby, it promotes better utilization of the wasted food material through processing, quality improvement etc. Till date, food processing based preservation has not been reported for these vegetables. Also, seasonal availability is another factor that needs to be addressed so as to facilitate the availability of such processed produces for consumption in the entire year. Therefore, various processing methods such as drying, fermentation, freezing, drying, smoking, blanching, frying, extrusion cooking and addition of food additives would be beneficial to transform them into various value added food products.

Needless to convey, such commercial avenues of underutilized endemic vegetables also meets the growing demands of nutritionally rich low cost food products and thereby ensures food security of the world. To do so, adequate scientific investigations need to be addressed to provide useful database for their potential utilization in the near future as key constituents of customized low cost vegetable based food products with good combinations of nutritional, medicinal and shelf life characteristics.

Among various underutilized vegetables, the emphasis is towards the following vegetables that are widely cultivated in the NE region of India:

- a) **Kolmou (*Ipomoea aquatica* Forssk.):** Kolmou is one of perennial leafy vegetables widely consumed by the people in this region. It belongs to convolvulaceae family. It is a wild vegetable grows mainly on wet soils and water bodies. It bears hollow stem with rooting at the nodal points. Both leaves and stems are edible and are used in various cuisines. Being a wild perennial leafy vegetable, Kolmou is known for adequately rich constitution of protein, carbohydrate, crude fibre, minerals (Satter et al. 2016),

polyphenol (Malakar and Choudhury 2015; Sivaraman and Muralidaran 2008) and antioxidants (Huang et al. 2005). From the medicinal perspective, it is traditionally used as laxative for sleeplessness, piles and headache related problems. With antimicrobial and anti-inflammatory properties (Malakar and Choudhury 2015), Kolmou is recommended in traditional naturopathy systems for the mitigation and treatment of diabetes, jaundice, constipation and high blood pressure.

- b) **Jatilao sak (*Lagenaria siceraria*):** the leafy vegetable is a commonly consumed leafy vegetable of a popular gourd plant called bottle gourd in the NE region of India. The plant has got large sized leaves bearing hairs. It is used in the form of oil fried product or as curry. With rich combinations of antioxidants, phytochemicals, vitamins and minerals, the leafy vegetable is well known to fight against ailments such as baldness, headache, mange, skin irritation, tumours, jaundice etc., (Badmanaban et al. 2010; Prajapati et al. 2010; Venkataraman et al. 2018)
- c) **Pui (*Basella alba*):** Belonging to the Baseliaceae family, Pui is a leafy vegetable with heart shaped leaves and purple stems. It is widely available during rainy season (especially August - December) of the year with pick. It is often consumed as an oil fried vegetable or as a soup (Baang et al. 2015). Possessing appropriate constituents that indicates its enhanced characteristics in terms of fibre, minerals, vitamin A, vitamin C and vitamin B content, the vegetable is well known for its antioxidant, anti-inflammatory and anticancer properties (Deshmukh and Gaikwad 2014). The vegetable has been indicated to be effective against heart, Alzheimer's, diabetes mellitus and atherosclerosis ailments (Özgen et al. 2006).

- d) Kolphul (*Musa balbisiana* Colla blossom):** Being a by-product of banana fruit cultivation, Kolphul is a large cone shaped entity covered with alternate purple coloured braces. It is used as a vegetable in curry, boiled or fried form across India particularly in North-Eastern region of India. It belongs to Musaceae family. It is available throughout the year. Kolphul has rich constitution in terms of desired nutrients such as protein, fat, fibre, vitamin C, mineral, antioxidants, phytochemicals and polyphenols. The vegetable has been reported to combat cardiovascular diseases, blood cholesterol (Wickramarachchi and Ranamukhaarachchi 2005), ulcer and constipation (Kumar et al. 2012b).
- e) Komora (*Benesiaca Hispida*):** Komora is a long oblong shaped non-leafy vegetable. It is used as a key ingredient in meat and fish dishes. It is widely available throughout the season. Traditionally, matured vegetable with a white waxy coating on its surface is stored that provides a long shelf life. It is popularly used to prepare a crystallized candied sweat “Murabba”. Komora has been analyzed to be a good source of carbohydrate, soluble fibre, vitamin C, minerals (Kumar et al. 2012a) and phytochemicals (Gupta and Premavalli 2010). From medicinal perspective, Komora has been analyzed to cure diabetes mellitus, respiratory diseases, urinary, gastrointestinal and heart diseases (Al-Snafi 2013).
- f) Kaskal (*Musa splendida*):** Kaskal is a starchy unripe banana vegetable being widely consumed in the NE India. The vegetable is widely available throughout the season and consumed in the form of various cuisines and chips. Containing various functional constituents such as saponins, tannins, alkaloids and flavonoids, Kaskal exhibits strong antioxidant activity (Eleazu et al. 2011). Also, unripe banana is as well rich in starch,

protein and fat (Odenigbo et al. 2013). Often, the unripe banana is recommended as a supplement to treat diabetes mellitus (Odenigbo et al. 2013).

g) Posola (*Musa balbisiana* Colla pseudostem): Posola refers to banana pseudo-stem. It is a popular vegetable being widely consumed across NE states of India. The vegetable is widely available all through the season. It forms distinct cuisines in Assamese culture. With high fibre content, Posola is well known natural remedy to mitigate constipation (Bhaskar et al. 2012). Posola has been characterized with high quantities of minerals such as calcium, potassium, iron and zinc (Aziz et al. 2011). Regular intake of Posola juice has been proven to be effective to mitigate stomach ulcer and kidney stones (Ma et al. 2016).

1.1.2 Preservation based on Horticultural Produce Processing

Microbial attack and degradation due to external factors such as transportation, chemical, heat, environmental and mechanical damages are often responsible towards large proportions of wastage of the perishable vegetable produces. Similar issues of perishability also exist for underutilized vegetables that are often being neglected in their potential role to fulfill the food security of a country. Thus, to minimize the wastage, adequate food processing and preservation strategies are to be applied for the abundant vegetable produces to translate them into value added quality food products. Further, in developing countries such as India, several demographic factors play a pivotal role to enhance the demand and consumption of ready to cook food products. These include extremely busy schedule of the working class, nuclear families desiring adequate nutrition with minimal household work associated to cooking, urbanization, ease of resource availability, enhanced income margins and greater awareness towards health factors.

Processing of horticultural produces based on cost effective preservation technologies assist towards reducing the gap that exists between the abundant produces of the farm community and processed vegetable based food products being demanded by the consumers. Thereby, cost effective food processing based preservation techniques emphasize upon the low cost transformation of raw horticultural produces into processed vegetables based customized products. However, to do so, a mature research methodology is often required that substantiates upon the deployment of effective research strategies in terms of low cost process technology selection, optimization of process parameters, optimal constitution of food products etc., This is also due to the fact that the partially processed vegetables are not accepted by the consumers as such products are not ready to consume with minimal processing at the consumer's end. Hence, ready to consume food products require greater emphasis on the food processing based food product formulation science and technology research. In summary, overall quality, shelf life and sensory characteristics of the processed leafy and non-leafy vegetables are often targeted through the application of suitable low cost food processing technologies to achieve customized food products.

1.1.3 Alternate Horticultural Produce Processing Technologies and their Cost Efficacy

Various technologies exist that facilitate the successful transformation of horticultural produces into value added food products. These refer to canning, fermentation, pasteurization, freezing, drying, smoking, blanching, frying and extrusion cooking as the commonly deployed processing technologies for food preservation. On the other hand, emerging process technologies have also been investigated for their processing efficacy from preservation and prolonged shelf life perspective. These include pulse electric processing, high pressure processing, ohmic heating, ultra-sonication, irradiation, microwave treatment and membrane processing. Rapid advances in

both conventional and emerging horticultural preservation technologies enables maintenance of adequate quality of varieties of customized food products. Needless to convey, research and development of these food processing methodologies for the fruit and vegetable sector has enhanced in the past ten years.

From a cost efficacy perspective, it is well known that traditional methods such as fermentation, pasteurization, drying, blanching etc., have been indicated to be highly effective in comparison with other techniques such as freezing and almost all emerging technologies. These technologies are also susceptible for scale up and this is a desired feature of abundant quantities of produces such as vegetables and fruit.

Among low cost food preservation technologies for horticultural produces, drying technology is one of the most effective and low cost method for the horticultural produce preservation and enhanced shelf life. The principle of drying process technology involves removal of moisture from a wet sample using hot fluid media such as air. The drying process ensures upon the moisture removal from a food material upto a precise level. The moisture removal takes place due to the difference in vapour pressure of the food materials and the surrounding environment being adjusted through the application of heat. Often, the drying process follows three stages namely constant rate period, first and second falling rate period. The constant drying rate period allows surface moisture removal and accounts for the initial drying phase. Following this, the inside moisture reaches the surface of the material being dried through the formation of some channels inside the moist food material. This eventually leaves as vapour from the surface. During this phase, initially there is a rapid reduction in drying rate due to the reduction in rate of moisture migration from inside to outside surface of a food material during the course of drying. Thereafter, slower reduction of drying rate onsets. This is due to lesser

availability of moisture on the food surface due to lesser diffusion of moisture from inside to the outside surface. Finally, the sample reaches equilibrium moisture content and hence no further variation in the sample moisture content can be observed. Thereby, drying technology facilitates a greater removal of moisture from the vegetables and thereby facilitates them to mitigate damages associated to such moisture content. These include microbial, physical, mechanical and chemical damages. However, it is a well-known fact that the nutritional constituents of fruits and vegetables are often heat sensitive and hence the appropriate choice of drying process and its process parameters is very important to achieve a functional horticultural produce based food product with better quality in terms of its nutritional content.

Among various drying techniques, tray drying, sun drying and oven drying are competitive on an economic basis with respect to other techniques such as freeze, vacuum, microwave, ohmic heating etc. Among these, tray drying is highly beneficial due to the drift of the air that drives effective removal of moisture from the dried food sample. Further, tray drying supplemented with intermittent air flow is a promising technology that facilitates drying of food materials with the customized option of pulsed air flow. Such pulsed air flow enables moisture removal during the flow phase and moisture migration from inner portions to the food material surface during stagnation period. Thereby, better nutritional characteristics could be obtained due to enabling the drying operation in a transient drying mode. Also, the pulsed air flow mode enables a proportional reduction in the electricity costs associated to the fan or blower that drives the circulation of air in the drying chamber.

1.1.4 Food Product Development Methodologies

Wide varieties of horticultural produces such as vegetables and fruits exist include wild, endemic and underutilized ones. A critical observation of the available horticultural produce based food products in the consumer market affirms that overall horticultural produces have a negligible share in the processed food products. Thus, to meet the growing demand for high quality low cost processed food products, ideologies can be explored in the context of food products being developed using underutilized horticultural produces. Such food products can offer alternate options for the consumers and thereby diverse their choice with respect to the existing set of processed food products. Hence, the vegetable based food product development requires a methodological thrust and emphasis towards newer food products based on the conventional product development principles such as concept design and feasibility study. Advancing upon these two instills confidence associated to the definitive development of a viable horticultural produce based food product. Further, such analysis provides useful benchmarks and insights towards the following phase of laboratory investigations. Such investigations target research based evaluations to characterize the process as well as the product. Needless to indicate, laboratory research investigations affirm towards the efficacy and optimization of raw materials, processing steps, intermediate and final products. Such analysis also often involves optimization of process technologies and their parameters followed with cost and economic analysis. Finally, during the advanced stage of product development, all desired specifications of the developed product can be met so as to meet the needs of the consumers. The developed food product is at this stage in the phase of a mature formulation. However, additives such as flavonoids and other nutritional constituents are often desired even at this stage to gain the wider acceptance of the consumer. Thereafter, with final product formulation being achieved through fine-tuned and

optimized combinations of processes and their parameters, the eventual step is to target the large scale production and address parametric fine tuning associated to scale up related issues. Thereafter, large scale production is being achieved and the developed product is marketed to reach the consumers so as to stabilize the product from general acceptance in the established market of food products. A striking feature of the overall methodology of food product development is that feedback based on sensory analysis and fine tuning based on research emphasis exists at all stages and at all levels. Thereby, each stage is flexible to accommodate suitable modifications to achieve a unique product that is being achieved through a holistic endeavor to meet all desired yet variegated specifications.

A food scientist and a technologist has a significant role to play in the above summarized food product development methodology. Concept design to target low cost formulations based on inexpensive food preservation and process technologies is the artistic hall mark of such adept and expert food technologist and scientist. Ranging from trial and error based formulations to advanced computer simulation based experiments, food scientists and technologists have a greater role to drive the product formulation and meet the holistic needs of the stringent specifications being set for a food product in the consumer market.

1.1.5 An Overview of Horticultural Produce based Food Product Formulations

From ancient civilizations, food product development was targeted through the mixing of various combinations of horticultural produces. Age old formulations refer to succulent cuisines, recipes, soups, pickles, sweets and fermented products. Horticultural produces occupy a key and unique position in such conventional food products. However, the age old traditional and indigenous food formulation science is often characterized to indicate approximate formulations and do not

endeavor towards precision in terms of nutritional and other characterizations that have been established to be the norm of the rapid advances in science and technology. Therefore, products such as soups need to be precisely customized in terms of qualitative and quantitative characterization parameters such as sensory characteristics, nutritional content, shelf life etc.

Modern science and technology assisted vegetable based food product development research is diverse and variegated. Customized food products for human consumption include vegetable paste and powders which are not regarded to be ready to consume products in comparison with other products such as vegetable based cereal mixes, ketchup, soups, chips, chutneys, sauces, pickles etc. A philosophical summary of various adopted approaches till date for such customized variegated vegetable based food products has been presented in **Table 1.1**. The table indicates the following inferences. Firstly, the vegetable base in the product formulation mix varies all the way from almost more than 100% for the case of chips and as low as 4% for the case of soup mix. Secondly, two alternate approaches have been followed to mature and converge upon optimal vegetable based food product formulation. These refer to either trial and error or response surface methodology (RSM) based approaches to identify optimal and superior formulations based on either sensory or nutritional characteristics or a combination of both. For instance Sharma et al. (2016) adopted a trial and error based approach and optimized a pumpkin based ketchup formulation with various thickening agents to achieve optimal set of proximate and sensory characteristics. Similar trial and error based investigations have been performed to optimize and achieve a superior formulation for a vegetable sauce (Singh et al. 2015) prepared with various vegetables. The superior formulation was achieved based on the combinational optimization of total soluble solids, acidity and sensory attributes.

Table 1.1: A summary of alternate vegetable based food product formulations and methodologies.

S. No.	Product	Constituent range	Methodology / Approach	Product characterization parameters	Reference
1	Ketchup	<ul style="list-style-type: none"> • Variable ingredients: tomato (0 – 1 kg), mushroom (0 – 1 kg) • Fixed ingredients: sugar (150 g), salt (15 g), garlic (10 g), red chilli powder (4 g), glacial acetic acid (3 mL), sodium benzoate (0.75 g), pectin (0.2%), cloves, black pepper, cinnamon, cardamom, cumin seeds (1.5 g each) 	<ul style="list-style-type: none"> • Trial and error approach • 5 formulations with various concentration of tomato and mushroom pulp • Storage study for 12 months 	<ul style="list-style-type: none"> • Proximate values, total soluble solids, total acidity, pH and vitamin C 	Kumar and Ray (2016)
2	Vegetable paste and powder	<ul style="list-style-type: none"> • Potato (175.4 g), carrot (4.2 g), kale (117.2 g), onion (270.3 g), tomato (300 g), soybean oil (0.5 g) 	<ul style="list-style-type: none"> • Linear programming technique using excel solver to maximize antioxidant activity of paste from fresh vegetables. • Optimization of paste drying (spouted bed- 80 – 100 °C and tray drying- 65 °C, 2 m/s) 	<ul style="list-style-type: none"> • Moisture content, total phenolic content, antioxidant activity and colour 	Larrosa et al. (2015)
3	Vegetable cereal mix	<ul style="list-style-type: none"> • Variable ingredients: <i>Gymnema sylvestre</i> leaves powder (3–8 g), <i>Trigonella foenumgraecum</i> powder (3 – 5 g) • Fixed ingredients: tomato powder (10 g), defatted soy flour (20 g) and semolina (10 g). 	<ul style="list-style-type: none"> • RSM • Independent variables- <i>Gymnema sylvestre</i> leaves powder, <i>Trigonella foenumgraecum</i> powder and soaking time • Reconstitution- 25g/100ml buttermilk 	<ul style="list-style-type: none"> • Fat, fibre, carbohydrate and overall acceptability 	Gupta et al. 2016
4	Soup	<ul style="list-style-type: none"> • Variable ingredients: <i>Solanum nigrum</i> leaf powder (1 – 5%), starch (25 – 35%), and spice mix (60 – 70%)- a blend of 17.5% onion powder, 17.5% garlic powder, 15% pepper, 17% salt, 25% milk solids and 8% oil 	<ul style="list-style-type: none"> • D-optimal mixture design based RSM • Independent variables- leaf power, starch and spices 	<ul style="list-style-type: none"> • viscosity, sensory, polyphenols, flavonoids, antioxidant content, proximate values, water retention capacity and swelling capacity 	Sugumar and Guha (2020)

S. No.	Product	Constituent range	Methodology / Approach	Product characterization parameters	Reference
		<ul style="list-style-type: none"> • Variable ingredients: green pea, chick pea and lentil (0 – 35 g), potatoes (22 – 39.50 g), barley (18 – 35.50 g), • Fixed ingredients: tomatoes (5 g), carrot (14 g), onion (1.2 g), garlic (0.75 g), black pepper (0.35 g), coriander (0.35 g), salt (3 g) and cumin (0.35 g) 	<ul style="list-style-type: none"> • Trial and error approach based on Four formulations based on different legumes (lentil, green pea, chicken pea, potato and barley) 	<ul style="list-style-type: none"> • Proximate, minerals, colour and sensory evaluation 	Abdel-Haleem and Omran (2014)
5	Chips	<ul style="list-style-type: none"> • Banana plantain (100%) 	<ul style="list-style-type: none"> • RSM • Frying temperature- 150 – 190 °C • Frying time- 2 – 4 min 	<ul style="list-style-type: none"> • Moisture content, oil content, breaking force, colour difference 	Adeyanju et al. (2016)
6	Chutney	<ul style="list-style-type: none"> • Variable ingredients: beetroot (666 g), apple (167 g), oil (50 g), cumin powder (8 g) • Fixed ingredients: onion (0 – 167 g), sugar (50 – 100 g), lemon juice (0 – 10 g), vinegar (0 – 50 g), chilli powder (15 – 20 g), salt (20 – 25 g) 	<ul style="list-style-type: none"> • Trial and error approach with various combinations of onion, sugar, lemon juice, vinegar and chilli powder and fixed beetroot apple and oil 	<ul style="list-style-type: none"> • Sensory parameters 	Sakshi et al. (2018)
7	Sauce	<ul style="list-style-type: none"> • Variable ingredients: potato (1 – 2 kg), carrot (0.5 – 2 kg), bottle gourd (1 – 15 kg), pumpkin (1 – 2 kg), beetroot (0.5 – 2 kg), cabbage (1 – 1.5 kg), cauliflower (1 – 2 kg), bean (1 – 2 kg), • Fixed ingredients: palak (1 kg), coriander leaves (0.25 kg), green chilli (0.1 kg) 	<ul style="list-style-type: none"> • Trial and error • 9 formulations with varied percentage of vegetables were studied for storage 	<ul style="list-style-type: none"> • Total soluble solids, acidity, sensory attributes 	Singh et al. (2015)
8	Pickle	<ul style="list-style-type: none"> • Variable ingredients: bitter gourd: bottle gourd (0:100) • Fixed ingredients: salt (12%), spice mix (mustard seed, fenugreek seed, asafoetida, turmeric, red chilli powder) 	<ul style="list-style-type: none"> • Optimization- Trial and error for varied blanched bitter gourd to bottle gourd ratio with fixed spice mix 	<ul style="list-style-type: none"> • Sensory parameters 	Pingale and Dabhade (2015)

On the other hand, the RSM based approach has been duly followed for vegetable based cereal mix (reconstituted with butter milk at 1:4 ratio) for precise variegated levels of the key ingredients and parameters (*Gymnema sylvestre* leaf powder, *Trigonella foenumgraecum* powder and soaking time). Considering proximate and overall acceptability, the authors converged upon the optimal formulation using the RSM design based approach (Gupta et al. 2016). Similar RSM methodology based investigations have been conducted to achieve *Solanum nigrum* leaves based soup formulation. The authors adopted D-optimal mixture based RSM design approach by considering various nutritional characteristics as response variables (Sugumar and Guha 2020). Adopting linear programming method using excel solver, Larrosa et al. (2015) optimized the antioxidant activity of a vegetable paste prepared using vegetables such as fresh pumpkin, potato, carrot, kale, onion and tomato vegetables. In other words, both trial and error and RSM based design approaches appear to be relevant for the identification of optimal horticultural produce based food product formulations.

1.1.6 Overview of Vegetable Soup Formulation

Among various horticultural produce based food product formulations, the Ph.D. thesis devotes towards vegetable soup formulations due to simplicity in formulation, scope for variegated product development such as thick soups, utilization of inexpensive equipment such as driers to enhance the low cost value addition of abundant farm produces. Traditionally, a soup is being prepared using various vegetable sources by adopting sequential procedures such as sorting, cleaning, chopping, hot water extraction of ingredients along with the addition of thickening agents and spices into a viscous liquid and subsequent heating and boiling of all ingredients to facilitate uniformity in taste and other characteristics (**Fig. 1.2**).

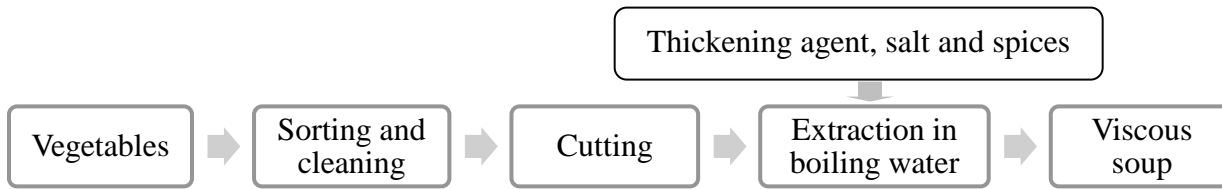


Fig. 1.2: Conventional vegetable soup preparation process flow diagram.

Thus, the mentioned traditional soup making process requires significant time to prepare a tasty soup. Also, standardization and retention of nutrients remains a critical issue that is often ignored in such traditional approaches. These issues can be overcome by targeting a processing based ready to eat and cook food products, using which adequate quantity of hot water can be added and the soup with precise characteristics of sensory and nutritional parameters can be prepared in not more than ten minutes. Such ready to cook food products are gaining attention due to the tech savvy consumers of today's food market who are characterized with demographical characteristics such as busy and time demanding schedule at office, ease to prepare quality food despite being very tired after returning to the residence past a hectic day at work, nuclear family and greater awareness towards nutritional health issues (Anita et al. 2016; Chaudhari and Solanke 2016; Farzana et al. 2017; Kokani et al. 2019)

Given their ease of preparation, sensory acceptability due to being a light but not heavy food and their rich nutritional, dietary and medicinal characteristics, vegetable soup products are becoming highly popular among various food products. Hence, both leafy and non-leafy vegetable mix based soup formulations have gained prominence in the commercial food product sector. Low cost processing based vegetable soup mix formulations target upon drying of vegetables in a hot air oven at fixed choices of temperature and time and subsequent mixing of precise proportions of various dried vegetables to achieve the desired formulation along with other ingredients such as thickening agents and spices. Needless to indicate, the available prior

art emphasizes upon either trial and error or RSM design based experimental investigations to identify and optimize the best vegetable mix formulation from alternate experimental data sets. The trial and error approach involves optimal formulation based on sensory or nutritional characteristics of alternate formulations. The alternate formulations are often chosen based on variegated proportions of the powdered vegetables (James and Matemu 2018) or thickening agents (Niththiya et al. 2014). However, few literature also targeted the trial and error based variation in the concentrations of all ingredients namely vegetable, thickening agent and spice mix (Sugumar and Guha 2020). On the other hand, other literature are also available that indicated optimization of the utilized thickening agents only but no other ingredients such as vegetable powders and spices (Abdel-Haleem and Omran 2014).

From product characterization perspective, various above mentioned vegetable soup formulations have been characterized either in terms of only sensory parameters (Sudarsan et al. 2017; Chavan et al. 2015) or both sensory and nutritional characteristics (Kokani et al. 2019). For the latter case, it is often targeted that the optimized soup formulation was achieved through sensory characteristics and the sensory based optimized soup formulation was evaluated for its nutritional characteristics. Further, few literature also indicate the simultaneous optimization of both nutritional and sensory parameters (Abdel-Haleem and Omran 2014; Jayasinghe et al. 2016; Sugumar and Guha 2020). On the other hand, shelf life (Niththiya et al. 2014; Jayasinghe et al. 2016) and rheological (Abdel-Haleem and Omran 2014) characteristics have been addressed by few authors for the optimal formulations that were deduced using the above mentioned methodologies.

In summary, the outlined approaches do not have significant rationality and hence cannot provide systematic and useful insights to enhance diversification of vegetable soup formulation

through an evolutionary framework. Such analysis is the need of the hour, given the fact that sensory and other nutritional characteristics are always to be considered holistic in due course of the identification of the formulation. Since the research methodology involved is very complex due to associated sensitivity of various parameters, it is therefore essential to envision and adopt a mature research methodology to obtain superior formulations that can further catalyze the horticultural produce based alternate soup formulation research. In this regard, it is important to note that the mathematical optimization based approach enables to find the optimal mix constitution of various vegetables based on the optimization of process variables such as drying time and drying temperature. Thereby, mathematical optimization can serve as a pre-formulation tool to guide the sensory and rheological investigations and further refine upon the soup formulation. In due course of the vegetable drying using operations such as tray drying, the statistical design based model equations obtained from the RSM analysis can serve as useful entities to formulate a mathematical model for the compositional optimization based on nutritional characteristics. Thus, nutritional parameters such as vitamin C, antioxidant activity etc. can be maximized to achieve a near optimum proportion of the vegetable mix formulation in due course of an enriched vegetable soup formulation. Thereafter, research needs to address the sensory and rheological characteristics of the optimized soup formulations so as to envisage the holistic maturity of the optimized vegetable soup formulation.

1.1.7 Summary and Targeted Perspectives

Despite being characterized to be a large production basket in the recent years, a significant proportion of horticultural produces are often wasted and underutilized due to the perishability and saturation of consumption against their abundant production. Among such abundant horticultural produces, vegetables such as Kolmou, Jatilao and Pui Kolphul, Komora, Kaskal and

Posola are notable in the NE region of India. High nutritional and medicinal values of these vegetables makes them highly potential entities for commercial utilization to mitigate the overwhelming demand for various ready to eat and cook food products in the existing scenario of consumers being characterized with busy schedule, nuclear family and greater awareness towards nutritional health.

Among various ready to cook or eat food products, vegetable soup products are among the most promising and popular products of greater demand in the food product consumer market. Due to rapid and established advances in low cost technologies, the nutritionally rich vegetable soup formulations can meet the existing market demand in terms of healthy ready to cook and eat food products. To further supplement such industrial, commercial and translational research in the horticultural sector, a mature research methodology is required to translate wild, endemic and regional leafy and non-leafy vegetables into sensory and nutritionally superior vegetable soup mix formulations. Considering this as the central theme of the Ph.D. thesis, the targeted perspectives of the research addressed in this volume are as follows:

- Identification of competent leafy and non-leafy vegetables
- Process-product characteristics of low cost drying technologies such as oven and tray drying to evaluate upon their nutritional optimization.
- Optimization of process variables associated to drying processes of leafy and non-leafy vegetables
- Mature research methodology associated to the formulation of leafy and non-leafy vegetable soup mix formulations using drying as preservation technology

- Optimal formulations of leafy and non-leafy soup recipes based on underutilized vegetable produces of the NE India.

The overall aim of the thesis is to propose and establish a mature research methodology and associated guidelines towards the development of underutilized leafy and non-leafy vegetables based nutritionally rich mixed soup formulations. The proposed methodology attempts to identify scalable low cost drying technologies and their optimal parameters and subsequent application of suitable experimental design and mathematical tools towards the optimal soup mix formulation development.

1.2 Prior Art

Based on the targeted perspectives summarized in section 1.1 of the Ph.D. thesis, a major thrust of the Ph.D. thesis is to evolve upon a research methodology and framework for the development of leafy and non-leafy vegetable soup formulations using endemic, underutilized wild and conventional abundant horticultural produces of the NE India. Thereby, the following five major areas of research have been identified to be relevant:

- a) Optimization of alternate drying methods to retain maximum nutritional characteristics of dried leafy vegetable samples (Kolmou, Jatilao and Pui).
- b) Optimization of alternate drying methods to retain maximum nutritional characteristics of dried non-leafy vegetable samples (Kolphul, Kaskal, Komora and Posola).
- c) Process parametric optimization of leafy vegetable drying process.
- d) Process parametric optimization of non-leafy vegetable drying process.
- e) Formulation and characterization of leafy vegetable based soup recipe.

- f) Formulation and characterization of non-leafy vegetable based soup recipe.

1.2.1 Efficacy of Alternate Drying Methods for the Optimal Retention of Nutritional Constituents in Leafy Vegetable Samples

Being sensitive to the drying environment, nutritional parameters of leafy vegetables vary significantly with respect to the type of drying method they are exposed to and their operating conditions and other parameters. The sensitivity of nutritional parameters of leafy vegetables is a complex function of the type of vegetable being dried, its constitution in terms of soluble and insoluble fiber content, sample thickness, sample size and drying conditions such as heat intensity and optional convective transport. Thereby, the final quality of a dried vegetable sample is very much dependent upon the type of drying method being deployed and its operating conditions and parameters. Till date, several studies have been conducted to provide useful insights in this area of research. A brief account of the available information in the literature have been presented in the following paragraphs.

Considering shadow, sun, oven (dried at 60 °C for constant weight) and cabinet drying (40 °C for 4 h) methods, Satwase et al. (2013) evaluated the influence of each chosen drying method with respect to the proximate values of dried drumstick leaves. Among all considered alternate drying methods, cabinet dried leafy vegetable sample possessed higher carbohydrate (26.55%), protein (24.80%), fat (9.82%) content and lower moisture content (5.3%).

Using *Allium hookeri* thwaites leafy samples, Wahengbam and Patel (2014) carried out investigations using oven (60 °C) and sun drying methods. The authors inferred that among both methods, sun drying is the best to indicate higher constitution of protein (6.30%), fat (1.03%), ash (1.78%), crude fibre (1.06%), total phenol (148 mg/100g), flavonoids (3.22 mg/100g) and

ascorbic acid (55.58 mg/100g) content but not carbohydrate content which was higher in the oven dried sample.

The influence of few chosen drying techniques on the nutritional characteristics of mint leaves (collected from Hisar city, India) was addressed by Sangwan et al. (2012). Thereby, the authors inferred that in comparison with the oven (50 °C for 6 – 8 h), solar (54 °C for 6 – 8 h) and microwave (800 W for 3 – 4 min) drying methods, the shade dried (at room temperature) leaves possessed better nutritional characteristics in terms of higher protein (19.79%), crude fibre (8.21%), fat (0.81%), ash (11.32%), vitamin C (28.76 mg/100g), beta carotene (16.63 mg/100g), polyphenol (10.49 mg/100g), Ca (1030 mg/100g), Fe (45.77 mg/100g) and Cu (0.97 mg/100g) content.

Adopting sun, shade and oven (60 °C for 1 h) drying techniques, Joshi and Mehta (2010) targeted the nutritional characteristics of dried drumstick leaves. Among the said drying methods, compared to sun and oven drying methods, the shade drying method indicated better retention of nutritional parameters in the dried samples. Thereby, the optimal nutritional characteristics of the dried drumstick leaf samples have been reported by the authors to be 28.48% carbohydrate, 7.03% fat, 12.1% fibre, 24 mg/100g iron, 218 mg/100g phosphorous, 39600 µg/100g beta carotene, 140 mg/100g vitamin C and 500 mg/100g oxalate.

Considering cabinet solar drying (30 ± 10 °C for 6 days), tunnel solar drying (30 ± 10 °C for 6 days) and sun drying (for 6 days), Namadi and Sarah (2017) addressed the proximate and mineral composition of pumpkin leaf samples. Among the chosen alternate drying processes, the tunnel dried leafy samples affirmed higher nutritional and mineral characteristics in terms of protein (13%), carbohydrate (67.4%), fibre (75), ash (8.5%), Ca (5.96 mg/100g), Mg (0.35

mg/100g) and Fe (1.27 mg/100g). However, higher fat content was obtained in the sun dried sample.

The influence of cabinet tray (55 °C), sun (31.8 ± 12 °C) and shade drying on the iron and vitamin C characteristics of cabbage, fenugreek and spinach leafy samples was addressed by Bharati and Asha (2010). The samples dried up to 8 – 10% moisture content level. Among the chosen methods, the cabinet tray drying was found to be effective to retain iron and vitamin C content. The corresponding iron and vitamin C content of the cabinet tray dried said leafy vegetables varied from 2.40 - 9.53 mg/100g and 13.42 - 52.57 mg/100g, respectively.

For curry leaf system, Kenghe et al. (2015) targeted nutritional and mineral content characteristics of shade (dried at 32.5 °C for constant weight), sun and tray (dried at 45, 55 and 65 °C till equilibrium) dried leafy samples. Among the said alternate drying methods, tray drying at 55 °C was inferred to be the best in terms of higher retention of ascorbic acid (3.56 mg/100g), calcium (46.65 mg/L) and iron content (1.34 mg/L).

For the fenugreek leaf system, Navale et al. (2015) carried out nutritional studies of open sun (36.2 ± 2.6 °C for 4 h) and cabinet solar (56.2 ± 3.5 °C for 2 h) dried leafy samples. Among both chosen methods, the cabinet solar dried samples possessed higher protein (1.1 mg/100g) and fat content (4.1 mg/100g) in comparison to those obtained with sun drying method (0.9 mg/100g and .3.9 mg/100g, respectively). Also, the cabinet solar drying enabled faster drying rate in comparison with open sun drying.

A critical summary of the literature data related to the optimization of drying methods and nutritional characteristics of leafy vegetable systems has been presented in **Table 1.2**.

1.2.2 Efficacy of Alternate Drying Methods for the Optimal Retention of Nutritional Constituents in Non-Leafy Vegetable Samples

Compared to the leafy vegetables, only few non-leafy vegetables have been analyzed from the perspective of optimal drying method for the retention of nutritional characteristics. A brief account of the available prior-art in this area of research has been outlined in the following paragraphs:

Ukegbu and Okereke (2013) addressed the effect of sun (drying temperature 24.7-35.6°C, drying time 4 - 6 days) and solar drying (drying temperature 26.90 – 36.3 °C, drying time 5 - 6 days) methods on the nutritional parameters of fluted pumpkin samples. The authors affirmed that the solar dried samples possessed better proximate characteristics in terms of carbohydrate (58.90%), protein (14.71%), fat (5.70%), ash (20.11%) and fiber (9.91%). Similarly, higher mineral and vitamin content and lower microbial load was apparent in the solar dried vegetable samples.

Targeting the efficacy of sun (dried for 4 days), oven (70 °C for 48 h) and solar (60 °C for 30 h) drying processes, Agoreyo et al. (2011) analyzed the nutritional characteristics of dried yam, banana plantain and cocoyam samples. For all investigated vegetable samples, the authors found that the solar drying was effective to retain higher carbohydrate (83.08 – 89.69%), lower ash (3.50 – 5.50%) and higher magnesium content (8.10 – 25.80%). However, the oven dried samples possessed higher protein (3.50 – 3.89%) and lipid (0.11 – 1.57%) content in comparison with the sun dried samples. Similarly, the sun dried samples possessed higher crude fiber content (6.88 – 10.43%) in comparison with the samples obtained from other two drying methods. In summary, the authors indicated that both oven and solar drying methods are equally important to retain nutritional parameters of said vegetables.

The efficacy of sun, solar, freezing (for 1 h) followed with mechanical drying (55 °C), vacuum (50 °C) and oven (55 °C) drying for the retention of nutritional and antioxidant characteristics of pumpkin (*Cucurbita maxima*) was targeted by Wijewardane et al. (2015). The sample was dried until a constant weight is achieved. Thereby, the authors inferred that better retention of fat (0.82%), ash (2.32%), beta- carotene (38.7 mg/100g) and total phenolic content (0.06 mgGA/g) was indicated by vacuum drying.

Considering cauliflower, cluster bean and okra samples, Bharati and Asha (2010) investigated the influence of cabinet tray (55 °C), sun (31.8 ± 12 °C) and shade drying on iron and vitamin C content of dried vegetable samples. Among chosen alternate drying methods, the cabinet tray drying method was found to be effective to retain iron (2.12 – 9.53 mg/100g) and vitamin C (12.43 – 52.57 mg/100g) content in all vegetable samples. **Table 1.3** outlines a brief summary of the critical research findings of the investigations that targeted nutritional and other parametric optimization of alternate drying methods.

1.2.3 Parametric Optimization of Leafy Vegetable Drying Systems

Drying process parameters such as drying temperature, drying time, air humidity, air velocity, dried sample characteristics, sample size and thickness do influence the optimal retention of heat sensitive constituents and moisture content of dried vegetable samples. Thus, it is very important to determine the optimization of such process parameters. To do so, two basic approaches are often followed and these refer to either trial and error based approach or response surface methodology (RSM) based statistical design of experiments approach. In the following paragraphs, a brief overview of various relevant investigations those have been addressed to affirm upon the optimization of drying process parameters for various leafy vegetable systems.

Considering fenugreek, coriander, spinach, mint, shepu and curry leaf samples, Pati et al. (2015) targeted the microwave oven based drying characteristics to optimize protein, calcium, chlorophyll and colour content of dried leafy vegetable samples. Based on trial and error, for an enhanced microwave system power from 135 to 675 W, the drying time (30 – 15 min for fenugreek, coriander, spinach and mint and 25 – 10 min and 25 – 15 min for shepu and curry leaves, respectively) reduced significantly by 64%. However, for all cases, corresponding protein content (21.23 – 11.45%), calcium content (309.35 – 245.40mg/100g) and colour value (52.43 – 40.55) reduced significantly with increasing microwave power. For 135 W microwave power case, the coriander and shepu leaf samples possessed higher chlorophyll content of 82.81% and 58.23%, respectively. Thereby, the authors affirm that higher microwave power is not conducive for the retention of desired characteristics of the dried samples.

Erbay and Icier (2009) targeted RSM based optimization of process variables associated to olive leaves and oven drying system. For this case, drying temperature (40 – 60 °C), drying time (240 – 480 min) and air velocity (0.5 – 1.5 m/s) were chosen as the independent variables. Corresponding moisture content, antioxidant, phenolic content and exergitic energy varied from 3.52 – 24.53%, 41.88 – 53.97%, 8.13 – 37.25% and 56.09 – 73.20%, respectively. Thereby, the authors inferred that the optimal process characteristics refers to 51.16 °C drying temperature, 298.68 min drying time and 1.01 m/s air velocity. Corresponding dried leafy samples possessed 6% moisture content, antioxidant retention of 58.12%, 10.25% phenolic content. Thereby, the drying process optimal exergitic efficiency has been evaluated to be 65.50%.

Few other researchers targeted Box Behnken design based RSM optimization of vacuum dried coriander leafy samples (Thirugnanasambandham and Sivakumar 2016). Independent parameters namely drying temperature, vacuum and loading density varied from 50 – 90 °C, 20 –

60 mm Hg and 0.25 – 0.75 kg/m². For such variation of independent variables, responses varied from 28 – 95%, 482.34 – 549.55 mg/100g and 6.54 – 13.08 g/100g for moisture removal, vitamin C and dietary fibre, respectively. Thereby, the authors inferred that the optimal process-product characteristics refer to 75 °C drying temperature, 28 mm Hg vacuum pressure, 0.63 kg/m² loading rate, 95% moisture removal, 13g/100g dietary fibre and 527 mg/100g vitamin C content.

Among microwave (350 – 100 W), air drying (50 – 175 °C) and vacuum drying (0.4, 50 and 100 mm Hg at 50 and 100 °C) processes, few authors targeted the process parametric optimization of the said processes for the drying of collard leaves (Alibas 2009). Based on extensive investigations, the authors reported drying time, vitamin C and overall colour to vary from 2.5 – 210 min, 90.04 – 37.44 mg/100g and 126.09 – 109.42, respectively. Thereby, the authors inferred that the microwave oven drying system being operated at 750 W and for 8 min drying time indicated best characteristics of dried leafy samples such as 90.04 mg/100g vitamin C and 126.09 colour content. The drying characteristics of the drying process systems followed Page model. Thereby, the model drying constant enhanced with microwave power, air temperature and both temperature and vacuum for microwave, air and vacuum drying processes, respectively. **Table 1.4** provides an outline of the process parametric optimization associated to various leafy vegetable samples.

1.2.4 Parametric Optimization of Non-Leafy Vegetable Drying Systems

As indicated in the previous sub-section 1.2.3, a similar research methodology has also been addressed by several research groups to address the parametric optimization of alternate drying

processes in terms of the nutritional characteristics of dried non-leafy vegetable systems. A brief account of the same has been presented in the following paragraphs.

Using trial and error approach, Adeboye et al. (2014) studied the influence of slice orientation (round and flat) and drying temperature (50 – 70 °C) on the proximate characteristics of green plantain (*Musa paradisiaca*) system in a cabinet drier. For round and flat samples, the inferred proximate parameters were protein (4.26 – 4.09% and 4.55 – 4.24%), fat (3.19 – 2.83% and 4.04 – 3.62%), ash (2.15 – 1.63% and 1.95 – 1.78%) and fibre content (0.82 – 0.69% and 1.25 – 0.89%). The highest protein (4.55%) and fibre content (1.25%) have been indicated by the control flat slices that were dried at 50 °C. The investigations by the authors affirmed that highest boundaries of the drying process in terms of energy, time and costs are indicated to be those apparent for round slices being dried at a drying temperature and time of 70 °C and 7 h, respectively.

The optimization of drying process parameters associated to convective air dried cauliflower system was targeted by Gupta et al. (2013) using response surface methodology. The authors considered size (3, 4 and 5 cm), temperature (55, 60 and 65 °C) and air velocity (40, 50 and 60 m/min) as degrees of freedom or critical independent variables. Based on characteristic parameters (drying time 425 – 735 min, rehydration ratio 5.39 – 7.35, vitamin C 246.20 – 290 mg/100g and browning index 0.11 – 0.18) of the dried cauliflower system, the optimum temperature, size and air velocity for the drying process system have been evaluated to be 60.10 °C, 3.35 cm and 59.28 m/min, respectively. Corresponding response variable characteristics refer to an optimal combination of 491.22 min drying time, 6.91 rehydration ratio, 289.86 mg/100 g vitamin C content and 0.14 browning index.

Adopting response surface methodology, Đào (2015) optimized process parameters associated to drum drying process and pumpkin vegetable system. Steam pressure (300 – 400 kPa) and rpm (0.5 – 1.5) were varied to evaluate the variation of moisture content (1.02 – 8.42%), crude fibre (6.28 – 10.44%), carotene content (0.70 – 0.92 mg/g), acceptability (4.57 – 7.67) and water absorption index (4.04 – 4.29 g/g). Thereby, the authors reported optimal process parametric choice of 313.54 kPa steam pressure and 1.27 rpm. Corresponding optimal response variable values refer to 8.359% moisture content, 10.398% crude fibre content, 0.864 mg/g carotene content and 4.220 g/g water absorption index.

Aruna and Nishadh (2014) studied the effect of tray and freeze drying temperature (60, 70, 80 °C and -40, -50, -60 °C, respectively) on vitamin C, polyphenol and anthocyanin content of radish based on trial and error methodology. Based on trial and error, the authors observed that the said characterization parameters (vitamin C 35.25 – 19.05 mg/100g, polyphenol 29 – 13.21 mg/100g and anthocyanin 35.82 – 11.35 mg/100g) reduced with increasing temperature for the tray drying process. Based on their extensive investigations, tray and freeze drying at 60 °C and -50 °C, respectively have been reported to be best for the radish drying process. Corresponding optimal nutritional characteristics refer to polyphenol, anthocyanin and vitamin C content of 29, 35.82 and 35.25 mg/100g and 27.93, 52, 42.45 mg/100g for tray and freeze drying cases, respectively.

Adopting a trial and error approach, Chahbani et al. (2018) investigated the influence of microwave drying on the drying kinetics, antioxidant activity, colour, phenolics and flavonoids of green pea vegetable system. The authors varied microwave power in the range of 100 – 450 W. Drying kinetics studies affirmed that the moisture diffusivity of the dried green peas system did vary from $1.69 - 5.36 \times 10^{-9} \text{ m}^2/\text{s}$. Corresponding activation energy of the drying process was

about 1.38 kW/kg. For an optimal microwave power of 100 W, the antioxidant activity, colour L^* , a^* and b^* have been evaluated for the system as 1840 IC_{50} mg/ml, 48, - 3 and 26, respectively. Various phenolic and flavonoid constituents ranged between 2 – 235 $\mu\text{g/g}$ and 1 – 880 $\mu\text{g/g}$, respectively.

For the buttermilk soaked banana blossom system, John et al. (2014) studied the tray drying system characteristics in the drying temperature range of 40 – 60 °C. Based on trial and error approach, the authors reported that the moisture diffusivity, moisture, protein, fat, ash, crude fibre, total phenol and flavonoid content of the process varied from 5.45 – 8.09×10^{-9} m^2/s , 9 – 8.9%, 1.78 – 1.93%, 0.31 – 0.58%, 1.35 – 1.42%, 19.76 – 17.63%, 5373.58 – 5470.16 mg GAE/100g and 281.32 – 335.59 mg QE/100 g. Corresponding activation energy of the process was about 50.06 kJ/mol. The investigations affirmed that the optimal process characteristics correspond to 60°C drying temperature and 195 min drying time. Corresponding optimal dried product characteristics refer to 8.9% moisture content, 1.93% protein, 0.58% fat, 1.42% ash, 17.63% crude fibre, 5470.16 mg GAE/100 g total phenolic content and 335.59 mg QE/100 g total flavonoid content.

A brief summary of the critical findings of all relevant literature summarized in the above paragraphs has been presented in **Table 1.5**.

1.2.5 Formulation and Characterization of Optimal Leafy Vegetable Soup Recipe

As mentioned earlier, several demographic factors of contemporary life style, nutritional awareness, nuclear family and enhanced income have propelled human interest towards the consumption of ready to cook vegetable leafy and non-leafy soups. These vegetable soup formulations are often dry and therefore have higher shelf life due to lowest moisture content.

Further, the research methodologies associate to arrive upon a particular combination of vegetable constituents as optimal choice in a soup system either based on a trial and error or statistical design approach. In the following paragraphs, a brief account of contemporary vegetable soup product development has been addressed from the perspectives of soup formulation and characterization.

Addressing the utility of sprouted horse gram and radish leaves, Sudarsan et al. (2017) targeted a vegetable soup formulation based on the organoleptic scores adopting trial and error approach. Among three formulations S1, S2 and control (radish leaf powder 0 – 12.5%, garlic and onion powder 2.5 – 5% each, coriander 1.25 – 5%, curry leaf powder 0 – 5% and salt 1.25 – 2.5%), the authors reported that the formulation S2 (sprouted horse gram 25%, radish leaves powder 12.5%, onion powder 2.5%, garlic powder 2.5%, coriander powder 1.25%, curry leaves powder 1.25%, pepper powder 2.5%, and salt 2.5%) possessed significantly higher values of colour (4.1), flavor (4.6), appearance (4.6), taste (4.4) and overall acceptability (4.7). Further, the sensory characteristics have been evaluated for a maximum score of 5. Also, after 1 month of storage study that was conducted in laminated pouches kept under room temperature conditions, the samples indicated to possess not a trace of pathogenic microorganisms.

Kokani et al. (2019) targeted the development of an instant leafy vegetable soup based on moringa leaves and garden cross seeds. The authors reported upon four formulations that constituted varied proportions of moringa leaves to garden cross seed powder ratio (T1 0:0, T2 6:6, T3 12:12 and T4 18:18). Thereby, optimal formulation was obtained through sensory evaluation. Following this, proximate parameters have been evaluated for the optimal formulation. Based on such trial and error based approach, among various formulations T1-T4, the reported optimum formulation refers to T1 (corn flour 10 g, sugar 30 g, salt 4 g, cumin

powder 10 g, ginger powder 10 g, onion powder 10 g, garlic powder 10 g, chilli powder 4 g, moringa leaves powder 6 g, and garden cress seed powder 6 g) formulation with an overall acceptability of 9/9. The proximate characteristics of the optimal vegetable soup formulation were 7.4% moisture, 8.33% fat, 8.2% ash, 35% protein, 41.07% carbohydrate and 450.24 kcal energy.

Niththiya et al. (2014) addressed the development of an instant soup mix that was targeted through the sensory analysis of alternate formulations (trial and error approach) prepared using locally available vegetables, variant constitution of palmyrah tuber powder (5 – 35 g) and tamarind paste (5 – 20 g). Thereby, the authors reported optimal leafy vegetable soup mix formulation of 35 g odiyal flour, 2 g rice grits, 4 g manioc, 4 g long beans, 0.25 g moringa leaves, 0.15 g carrots, 10 g tamarind, 1 g chilli powder, 5 g salt, 0.75 g onion and 0.82 g garlic. For a reconstitution ratio of 1:12.5 for solids to water ratio, the overall acceptability of the soup was 4.3/5. From proximate analysis perspective, the soup mix was analyzed to contain 3.24% moisture, 79.32% carbohydrate, 5.9% protein, 1.16% fat, 5.58% ash and 4.8% fibre content. After storing the soup mix samples in high density polyethylene bags, they have been inferred to have insignificant degradation in their quality for a period of about two months.

Using a combination of solar dried leafy samples of *Moringa oleifera*, *Ipomoea batatas* and *Daucus carota*, James and Matemu (2018) developed an optimal vegetable mix formulation. The authors optimized vegetable proportions in the vegetable mix from the perspectives of maximum retention of mineral content and beta-carotene based on trial and error approach. Among several alternate formulations (with vegetable proportions varied as F1 (1:1:1), F2 (2:1:1), F3 (1:1:2) and F4 (1:2:1), the formulation F2 with the chosen vegetable ratio 2:1:1 for the said sequence of vegetables, respectively has been reported by the authors to be a superior

formulation with high constitution of zinc (2.15 mg/100 g), iron (58.54 mg/100 g) and beta-carotene (4.16 mg/100 g) content.

Adopting D-optimal mixture design based RSM approach, Sugumar and Guha (2020) developed an optimal leafy vegetable soup formulation using *S. nigrum* leaves. The formulation was targeted by considering leafy powder percentage (1 – 5%), starch (25 – 35%) and spices constitution (60 – 70%) as critical independent variables or degrees of freedom. The optimal soup formulation was evaluated in terms of several nutritional and other characterization parameters such as viscosity, organoleptic parameters, polyphenols, flavonoids, antioxidant content, antioxidant activity, proximate values, water retention capacity and swelling capacity. Based on extensive investigations, the optimum vegetable soup formulation was reported to have been prepared with 4% leaf powder, 30% starch and 66% spice mix (onion, pepper, garlic, salt, milk solids and vegetable oil). The optimal vegetable soup has been characterized with an optimal combination of 1942 cp viscosity, 6.42 overall acceptability, 32 mg GAE/g polyphenol content, 10 mM tocopherol equivalence/g antioxidant content, 31.74% crude protein, 27.3% carbohydrate content and 9% crude fibre content.

Joshi et al. (2020) developed an instant leafy vegetable soup formulation using powders of radish, turnip, carrot and cauliflower leaves (2.5, 3.75 and 5% each) based on trial and error approach. The optimal constitution of the soup was targeted based on its optimization in terms of antioxidant activity and organoleptic response characteristics. The best formulation corresponded to a combination for 5 g radish and 5 g turnip leaf powder blended with 35g lentil powder, 5g corn flour, 18g tomato powder, 10g garlic powder, 11g onion powder, 1.5g black pepper, 1.5g red chilli, 8g salt and 5g ghee. Also, for the radish and turnip leaves based formulations, the

antioxidant activity and overall acceptability characteristics have been evaluated to be (44.80% and 7.07) and (41.58% and 7.62), respectively.

A critical summary of the above elaborated literature with respect to the formulation and characterization of leafy vegetable soup mix formulation and characterization has been presented in **Table 1.6** of the Ph.D. thesis.

1.2.6 Formulation and Characterization of Optimal Non-Leafy Vegetable Soup Recipe

The following paragraphs summarize the available prior art in the field of formulation and characterization of non-leafy vegetable soup mix recipe.

Jayasinghe et al. (2016) studied the development of instant vegetable soup mix formulation based on the seaweeds. Based on proximate, mineral content, viscosity and sensory analysis, the authors compared two formulations prepared with 2% carrageenan and 3% agar seaweeds. Subsequently, they have been compared with commercially available soups. The vegetable soup mix formulation included other ingredients such as 80% vegetables (pumpkins, celery, tomato, leeks, carrot, potato, mushrooms and onions), 10% grain, 2.5% dried ulva powder, 3.5% legumes and preservatives. Among both formulations, the formulation with carrageenan 2% was inferred to be the best with better combinations of proximate, mineral contents and overall acceptability (43.78/50). The optimal seaweed formulation was stable against mould and yeast for storage duration of 3 and 6 months in ambient and deep freezer conditions, respectively.

Abdel-Haleem and Omran (2014) investigated the development and characterization of a vegetarian soup by considering four alternate soup formulation (trial and error approach). These

formulations have been based on a combination of various legumes (green pea, chick pea and lentil 0 – 35% each, potatoes 22 – 39.50% and barley 18 – 35.50%). The optimal soup formulation was determined based on proximate, minerals, colour and sensory characteristics. Based on extensive investigation, the authors inferred that the optimal formulation corresponds to that being prepared with 35% lentil, 22% potato, 18% barley, 5% tomato, 14% carrot, 1.2% onion, 0.75% garlic, 3% salt, 0.35% black pepper, 0.35% cumin and 0.35% coriander. The overall acceptability of the said formulation was 45.85/50. Corresponding proximate characteristics of the soup referred to 74.96%, 3.29%, 6.16%, 15.59%, 1.68%, 5.06 mg/100g and 2.50 mg/100g for carbohydrate, fat, ash, protein, crude fibre, Fe and Zn content, respectively. Also, the reconstituted soup was analyzed to behave as a non-Newtonian pseudo-plastic fluid.

Targeting the development of white button mushroom based vegetable soup formulations, Kumar (2015) carried out proximate analysis, sensory characteristics and shelf life studies to infer upon the optimal soup formulation using trial and error based approach. For varied proportions of the mushroom (dehydrated mushroom piece and powder varied from 5 – 10 g each) and skim milk powder (25 – 35 g) constitutions, proximate and sensory parameters have been targeted by the authors. The shelf life studies have been addressed in terms of moisture content, overall acceptability and microbial load. Among three alternate formulations A – C, the developed formulation with 5g dehydrated mushroom pieces, 5g dehydrated mushroom powder, 35g skim milk powder, 25g wheat flour, 20g fat, 5.5 g salt, 0.1g white pepper, 1.5 g onion, 1.5g garlic and 1.5g sugar has been evaluated to be optimal formulation. The said formulation was evaluated to possess 71.82% carbohydrates, 13.86% protein content, 5.84% fat content, 3.64% ash content and 0.88 crude fibre content. The overall acceptability of the optimal soup formulation was 8.55/9. The stored sample indicated marginal reduction in the proximate and

overall acceptability characteristics. Further, during such storage studies, the total plate count has been evaluated to be below the hazardous level of the microorganism load.

Chavan et al. (2015) targeted the optimization of tomato soup premix base formulation using response surface methodology. During their investigations, the authors considered tomato powder (30 – 34 g), whey powder (38 – 42 g), sugar (9 – 11 g) and salt (10 – 12 g) constitution as degrees of freedom or critical independent variables. Thereby, the authors identified the optimal soup formulation based on sensory characteristics. The reported optimal tomato soup premix base formulation referred to 34 g tomato powder, 42 g whey powder, 9.87 g salt, 11.13 g sugar, 2 g skim milk, 2 g cream powder, 1 g citric acid and 2 g yeast extract. For a maximum scale value of 9, the optimal sensory characteristics refer to 6.51, 6.66, 8.14, 6.68 and 5.57 for taste, flavor, texture, appearance and odour, respectively.

A critical summary of the available literature data in the specific field of non-leafy vegetable soup formulation and characterization has been presented in **Table 1.7** of the Ph.D. thesis.

Table 1.2: Literature data summary associated to the efficacy of alternate drying methods and leafy vegetable systems.

Sl.no.	Author	Target	Methodology	Parameters studied	Optimum drying technique and nutrient content
1	Satwase et al. (2013)	Evaluation of effect of shadow, sun, oven and cabinet drying on proximate composition and drying characteristic of Drumstick leaves	<ul style="list-style-type: none"> • Sun drying- until equilibrium is achieved • Shade drying- until equilibrium is achieved • Cabinet tray drying- 40 °C for 4 hrs • Oven drying-60 °C until complete drying 	<ul style="list-style-type: none"> • Moisture, carbohydrate, protein, fat, fibre, ash 	<ul style="list-style-type: none"> • Cabinet drying • Carbohydrate (26.55%), protein (24.80%), fat (9.82%) and lower moisture content (5.3%).
2	Kenghe et al. (2015)	Effect of shade and tray drying on nutrient values of Curry leaves	<ul style="list-style-type: none"> • Shade drying- room temp. (32.5 °C) for constant weight • Tray drying (45, 55 and 65 °C) for constant weight 	<ul style="list-style-type: none"> • Moisture, iron, calcium, ascorbic acid 	<ul style="list-style-type: none"> • Tray drying at 55 °C • ascorbic acid (3.56 mg/100g), calcium (46.65 mg/L), iron (1.34 mg/L)
3	Sangwan et al. (2012)	Effect of different drying techniques on nutrient parameters of Mint leaves	<ul style="list-style-type: none"> • Shade drying- room temp for constant weight. • Oven drying-50±5 °C for 6-8 h. • Microwave drying- 800W for 3-4 mins • Solar drying- 54 °C for 6-8 h. 	<ul style="list-style-type: none"> • Moisture, crude fibre, ash, protein, fat • Beta-carotene, ascorbic acid • Ca, Fe, Cu 	<ul style="list-style-type: none"> • Shade drying • protein (19.79%), crude fibre (8.21%), fat (0.81%), ash (11.32%), vitamin C (28.76 mg/100g), beta carotene (16.63 mg/100g), polyphenol (10.49 mg/100g), Ca (1030 mg/100g), Fe (45.77 mg/100g) and Cu (0.97 mg/100g)
4	Bharati and Asha (2010)	Effect of cabinet tray drying, sun drying and shade drying on iron and vitamin C content of three leafy vegetables (Cabbage, Fenugreek leaves and Spinach).	<ul style="list-style-type: none"> • Sun drying- 31.8 – 43.8 °C • Shade drying • Tray drying- 55 °C • Samples were dried up to 8 – 10% moisture level 	<ul style="list-style-type: none"> • Iron and vitamin C 	<ul style="list-style-type: none"> • Tray drying • Iron (2.40 - 9.53 mg/100g) and vitamin C (13.42 - 52.57 mg/100g)

Table 1.3: Literature data summary associated to the efficacy of alternate drying methods and non-leafy vegetable systems.

Sl.no.	Author	Target	Methodology	Parameters studied	Optimum drying technique and nutrient content
1	Ukegbu and Okereke (2013)	Effect of sun and solar drying methods on nutrient constituents of Okra and Fluted pumpkin.	<ul style="list-style-type: none"> • Sun drying- (24.7-35.6°C) for 4 - 6 days. • Solar drying -26.90-36.3°C for 5 - 6 days. 	<ul style="list-style-type: none"> • Moisture, dry matter, carbohydrate, protein, fat, crude fibre, ash, energy • P, K, Ca, Mg, Na • Beta carotene, vitamin C • Microbial load 	<ul style="list-style-type: none"> • Solar drying • Carbohydrate (58.90%), protein (14.71%), fat (5.70%), ash (20.11%) and fiber (9.91%)
2	Agoreyo et al. (2011)	Analysis of nutritional composition of Yam, Banana plantain and Cocoyam dried by sun, oven and solar dryer.	<ul style="list-style-type: none"> • Sun drying- 10 a.m. to 4.30 p.m for 4 days • Oven drying- 70 °C for 48 h • Solar drying- 60 °C for 30 h 	<ul style="list-style-type: none"> • Moisture, carbohydrate, protein, lipid, crude fibre, ash • Ca, Mg 	<ul style="list-style-type: none"> • Solar dried: higher carbohydrates (83.08 – 89.69%), ash (3.50 – 5.50%) and magnesium (8.10 – 25.80%). • Protein (3.50 – 3.89%) and lipid (0.11 – 1.57%) were better retained in oven drying
3	Wijewardane et al. (2015)	Effect of drying methods on nutritional properties and antioxidants in dehydrated powdered vegetables (Pumpkin, Tampala, Sweet potato and Hibiscus)	<ul style="list-style-type: none"> • Sun drying • Solar drying • Oven drying-55 °C • Vacuum drying-50 °C • Freezing and drying 50 °C • The samples were dried until an equilibrium is achieved 	<ul style="list-style-type: none"> • Moisture, fat, protein, crude fibre, ash • Beta carotene, total phenolic content 	<ul style="list-style-type: none"> • Vacuum drying • Fat (0.82%), ash (2.32%), beta-carotene (38.7 mg/100g) and total phenolic (0.06 mgGA/g)
4	Bharati and Asha (2010)	Effect of shade, sun and cabinet tray drying on vitamin C and iron content of Cauliflower, Cluster bean and Okra.	<ul style="list-style-type: none"> • Cabinet tray drying- 55 °C • Sun drying- 31.8 to 43.8 °C • Shade drying • All drying carried out up to 8-10% moisture content 	<ul style="list-style-type: none"> • Iron and vitamin C 	<ul style="list-style-type: none"> • Cabinet tray drying • Iron (2.12 – 9.53 mg/100g) and vitamin C (12.43 – 52.57 mg/100g)

Table 1.4: Literature data summary of drying process parametric optimization and leafy vegetable systems.

Sl.no.	Author	Target	Methodology	Parameters studied / obtained range	Key findings
1	Pati et al. (2015)	Drying of Green Leafy Vegetables (Fenugreek, Coriander, Spinach, Shepu, Mint and Curry leaves) using microwave oven dryer	<ul style="list-style-type: none"> Microwave oven drying power ranging from 135 to 675 W 	<ul style="list-style-type: none"> Protein (21.23 – 11.45%), calcium (309.35 – 245.40mg/100g), chlorophyll content (4.06 – 1.33 mg/g) and colour L value (52.43 – 40.55) 	<ul style="list-style-type: none"> Protein content decreased with increase in power from 135 to 675 W. However drying time decreased significantly by 64%. Greater retention of calcium was at lower microwave output power. Coriander and shepu showed higher chlorophyll content (82.81% and 58.23%, respectively) at 135 W.
2	Erbay and Icier (2009)	RSM based optimization of process variables for hot air dried olive leaves.	<ul style="list-style-type: none"> RSM Drying temperature (40 – 60 °C) Drying time (240 – 480 min) Air velocity (0.5 – 1.5 m/s) 	<ul style="list-style-type: none"> Moisture content (3.52 – 24.53%), antioxidant capacity loss (41.88 – 53.97%), phenolic content (8.13 – 37.25%) and exergetic efficiency (56.09 – 73.20%) 	<ul style="list-style-type: none"> Optimal parameters- temperature 51.16 °C, drying time 298.68 min, air velocity 1.01 m/s, moisture content 6%, antioxidant capacity loss 41.88%, phenolic content 10.25% and exergetic efficiency 65.50%
3	Thirugnanasambandham and Sivakumar (2016)	Box Behnken design based RSM optimization of vacuum dried coriander leaves	<ul style="list-style-type: none"> Box Behnken design based RSM Temperature (50 – 90 °C) Vacuum (20 – 60 mmHg) Loading density (0.25 – 0.75 kg/m²) 	<ul style="list-style-type: none"> Moisture removal (28 – 95%), dietary fibre (482.34 – 549.55 mg/100g) and vitamin C (6.54 – 13.08 g/100g) 	<ul style="list-style-type: none"> Optimal process variables- temperature 75 °C, vacuum 28 mm Hg, loading rate 0.63 kg/m², moisture removal 95%, dietary fibre 13 g/100g and vitamin C 527 mg/100g
4	Alibas (2009)	Optimization of process parameters for dried collard leaves	<ul style="list-style-type: none"> Microwave drying (350 – 100 W) Air drying (50 – 175 °C) Vacuum drying (0.4, 50 and 100 mm Hg at 50 and 100 °C) 	<ul style="list-style-type: none"> Ascorbic acid (90.04 – 37.44 mg/100g), drying time (2.5 – 210 min) and colour value (126.09 – 109.42) 	<ul style="list-style-type: none"> Optimum values- microwave drying at 750 W with ascorbic acid 37.44 mg/100g, drying time 8 min and colour value 126.09 Best fit model- Page model

Table 1.5: Literature data summary of drying process parametric optimization and non-leafy vegetable systems.

Sl.no.	Author	Target	Methodology	Parameters studied / obtained range	Key findings
1	John et al. (2014)	Evaluation of drying characteristics using thin layer drying	<ul style="list-style-type: none"> • Trial and error approach • Tray drying- fixed air velocity of 1 m/s, • Temperatures- 50, 60 and 70 °C 	<ul style="list-style-type: none"> • Drying time 300 – 195 min, moisture (9 – 8.9%), protein (1.78 – 1.93%), fat (0.31 – 0.58%), ash (1.35 – 1.42%), fibre (19.76 – 17.63%), phenol (5373.58 – 5470.16 mg GAE/100g) and flavonoids (281.32 – 335.59 mg QE/100 g) 	<ul style="list-style-type: none"> • Optimal values- 60°C drying temperature, 195 min drying time, 8.9% moisture, 1.93% protein, 0.58% fat, 1.42% ash, 17.63% crude fibre, 5470.16 mg GAE/100g phenols and 335.59 mg QE/100g flavonoid content
2	Gupta et al. (2013)	Optimization of convective air drying for the drying of Cauliflower.	<ul style="list-style-type: none"> • Convective air drying • Box-Behnken design • Temperatures-55, 60 and 65 °C • Sizes- 3, 4, and 5 cm • Airflow- 40, 50, and 60 m/min 	<ul style="list-style-type: none"> • Drying time (425 – 735 min), rehydration ratio (5.39 – 7.35), vitamin C (246.20 – 290 mg/100g) and browning index (0.11 – 0.18) 	<ul style="list-style-type: none"> • The optimum values for the drying process- 60.10 °C, 3.35 cm and 59.28 m/min • Drying time 491.22 min, 6.91 (rehydration ratio, 0.14 (browning), 289.86 mg/100 g (Vitamin C) and desirability factor of 0.787.
3	Aruna and Nishadh (2014)	Effect of temperature on some biochemical qualities of Radish	<ul style="list-style-type: none"> • Tray drying (60, 70 and 80 °C) • freeze drying (-40, -50 and -60 °C) 	<ul style="list-style-type: none"> • For tray and freeze drying, vitamin C (35.25 – 19.05 and 23 – 42.45 mg/100g), polyphenol (29 – 13.21 and 12.7 – 27.93 mg/100g) and anthocyanin (35.82 – 11.35 and 14.05 – 52 mg/100g), respectively 	<ul style="list-style-type: none"> • Components decreased with increase in temperature • Tray drying at 60 °C and freeze drying at -50 °C were found to be best for the drying radish.
4	Dao (2015)	Process parameter optimization of drum drying for production Pumpkin powder.	<ul style="list-style-type: none"> • Drum drying • CCD-RSM • Steam pressure- 300-400 kPa and rotational speed of 0.5-1.5 rpm. 	<ul style="list-style-type: none"> • Moisture content (1.02 – 8.42%), crude fibre (6.28 – 10.44%), carotene content (0.70 – 0.92 mg/g), acceptability (4.57 – 7.67) and water absorption index (4.04 – 4.29 g/g). 	<ul style="list-style-type: none"> • Optimum combination: 313.54 kPa steam pressure, 1.27 rpm, 8.36% moisture, 10.40% crude fibre, 0.86 mg/g carotene, 4.22 g/g water absorption index
5	Adeboye et al. (2012)	Effect of slice orientation and drying temperature on proximate parameters of Green plantain	<ul style="list-style-type: none"> • Cabinet drying • Temperature (50 – 70 °C) 	<ul style="list-style-type: none"> • Protein (4.09 – 4.55%), fat (2.83 – 4.04%), fibre (0.69 – 1.25%) and ash (1.63 – 2.15%) 	<ul style="list-style-type: none"> • Highest protein (4.55%) and fibre (1.25%) were found in control flat slices dried at 50 °C

Table 1.6: Prior art data summary of optimal leafy vegetable soup formulation systems.

S.No.	Author	Target	Methodology	Best Formulation in terms of parameters studied	Best product characterization parameters
1	Sudarsan et al. (2017)	Development and formulation of instant soup mix from radish leaves and sprouted horse gram.	<ul style="list-style-type: none"> Horse gram- soaking (10h)- cabinet drying and powdered Formulations- S1, S2 and control with varied radish leaf powder (0 – 12.5%), garlic and onion powder (2.5 – 5% each), coriander (1.25 – 5%), curry leaf powder (0 – 5%) and salt (1.25 – 2.5%) Sensory evaluation Water : soup mix (100ml : 1 tea spoon) 	<ul style="list-style-type: none"> S2 (Sprouted horse gram 25%, Radish leaves powder 12.5%, Onion powder 2.5%, Garlic powder 2.5%, Coriander powder 1.25%, Curry leaves powder 1.25%, Pepper powder 2.5%, and Salt 2.5%) 	<ul style="list-style-type: none"> Colour (4.1), flavour (4.6) appearance (4.6), taste (4.4) and overall acceptability (4.7) No pathogens for 1month in laminated pouches under room condition.
2	Kokani et al. (2019)	Development and Standardization of Moringa Leaves Instant Soup Mix Powder Incorporated With Garden Cress Seeds	<ul style="list-style-type: none"> Four formulations based on moringa leaves and garden cress seed powders viz., T₀ (0:0), T₁ (6:6), T₂ (12:12) and T₃ (18:18). Reconstitution: 100g / 600ml Optimized based on Sensory evaluation Evaluation of colour, texture and proximate parameters for best formulation 	<ul style="list-style-type: none"> T₁ (Corn flour 10g , Sugar 30g, Salt 4g, Cumin powder 10g, Ginger powder 10g, Onion powder 10g, Garlic powder, Chilli powder 4g, Moringa leaves powder 6g, and garden cress seed powder 6g 	<ul style="list-style-type: none"> Colour (8.5), Flavour (8), Taste 8), Consistency (9), Appearance (8) and Overall acceptability (9) Moisture (7.4%), Fat (8.33%), Ash (8.2%) Protein (35%), Carbohydrate (41.07%)
3	Sugumar and Guha (2020)	<i>Solanum nigrum</i> leaves based soup formulation was optimized using D-optimal mixture design	<ul style="list-style-type: none"> D-optimal mixture design RSM Independent variables- leaf power (1 – 5%), starch (25 – 35%) and spices (60 – 70%) Responses- viscosity, sensory, polyphenols, flavonoids, antioxidant content, antioxidant activity, proximate values, water retention capacity and swelling capacity 	<ul style="list-style-type: none"> 4% leaf powder, 30% starch and 66% spice mix (onion, pepper, garlic, salt, milk solids and vegetable oil) 	<ul style="list-style-type: none"> viscosity 1942 cp, overall acceptability 6.42, polyphenol 32 mg GAE/g, antioxidant content 10 mM tocopherol equivalence/g, crude protein 31.74%, carbohydrates 27.3% and crude fibre 9%
4	Joshi et al. (2020)	Formulation and characterization of an instant soup mixes from leaf powders of radish, turnip, carrot and cauliflower	<ul style="list-style-type: none"> Trial and error with various % of radish, turnip, carrot and cauliflower leaf powder (2.5, 3.75 and 5% each) Characterization- antioxidant activity and sensory parameters Reconstitution- 25g/280mL 	<ul style="list-style-type: none"> Formulations with 5 g radish and 5 g turnip leaf power Other ingredients- lentil powder 35 g, corn flour 5 g, tomato powder 18 g, garlic powder 10 g, onion powder 11 g, black pepper 1.5 g, red chilli 1.5 g, salt 8 g and ghee 5 g. 	<ul style="list-style-type: none"> antioxidant activity and overall acceptability for the developed soup formulations found to be (44.80% and 7.07) and (41.58% and 7.62) for radish and turnip leaves based formulations

Table 1.7: Prior art data summary of optimal non-leafy vegetable soup formulation systems.

S.No.	Author	Target	Methodology	Best Formulation in terms of parameters studied	Best product characterization parameters
1	Jayasinghe et al. (2016)	Instant vegetable soup formulation incorporated with seaweed.	<ul style="list-style-type: none"> • Trial and error for formulations with 2% carrageenan or 3% agar were compared with commercially available soup. • Characterization- proximate, minerals, viscosity and sensory • Shelf life evaluation 	<ul style="list-style-type: none"> • 80% vegetables (pumpkins, celery, tomato, leeks, carrot, potato, mushrooms and b-onions), 10% grain, 2.5% dried ulva powder, 3.5% legumes and 2% carrageenan 	<ul style="list-style-type: none"> • Higher proximate and mineral contents • Overall acceptability of 43.78/50 • Stable against mould and yeast for 3 and 6 months at ambient and deep freezer, respectively.
2	Abdel-Haleem and Omran (2014)	Characterization of vegetarian soup based on various legumes.	<ul style="list-style-type: none"> • Trial and error approach • Four formulations based on different legumes- green pea, chick pea and lentil (0 – 35 g), potatoes (22 – 39.50 g) and barley (18 – 35.50 g) • Characterization- Proximate, minerals, colour and sensory evaluation 	<ul style="list-style-type: none"> • Lentil 35%, Potato 22%, Barley 18%, Tomato 5%, Carrot 14%, Onion 1.20%, Garlic 0.75%, Salt 3%, Black pepper 0.35%, Cumin 0.35% and Coriander 0.35% 	<ul style="list-style-type: none"> • Overall acceptability- 45.85/50 • Carbohydrate (74.96%), fat (3.29%), ash (6.16%), protein (15.59%), crude fibre (1.68%), Fe (5.06 mg/100g) and Zn (2.50 mg/100g)
3	Kumar (2015)	Development and shelf life study of soup powder incorporated with white button mushroom.	<ul style="list-style-type: none"> • Trial and error for formulations with varied mushroom (dehydrated mushroom piece and powder varied from 5 – 10 g each) and skim milk powder (25 – 35 g) constitutions • Characterization- proximate and sensory parameters • storage study for moisture content, overall acceptability and microbial load 	<ul style="list-style-type: none"> • Dehydrated mushroom pieces 5 g, dehydrated mushroom powder 5 g, skim milk powder 35 g, wheat flour 25 g, fat 20 g, salt 5.5 g, white pepper 0.1 g, onion 1.5 g, garlic 1.5 g and sugar 1.5 g 	<ul style="list-style-type: none"> • Carbohydrates 71.82%, proteins 13.86%, fat 5.84%, ash 3.64% and crude fibre 0.88% • Sensory out of 10- flavour (6.7), color (5.6), texture (6.8), overall acceptability 8.55/9 • Slight decrease in proximate and acceptability for 1 year • Total plate count was below hazardous level
4	Chavan et al. (2015)	Optimization of tomato soup premix base formulation using RSM	<ul style="list-style-type: none"> • RSM • Independent variables- tomato powder (30 – 34 g), whey powder (38 – 42 g), sugar (9 – 11 g) and salt (10 – 12 g) • Responses- sensory parameters 	<ul style="list-style-type: none"> • 34 gm tomato powder, 42 g whey powder, 9.87 g salt, 11.13 g sugar, 2 g skim milk powder, 2 g cream powder, 1 g citric acid and 2 g yeast extract 	<ul style="list-style-type: none"> • Taste (6.51), flavor (6.66), texture (8.14), appearance (6.68) and odour (5.57) out of 9

1.3 Lacunae and Possible Scope for Further Research

The summarized prior-art is affirming towards the following lacunae and thereby the possible scope for further research:

- a) Optimization of alternate drying methods and chosen leafy (Kolmou, Jatilao and Pui) and non-leafy (Kolphul, Kaskal, Komora and Posola) vegetables.
- b) Process parametric optimization of best drying technology for chosen leafy (Kolmou, Jatilao and Pui) and non-leafy (Kolphul, Kaskal, Komora and Posola) vegetables.
- c) Formulation and characterization of vegetable soup mix recipes based on chosen leafy (Kolmou, Jatilao and Pui) and non-leafy (Kolphul, Kaskal, Komora and Posola) vegetables.

1.3.1 Efficacy of Alternate Drying Systems for Chosen Leafy and Non-Leafy Vegetable Systems

The available prior art affirms that several literature did address the nutritional content based optimization of alternate drying methods for selected leafy and non-leafy vegetable species. Proximate, mineral and vitamin constitution of the dried vegetable samples have been considered as the bias to judge and evaluate upon the optimization of the alternate drying methods. The precise lacunae of relevant literature are as follows. While Satwase et al. (2013) studied the influence of drying on the proximate parameters of shadow, sun, cabinet and oven dried drumstick leaf samples, the authors did not target upon the minerals, vitamins, antioxidant, physical parameters and phytochemical content of the leafy vegetable system. These are of significance in the development of nutritionally rich vegetable mix soup formulations. Similarly, Joshi and Mehta (2010) addressed the proximate and mineral content (calcium, iron and

phosphorous) of dried drumstick leaves by targeting sun, shade and oven drying techniques. However, the authors did not target their influence on other characteristics such as essential minerals, antioxidant, drying characteristics, phytochemicals and physical parameters (such as colour, texture etc.). Some of these are relevant towards nutritionally rich constitution of the optimized vegetable soup mix recipe.

Similar insights are apparent for the non-leafy vegetable related studies for the optimization of alternate drying methods. Most authors targeted the best process in terms of nutritional evaluation. For example, proximate (carbohydrate, protein, ash and fiber content) and mineral (Zn, Ca, Mn, Mg and Fe) characteristics have been addressed by a research group for the efficacy of tunnel solar, cabinet solar and sun drying methods and pumpkin leaves (Namadi and Sarah 2017). However, the authors did not elaborate upon other essential minerals, drying characteristics, phytochemicals, antioxidant and physical parameters. Similarly, Agoreyo et al. (2011) analyzed the effect of sun, oven and solar drying on the proximate characteristics and few minerals (calcium and magnesium) for banana plantain. However, the authors did not elaborate upon minerals other than Mg and Ca and other desired characteristics such as phytochemical and vitamin content, physical parameters, microbial load and antioxidant activity. Many of these are of significance in terms of the nutritional richness of the mix vegetable soup formulation that may be developed using some of the already investigated vegetables. Adopting a similar research methodology, the influence of cabinet tray, sun and shade drying techniques on iron and vitamin C content of cauliflower, cluster bean and okra was addressed by Bharati and Asha (2010). However, the authors also did not direct their investigations towards proximate, phytochemicals and antioxidant characteristics and other desired physical parameters such as colour and texture.

Among alternate drying methods, tray drying has been considered to be an effective and low cost method to address the abundant produces of leafy and non-leafy vegetable samples. In this regard, a critical analysis of tray drying systems for such samples needs to be carried out to identify the relevant lacunae and possible scope for further research. In the context of wider varieties of leafy and non-leafy vegetable samples, the available literature confirms to the fact that much research has been addressed towards efficacy of tray methods in comparison with other drying methods. For instance, Satwase et al. (2013) and Kenghe et al. (2015) considered tray drying along with other drying methods for the drying of curry and drumstick leafy samples, respectively. Similarly, tray drying characteristics were duly addressed for cauliflower, cluster bean and okra (Bharati and Asha 2010). Hence, there is a need to devote many experimental investigations to affirm the efficacy of tray drying process towards several abundant leafy and non-leafy produces of India and especially NE India.

In summary, for the chosen non-leafy (Komora, Kolphur, Kaskal and Posola) and leafy vegetables (Kolmou, Jatilao and Pui), the optimization of alternate drying methods has not been addressed in the literature. In this regard, it is important to note that, for banana blossom and banana pseudo-stem, nutritional characteristics have been reported for a fixed drying temperature condition using hot air drying system by a research group (Ma et al. (2016)). However, a comparative assessment with other alternate methods has not been addressed by this research group or any other research group. Thereby, sensitivities associated to variation in the nutritional content of chosen leafy (Kolmou, Jatilao and Pui) and non-leafy vegetable (Kolphul, Kaskal, Komora and Posola) samples can be addressed for alternate drying methods.

1.3.2 Parametric Optimization of Optimal Drying Process and Chosen Leafy and Non-Leafy Vegetable Systems

The available prior art in the said area of research emphasizes towards optimization related investigations for the best drying process parameters for few leafy and non-leafy vegetables that are not of prominence as far as the NE region of India is concerned. Such underutilized yet abundant leafy and non-leafy produces of the region have been targeted in the thesis.

The notable prior art in the specific area of drying process optimization for selected leafy and non-leafy vegetables are as follows. Erbay and Icier (2009) and Thirugnanasambandham and Sivakumar (2016) addressed the RSM based optimization of process parameters associated to hot air dried olive leaves and vacuum dried coriander leaves, respectively. In due course of their investigations, the authors did not emphasize upon the optimization of the process parameters with respect to the maximum retention of desirable characteristics such as vitamin C, flavonoid and phytochemical content.

Similarly, among non-leafy vegetables, the RSM based optimization approach has been effectively targeted for the optimization of process parameters associated to air jet impingement dried Okra (Wang et al. 2016) and cauliflower (Gupta et al. 2013). In these articles, the authors regarded few physical and chemical responses (soy isoflavones, antioxidant activity and vitamin C) as the key biases for which parametric optimization was sought. However, the research groups did not elaborate upon the optimization of the process parameters with respect to the phenolic content, flavonoids, saponins, carotene, vitamins, minerals and other phytochemical constituents of the dried samples. Similar lacunae exist in other works that addressed the RSM based optimization of process parameters associated to the vacuum dried red currant samples (Šumić et al. 2016). Further, it is important to note that while the authors considered drying

temperature, pressure and time as critical independent variables, they did not consider sample thickness and loading as additional degrees of freedom.

On the other hand, in the specific field of leafy vegetable drying process optimization, few available prior art indicated that the trial and error based approaches targeted the process parametric optimization associated to hot air dried collard leaves (Alibas 2009), microwave dried green peas (Chahbani et al. 2018) and hot air dried banana blossom (John et al. 2014). However, such investigations did not address upon the efficacy of the process parameters with respect to the retention of vitamins and other phytochemical constituents. Other than these, the trial and error based approaches have been addressed by another research group with respect to optimization of oven drying temperature for the drying of *Moringa oleifera* leafy samples (Alakali et al. 2015). However, the authors did not consider the influence of other drying parameters in due course of optimization. Also, the authors did not consider the efficacy of the process parameters for good vitamins, phytochemicals and physical characteristics in the dried leafy samples.

Similarly, in the specific field of trial and error based optimization of process parameters associated to the drying of non-leafy vegetable samples, very few literatures are available. Adeboye et al. (2012) addressed the influence of slice orientation and drying temperature on proximate characteristics of green plantain (*Musa paradisiaca*) and cabinet drier system. However, the authors did not elaborate upon the criticality of drying time and air velocity. Also, often sought response variables such as vitamins, minerals and physical parameters were also ignored by the authors.

In the specific research area of tray drying process parametric optimization of leafy and non-leafy vegetable samples, very few literatures exist. Among these, while Aruna and Nishadh (2014) targeted the optimization of drying temperature on the vitamin C, polyphenol and anthocyanin content of radish. However, the authors did not elaborate upon its optimization with respect to other nutritional and physical parameters. Also, the optimization of drying time, sample thickness and blower speed (air velocity) was not targeted by the authors. Further, Smita and Karuna (2014) addressed the cabinet tray drying process parametric optimization associated to bottle gourd powder production. The authors did not address blower (fan) speed and slice thickness as critical independent variables and did not account upon the optimization of critical dependent variables such as proximate constituents, phytochemicals and microbial load.

In summary, it can be inferred that the available literature has the following lacunae in terms of the process parametric optimization associated to the drying of leafy and non-leafy vegetable system:

- a) Lack of integrated and correlated approach for the synthesis of trial and error based optimization (drying kinetics approach) and statistical design (RSM) based optimization approach in vegetable drying system studies. This is especially applicable for the intermittent air flow assisted tray dried targeted leafy and non-leafy vegetable samples.
- b) Process parametric optimization associated to any drying process for the chosen leafy (Kolmou, Jatilao and Pui) and non-leafy (Kolphul, Kaskal, Komora and Posola) vegetable systems and for the desired response variables that includes moisture content, vitamin C and antioxidant activity.

1.3.3 Design and Development of Leafy and Non-Leafy Vegetable based Soup with Optimal Nutritional and Sensory Characteristics

The available prior art affirms various optimal vegetable soup mix formulations that have been addressed till date. However, most literature targeted the soup mix formulation optimization on a trial and error approach based on 2 – 5 pre-defined formulations. Further, the authors only considered either only sensory characteristics or both sensory and nutritional characteristics of the formulations prepared with varied constitution of vegetables, thickening agents and additives. Among leafy vegetable based soup formulations, Sudarsan et al. (2017) reported an optimal instant soup mix based on sprouted horse gram and radish leaves. The soup formulation optimization was addressed in terms of response variables such as colour, flavour, appearance, taste and shelf life. However, the authors did not target nutritional optimization and associated rheological characteristics. Kokani et al. (2019) standardized a vegetable soup mix formulation based on moringa leaves and garden cross seeds. Sensory characteristics have been considered to judge upon the soup mix optimization. Thereafter, for the optimal soup mix formulation, proximate analyses were conducted. Thus the authors did not address optimization of nutritional characteristics such as vitamins, minerals and phytochemicals. Also, rheological analysis and storage studies have not been addressed by the authors. An optimal soup mix formulation has been reported based on solar dried *M. oleifera*, *I. batata* and *D. carota* leaves (James and Matemu 2018). While the authors achieved soup mix optimization based on optimal constitution of iron, zinc and beta-carotene, the authors did not consider optimization of other essential minerals, vitamins, proximate parameters, sensory, phytochemicals, mechanical and rheological characteristics. Also, details with respect to the optimization of drying process parameters for the chosen responses have not been presented by the authors.

On the other hand, the RSM based optimization of soup mix formulation was duly addressed for the soup being prepared with *S. nigrum* leaves using D-optimal mixture design approach (Sugumar and Guha 2020). Considering leafy powder, starch and spices as degrees of freedom or critical independent variables, the authors targeted viscosity, sensory parameters, polyphenols, flavonoids, antioxidant capacity, antioxidant content, proximate values, water retention capacity and swelling capacity as the relevant response variables. However, for reconstituted soup, the authors only considered viscosity and sensory characteristics and did not address upon the optimization of other chosen responses. Also, the authors did not evaluate other characteristics such as minerals, phytochemicals, static and dynamic rheological characteristics.

Similarly, only few research groups addressed the optimization of vegetable soup mix formulations using trial and error based approaches. Using pumpkin, tomato, celery, leeks, potato, carrot, mushroom, onion and seaweed, Jayasinghe et al. (2016) achieved optimal vegetable soup mix formulation based on detailed characterizations such as proximate, minerals, viscosity and sensory parameters. However, the authors did not elaborate upon the optimization of phytochemicals and vitamins. Also, rheological studies have not been reported for the reconstituted soup formulations. Similar lacunae also exists for optimal vegetable soup mix formulation achieved using tomato, potato, carrot and other varieties of legumes (Abdel-Haleem and Omran 2014). Similarly, very few literatures addressed the RSM based optimization of non-leafy vegetable soup mix. Using such research methodology, the optimization of tomato soup premix was addressed by Chavan et al. (2015). With varied proportion of ingredients, the authors considered only sensory characteristics as the key response variables. However, various nutritional and rheological parameters were not addressed for the developed product.

A critical analysis of the available literature indicates that a rational approach is very much missing towards the development and characterization of leafy and non-leafy soup recipes. Mathematical optimization based optimization of nutritional parameters associated to dried vegetables is missing prior to the sensory and rheological evaluations that were often addressed in the above mentioned prior art. Also, most literature targeted a trial and error based approach to judge upon the vegetable soup mix optimization among several formulations. This didn't consider the associated sensitivity and complexity of parameters. Research methodology associated to the optimization of vegetable soup formulations is highly complex. Often, sensory analysis is targeted along with nutritional and rheological fitness of the said product. Due to having its origins in horticultural produces, the soup formulation is a complex repository for the optimization of a large set of characterization parameters. These include nutritional, sensory and rheological characteristics. Given the complexity of combinatorial optimization of vegetable mix, thickening agent, solids to water ratio and spice mix, the identification of optimal soup formulation is a tedious task due to the optimization desired in all targeted attributes of the product. Therefore, a mature research methodology that effectively reduces the engagement of the subjects along with acceptable framework that does not jeopardize upon the quality of the product is highly interesting and motivating for continued emphasis upon low cost vegetable soup formulations. This is not the case with the available literature. Hence, a matured mathematical optimization based approach for the assessment of nutritional constituents of the dried vegetables is the need of the hour to indicate newer frontiers and directions towards nutritionally rich optimal vegetable soup mix formulations along with sensory characteristics. Finally, as far as chosen leafy (Kolmou, Jatilao and Pui) and non-leafy (Kolphul, Kaskal,

Komora and Posola) vegetables are concerned, the literature does not report any investigations towards the achievement of optimal non-leafy and leafy soup formulations.

1.4 Objectives of the Ph.D. Thesis

Based on the identified gaps in literature, the objectives of the Ph.D. thesis have been outlined as follows:

- a) Optimization of alternate drying technologies (oven and tray drying) based on nutritional characteristics (proximate parameters, vitamin C and antioxidant activity) of chosen leafy (Kolmou, Jatilao and Pui leaves) and non-leafy vegetables (Komora, kaskal, Kolphul and Posola).
- b) Process parametric optimization of best drying process (such as drying temperature and time among tray and oven drying processes) based on nutritional characteristics (moisture content, vitamin C and antioxidant activity) of selected leafy (Kolmou, Jatilao and Pui leaves) vegetables.
- c) Process parametric optimization of best drying process (such as drying temperature and time among tray and oven drying processes) based on nutritional characteristics (moisture content, vitamin C and antioxidant activity) of selected non-leafy (Komora, Kaskal, Kolphul and Posola) vegetables.
- d) Mathematical optimization coupled sensory analysis based formulation, optimization and characterization of vegetable soup mix recipe based on selected leafy (Kolmou, Jatilao and Pui leaves) vegetables.

- e) Mathematical optimization coupled sensory analysis based formulation, optimization and characterization of vegetable soup mix recipe based on selected leafy (Komora, Kaskal, Kolphul and Posola) vegetables.

1.5 Organization of the Thesis

Considering the above mentioned lacunae and scope for further research and the objectives set for the Ph.D. thesis, the ultimate objective of the thesis is to envision upon the greater utility of underutilized abundant produces of leafy and non-leafy vegetables of NE India into customized vegetable soup mix products with optimal combinations of desired characteristics such as good shelf life, enriched nutritional content and very good sensory characteristics. Addressing these issues, the Ph.D. thesis has been organized into seven chapters. A brief account of these chapters has been addressed as follows in the following paragraphs.

Chapter 1 address the relevance of customized research towards the evaluation of nutritional characteristics, alternate drying methods for chosen leafy (Kolmou, Jatilao and Pui) and non-leafy (Kolphul, Kaskal, Komora and Posola) vegetables. The available prior art affirms that significant research gap exists in terms of applying oven/tray drying techniques for the evaluation of drying characteristics of chosen leafy and non-leafy vegetables. Such investigations are very much essential prior to the development of optimal vegetable mix based soup formulations. Thereby, the objectives set for the Ph.D. thesis encourage significant yet useful insights into the drying characteristics of leafy and non-leafy vegetables and associated sensitivities of nutritional parameters of the said dried vegetable samples with respect to the drying process parameters such as drying temperature and time.

Elaborating upon the details of the followed methodology, **Chapter 2** addresses the customized approaches that were duly followed to achieve the set thesis objectives. Firstly, these refer to the procedures followed for the evaluation of optimization of drying techniques to achieve best retention of desired nutritional characteristics of dried vegetable samples. Thereafter, trial and error and statistical design based approaches have been described to gain useful insights into the drying kinetics and other characteristics of tray dried vegetable samples due to the very fact that tray drier provides better performance than oven drying to achieve better retention of the desired nutritional characteristics. Finally, the methodology devotes towards the approaches adopted to integrate mathematical optimization into sensory analysis based vegetable soup mix formulations. Relevant modeling strategies have been elaborated with respect to the associated research methodology for both leafy and non-leafy vegetable soup formulations.

Chapter 3 summarizes key research findings associated to the oven and tray drying based nutritional characteristics of chosen leafy (Kolmou, Jatilao and Pui) and non-leafy (Kolphul, Kaskal, Komora and Posola) samples. Thereafter, a comparative assessment of both processes was targeted to evaluate upon the efficacy of tray drying process in comparison with oven drying process for the retention of said nutritional characteristics.

Chapter 4 summarizes the results and discussion associated to the process parametric and response variable optimization of leafy vegetables (Kolmou, Jatilao and Pui). Both trial and error and statistical design approaches have been followed to identify the best process variables. Thereby, synergy between these two approaches has been dealt in the relevant sections. Fitness of best drying kinetic models, moisture diffusivity and activation energy determination have been duly addressed to evaluate upon the associated sensitivities of response variable characteristics (moisture content, antioxidant activity and vitamin C content) with respect to

drying time and temperature as independent variables of the tray drying process. Thereby, optimal set of process and response variable characteristics have been identified for experimental and optimization based approaches. Central composite rotatable design approach was followed in the investigations addressed with the RSM design approach. The RSM optimization involved analysis of variance based best fit model evaluation and subsequent numerical optimization. Finally, proximate characteristics of dried leafy vegetable samples have been summarized in the chapter.

Chapter 5 addresses results and discussions associated to trial and error and statistical design (RSM) based approaches for the process parametric optimization associated to chosen non-leafy vegetable samples (Kolphul, Kaskal, Komora and Posola). The sections and contents of the chapter are similar to those elaborated for chapter 4 of the Ph.D. thesis.

Chapter 6 elaborates upon results and discussion associated to the formulation and characterization of leafy vegetable mix (Kolmou, Jatilao and Pui) based soup product. As explained previously, a near optimal vegetable soup mix formulation was targeted prior to the sensory analysis by targeting a non-linear programming based approach. The model expressions obtained in the analysis targeted in chapters 4 and 5 have been utilized to build the mathematical mode. Thereafter, the chapter addresses results obtained from both individual and co-drying approaches. Finally, the chapter addresses results obtained from sensory characterization based optimization approaches followed with rheological characteristics of optimal leafy vegetable soup formulation.

Chapter 7 summarizes results and discussion associated to the non-leafy vegetable (Kolphul, Kaskal, Komora and Posola) based soup formulation. The contents of the chapter are similar to those presented in chapter 6 of the Ph.D. thesis. Thereby, sensory analysis was targeted

after carrying out preliminary vegetable soup mix optimization studies that were targeted through non-linear programming based approaches. Finally, rheological characteristics have been addressed for the optimal non-leafy vegetable soup mix formulations.

Chapter 8 devotes towards the conclusions of the carried out research and the best results obtained from the investigations of the Ph.D. thesis. Thereafter, the chapter presents a dedicated section to elaborate upon the possible direction for further research in the specific field of leafy and non-leafy vegetable soup formulations based on abundant, endemic and underutilized horticultural produces of NE India.

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Chapter 2:

Materials and Methods



Materials and Methods

In this chapter, further details with respect to the targeted experimental, analytical and optimization methodologies have been deliberated with respect to the fulfillment of the mentioned objectives of the Ph.D. thesis. Section 2.1 presents a brief summary of relevant materials and sample preparation procedures. Following this, section 2.2 addressed methodologies associated to the identification of optimal drying method based on nutritional content of the dried samples. In section 2.3, procedures associated to kinetic studies and trial and error based approach have been summarized to obtain optimal drying process parameters. These refers to drying kinetics, fitness of drying models, determination of diffusivities and activation energies of dried samples, variation of nutritional parameters with drying temperature and time. Section 2.4 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 2.5 deliberates upon the procedures associated to the development and characterization of leafy and non-leafy vegetable mix based soup formulations. The section presents a mature methodology based on mathematical optimization based vegetable soup mix optimization and subsequent strategies associated to the sensory analysis based vegetable soup formulation optimization. Eventually, the characterization procedures also considered rheological characterization for optimal water based reconstituted vegetable mix soup system.

2.1 Materials

2.1.1 Raw Materials

Fresh leafy and non-leafy vegetables were purchased from Shingimari, Kamrup, Assam, India (26.22° N and 91.62° E) and were thereafter placed in enclosed polythene pouches. The vegetables being investigated in the thesis refers to Kolmou (*Ipomoea aquatica* Forssk.), Jatilao leaves (*Lagenaria siceraria* leaves) and Pui (*Basella alba*) among leafy vegetables and Kaskal (*Musa splendida*), Kolphul (*Musa balbisiana* Colla), green Komora (*Benincasa hispida*) and Posola (*Musa balbisiana* Colla. pseudo-stem) among non-leafy vegetables. Among these vegetables, matured Kolmou and Pui samples are abundantly available during rainy season (August - November). On the other hand, other vegetables are available throughout the year.

2.1.2 Other Chemicals

2,2-diphenyl-1-picrylhydrazyl (DPPH) extrapure, 2, 6-dichlorophenol indophenol sodium salt (DCPIP), Bovine Serum Albumin and anthrone extrapure were purchased from SRL Pvt. Ltd., India; oxalic acid dehydrate, sodium hydroxide pellets, dextrose, sulfuric acid (98%), petroleum ether and absolute methanol were purchased from Merck India and Bradford Reagent, sodium bicarbonate were procured from Sigma Aldrich, India and Rankem, respectively.

2.1.3 Sample Preparation

After sorting, the vegetable of specific investigation was washed in running water to remove materials adhering to its surface. Following this, the vegetable was rinsed to remove excess water. For leafy vegetable case, a single layer of detached leaves (from stalk) was subjected to drying by placing it on a tray. Average size of Kolmou, Jatilao and Pui vegetables were 8.5 cm ×

3 cm × 0.02 cm, 11 cm × 13.5 cm × 0.027 cm and 11.5 cm × 8.5 cm × 0.029 cm, respectively. On the other hand, for non-leafy vegetables, Kaskal, Posola and Komora were cut into circular slices with 1 mm thickness and of 3.5, 5 and 8 cm average diameter, respectively. For the non-leafy vegetable case, a single layer of the slices was subjected to drying by placing them on a tray. For the Kolphul case, the outer two to three bracts were discarded. Thereafter, the subsequent blossoms were cut vertically into two equal halves and kept on a tray as a single layer for drying. The Kolphul sample size was 8.6 cm × 0.5 cm × 0.1 cm.

2.2 Optimization of Alternate Drying Methods (Oven and Tray Drying) for the Maximum Retention of Nutritional Constituents

2.2.1 Drying Methods

For the specific leafy or non-leafy vegetable being investigated, drying experiments were conducted using laboratory scale oven (model: assembled, make: REICO) or intermittent airflow assisted tray dryer (model: assembled, make: International Commercial Traders, Kolkata, India). Targeting the fulfillment of the first objective of the Ph.D. thesis, the oven drying temperature and time has been chosen based on few trials and available literature data as 60 °C and 4 h for leafy vegetables whereas 10 h for non-leafy vegetables. For the tray drying process as well similar drying temperature and time have been set. Along with these values, the tray drier was operated at a constant air velocity of 4.5 m/s and a pulse of 20/20 sec (on/off mode) to enable intermittent airflow. The dried samples were subsequently grinded to foster further analysis.

2.2.2 Nutritional and Proximate Analysis

The oven and tray dried samples were subjected to several nutritional and proximate characterizations. These have been presented in the following subsections:

2.2.2.1 Yield

Based on sample weight measured before and after oven/tray drying, the yield was evaluated using the expression:

$$Yield(\%) = \frac{W_s}{W_r} \times 100 \quad (2.1)$$

where, W_r and W_s correspond to the fresh and oven / tray dried sample weights, respectively.

2.2.2.2 Moisture Content

The moisture content of a sample was determined after drying it at 105 °C in an oven for about 12 h (AOAC 2010). Thereby, using measured weights of the sample prior to drying and after drying, the moisture content of the sample was determined using the expression:

$$MC(\%) = \frac{W_s - W_d}{W_s} \times 100 \quad (2.2)$$

where, W_d and W_s correspond to the sample weight measured before and after drying (at 105 °C), respectively.

2.2.2.3 Ash Content

Ash content of the sample was determined by following the AOAC 2010 method (AOAC 2010).

This referred to incineration of the sample at 600 °C for 6 h in a muffle furnace (model:

assembled, make: REICO). Thereby, based on the measured weights of the sample and ash, the ash content was evaluated using the expression:

$$\text{Ash}(\%) = \frac{W_a}{W_s} \times 100 \quad (2.3)$$

where, W_a and W_s correspond to the weights of ash and sample, respectively.

2.2.2.4 Crude Fibre Content

A acid-base hydrolysis method summarized by Sadasivam and Manikam (1992) was duly followed to determine crude fibre content of the sample. The relevant procedure has been elaborated as follows. Firstly, a mixture of 1g dried sample and 100 mL 1.25% H_2SO_4 was boiled for 30 minutes under constant stirring conditions using a magnetic hot plate stirrer setup. Thereafter, the sample was filtered using muslin cloth and was washed several times with boiling water to wash away the free acids. The residue obtained was subjected to similar treatment with 1.25% NaOH. Thereafter, the processed residue was washed with a boiling mixture of 50 mL water, 25 mL of alcohol and 1.25% H_2SO_4 . Thereby, the residue was washed again with boiling water and was dried at 130 ± 2 °C for 2 h in an oven. After cooling in a desiccator, the sample was subjected to ignition at 600 °C for 30 minutes in a muffle furnace. After subsequent cooling, the sample weight was measured. Finally, the crude fibre content was determined using the expression:

$$\text{Crude Fibre}(\%) = \frac{W_l}{W_s} \times 100 \quad (2.4)$$

where, W_l and W_s correspond to the weight loss in due course of ignition and initial sample weight, respectively.

2.2.2.5 Fat Content

The fat content was evaluated through the extraction of the dried sample with petroleum ether in a Soxhlet apparatus (AOAC 2010). Thereby, the sample was kept aside to enable evaporation of petroleum ether. In other words, the left over sample refers to the fat content of the original sample. Thereby, percentage fat content was determined using the expression:

$$Fat (\%) = \frac{W_f}{W_s} \times 100 \quad (2.5)$$

where, W_f and W_s correspond to extract and sample weights, respectively.

2.2.2.6 Soluble Protein Content

Bradford's method was adopted to determine the soluble protein content of the sample (Bradford 1976). The procedural hierarchy was as follows. Firstly, the dried sample (100 mg) was extracted with distilled water (100 mL). Thereafter, 1mL extract was mixed with 5 mL Bradford reagent. Thereby, the green coloured mixture was subjected to absorbance measurement at 595 nm using a UV-Visible spectrophotometer (Model No.: UV-2600, Make: Shimadzu, Singapore). Bovine serum albumin (BSA) was used to prepare standard solutions and calibration curves. The soluble protein content was expressed in terms of g/100g.

2.2.2.7 Carbohydrate Content

The carbohydrate content of the sample was evaluated using Clegg anthrone method (Okonwu and Enyinnaya 2016). The procedural hierarchy involved prior mixing of 1 g sample with 10 mL distilled water followed with further mixing with 13 mL of 62% perchloric acid. Thereafter, the mixture was continuously stirred for 20 minutes. Eventually, the mixture volume was adjusted to 250 mL using distilled water. Following this, using a Whatman filter paper (no. 1), the mixture

was filtered. Thereby, to 10 mL of the filtrate, distilled water was added to enhance its volume to 100 mL. Eventually, 1 mL of diluted filtrate was mixed with 5 mL of anthrone reagent. Similarly, a blank solution was prepared by mixing 1 mL distilled water and 5 mL anthrone reagent. Subsequently, both control and experimental samples were incubated for 12 minutes at 100 °C in a water bath. Finally, the absorbance of the samples was measured at 630 nm. For the sample, the % carbohydrate content was calculated using the expression:

$$\text{Carbohydrate (\%)} = \frac{A_s}{A_{std}} \times 25 \quad (2.6)$$

where, A_s and A_{std} correspond to the absorbance of the diluted and blank samples, respectively.

2.2.2.8 Crude Protein Content

The crude protein content was determined using a differential method and with the expression:

$$\text{Crude protein (\%)} = 100 - (\text{moisture} + \text{carbohydrate} + \text{fat} + \text{crude fibre}) \quad (2.7)$$

2.2.2.9 Vitamin C Content

The vitamin C content of a sample was evaluated using titration method mentioned in the literature (Sadasivam and Manickam 1992; Anjali et al. 2012; Ravula et al. 2017). A brief account of the procedure was as follows. Initially, 100 mg dried sample was blended with 10 mL 4% oxalic acid using mortar and pestle. Thereby, 5 mL of the extract was mixed with 10 mL of 4% oxalic acid. Subsequently, the mixture was titrated with 2, 6-dichlorophenol indophenol dye solution. Using 1 mL of 100 ppm ascorbic acid, standard solution and 5 mL 4% oxalic acid mixture, a blank run was conducted. Using measured titration volume values, the vitamin C content was determined using the expression:

$$\text{Vitamin } C \left(\frac{\text{mg}}{100\text{g}} \right) = \frac{0.5 \times V_2 \times 10 \times 100}{V_1 \times 5 \times W_s} \quad (2.8)$$

where, V_1 , V_2 and W_s correspond to mL of ascorbic acid consumed, sample extract consumed and sample weight, respectively.

2.2.2.10 Antioxidant Activity

The antioxidant activity was evaluated using 2,2-diphenyl-1-picrylhydrazyl (DPPH) scavenging assay method elaborated in the relevant prior art (Barimah et al. 2017; Sana et al. 2014; Sochor et al. 2010). A brief account of the procedure was as follows. Firstly, 10 mg dried sample was extracted with 20 mL of absolute methanol (in a mortar and pestle). Thereby, the extract was subjected to sonication (model: Elmasonic S 30 H, make: Elma) for 30 minutes. Following this, filtration was carried using Whatman no.1 filter paper. Thereafter, about 1 mL of extract was mixed with 3 mL of 0.002% methanolic DPPH solution. Similarly, a control sample was prepared with 1 mL absolute methanol and 3 mL methanolic DPPH solution. After thorough and vigorous shaking, both samples were incubated in a dark environment for 30 minutes. Finally, for both experimental and control samples, the absorbance was measured at 517 nm using UV spectrophotometer against the blank (absolute methanol). The antioxidant activity of the sample was eventually determined using the expression:

$$AA = \frac{A_c - A_s}{A_c} \times 100 \quad (2.9)$$

where, A_c and A_s correspond to the absorbance of control and experimental samples, respectively.

2.3 Drying Kinetics of Leafy and Non-Leafy Vegetables

2.3.1 Drying Methods

The leafy and non-leafy vegetable samples were subjected to drying in a laboratory based tray drier (Assembled, International Commercial Traders, Kolkata, India) that facilitated with intermittent airflow. The tray drier was operated for a constant average air velocity of 4.5 m/s and 20/20 s periodicity of run and stagnation. Such conditions foster realization of equilibrium conditions. The stationary phase of operation during intermittent drying process is very likely to detriment moisture removal from the processed vegetable samples. Thus, higher flow rate has been ensured to achieve better drying characteristics. The periodic airflow would facilitate moisture removal through vaporization based removal from the sample surface during the onset phase. However, since moisture may accumulate during the stagnant phase of the air flow, higher velocity of the air fosters effective removal of the moisture and to a greater extent. Thus, the reported data in this work aims to provide ideal operational scenarios of tray dried mentioned horticultural produces. Drying kinetics was conducted in the temperature range of $40 - 80\text{ }^{\circ}\text{C} \pm 1$. These experiments were carried out until constant sample weight was achieved. However, investigations targeting the variations of vitamin C and antioxidant activity were conducted in the temperature range of $50 - 70\text{ }^{\circ}\text{C} \pm 1$. This was due to the following justification. Preliminary investigations affirmed that the lower temperature ($40\text{ }^{\circ}\text{C}$) takes very long duration to dry the samples completely. On the contrary, higher temperature ($80\text{ }^{\circ}\text{C}$) was detrimental to vitamin C and antioxidant activity. Further, since moisture diffusivity and activation energy requires at least five data points for an optimal regression methodology, moisture kinetics were carried out

additionally for 40 and 80 °C cases. For all cases, the experimental investigations have been carried out by considering a temperature interval of 10 °C.

2.3.2 Drying Characteristics Curve

The variation of moisture with drying time and temperature was evaluated through a periodic measurement of sample weight during the entire duration of drying. Such experiments were terminated upon achieving a constant sample weight. Thereby, drying characteristics curves have been prepared for variant drying temperature of 40 – 80 °C. The curves have been represented as time dependent variation of moisture ratio (MR), expressed as:

$$MR = \frac{M_t - M_e}{M_0 - M_e} \quad (2.10)$$

where, MR , M_0 , M_t and M_e refers to moisture ratio, initial moisture content, moisture content at any time and equilibrium moisture content, respectively for a fixed choice of drying temperature.

As mentioned previously, the variation in vitamin C and antioxidant activity with drying time were evaluated in the temperature range of 50 – 70 °C. For this purpose, the samples were analyzed periodically for time duration of 1 h and 2 h for leafy and non-leafy vegetable samples, respectively. Such analyses were terminated after achieving constant weight of the sample.

2.3.3 Identification of Best Fit Drying Model

The MR data with respect to drying time for various drying temperature cases were subjected to fitness towards seven empirical and non-empirical drying models (**Table 2.1**). Among the chosen models, the best fit model was evaluated in terms of combinations of highest R^2 and lowest residual sum of squares (RSS) and reduced chi-squared values.

Table 2.1 A summary of alternate drying models being considered to represent pertinent tray drying kinetics of leafy and non-leafy vegetable systems.

S. No.	Model	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	Wang and Singh	$MR = M_0 + at + bt^2$
6	Singh et al. (2014)	$MR = \exp(-kt) - akt$
7	Silva et al. (2012)	$MR = \exp(-at + bt^{1/2})$

2.3.4 Moisture Diffusivity and Activation Energy

The moisture diffusivity and activation energy of various tray dried vegetable sample systems were determined according to the procedure mentioned in the relevant prior art (Wang et al. 2018; Olawoye et al. 2017; Esmaelian et al. 2016; Limpaboon 2011; Gharehbeglou et al. 2014; Mghazli et al. 2017; Doymaz 2012). Considering each sample as a series of infinite slabs, Fick's 2nd law of diffusion was applied to determine the moisture diffusivity. The Fick's 2nd law of diffusion can be expressed as:

$$\frac{\partial M}{\partial t} = D_{diff} \frac{\partial^2 M}{\partial x^2} \quad (2.11)$$

where, D_{diff} , M and x refer to the diffusivity, moisture content and thickness of sample, respectively.

The solution to the above partial differential equation was given by Crank equation (Crank 1975). The author obtained the said solution by considering constant temperature, negligible shrinkage for the assumed diffusion based transport phenomenon. Thereby, Crank equation can be written as:

$$MR = \frac{8}{\pi^2} \sum_{n=0}^{\infty} \frac{1}{(2n+1)^2} \exp\left[-(2n+1)^2 \frac{\pi^2 Dt}{4L^2}\right] \quad (2.12)$$

where, MR , D and t correspond to moisture ratio, effective moisture diffusivity and drying time, respectively.

Neglecting the higher order terms, the simplest form of equation can be written as:

$$MR = \frac{8}{\pi^2} \exp\left[-\frac{\pi^2 Dt}{4L^2}\right] \quad (2.13)$$

Thus, logarithmic plot of eq. (2.13) shall affirm a straight line (slope and intercept of $-\frac{\pi^2 D}{4L^2}$ and

$\ln \frac{8}{\pi^2}$, respectively). Thereby, moisture diffusivity can be determined from the slope of the plot.

Similarly, the activation energy was determined using Arrhenius equation:

$$D = D_0 \exp\left(-\frac{E_a}{RT}\right) \quad (2.14)$$

where, E_a , R and T correspond to the activation energy, gas constant and drying temperature (K), respectively.

2.4 Response Surface Methodology based Drying of Leafy and Non-Leafy Vegetables

2.4.1 Drying Method

The drying methodology was similar to that presented in section 2.3.1 of the Ph.D. thesis. The precise combinations of drying temperature and time were based on central composite design (CCD) approach of the RSM. These data sets were deduced using Design expert 7.0 software.

The bias for the software was set based on few preliminary drying studies at lowest and highest combinations of drying temperature and time.

2.4.2 Experimental Design

For the RSM design approach, rotatable CCD based design was deployed to design drying experiments (The CCD based RSM design provides an efficient statistical design based model to gain useful information with respect to the influences of the chosen degrees of freedom and associated errors. The CCD operational modes involves either rotatable or face-centered or inscribed design options and thereby facilitates reasonable selection of options to supplement desired combinations of operability and interested regimes of variable range search. The rotatable CCD facilitates consideration of points outside the set boundaries and hence a better scenario to achieve optimization of the process variables. On the other hand, since the number of experiments at the central level is sufficiently large, it thereby ensures better accuracy of the developed model.) in the drying temperature range of 50 – 70 °C and drying time range of 90 – 300 min, 150 – 390 min, 240 – 420 min, 390 – 720 min, 240 – 450 min, 360 – 630 min and 240 – 420 min for Kolmou, Jatilao, Pui, Kolphul, Kaskal, Komora and Posola, respectively. The drying time range for the CCD based RSM design was set based on the detailed drying kinetics and preliminary investigations being carried out for each vegetable to achieve constant weight, vitamin C and antioxidant activity characteristics in due course of longer periods of tray drying. These have been delineated in the kinetics studies section of the PhD thesis. According to the CCD based RSM design, 14 alternate data sets (with a replicate of 6 data sets at the central level) have been generated for which responses such as moisture content, vitamin C and antioxidant activity were assessed. For each case, triplicate experiments were conducted for each combination of drying time and temperature. This was considered to ensure that the operational

and measurement errors do not propagate in the determination of best fit model. Thereafter, average values of the variables have been evaluated along with their standard deviations. Thereby, repeatability has been assured of the measured data and confidence levels have been ensured to supplement RSM analysis with minimum possible error from the experimental investigations perspective. For the response variables, appropriate generalized model can be expressed as:

$$Y = A_0 + \mu_1 A + \mu_2 B + \mu_3 AB + \mu_4 A^2 + \mu_5 B^2 + \varepsilon \quad (2.15)$$

where, A_0 denotes a constant, A and B are drying temperature and time, μ_1 and μ_2 represent linear coefficients, μ_3 and μ_4 correspond to quadratic coefficients, μ_5 refers to the coefficient of interaction term variable. ε refers to the error associated to represent the experimental response data.

2.4.3 Statistical Analysis

With a multiple regression based modeling approach of dependent (responses) and independent (drying temperature and time) variables, the statistical data was obtained using design expert 7.0 software. Among various alternate models, the best fit model was chosen based on a high model F-value and a low p-value. The analysis of variance (ANOVA) approach facilitated the generation of useful and statistical parameters for each response and for all samples. Thereby, the statistical parameters such as F and p-values, lack of fit, R^2 , adjusted R^2 , predicted R^2 value, C.V. and PRESS value provided a bias for the selection of best fit model. These biases have been considered as follows for the best fit model.

- a) High F-value and low for all model terms
- b) Non-significant lack of fit affirmed by low F-value

- c) Closeness of all R^2 values
- d) C.V. values greater than 4

2.4.4 Optimization and Validation

For alternate responses, the best fit model was subjected to optimization using numerical optimization tool of design expert 7.0 software. The optimization targeted the simultaneous minimization of moisture content and maximization of vitamin C and antioxidant activity through the optimization of drying temperature and time. The optimal set of independent variable and response values have been validated experimentally by conducting triplicate runs. Thereby, the validity of optimal value was ensured through evaluated standard.

2.4.5 Response Analysis

Response variables namely moisture content, vitamin C and antioxidant activity have been evaluated by following the procedure summarized in sections 2.2.2.2, 2.2.2.9 and 2.2.2.10 of the Ph.D. thesis, respectively.

2.5 Formulation and Characterization of Leafy and Non-Leafy Vegetable based Soup Recipe

2.5.1 Evaluation of Optimum Leafy Vegetable Mix Formulation

The best non-linear expressions to represent tray drying characteristics of chosen leafy vegetables can be adopted for the design and development of leafy vegetable soup formulations. A non-linear programming (NLP) model could be developed that targets the nutritional optimization of the dry leafy vegetable soup mix. Since vegetables can be dried independently or

together, two alternate solution strategies exist to achieve nutritionally optimal dry leafy vegetable mix. To do so, either individually dried or co-dried optimal dried vegetable mix formulations could be obtained using a non-linear programming solver. In this work, generalized reducing gradient method (GRG) was adopted in Microsoft Excel to achieve the desired optimal dry leafy vegetable mix formulation. Upper and lower bounds have been set for the time and temperature based on the realistic assumptions and experimentation based insights. The objective function refers to the linear weighted function of desired maximum attributes (vitamin C and antioxidant activity) and minimum attributes (moisture content).

The drying temperature and time variables (independent variables) for each leafy vegetable have been denoted as T_k , T_j and T_p and t_k , t_j and t_p for Kolmou, Jatilao and Pui leafy vegetables, respectively. Accordingly, the objective function was set as:

$$Y = w_1E + w_2F - w_3G \quad (2.16)$$

where, E , F and G refer to vitamin C (mg/100g), antioxidant activity (%) and moisture content (%), respectively and w_1 , w_2 and w_3 correspond to positive weight factors. The positive signs for E and F functions and negative sign of G function affirm target towards the maximization and minimization of respective terms during optimization. Further, based on theory of ideal mixing Larrosa et al. (2015), these dependent variables have been expressed as follows:

$$E = \frac{w_k E_k + w_j E_j + w_p E_p}{w_k + w_j + w_p} \quad (2.17)$$

$$F = \frac{w_k F_k + w_j F_j + w_p F_p}{w_k + w_j + w_p} \quad (2.18)$$

$$G = \frac{w_k G_k + w_j G_j + w_p G_p}{w_k + w_j + w_p} \quad (2.19)$$

where, w_k , w_j and w_p refers to % weight of dried Kolmou, Jatilao and Pui, respectively in the dried vegetable mix. According to the above notation, (E_k , E_j and E_p), (F_k , F_j and F_p) and (G_k , G_j and G_p) correspond to vitamin C, antioxidant activity and moisture content of the respective sample. These can be determined from the ANOVA based analysis of obtained CCD based RSM data for the associated independent variables presented in eq. 1. i.e.

$$E_k, E_j, E_p, F_k, F_j, F_p, G_k, G_j, G_p = f_i(T, t) \quad (2.20)$$

where, $f_i(T, t)$ corresponds to best fit model and $i \in k, j$ and p .

To maximize the objective function mentioned in eq. 2.16, the following constraints will be applicable:

Upper and lower bounds of T_i and t_i where $i \in k, j$ and p (based on experimental investigations addressed prior to optimization):

$$50 \leq T_k, T_j, T_p \leq 70 \quad (2.21)$$

$$90 \leq t_k \leq 300 \quad (2.22)$$

$$150 \leq t_j \leq 390 \quad (2.23)$$

$$240 \leq t_p \leq 420 \quad (2.24)$$

Weight proportion constraint:

$$10 \leq w_k, w_j, w_p \leq 80 \quad (2.25)$$

Total weight constraint:

$$w_k + w_j + w_p = 100 \quad (2.26)$$

Maximum moisture content of any leafy vegetable in the mix:

$$G_k, G_j, G_p \leq 5 \quad (2.27)$$

Upper and lower bounds for E_i and F_i

$$80 \leq E_i \leq 90, \text{ where } i \in k, j \text{ and } p \quad (2.28)$$

$$100 \leq F_i \leq 140, \text{ where } i \in k, j \text{ and } p \quad (2.29)$$

The above summarized bounds have been set to formulate a non-linear programming model with tighter specifications. Thereby, minimum weightage to the vegetables was desired to restrict poor quality parameters. These boundary conditions are set to facilitate the highly non-linear system of equations with tighter specification of bonds and provide minimum weightage to vegetables with poor desired quality parameters.

For co-drying, since same drying temperature and time are applicable for all leafy vegetables, the above summarized non-linear programming optimization model can be further relaxed by imposing the following constraints:

$$50 \leq T_k = T_j = T_p \leq 70 \quad (2.30)$$

$$90 \leq t_k = t_j = t_p \leq 420. \quad (2.31)$$

In summary, eq. (2.16 – 2.29) and eq. (2.16 – 2.31) refer to the NLP model summary for individual and co-drying cases, respectively.

2.5.2 Evaluation of Optimal Non-Leafy Vegetable Mix Formulation

The NLP model formulation for the nutritional optimization of non-leafy vegetable dry mix is similar to the methodology presented in the previous subsections. The following paragraph and equations summarize the same. The model terms are similar with sub-scripts being replaced as a , b , c and d to represent Kolphul, Kaskal, Komora and Posola, respectively. The objective function

and mix responses are similar to eq. (2.16) and eq. (2.17 – 2.19), respectively. Bounds have been set as follows:

Drying temperature and time constraints:

$$50 \leq T_a, T_b, T_c, T_d \leq 70 \quad (2.32)$$

$$390 \leq t_a \leq 720 \quad (2.33)$$

$$240 \leq t_b \leq 450 \quad (2.34)$$

$$240 \leq t_c \leq 420 \quad (2.35)$$

$$360 \leq t_d \leq 630 \quad (2.36)$$

Weight proportion constraint:

$$10 \leq w_a, w_b, w_c, w_d \leq 80 \quad (2.37)$$

Total weight constraint:

$$w_a + w_b + w_c + w_d = 100 \quad (2.38)$$

Maximum moisture content for each dried vegetable:

$$G_a, G_b, G_c, G_d \leq 6 \quad (2.39)$$

Constraints for E_i and F_i are:

$$50 \leq E_i \leq 90, \text{ where } i \in a, b, c \text{ and } d \quad (2.40)$$

$$100 \leq F_i \leq 300, \text{ where } i \in a, b, c \text{ and } d \quad (2.41)$$

For co-drying case, additional constraints have been imposed as:

$$50 \leq T_a, T_b, T_c, T_d \leq 70 \quad (2.42)$$

$$240 \leq t_a = t_b = t_c = t_d \leq 720. \quad (2.43)$$

In summary eq. (2.16, 2.32 – 2.41) and eq. (2.16, 2.32 – 2.43) represent NLP model for optimal non-leafy vegetable dry mix formulation through individual and co-drying approaches, respectively.

2.5.3 Sensory Evaluation

Based on the optimal mix formulation of leafy and non-leafy vegetables achieved from the mathematical optimization, optimal soup formulations (F_M and F_{M1} for leafy and non-leafy based soup formulations, respectively) were prepared. Control formulations such as F_K , F_J and F_P for leafy vegetable based soup formulations were as well prepared with 100 % weight (5 g each) of either Kolmou or Jatilao or Pui, respectively. Similarly, for the non-leafy vegetable based soup formulation, formulations with 100% weight of either Kolphul or Kaskal or Komora or Posola (F_A , F_B , F_C and F_D) have been considered as control samples. Based on the obtained sensory scores, the control formulation serves as a useful reference to reduce or enhance a particular vegetable in the mix formulations (F_M and F_{M1}). **Table 2.2** presents the formulations targeted with other combinations of ingredients (fixed choices of other ingredients). These specific values were based on earlier literature (Srivastava et al. 2019; Chaudhari and Solanke 2016; Sugumar and Guha 2020; Dhiman et al. 2017) and few preliminary trials. Further, based on varied leafy vegetable mix to corn flour ratio, formulations (F_1 (20:80), F_2 (30:70), F_3 (40:60) and F_4 (50:50) have been achieved for sensory analysis. Similar investigation were conducted for non-leafy vegetable based soup formulations achieved through variant non-leafy vegetable mix to corn flour ratio (20:80 (F_5), 30:70 (F_6), 40:60 (F_7) and 50:50 (F_8). For all cases, the sensory evaluation was performed for soups prepared through a reconstitution of 20 g soup powder (vegetable mix + corn flour + other ingredients) with 400 mL boiling water and subsequent boiling for 10 min. In summary, reconstituted soups for control and mix soup samples with fixed choices of corn flour

and other ingredients (F_M , F_{M1} , F_K , F_J , F_P , F_A , F_B , F_C and F_D) and variant dry vegetable mix to corn flour ratio ($F_1 - F_8$) have been evaluated for their sensory characteristics.

Table 2.2: A summary of other ingredient compositions in alternate vegetable soup formulations.

Ingredients/Formulation	Amount (g)
Corn flour (g)	5
Ginger powder (g)	0.5
Onion powder (g)	0.5
Garlic powder (g)	0.5
Milk powder (g)	1
Salt (g)	3
Black pepper (g)	0.5
Vegetable oil (mL)	2
Carrot flakes (g)	2

Hedonic scale (9 - Pointer) was followed to evaluate the sensory characteristics such as texture, taste, flavour, appearance, consistency, mouth feel, after feel and overall acceptability. Similarly, such methodology was adopted in the available prior art (Kokani et al. 2019; Alozie and Ene-Obong 2018; Anita et al. 2016; Singh et al. 2017). A laboratory based sensory analysis was carried out with ten experienced experts from Indian Institute of Guwahati, India (5 males and 5 females of age group between 25 – 50 years). Also, these subjects have been trained with the sensory based preliminary investigations for soup products to instil confidence upon investigated sensory characteristics of alternate soup formulations. Each number of the hedonic scale represent likeliness starting from 1 (dislike extremely) to 9 (like extremely). Sensory responses corresponding to numbers (2, 3, 4, 5, 6, 7 and 8) on the hedonic scale, respectively represents dislike very much, dislike moderately, dislike slightly, neither like nor dislike, like slightly, like moderately and like very much, respectively., respectively

2.5.4 Rheological Characterizations of Soup Samples

Rheological investigations were conducted for the best reconstituted soup (inferred from sensory evaluation) using a Rheometer (Physica MCR 301, Anton Paar) equipped with CP50 (cone and plate geometry) probe. Steady state rheology was examined for varied shear rates of $0.1 - 100 \text{ s}^{-1}$ and temperatures ($30 - 50 \text{ }^\circ\text{C}$). Thereafter, using Power law and Herschel-Bulkley models, model fitness investigations were carried out for the obtained shear rate versus shear stress data. Thereby, the relevant flow behaviour of the samples was characterized (Chavan et al. 2015). These models can be expressed as:

$$\text{Power law: } \tau = K(\dot{\gamma})^n \quad (2.44)$$

$$\text{Herschel-Bulkley model: } \tau = \tau_0 + K(\dot{\gamma})^n \quad (2.45)$$

where, τ , τ_0 , K , $\dot{\gamma}$ and n denote shear stress (Pa), yield stress (Pa), flow consistency index ($\text{Pa}\cdot\text{s}^n$), shear rate (s^{-1}) and flow behaviour index, respectively.

The time dependent rheological study was conducted by considering viscosity and shear stress as desired response variables. These measurements were conducted for 10 s^{-1} shear rate, 300 sec time duration and at $30 \text{ }^\circ\text{C}$ temperature.

On the other hand, the sample was also subjected to dynamic flow based rheological evaluation (frequency varied from $0.01 - 10 \text{ Hz}$ and for a constant strain of 0.05%). The measurements were carried at $30 \text{ }^\circ\text{C}$. Thereby, relevant dynamic flow parameters such as storage modulus (G'), loss modulus (G'') and loss tangent (G'/G'') have been determined in such investigations.

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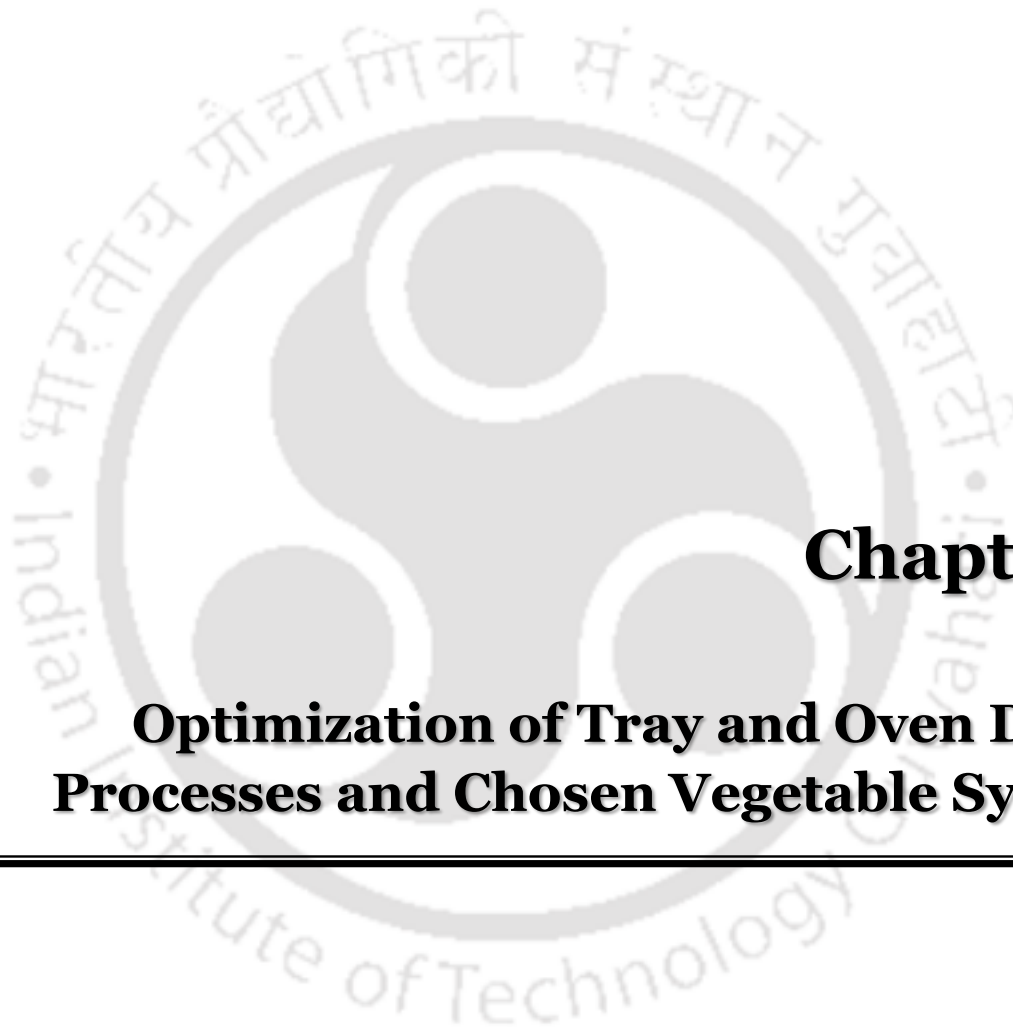
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Chapter 3:

Optimization of Tray and Oven Drying Processes and Chosen Vegetable Systems



Optimization of Tray and Oven Drying Processes and Chosen Vegetable Systems

In this chapter, the results associated with the optimization of alternate drying methods have been summarized from the perspective of the maximum retention of nutritional parameters. Due to their cost efficacy and scalability, oven and intermittent airflow assisted tray drying have been chosen as the alternate drying methods. While section 3.2 focuses towards the influence of oven and tray drying on the yield, sections 3.3, 3.4, 3.5 and 3.6, respectively present findings associated to the moisture content, ash content, crude fibre and fat content of the dried leafy and non-leafy vegetable samples. Thereafter, sections 3.7, 3.8, 3.9, 3.10 and 3.11, respectively summarize upon the findings related to soluble protein, carbohydrate, crude protein, vitamin C and antioxidant activity of dried leafy and non-leafy vegetable samples. Finally, section 3.12 presents an overall summary of the research findings in the context of best drying process among tray and oven drying methods.

3.1 Introduction

In this chapter, a comparative assessment of oven and intermittent airflow assisted tray drying methods has been targeted for the chosen leafy and non-leafy vegetables. The chosen leafy vegetables refer to Kolmou (*Ipomoea aquatica* Forssk.), Jatilao leaves (*Lagenaria siceraria* leaves) and Pui (*Basella alba*). On the other hand, the non-leafy vegetables being addressed in the thesis refer to Kolphul (*Musa balbisiana* Colla blossom), Posola (*Musa balbisiana* Colla

pseudo-stem), Kaskal (*Musa splendida*) and Komora (*Benincasa hispida*). Laboratory scale oven (model: assembled, make: REICO) and intermittent airflow assisted tray dryer (model: assembled, make: International Commercial Traders, Kolkata) have been deployed to dry leafy and non-leafy vegetable samples at 60 °C for 4 and 10 h, respectively. Further, the tray drying process involved drying of leafy and non-leafy vegetables at a constant air velocity of 4.5 m/s that was facilitated through automated intermittent airflow (20 sec in run and 20 sec in off-mode). Therefore, the dried samples were subjected to nutritional characterization. The evaluated nutritional characteristics include yield, moisture content, ash content, crude fibre content, fat content, soluble protein content, carbohydrate content, crude protein content, vitamin C and antioxidant activity.

3.2 Yield Characteristics

The Yield characteristic has been expressed in terms of the constant dried sample weight being obtained in due course of the termination of the drying process. Thereby, yield has been expressed as the ratio of dried sample weight per 100 g of the fresh sample. For all chosen vegetables, the yield data for oven and tray dried samples have been depicted in **Table 3.1 (a)** and **(b)**. The yield values varied from 11.41 – 14.06% and 10.99 – 13.29% for tray dried and oven dried leafy samples, respectively. Among chosen leafy vegetables, Jatilao possessed the highest yield (14.06% and 13.29% for oven and tray dried samples) and lowest yield (11.41% and 10.99% for oven and tray dried samples, respectively) correspond to Pui samples. Higher yield characteristic of Jatilao is due to its higher dry matter content. On the contrary, lower dry matter and higher moisture content of fresh Pui sample resulted in lower yield characteristic of the sample. The yield data varied from 4.25 – 15.86% and 4.61 – 16.64% for oven and tray dried

non-leafy vegetables, respectively. The yield trend was as per the following order for oven dried non-leafy vegetable samples: Komora < Posola < Kolphul < Kaskal for oven dried samples. Thus, highest and lowest yield was obtained for Kaskal (15.86%) and Komora (4.25%), respectively. For the tray dried non-leafy vegetables, the yield was as per the following order: Komora < Posola < Kolphul < Kaskal. Among these, Kaskal and Komora gave highest (16.64%) and lowest (4.61%) yields. The increasing trend of yield characteristics for the case has been due to enhancement in dry matter content of samples. On the contrary, the moisture content reduced with sample.

Table 3.1: Yield characteristics of tray and oven dried (a) leafy and (b) non-leafy vegetable systems.

(a)		
Sample/Drying method	Oven	Tray
Kolmou	13.3 ± 0.16	13.94 ± 0.21
Pui	10.99 ± 0.53	11.41 ± 0.32
Jatilao	13.15 ± 0.29	14.06 ± 0.36
(b)		
Sample/Drying method	Oven	Tray
Kolphul	9.26 ± 0.18	10.01 ± 0.09
Kaskal	15.86 ± 0.47	16.64 ± 0.28
Komora	3.35 ± 0.13	4.26 ± 0.4
Posola	5.45 ± 0.25	5.58 ± 0.14

The yield characteristics have been significantly influenced with the drying environment. Tray drying facilitated with intermittent airflow facilitated better retention of sample constituents and thereby promoted higher yield. During intermittent airflow facilitated tray drying, better heat penetration enabled uniform drying of products and better retention of sample constituents. The intermittent mode of airflow operation facilitates removal of moisture from samples during

circulation phase and movement of the moisture to the sample surface during stagnation phase. The direct heating effect was also reduced due to air circulation and due to this reason; moisture removal was comparatively higher for the case with respect to oven dried samples.

3.3 Moisture Content

Table 3.2 (a) and (b) depicts the variations in the moisture content (MC) of leafy and non-leafy vegetables for oven and tray drying processes, respectively. The initial moisture content of Kolmou, Jatilao, Pui, Kolphul, Kaskal, Komora and Posola were 86.65, 87.02, 89.78, 90.72, 84.09, 96.05 and 94.49% (standard deviation < 2), respectively. The table data affirms significant variation in the MC for oven and tray dried samples. For all cases, better moisture removal occurred during tray drying. The moisture content of oven and tray dried samples varied from 3.05 – 11.51% and 1.56 – 10.44%, respectively. The variation in moisture content of dried leafy and non-leafy vegetables is due to their variant composition, structure, texture, dry matter and moisture content. Among leafy vegetables, highest and lowest moisture content was found for the dried Pui (11.51%) and Kolmou (4.21%) among vegetables. Among non-leafy vegetables, oven dried Komora (8.54%) and tray dried Posola (1.56%) possessed highest and lowest moisture content, respectively. In summary, better moisture removal was apparent in tray drying but not oven drying of the samples. The final moisture content of the leafy and non-leafy vegetables varied from 4.21 – 11.51% and 1.56 – 8.54%, respectively. The variant moisture content of tray dried samples is attributed to variation in their composition, structure, texture, dry matter and moisture content. For better shelf life and storage of leafy and non-leafy vegetables, the final moisture content has been recommended to be about 4 – 10% in the literature. Thus, for most vegetables, the final moisture content of the leafy and non-leafy vegetables has been in the

appropriate range associated to long term storage. Only for the Pui vegetable, the higher final moisture content of 11.51% can be reduced through additional drying time of 10%. In summary, all vegetables after tray drying can be inferred to be safe for long term storage. The available prior-art for moisture content of the targeted samples affirms lower moisture removal than that being reported in the thesis for tray dried samples (Arinola et al. 2009; Ma et al. 2016). This could be due to variations in time and temperature combinations, adopted drying method and source of the samples.

Table 3.2: Moisture content characteristics of oven and tray dried (a) leafy and (b) non-leafy vegetable systems.

(a)		
Sample/Drying method	Oven	Tray
Kolmou	5.79 ± 0.08	4.21 ± 0.12
Pui	11.51 ± 0.43	10.44 ± 0.28
Jatilao	9.11 ± 0.12	7.68 ± 0.30
(b)		
Sample/Drying method	Oven	Tray
Kolphul	8.08 ± 0.17	7.34 ± 0.11
Kaskal	3.54 ± 0.04	4.36 ± 0.13
Komora	8.54 ± 0.19	7.30 ± 0.09
Posola	3.05 ± 0.07	1.56 ± 0.15

Also, except for Kaskal that confirmed better moisture removal through oven drying, all other leafy and non-leafy vegetables exhibited lower moisture content for tray dried but not oven dried samples. These moisture content trends are comparable with Satwase et al. (2013). The exceptional data of Kaskal is compatible with the observations of Arinola et al. (2009) with respect to conducted oven, tray, sun and fluidized bed drying of unripe banana plantain.

Similarly, Sakhale et al. (2007) and Kenghe et al. (2015) inferred that tray dried curry leaves possessed lower moisture content than sun and shade dried products.

The primary reason for such trends is due to the fact that while oven drying is deprived of air circulation and facilitates dry heat, the tray drying facilitates drying as well as the better environment in terms of appropriate lower humidity in the circulated air in the drying process. Intermittent airflow facilitates moisture removal during flow phase of air. However, during the stagnant phase that does not involve the flow of air, the moisture is very likely to get accumulated at the surface through transport from inner portions of the sample. Thus, due to better driving force for moisture transport within the sample during intermittent air flow assisted tray drying, better moisture removal efficiency can be achieved. On the other hand, for the Kaskal sample, deviant drying characteristics have been obtained i.e., for starchy banana (Kaskal) and tray drying case, moisture removal was marginally lower for similar drying time as that of the oven drying. Arinola et al. (2009) also reported better efficiency of oven drying in comparison with tray drying of unripe banana. The exceptional case of Kaskal is possibly due to the presence of water in the form of gummy fluid on its surface which is largely unaffected even with the circulation of air. On the other hand, during oven drying, dry heating facilitated effective moisture removal from Kaskal in comparison with tray drying. Thus, oven drying facilitated greater penetration of heat into the sample and this was not the case for the tray drying system. Also, for the tray drying case, the circulating air is possibly picking up the heat and thereby does not allow significant moisture removal from the kaskal sample surface. Therefore, for the Kaskal vegetable, compared to oven drying, tray drying process facilitated marginally lower moisture removal for similar conditions of drying time and temperature.

3.4 Ash Content

Table 3.3 (a) and **(b)**, respectively present the ash content data of tray and oven dried leafy and non-leafy vegetables. Better retention of ash content can be observed for all samples being processed with intermittent airflow based tray drying process. Among all leafy vegetables, Pui possessed high ash content (15.26% and 16.32%) for both drying processes. On the other hand, among non-leafy vegetables, oven and tray dried posola (11.72% and 12.42%, respectively) possessed the highest ash content. Irrespective of drying method, lowest ash content was found in Kaskal (4.20% and 4.55% in oven and tray drying). Among the leafy vegetables, Kolmou possessed the lowest ash content for both oven (10.08%) and tray (10.98%) dried cases.

Table 3.3: Ash content characteristics of oven and tray dried **(a)** leafy and **(b)** non-leafy vegetable systems.

(a)		
Sample/Drying method	Oven	Tray
Kolmou	10.08 ± 0.12	10.98 ± 0.07
Pui	15.26 ± 0.27	16.32 ± 0.18
Jatilao	12.13 ± 0.21	13.88 ± 0.13
(b)		
Sample/Drying method	Oven	Tray
Kolphul	9.25 ± 0.38	10.84 ± 0.16
Kaskal	4.20 ± 0.08	4.55 ± 0.14
Komora	7.22 ± 0.36	8.22 ± 0.45
Posola	11.72 ± 0.09	12.42 ± 0.15

The high ash content after tray drying for the mentioned samples is due to lower moisture content in tray dried samples and the combined effect of heat and intermittent air flow during tray drying. However, much variation did not exist in the ash content of tray and oven dried samples. This is due to the reason that the ash content of vegetables is marginally influenced

with airflow and heat. Satwase et al. (2013) and Arinola et al. (2009) also reported marginal variations in the ash content of tray and oven dried drumstick leaves and unripe banana plantain samples. This indicates a relatively inertness of the parameter with respect to the prevalent drying environment. Therefore, the variation in ash content with drying method was insignificant.

3.5 Crude Fibre Content

The crude fibre data for the oven and tray dried samples has been summarized in **Fig. 3.1 (a)** and **(b)**. For the tray dried samples, the crude fibre content varied from 1.08 – 15.67% with the highest crude fibre obtained for Jatilao (12.15%) and Posola (15.67%) among leafy and non-leafy vegetables, respectively. Incidentally, lowest fibre content was obtained for Poi (6.53%) and Kaskal (1.08%). On the other hand, the crude fibre content of oven dried vegetables varied from 1.36 to 15.86% with the highest content being analyzed for Jatilao (12.55%) and Posola (15.86%) among leafy and non-leafy vegetables, respectively. These values are in agreement with literature reported values for other vegetables as well. The oven dried samples had higher crude fibre content than tray dried samples and this is in agreement with the trends reported by Satwase et al. (2013). Similar to the results of Arinola et al. (2009), Kaskal possessed similar values of crude fibre content for tray (1.08%) and oven (1.36%) dried samples. Despite lower values of crude fibre content for tray dried samples, the equivalent fibre content is high for all cases and this is due to higher yield and lower moisture content of the samples. This confirms superiority of tray drying with respect to oven drying in both relevant prior art and this work. However, the variation in crude fibre content of oven and tray dried samples was not significant.

This is due to non-significant sensitivity of the variable towards heat and drying environment for both cases.

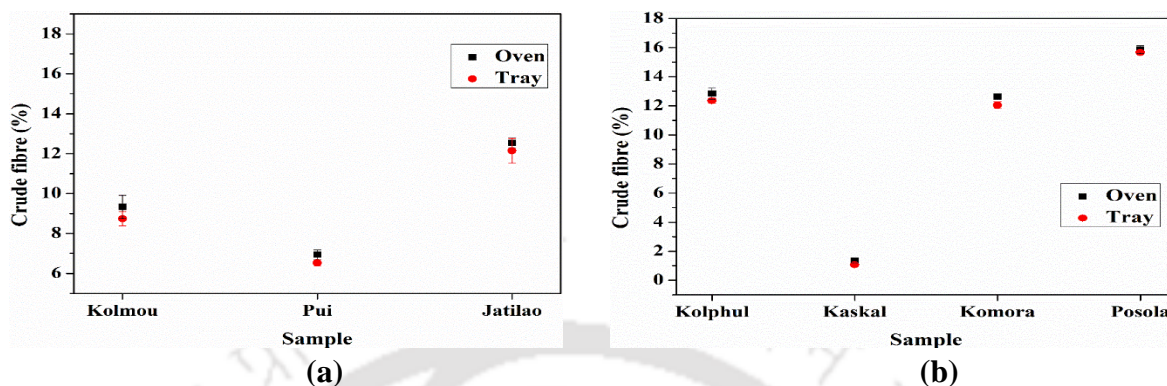


Fig. 3.1: Crude fibre characteristics of oven and tray dried (a) leafy and (b) non-leafy vegetables.

3.6 Fat Content

As shown in **Fig. 3.2 (a)** and **(b)**, the fat content of leafy and non-leafy vegetables is in the order of Jatilao > Kolmou > Pui and Kolphu > Posola > Komora > Kaskal, respectively. The highest fat content was obtained for Jatilao (8.67% and 8.06% for tray and oven dried samples) followed by Kolphul (8.57% and 8.22% corresponding values, respectively). The lowest fat content was obtained for Pui (3.39 and 3.87% for oven and tray drying process) in the leafy vegetables and Kaskal (2.68 and 2.93% corresponding values, respectively). Due to variations in the sample sources and time-temperature combinations, the obtained values for tray dried samples for few vegetables have been marginally different from the values reported in the literature. This affirms higher fat values for tray dried samples reported in this work in comparison with the literature reported values (Satter et al. 2016; Tongco et al. 2015; Arinola et al. 2009; Ma et al. 2016). The mode of drying had a relatively negligible effect on the fat content and the fat content remained intact irrespective of the deployed drying methods. This is due to the fact that the fat content of the samples did not deteriorate during the drying process. Thus, the is relatively independent of

heat and airflow. The possible reason for marginally higher values for the tray dried samples is due to their comparatively lower moisture content and higher yield with respect to oven dried samples.

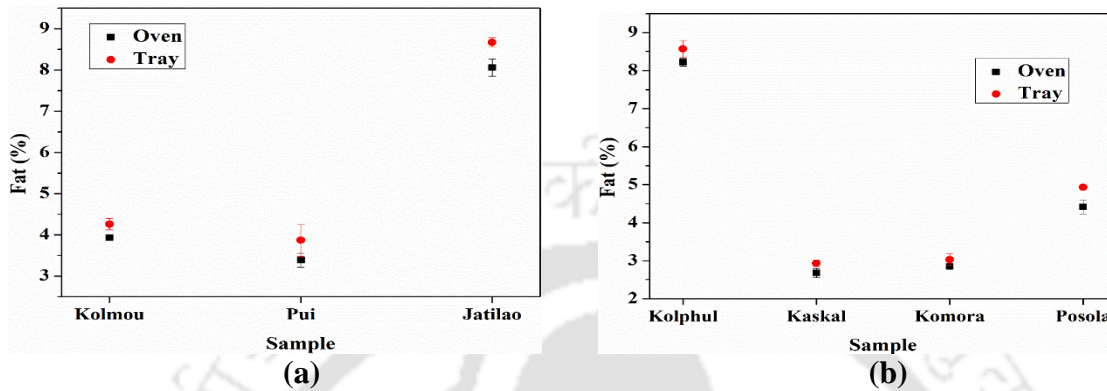


Fig. 3.2: Fat content characteristics of oven and tray dried (a) leafy and (b) non-leafy vegetables.

3.7 Soluble Protein Content

A comparative analysis of tray and oven dried samples with respect to soluble protein content has been depicted in **Fig. 3.3 (a) and (b)**. For leafy vegetables, soluble protein content varied from 9.91 – 14.62% and 8.44 – 12.56% for the tray and oven dried samples, respectively. On the other hand, for non-leafy vegetables, these values correspondingly varied from 2.99 – 8.26% and 2.18 – 5.98%, respectively. Among the leafy vegetables, Jatilao (14.62% and 12.56% in tray and oven drying) and Pui (9.91% and 8.44% in tray and oven drying) exhibited highest and lowest soluble protein content. Among non-leafy vegetables, Kolphul (8.26% in the tray and 5.98% in oven drying) exhibited the highest soluble protein content. Further, it can be analysed that tray dried Kaskal (2.99%) and oven dried Posola (2.18%) possessed lowest soluble protein content. In summary, the soluble protein content has been found to be higher for tray dried but not oven dried samples. Similar inferences have been presented by Satwase et al. (2013) for the protein content of drumstick leaves.

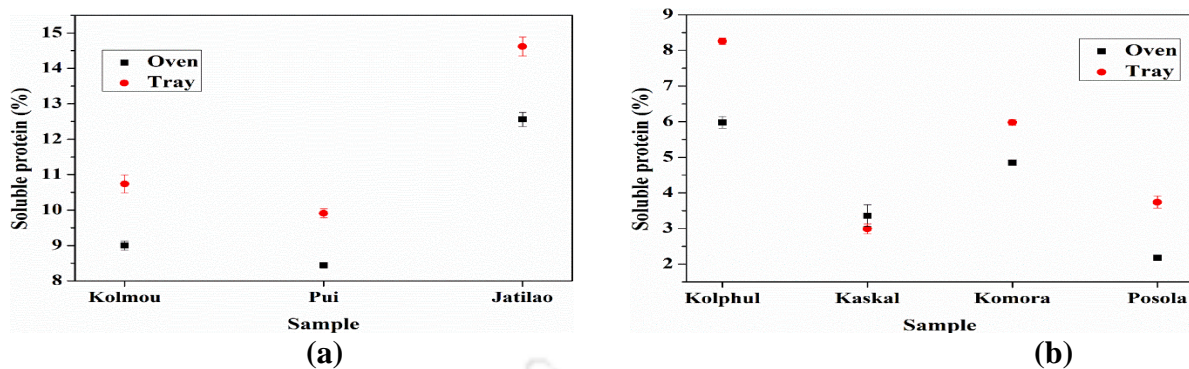


Fig. 3.3: Soluble protein content characteristics of oven and tray dried (a) leafy and (b) non-leafy vegetables.

The uniform and intermittent circulation of air is responsible for the tray drying process to facilitate higher soluble protein content in the vegetables. Intermittent circulation of air facilitates rapid and effective drying of the samples. For a component to come out of intra and inter-cellular matrix, drying time should be sufficient. Such drying time is highly dependent on the nutritional and other constituents of the sample. For the tray drying case, the drying has been rapid and effective in comparison with oven drying. Such an effect is likely to have minimized the oozing of the soluble protein to reach the sample surface and hence minimal loss of the variable. Thus, the loss of soluble protein content was comparably lower for tray dried samples than for the oven drying case. Also, for the oven drying case, dry heat might have greater degradation effect on soluble protein due to its solubility and heat sensitivity. On the other hand, the dry matter content of the tray dried samples was higher in comparison with the oven dried sample case. Thus, considering all these effects, the observed data trend is herewith justified.

3.8 Carbohydrate Content

The effect of drying methods on the carbohydrate content of the samples has been depicted in **Fig. 3.4 (a) and (b)**. The carbohydrate content of oven dried leafy and non-leafy vegetables were

found to vary from 41.22 – 51.08% and 53.95 – 82.06%, respectively. For the tray dried samples, these values, respectively varied from 39.61 – 49.27% and 50.52 – 81.37%. Highest carbohydrate content for tray and oven drying method was found for Kaskal vegetable (82.06% and 81.37%, respectively). Jatilao (41.22% and 39.61%, respectively) possessed lowest carbohydrate content. Among the leafy vegetables, Kolmou (51.08% and 49.27% in tray and oven drying) possessed highest carbohydrate content in respective drying methods. In general, oven dried samples had higher values than the tray dried samples. This is possibly due to the loss of other components during oven drying to simultaneously enhance the % carbohydrate content of the oven dried sample. On the other hand, carbohydrate was relatively inert towards heat and drying conditions that caused its minimal degradation. Therefore, loss of other nutritional components led to its higher value per 100 g of dried sample. Similar results have been reported by Satwase et al. (2013) and Arinola et al. (2009) for drumstick leaves and banana plantain samples, respectively.

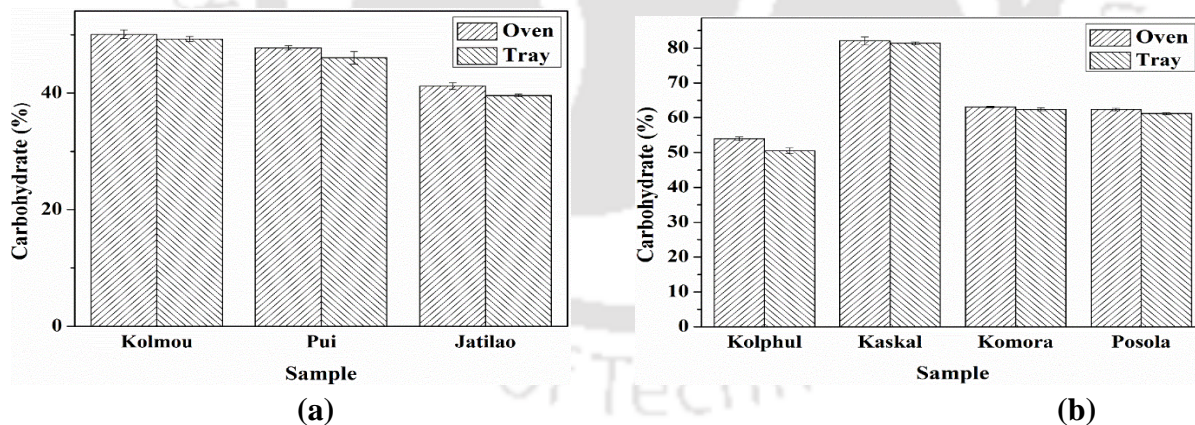


Fig. 3.4: Carbohydrate content characteristics of tray and oven dried (a) leafy and (b) non-leafy vegetables.

3.9 Crude Protein Content

Fig. 3.5 (a) and (b) depict the results obtained for crude protein content. The crude protein content of tray and oven dried leafy vegetables varied from 16.80 – 22.54% and 15.12 – 19.80%, respectively. For the non-leafy vegetables, corresponding values varied from 4.23 – 10.38% and 2.59 – 7.68%, respectively. Among tray dried samples, Kolmou (22.54%) followed by Jatilao (18.02%) possessed the highest crude protein content. Similarly, among oven dried samples, Kolmou (19.80%) and Jatilao (16.94%) possessed highest crude protein content. Among non-leafy vegetables, lower crude protein content was measured for Posola (2.59 and 4.23% for oven and tray dried samples). The obtained data have been consistent with literature reported values (Satter et al. 2016; Tongco et al. 2015; Awedem et al. 2015; Arinola et al. 2009; Ma et al. 2016).

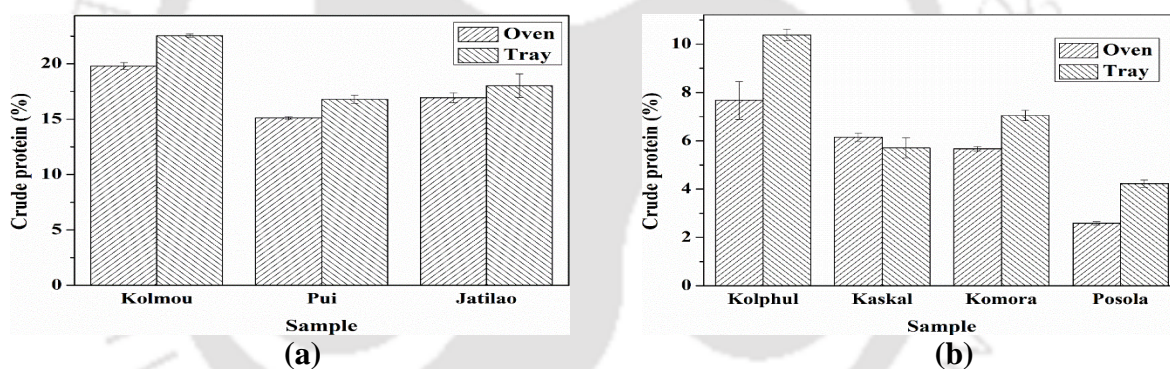


Fig. 3.5: Crude protein characteristics of tray and oven dried (a) leafy and (b) non-leafy vegetables.

It has been inferred that the crude protein content of the chosen vegetables have been higher than literature reported values. Thus, it is apparent that tray drying performed better to enable higher protein content of the samples. The higher value for this case is due to the greater retention of soluble protein than that being retained for the oven drying case. For the oven drying case, due to detrimental effect of soluble protein, the value for crude protein got reduced significantly. This is in agreement with the results reported by Satwase et al. (2013) for

drumstick leaves. On the other hand, oven dried Kaskal exhibited higher protein content and the obtained value is in good agreement with that reported for banana plantain by Arinola et al. (2009).

3.10 Vitamin C Content

Fig. 3.6 (a) and (b) depict the vitamin C content of oven and tray dried samples. For leafy vegetables, the characteristics varied from 56.96 – 123.94 mg/100g and 45.58 – 101.41 mg/100g for tray and oven dried samples, respectively. Corresponding values for non-leafy vegetables varied from 42.25 – 304.37 mg/100g and 33.80 – 280.28 mg/100g, respectively. Among the samples, highest vitamin C content was found for tray dried Komora (304.37 mg/100g). The obtained vitamin C value was in good agreement with the reported value in the data base (USDA, 2014). According to this database, the vitamin C content of Komora (ash gourd) was 13 mg per 100 g of fresh edible portion of Komora (equivalent to about 325 mg/100 g, dry basis). On the other hand, among leafy vegetables, the highest vitamin C content was achieved for tray dried Pui sample (123.94 mg/100g) followed with Jatilao sample. Among all vegetables, oven dried Kolmou (55.58 mg/100g) and Posola (33.80 mg/100g) possessed the lowest vitamin C content.

In general, compared to oven dried samples, tray dried leafy and non-leafy vegetables possessed higher vitamin C content in comparison with oven dried samples. Since vitamin C is highly sensitive to heat, significant variations in the value is apparent for all samples with variant drying mode and hence conditions. Intermittent airflow mode better facilitated vitamin C retention for all samples. During the flow phase, hot air picks up the moisture in the sample and does not significantly influence the heat sensitive constituents in the vegetables. The comparative

trends reported in this work have been in good agreement with the trends reported by Saini et al. (2014). The authors reported that tray dried *Moringa oleifera* leaves possessed higher ascorbic acid content than the oven dried samples. Similar views have been presented by Kenghe et al. (2015) for tray and shade dried curry leaves and comparative efficacy of the former process.

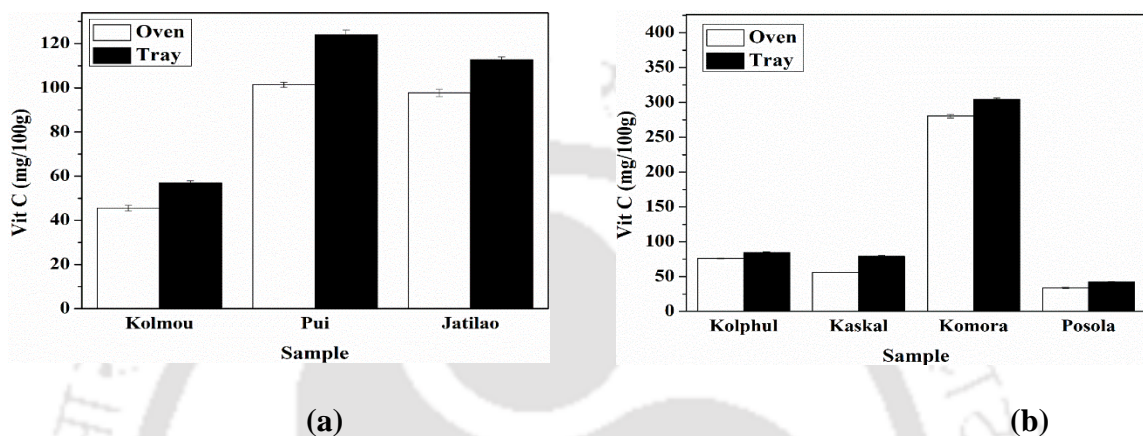


Fig. 3.6: Vitamin C content characteristics of oven and tray dried (a) leafy and (b) non-leafy vegetables.

3.11 Antioxidant Activity

Antioxidant activity is a very important parameter for consideration in food product formulation.

Fig. 3.7 (a) and (b) depicts the variation of antioxidant activity of various tray and oven dried leafy and non-leafy vegetables, respectively. These trends are similar to those reported for vitamin C content. Similar to the vitamin C content trend, the antioxidant activity has been observed to be higher for tray dried samples in comparison with the oven dried samples for all dried leafy and non-leafy vegetables. For leafy vegetables, antioxidant activity varied from 23.08 – 92.34% and 21.81 – 85.23% for tray and oven drying cases, respectively. On the other hand, for non-leafy vegetables, the corresponding variations were 86.38 – 21.68% and 79.26 – 17.80%, respectively. Highest antioxidant activity was obtained for tray dried Kolmou (92.34%) and

Jatilao (89.35%) followed by Kolphul (86.38%). Among all leafy vegetables, Kolmou (92.34% and 85.23% in tray and oven drying) possessed the highest antioxidant activity. Similarly, among chosen non-leafy vegetables, Kolphul possessed the highest values (86.38% and 79.26% in tray and oven drying). Among all vegetables, Pui (23.08% and 21.81%), Posola (21.68% and 17.80%) and Komora (21.36% and 17.82%), possessed lower antioxidant activity.

The uniform heating coupled with intermittent air circulation facilitated better retention of antioxidant activity in tray dried samples. Effective moisture movement within the sample to reach the surface during stagnation phase (no airflow) and moisture removal during air circulation phase enabled better retention of antioxidant constituents during tray drying operation. The tray drying operation under intermittent airflow condition enables partial transfer of heat from air to the sample surface to effectively remove moisture but retain heat sensitive nutrients due to intermittent airflow conditions. The obtained results are in good agreement with those reported by Saini et al. (2014). Also, it can be noted that Ahluwalia et al. (2012) reported that the tray dried marigold petals possessed higher antioxidant activity in comparison to vacuum, fan and solar dried samples.

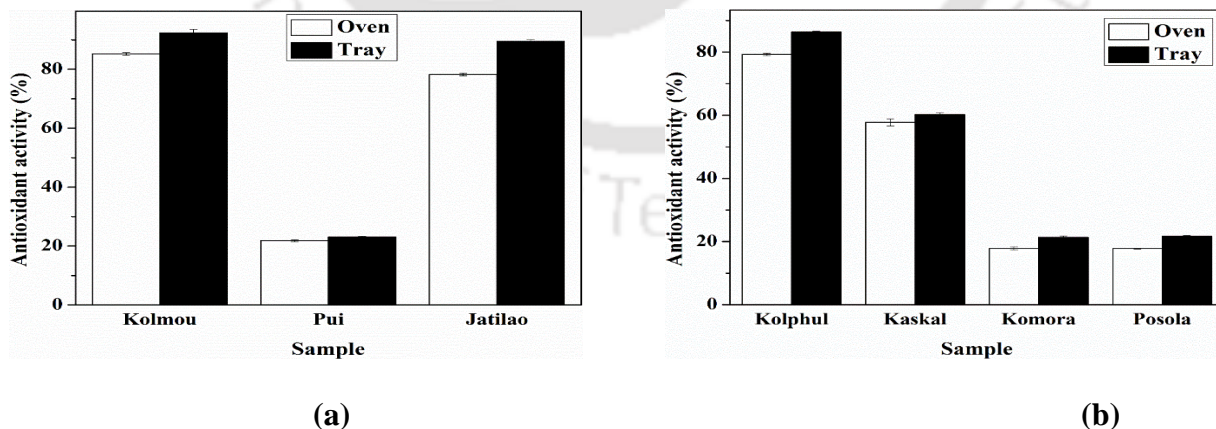


Fig. 3.7: Antioxidant activity characteristics of oven and tray dried (a) leafy and (b) non-leafy vegetables.

For all nutritional parameters, **Table 3.8** and **3.9**, respectively summarizes a comparative summary of obtained data with available prior art data for leafy and non-leafy vegetables, respectively. Thus, it is evident that the protein, crude fibre, ash and fat content have been comparably higher than the literature reported values for few cases. Since triplicate runs were conducted and average value has been reported, experimental error has been ruled out. Thus, the higher fat and protein content of the mentioned samples is probably due to the combinational variations of cultivated species, drying method used and climate of the NE India.

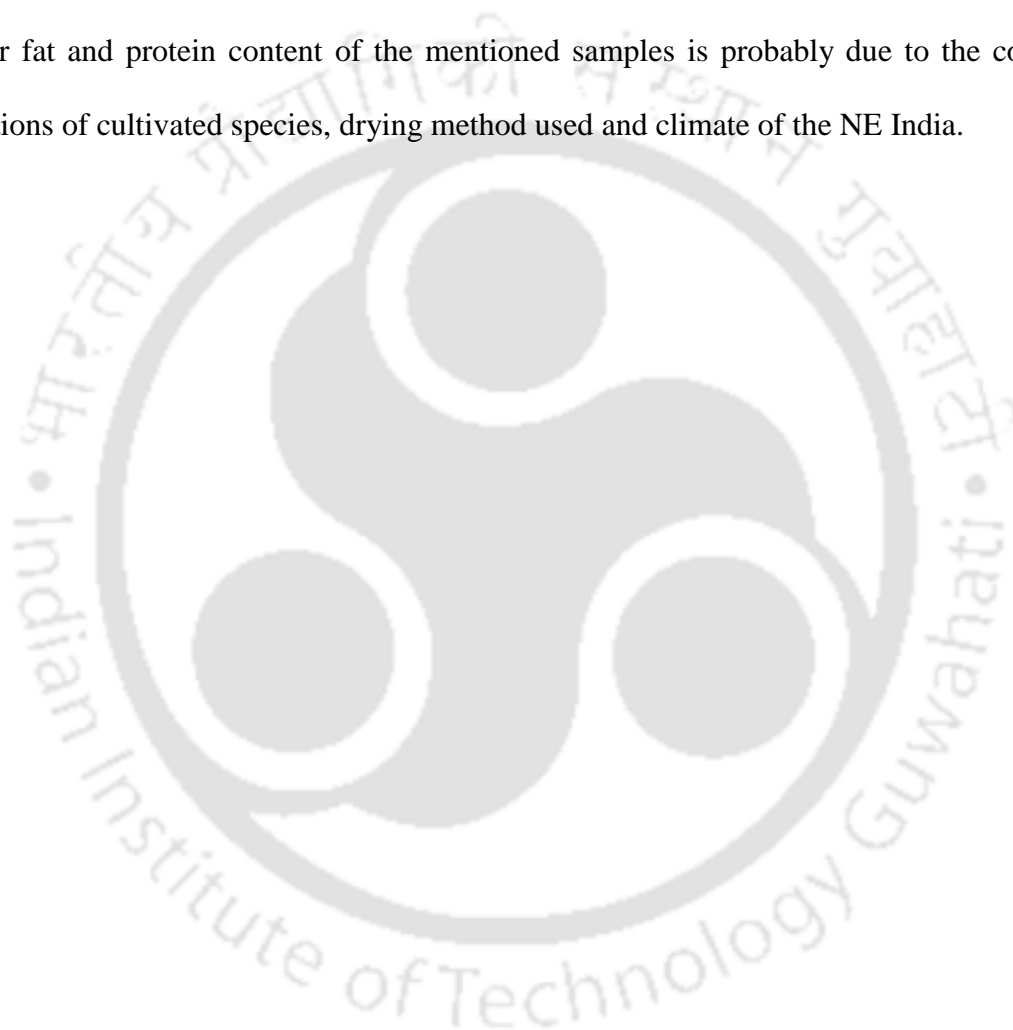


Table 3.8: Parametric and response summary data of tray and oven dried leafy vegetable systems.

Sl.no.	Sample	Drying methods	Optimal Drying and drying conditions	Optimal drying characteristics										Reference
				Yield (%)	Moisture (%)	Carbohydrate (%)	Protein (%)	Soluble protein (%)	Fat (%)	Fibre (%)	Ash (%)	Vit C (mg/100g)	Antioxidant activity (%)	
1	Kolmou	Oven and tray drying	Tray drying- 60 °C for 4 hrs	13.94	4.21	49.27	22.54	10.74	4.26	8.74	10.98	56.96	92.34	This work
	Kolmou	Oven	55 °C for constant weight	-	-	52.78	21.45	-	3.34	9.26	9.11	-	-	(Satter et al. 2016)
2	Pui	Oven and tray drying	Tray drying- 60 °C for 4 hrs	11.41	10.44	46.05	16.8	9.91	3.87	6.53	16.32	123.94	23.08	This work
	Pui	Cabinet drying	55 °C for 10 hours	-	-	50.62	17.55	-	1.58	7.23	15.49	-	-	(Tongco et al. 2015)
3	Jatilao	Oven and tray drying	Tray drying- 60 °C for 4 hrs	14.06	7.68	39.61	18.02	14.62	8.67	12.15	13.88	112.68	89.45	This work
	Jatilao	Oven drying	45 °C until constant weight	-	-	-	-	-	-	-	14.50	89.0	-	(Karmakar et al. 2013)

Table 3.9: Parametric and response summary data of tray and oven dried non-leafy vegetable systems.

Sl. no.	Sample	Drying methods	Optimal Drying and drying conditions	Optimal drying characteristics										Reference
				Yield (%)	Moisture (%)	Carbohydrate (%)	Protein (%)	Soluble protein (%)	Fat (%)	Fibre (%)	Ash (%)	Vit C (mg/100g)	Antioxidant activity (%)	
1	Kolphul	Oven and tray drying	Tray drying-60 °C for 10 hrs	10.01	7.34	50.52	10.38	8.26	8.57	12.35	10.84	84.51	86.38	This work
	Kolphul (Musa AAB-Zip Ekon)	Oven drying	55 °C for 48 hrs	-	-	62.19	8.89	-	8.81	-	9.88	-	-	(Awedem et al. 2015)
2	Kaskal	Oven and tray drying	Tray drying-60 °C for 10 hrs	16.64	4.37	81.37	5.71	2.99	2.93	1.08	4.55	78.87	60.24	This work
	<i>Musa paradisiaca</i>	Oven, fluidized bed, tray and sun	Oven drying-70 °C for 5 hrs	-	3.24	87.64	3.82	-	1.15	1.04	3.11	-	-	(Arinola et al. 2009)
3	Komora	Oven and tray drying	Tray drying-60 °C for 10 hrs	4.26	7.30	62.35	7.05	5.98	3.03	12.04	8.22	304.37	21.36	This work
	Komora	-	-	-	-	-	-	-	-	-	-	-	-	-
4	Posola	Oven and tray drying	Tray drying-60 °C for 10 hrs	5.58	1.56	61.19	4.23	3.74	4.93	15.67	12.42	42.25	21.68	This work
	Banana pseudostem	Cabinet drying	50 °C	-	4.4	64.4	3.4	-	3.4	-	14	-	-	(Ma et al. 2016)

3.12 Summary

In this chapter, a comparative analysis of the nutritional characteristics of tray and oven dried leafy and non-leafy vegetables were targeted. To do so, all important nutritional characteristics have been targeted. Among oven and tray drying, later provided the best combination of nutritional parameters. The tray drying process is therefore advantageous for food processing and food product formulations based on NE India's horticultural produce. In general, the tray dried samples possessed higher proximate, vitamin C and antioxidant values. For each leafy and non-leafy vegetable being chosen, the moisture content, vitamin C and antioxidant activity significantly varied for oven and tray dried samples for each leafy/non-leafy vegetable. Among all considered samples, tray dried Kolmou possessed highest antioxidant activity. Vitamin C content was analyzed to be the highest for tray dried Komora. In summary, the carried out research is anticipated to serve as an effective guideline to promote food processing in NE India with specific emphasis on ready to eat/cook leafy and non-leafy vegetable soups with optimized nutritional content. For this purpose, tray drying has the edge in comparison with oven drying process.

In the following chapters, a detailed account of process parametric optimization has been addressed to further substantiate towards the development of customized leafy and non-leafy vegetable soup formulations that are being targeted as the ultimate objectives of the Ph.D. thesis.

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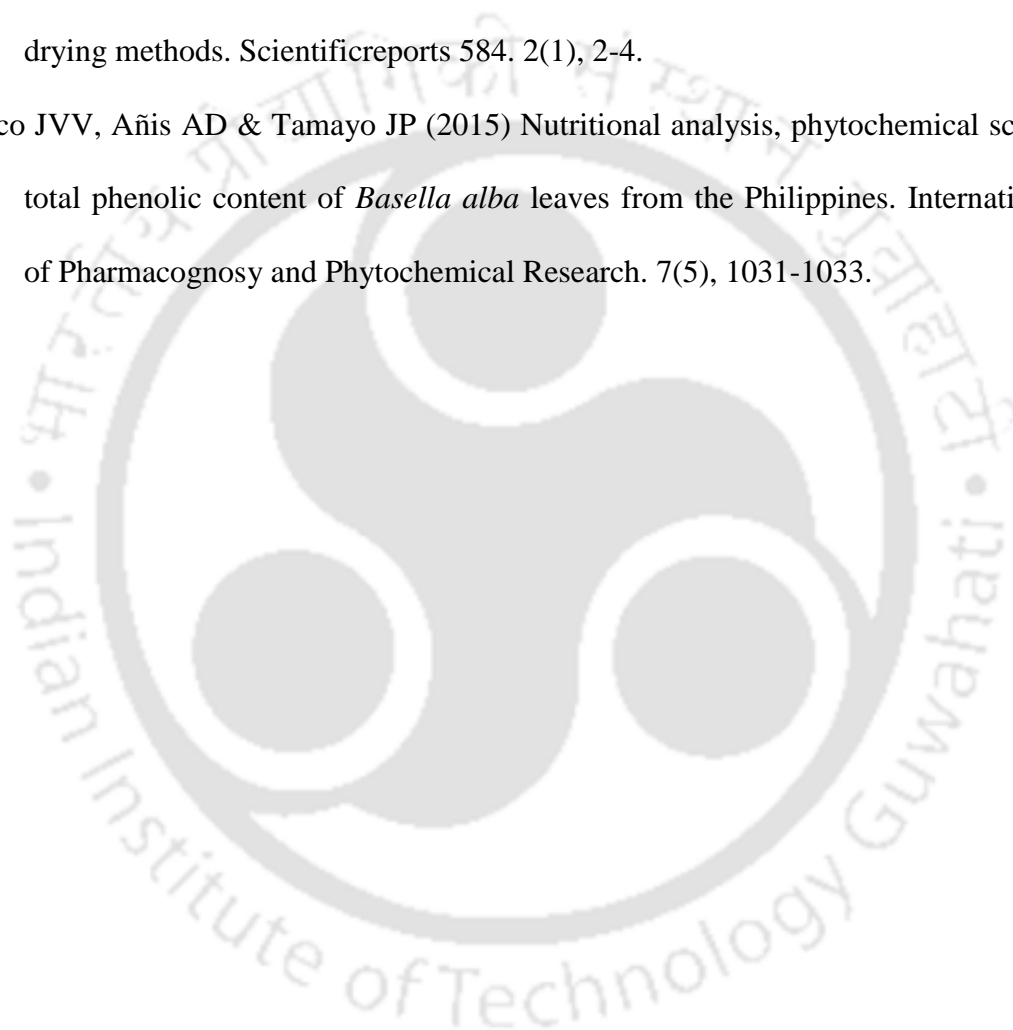
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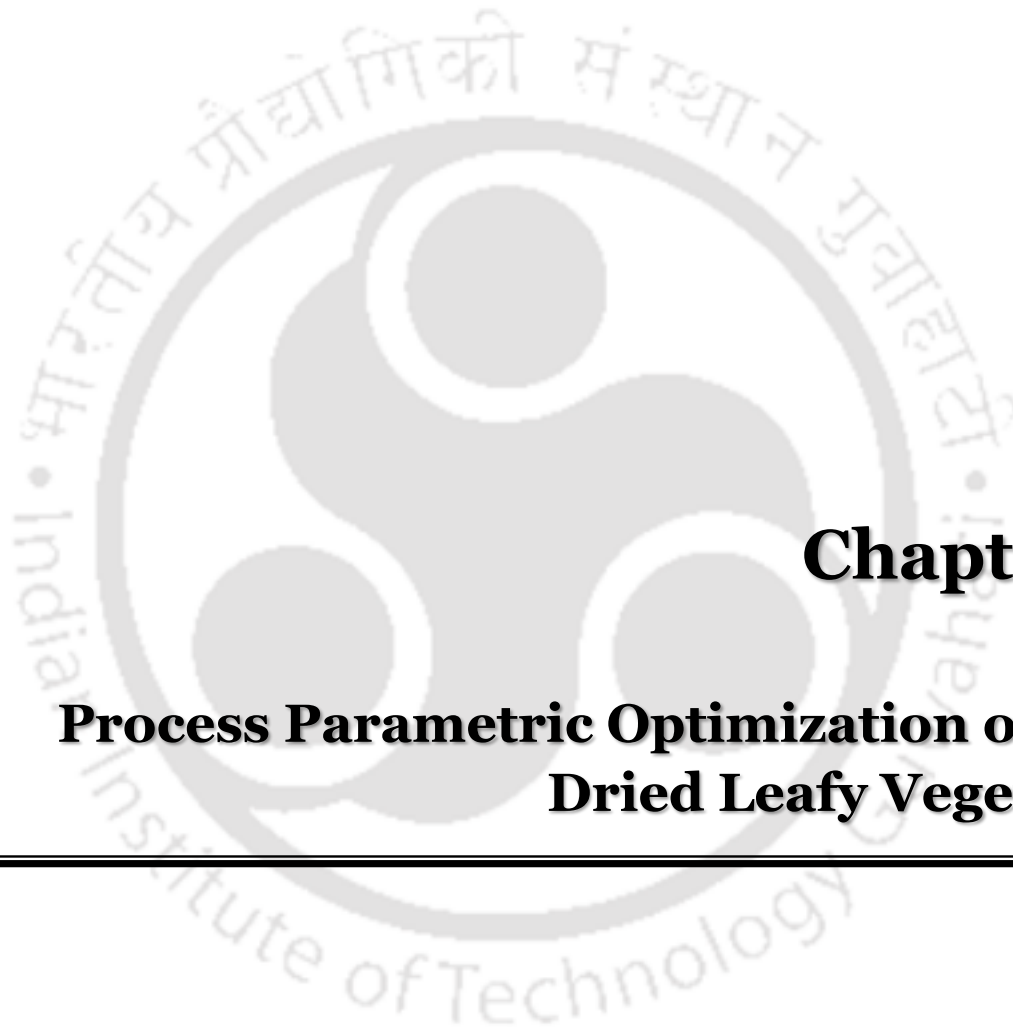
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Chapter 4:

Process Parametric Optimization of Tray Dried Leafy Vegetables



Process Parametric Optimization of Tray Dried Leafy Vegetables

After presenting a brief orientation to the targeted objectives of the chapter in section 4.1, the following section 4.2 presents findings associated to the trial and error based optimization of process variables and dried leafy vegetable characterizations. The findings refer to drying kinetics based evaluation of moisture and time dependent variation of vitamin C and antioxidant activity characteristics. Thereafter, in section 4.3, the findings associated to statistical design based optimization of drying process for dried vegetable characteristics have been elaborated. These include identification of best fit model based on analysis of variance approach, response surface plots, numerical optimization and validation of best data. Finally, in section 4.4, proximate characteristics of optimally tray dried leafy vegetables have been presented along with chapter summary in section 4.5.

4.1 Introduction

The available prior art targeted either drying kinetics or RSM based optimization. Thus, a comparative assessment of both approaches was not targeted for any leafy vegetable drying system. Also, the chosen leafy vegetables have not been investigated for either kinetic or RSM based process parametric optimization studies. Based on these primary lacunae, the thesis targeted the combinatorial optimization of process variables during periodic airflow supplemented tray drying of Kolmou (*Ipomoea aquatica* Forssk), Jatilao (*Lagenaria siceraria*)

and Pui (*Basella alba*) leaves. The kinetics based evaluation involved studies associated to fitness of drying models, determination of moisture diffusivity with activation energy and overall influence of drying time and temperature on vitamin C and antioxidant activity characteristics of chosen dried leafy vegetable samples. On the other hand, statistical design based investigations targeted fitness of various models based on the analysis of variance (ANOVA) approach and subsequent numerical optimization of process-product characteristics through design expert 7.0 software. For drying experiments, drying temperature and time range were set based on preliminary trials and as 40 – 80 °C and 90 – 300 min (Kolmou), 150 – 390 min (Jatilao) and 240 – 420 min (Pui), respectively. Finally, proximate evaluation was targeted for optimally tray dried chosen leafy vegetable samples. In the following section, trial and error followed with statistical design based process-product characteristics have been elaborated.

4.1 Kinetics based Characterization of Process Parameters and Responses

4.1.1 Drying Kinetics

As illustrated, the moisture ratio (MR) variation with time depicted exponential decline for all chosen leafy vegetables (**Fig. 4.1 (a) – (c)**). Also, it is apparent for all cases that the moisture ratio reduced at a higher rate during initial time frame. This is in agreement with the theoretical insights that higher difference in vapour pressure between the samples and environment facilitated faster drying rate (Kaleemullah and Kailappan 2007). For Kolmou, this phase persisted for a drying time from 0 – 315 min, 0 – 150 min, 0 – 90 min, 0 – 60 min and 0 – 45 min for drying temperatures of 40, 50, 60, 70 and 80 °C, respectively. For Jatilao, the MR value significantly reduced up to 195, 150, 105, 75 and 45 min. Similarly, for Pui, the corresponding drying time values were 510, 390, 180, 165 and 120 min, respectively. Thus, for this phase, the

corresponding MR reduced significantly from 1 – 0.103, 1 – 0.094, 1 – 0.084, 1 – 0.080 and 1 – 0.046 for Kolmou, 1 – 0.101, 1 – 0.064, 1 – 0.059, 1 – 0.06 and 1 – 0.064 for Jatilao and 1 – 0.099, 1 – 0.052, 1 – 0.071, 1 – 0.035 and 1 – 0.026 for Pui leafy vegetable. This rapid reduction in MR is due to the higher moisture migration rate from inner portions to the outer surface of the sample. Also, high MR reduction rate was observed initially for higher drying temperatures. This is due to the greater vapour pressure difference between the leafy vegetable and surrounding environment to enhance moisture transfer from the sample to air (Kaleemullah and Kailappan 2007). Following this, due to lesser migration rate of moisture from the inner portion to outer surface, the drying rate reduced significantly. Hence, MR reduced significantly to reach a near equilibrium value.

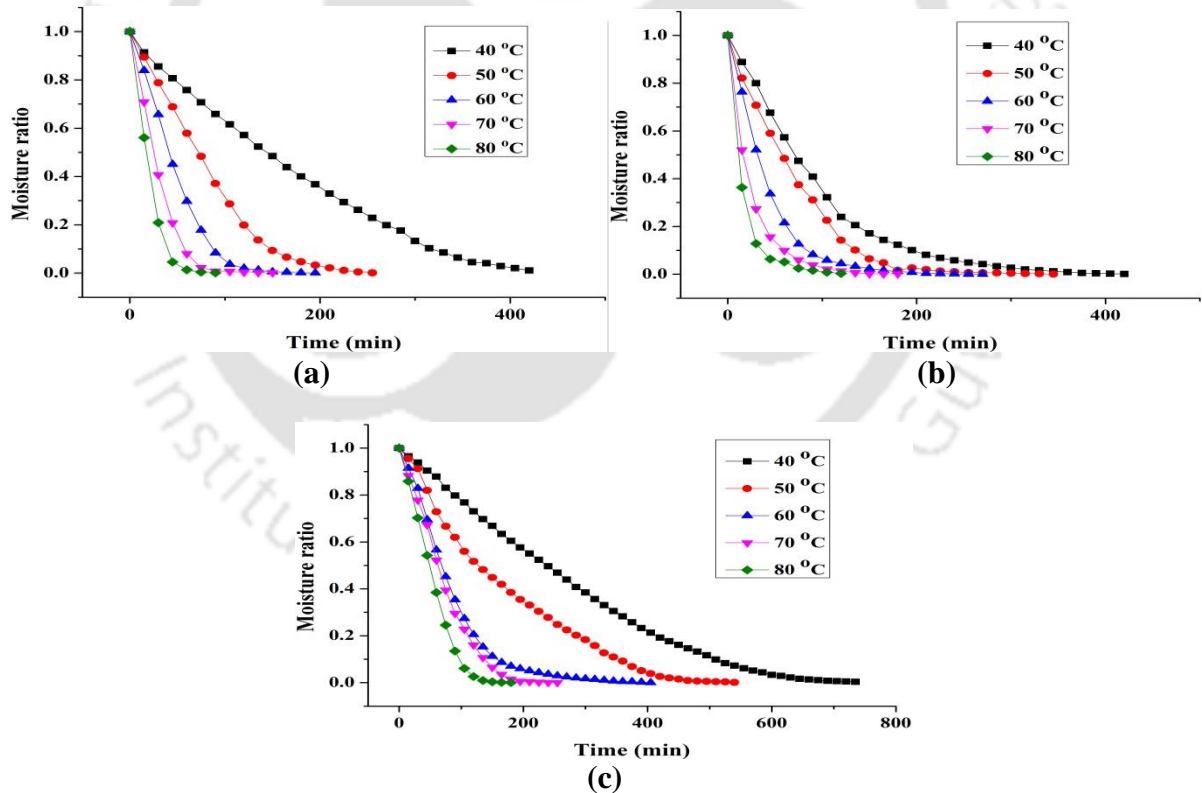


Fig. 4.1: Variation of MR with time and temperature for alternate leafy vegetable systems (a) Kolmou (b) Jatilao (c) Pui.

For, Kolmou, a fairly constant MR values (0.0105, 0.0016, 0.001, 0.0019 and 0.0015) have been obtained for after reaching a drying time of 405 min, 240 min, 180 min, 135 min and 75 min at 40, 50, 60, 70 and 80 °C drying temperature, respectively. For Jatilao, corresponding MR values (0.0012, 0.0008, 0.0002, 0.0015 and 0.0035) reached saturation after 435 min, 360 min, 285 min, 195 min and 135 min of drying time. Similarly, for Pui, these values were 0.0037, 0.0014, 0.0006, 0.0004 and 0.0005 for 735, 540, 405, 255 and 180 min drying time, respectively. The equilibrium MR value that persisted at the end of drying was due to the achievement of equilibrium moisture content (EMC). This refers to no further variation in the moisture content of the sample and thereby, a constant weight of the sample. The trends for the variation of MR with drying time are similar to various food materials being dried using hot air drying system (Ashtiani et al. 2017; Bensebia and Allia 2015).

4.1.2 Fitness of Alternate Drying Models

Table 4.1 (a), (b) and (c), respectively summarize model parameter values of alternate drying models (representing MR versus t data) for Kolmou, Jatilao and Pui samples. For Kolmou, among alternate drying models, Page model was found to be the best fit model for 50, 60, 70 and 80 °C cases with high R^2 (0.9976, 0.9992, 0.9996 and 0.9998, respectively) and low chi-squared value (2.79×10^{-4} , 9.86×10^{-5} , 4.28×10^{-5} and 2.64×10^{-5} , respectively). However, for the 40 °C case, the data was best represented using Wang and Singh model ($R^2 = 0.9991$, reduced chi-square = 7.87×10^{-5}). For Jatilao, Page model with high R^2 (0.9989, 0.9962, 0.9991 and 0.9997, respectively) and low chi-squared values (9.80×10^{-5} , 3.42×10^{-5} , 7.09×10^{-5} and 2.90×10^{-5} , respectively) was the best fit model for 40, 50, 60, and 70 °C drying temperature cases, respectively. However, for 80 °C case, the drying characteristics can be better represented with the logarithmic model (R^2 0.9988, reduced chi-square 1.29×10^{-4}). Further, for the 60 °C case,

Silva et al. model (da Silva et al. 2012) was also a good fit model. On the other hand, for the Pui vegetable case, the drying characteristics curves at 60, 70, and 80 °C could be best represented through the Page model (high R^2 and low reduced chi-squared and RSS values). For the same vegetable and for 40 and 50 °C cases, Wang and Singh model and Logarithmic model were the best fit models, respectively. The drying rate constant was found to increase with increasing drying temperature.



Table 4.1: Fitness parameters of alternate drying models to represent drying characteristics of (a) Kolmou (b) Jatilao (c) Pui vegetables.

(a)							
Model	Model equation	Parameters	40 °C	50 °C	60 °C	70 °C	80 °C
Newton	$MR = \exp(-kt)$	k	0.00565	0.01203	0.0206	0.03315	0.00406
		R^2	0.96844	0.96217	0.96745	0.98179	0.98479
		RSS	0.08191	0.0738	0.0504	0.02135	0.01338
		χ^2	0.00293	0.00434	0.00388	0.00213	0.00223
Page	$MR = \exp(-kt^n)$	k	0.00116	0.0013	0.00234	0.00696	0.01055
		n	1.2988	1.48431	1.53376	1.43162	1.47562
		R^2	0.9898	0.99757	0.99918	0.99963	0.99982
		RSS	0.02552	0.00446	0.00117	3.85×10^{-4}	1.32×10^{-4}
Henderson and Pabis	$MR = a \exp(-kt)$	χ^2	9.45×10^{-4}	2.79×10^{-4}	9.86×10^{-5}	4.28×10^{-5}	2.64×10^{-5}
		a	1.06019	1.09643	1.08412	1.04571	1.02445
		k	0.00599	0.01307	0.02205	0.03437	0.05022
		R^2	0.9722	0.96991	0.97203	0.98216	0.98263
Logarithmic	$MR = a \exp(-kt) + c$	RSS	0.06952	0.05523	0.03998	0.01883	0.01273
		χ^2	0.00257	0.00345	0.00333	0.00209	0.00255
		a	1.37949	1.20953	1.13619	1.07535	1.06699
		c	-0.38654	-0.15437	-0.07228	-0.03815	-0.04891
Wang and Singh	$MR = M_0 + at + bt^2$	k	0.00314	0.00938	0.00196	0.03106	0.04427
		R^2	0.99831	0.98728	0.98064	0.98524	0.98539
		RSS	0.00408	0.02189	0.02536	0.01385	0.00857
		χ^2	1.57×10^{-4}	0.00146	0.00231	0.00173	0.00214
Singh et al. (2014)	$MR = \exp(-kt) - akt$	M_0	0.98292	1.03176	1.00474	0.94427	0.97086
		a	-0.00393	-0.00916	-0.01402	-0.01863	-0.003004
		b	3.77×10^{-6}	2.03×10^{-5}	4.70×10^{-5}	8.58×10^{-5}	2.20×10^{-4}
		R^2	0.99915	0.9964	0.98783	0.96571	0.97907
Singh et al. (2014)	$MR = \exp(-kt) - akt$	RSS	0.00205	0.0062	0.01594	0.03217	0.01227
		χ^2	7.87×10^{-5}	4.13×10^{-4}	0.00145	0.00402	0.00307
		k	0.00403	0.00986	0.01853	0.03157	0.04627
		a	0.12068	0.05626	0.02049	0.0079	0.01058
Silva et al. (2012)	$MR = \exp(-at + bt^{1/2})$	R^2	0.99803	0.98287	0.9755	0.98353	0.98627
		RSS	0.00493	0.03145	0.03501	0.01738	0.01007
		χ^2	1.83×10^{-4}	0.00197	0.00292	0.00193	0.00201
		a	0.00749	0.01874	0.03464	0.05589	0.09378
Silva et al. (2012)	$MR = \exp(-at + bt^{1/2})$	b	-0.02485	-0.06383	-0.10266	-0.13209	-0.21553
		R^2	0.98068	0.98813	0.99382	0.99816	0.99933
		RSS	0.04834	0.02179	0.00883	0.00194	4.92×10^{-4}
		χ^2	0.00179	0.00136	7.36×10^{-4}	2.16×10^{-4}	9.84×10^{-5}

(b)

Model	Model equation	Parameters	40 °C	50 °C	60 °C	70 °C	80 °C
Newton	$MR = \exp(-kt)$	k	0.01077	0.01423	0.02425	0.04177	0.06619
		R^2	0.99077	0.98544	0.99381	0.9987	0.99776
		RSS	0.02239	0.02985	0.00922	0.00132	0.00191
		χ^2	8.0×10^{-4}	0.0013	5.12×10^{-4}	1.1×10^{-4}	2.38×10^{-4}
		k	0.00395	0.00445	0.01063	0.05635	0.09705
Page	$MR = \exp(-kt^n)$	n	1.21282	1.25881	1.21123	0.91299	0.87491
		R^2	0.99887	0.99617	0.99914	0.99966	0.99841
		RSS	0.00265	0.00752	0.00121	3.19×10^{-4}	0.00118
		χ^2	9.80×10^{-5}	3.42×10^{-5}	7.09×10^{-5}	2.90×10^{-5}	1.69×10^{-4}
		k	1.06142	1.04863	1.03781	0.99277	0.9975
Henderson and Pabis	$MR = a \exp(-kt)$	k	0.01139	0.01484	0.02507	0.04148	0.06606
		R^2	0.99386	0.98698	0.9948	0.99864	0.99745
		RSS	0.01435	0.02555	0.00732	0.00126	0.0019
		χ^2	5.32×10^{-4}	0.00116	4.31×10^{-4}	1.15×10^{-4}	2.71×10^{-4}
		a	1.07136	1.06776	1.04204	0.98716	0.98434
Logarithmic	$MR = a \exp(-kt) + c$	c	-0.02079	-0.03397	-0.00687	0.00826	0.01544
		k	0.0107	0.01345	0.02455	0.04268	0.06957
		R^2	0.99502	0.9906	0.99477	0.99902	0.99878
		RSS	0.01121	0.0176	0.00692	8.27×10^{-4}	7.77×10^{-4}
		χ^2	4.31×10^{-4}	8.38×10^{-4}	4.32×10^{-4}	8.27×10^{-4}	1.29×10^{-4}
Wang and Singh	$MR = M_0 + at + bt^2$	M_0	0.92117	0.91188	0.82515	0.78178	0.81149
		a	-0.0061	-0.00771	-0.00964	-0.01369	-0.0215
		b	9.78×10^{-6}	1.54×10^{-5}	2.59×10^{-5}	5.50×10^{-5}	1.30×10^{-4}
		R^2	0.96964	0.97317	0.9002	0.8559	0.81767
		RSS	0.06837	0.05023	0.1321	0.12191	0.11629
Singh et al. (2014)	$MR = \exp(-kt) - akt$	χ^2	0.00263	0.00239	0.00826	0.01219	0.01938
		k	0.01037	0.01356	0.02404	0.04212	0.06738
		a	0.00617	0.00764	0.00116	-0.00103	-0.00204
		R^2	0.99241	0.98856	0.99367	0.99885	0.99851
		RSS	0.01775	0.02245	0.0089	0.00107	0.00111
Silva et al. (2012)	$MR = \exp(-at + bt^{1/2})$	χ^2	6.58×10^{-4}	0.00102	5.24×10^{-4}	9.75×10^{-5}	1.58×10^{-4}
		a	0.01425	0.01868	0.03318	0.0346	0.04961
		b	-0.03499	-0.04007	-0.05997	0.03871	0.07515
		R^2	0.99851	0.9925	0.99952	0.99952	0.99819
		RSS	0.00349	0.0147	6.68×10^{-4}	4.48×10^{-4}	0.00134
χ^2	1.29×10^{-4}	6.68×10^{-4}	3.93×10^{-5}	4.07×10^{-5}	1.92×10^{-4}		

(c)

Model	Model equation	Parameters	40 °C	50 °C	60 °C	70 °C	80 °C
Newton	$MR = \exp(-kt)$	k	0.00355	0.00576	0.01178	0.01348	0.01808
		R^2	0.95916	0.98299	0.97669	0.96353	0.95501
		RSS	0.20025	0.05577	0.05898	0.07158	0.06943
		χ^2	0.00409	0.00155	0.00218	0.00421	0.00579
Page	$MR = \exp(-kt^n)$	k	3.19×10^{-4}	0.00213	0.00165	0.00142	0.00148
		n	1.41689	1.18548	1.42746	1.50086	1.59603
		R^2	0.9952	0.9924	0.99846	0.99826	0.9974
		RSS	0.02306	0.02423	0.00376	0.00321	0.00368
Henderson and Pabis	$MR = a \exp(-kt)$	χ^2	4.80×10^{-4}	6.92×10^{-4}	1.45×10^{-4}	2.01×10^{-5}	3.34×10^{-4}
		a	1.10957	1.06194	1.102	1.09843	1.09451
		k	0.00393	0.0061	0.01285	0.01463	0.01952
		R^2	0.97118	0.98664	0.98441	0.97107	0.96085
Logarithmic	$MR = a \exp(-kt) + c$	RSS	0.13843	0.04258	0.03799	0.05344	0.05538
		χ^2	0.00288	0.00122	0.00146	0.00334	0.00503
		a	1.32007	1.13403	1.11511	1.17932	1.21217
		c	-0.27978	-0.11601	-0.02455	-0.11557	-0.1522
Wang and Singh	$MR = M_0 + at + bt^2$	k	0.00233	0.00461	0.012	0.01121	0.01411
		R^2	0.99597	0.99698	0.98598	0.98575	0.9802
		RSS	0.01895	0.00934	0.03286	0.02468	0.02546
		χ^2	4.03×10^{-4}	2.75×10^{-4}	0.00131	0.00165	0.00255
Singh et al. (2014)	$MR = \exp(-kt) - akt$	M_0	1.02483	0.97009	0.94467	1.02579	1.04042
		a	-0.00268	-0.00389	-0.00682	-0.00991	-0.01376
		b	1.74×10^{-6}	3.93×10^{-6}	1.17×10^{-5}	2.35×10^{-5}	4.47×10^{-5}
		R^2	0.9991	0.99406	0.95866	0.9957	0.99464
Singh et al. (2012)	$MR = \exp(-at + bt^{1/2})$	RSS	0.00424	0.01838	0.09687	0.00744	0.0069
		χ^2	9.03×10^{-5}	5.41×10^{-4}	0.00387	4.96×10^{-4}	6.90×10^{-4}
		k	0.00254	0.00491	0.01129	0.01135	0.01463
		a	0.11192	0.03787	0.0071	0.03912	0.05212
Silva et al. (2012)	$MR = \exp(-at + bt^{1/2})$	R^2	0.99337	0.99649	0.97865	0.98025	0.97531
		RSS	0.03182	0.01117	0.05202	0.03648	0.03493
		χ^2	6.63×10^{-4}	3.19×10^{-4}	0.002	0.00228	0.00318
		a	0.00525	0.00726	0.01848	0.02144	0.03056
Silva et al. (2012)	$MR = \exp(-at + bt^{1/2})$	b	-0.02911	-0.02074	-0.06383	-0.07203	-0.09733
		R^2	0.98422	0.9903	0.99709	0.99008	0.98701
		RSS	0.07581	0.03092	0.00709	0.01833	0.01837
		χ^2	0.00158	8.84×10^{-4}	2.73×10^{-4}	0.00115	0.00167

4.1.3 Moisture Diffusivity and Activation Energy

For alternate drying temperature case, the $\ln MR$ versus time plots have been prepared with Kolmou, Jatilao and Pui drying data (**Fig. 4.2 (a) – (c)**). Based on the slope of the linear plots (with good fitness of R^2 as 0.9444 – 0.9932, 0.9873 – 0.9969 and 0.9336 – 0.9889 for Kolmou, Jatilao and Pui, respectively), the moisture diffusivity of the leafy vegetable tray drying process were determined. For Kolmou, the moisture diffusivity enhanced from 6.36×10^{-13} – 5.16×10^{-12} m^2/s for an increase in drying temperature from 40 – 80 °C. For Jatilao, the values correspondingly varied from 1.79×10^{-12} – 5.36×10^{-12} m^2/s . Similarly, corresponding values of moisture diffusivity for Pui varied from 9.94×10^{-13} – 6.23×10^{-12} m^2/s . The reduction in moisture diffusivity with drying temperature is due to enhanced moisture transfer being driven with an increase in the vapour pressure difference. These values are within the general range of the moisture diffusivity values (10^{-8} – 10^{-12}) being reported for food materials (Zogzas et al. 1996). The obtained moisture diffusivity is comparable with that of rosemary leaves (0.2 – 9.4×10^{-12} m^2/s) (Bensebia and Allia 2015). A detailed account of the data has been summarized in **Table 4.2 (a) – (c)**. Thus, the moisture diffusivity enhanced with temperature varied from 40 – 80 °C. The enhancement was due to higher heat transfer coefficient at higher temperature.

The activation energy for the process was obtained using corresponding $\ln D$ and $1/T$ plots (**Fig. 4.3 (a) – (c)**). Accordingly, these have been obtained as 45.21, 25.93 and 43.15 kJ/mol for Kolmou, Jatilao and Pui, respectively and are in good agreement with the general range (12 – 110 kJ/mol) reported for food materials (Mwithiga and Olwal 2005). The obtained values have been comparable with those obtained for rosemary (Ashtiani et al. 2017) and peppermint leaves (Bensebia and Allia 2015) being dried using hot air drying system.

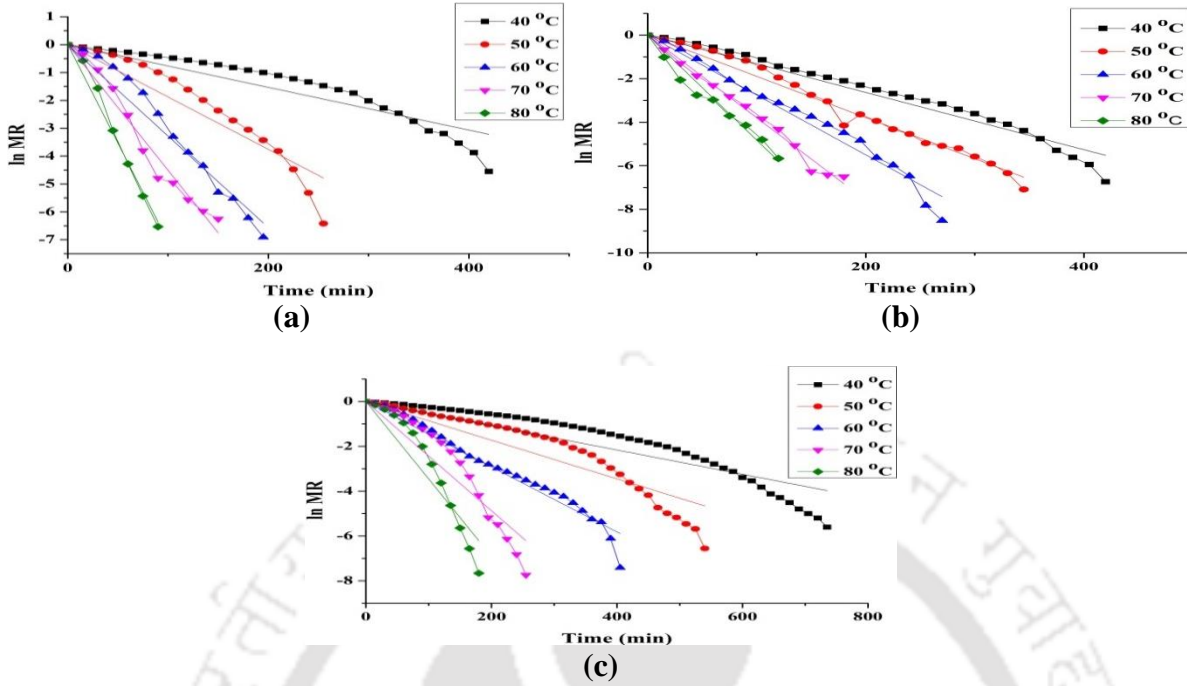


Fig. 4.2: ln MR versus time plots for (a) Kolmou (b) Jatilao and (c) Pui vegetable systems.

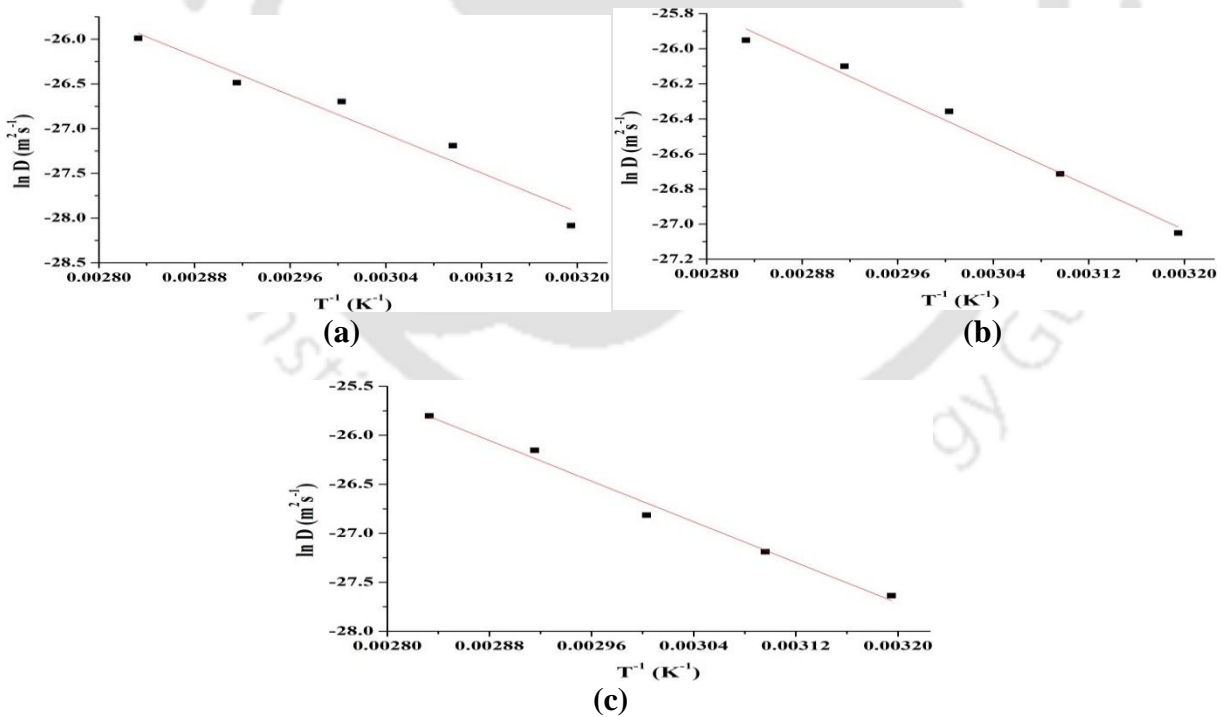


Fig. 4.3: Arrhenius plots representing moisture diffusivity of (a) Kolmou (b) Jatilao and (c) Pui vegetable systems.

Table 4.2: Evaluated and literature data of moisture diffusivity for (a) Kolmou (b) Jatilao and (c) Pui vegetable systems.

(a)					
Sample	Drying method	Temperature (°C)	Diffusivity (m ² /s)	Activation energy (kJ/mol)	Reference
Kolmou	Intermittent airflow assisted tray drying	40	6.356×10 ⁻¹³		This work
		50	1.555×10 ⁻¹²		do
		60	2.542×10 ⁻¹²	45.21	do
		70	3.144×10 ⁻¹²		do
		80	5.159×10 ⁻¹²		do
Peppermint leaves	Hot air drying	30 -50	1.096 - 2.486×10 ⁻¹¹	21.476–27.784	Ashtiani et al. (2017)
Rosemary leaves	Oven drying	Ambient - 60	0.2–9.4×10 ⁻¹²	66.3	Bansebia and Allia (2015)
(b)					
Sample	Drying method	Temperature (°C)	Diffusivity (m ² /s)	Activation energy (kJ/mol)	Reference
Jatilao	Intermittent airflow assisted tray drying	40	1.787×10 ⁻¹²		This work
		50	2.502×10 ⁻¹²		do
		60	3.574×10 ⁻¹²	25.93	do
		70	4.621×10 ⁻¹²		do
		80	5.361×10 ⁻¹²		do
Peppermint leaves	Hot air drying	30 -50	1.096 - 2.486×10 ⁻¹¹	21.476 – 27.784	Ashtiani et al. (2017)
Rosemary leaves	Oven drying	Ambient - 60	0.2 – 9.4×10 ⁻¹²	66.3	Bansebia and Allia (2013)
(c)					
Sample	Drying method	Temperature (°C)	Diffusivity (m ² /s)	Activation energy (kJ/mol)	Reference
Pui	Intermittent airflow assisted tray drying	40	9.945×10 ⁻¹³		This work
		50	1.557×10 ⁻¹²		do
		60	2.263×10 ⁻¹²	43.15	do
		70	4.382×10 ⁻¹²		do
		80	6.226×10 ⁻¹²		do
Peppermint leaves	Hot air drying	30 -50	1.096 - 2.486×10 ⁻¹¹	21.476–27.784	Ashtiani et al. (2017)
Rosemary leaves	Oven drying	Ambient - 60	0.2–9.4×10 ⁻¹²	66.3	Bansebia and Allia (2013)

4.1.4 Vitamin C Characteristics

Figure 4.4 (a) – (c) depict the variation of vitamin C with drying time for various temperature cases and for the chosen leafy vegetables. For all cases, the vitamin C content enhanced with drying time to reach a maximum and thereafter, reduced. Also, for lower temperatures, it is apparent that the maximum value persisted for certain period and thereafter reduced. For Kolmou, at 50 °C, the vitamin C content varied from 2.68 – 47.29 mg/100g for a variation in drying time from 0 – 300 min. For the 70 °C case, the response enhanced from 2.68 – 50.25 mg/100g for 0 – 180 min drying time variation and eventually reduced to 37.12 mg/100g for 300 min of drying time. At 60 °C, the response varied from 2.68 – 56.14 and 56.28 – 53.08 mg/100g for drying time variations from 0 – 240 min and 240 – 300 min, respectively.

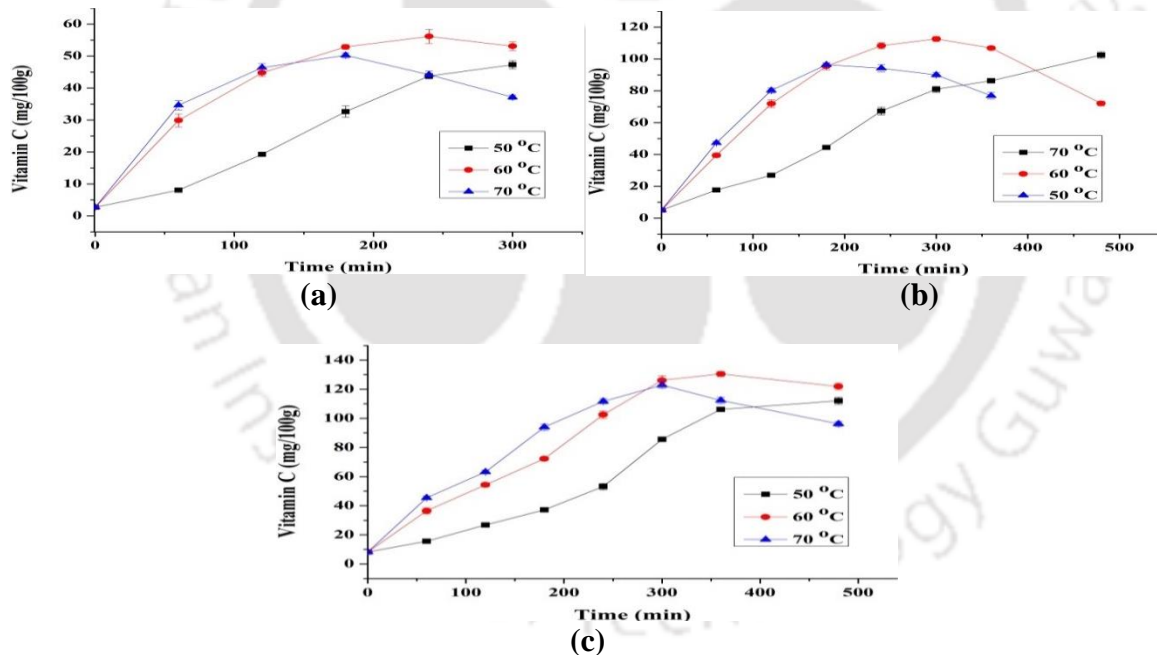


Fig. 4.4: Time dependent variation of Vitamin C content of tray dried (a) Kolmou (b) Jatilao and (c) Pui vegetable systems.

For Jatilao and at 50 °C, the vitamin C content varied from 5.07 – 102.47 mg/100g for a drying time variation from 0 – 480 min. However, for the case of higher temperature of 70 °C,

the vitamin C content enhanced from 5.07 – 96.36 mg/100g for 0 – 180 min drying time variation and thereafter reduced to 76.84 mg/100g for 360 min of drying. For 60 °C case, the response variations have been from 5.07 – 112.58 and 112.58 – 72.06 mg/100g for a variation in drying time from 0 – 300 min and 300 – 480 min, respectively.

For Pui and at 50 °C, the response varied from 8.23 – 116.03 mg/100g for a variation in drying time from 0 – 480 min. At 60 and 70 °C, the response varied from 8.23 – 128.46 – 121.92 mg/100g and 8.23 – 122.8 – 96.25 mg/100g for a variation in drying time from 0 – 360 – 480 min and 0 – 300 – 480 min, respectively.

At a lower temperature of 50 °C, the vitamin C content increased with increasing drying time. This is due to lower sensitivity of vitamin C constituents with temperature that did not enable greater degradation. Also, the dry matter content of the sample enhanced with drying time. Hence, due to these coupled effects, the vitamin C increased with drying time at the mentioned lower temperature. For the higher temperatures of 60 and 70 °C, a mixed effect has been apparent. With increasing drying time, the Vitamin C content increased to a certain level and thereafter reduced to a lower value. This affirms greater sensitivity of vitamin C constituents with temperature that underwent degradation only after a prolonged exposure to the drying environment. However, this is likely to be not the case for moderately higher drying time condition as the parametric sensitivity has been not very significant. Also, the vitamin C content reached early to its peak value at 70 °C in comparison with that being obtained at 60 °C. This is in agreement with the above presented hypothesis that affirms that with temperature enhancement, the degradation is significant for the vitamin C constituents. In summary, prolonged time duration and higher drying temperature detrimentally influenced vitamin C content. For all cases, the time dependent enhancement in vitamin C content is due to enhanced

dry matter content in the sample. For few cases, the reduction in vitamin C content is due to deterioration of heat sensitive vitamin C constituents at higher combinations of drying temperature and time (Santos and Silva 2008). It is well known that under conditions stated above, ascorbic acid undergoes oxidation to produce dehydro-ascorbic acid. Thereafter, this gets hydrolyzed to 2, 3-diketogolonic acid. Such hydrolysis further promotes oxidation and polymerization to eventually accelerate various inactive compounds in terms of desired constitution such as the vitamin C (Thankitsunthorn et al. 2009).

4.1.5 Antioxidant Activity Characteristics

The antioxidant activity trends with drying temperature and time variation are similar to those being discussed for the vitamin C. **Fig. 4.5 (a), (b) and (c)** depict these variations for Kolmou, Jatilao and Pui samples, respectively. Initially, an increase in antioxidant activity was not significant for all temperature cases. Thereafter, the response rapidly increased to reach a highest value and eventual reduction. For the Kolmou and 50 °C, the antioxidant activity enhanced from 1.72 – 76.18% for a variation in drying time from 0 – 300 min. For the same case, the response only enhanced marginally from 70.95% (240 min) to 76.18% (300 min) of drying time. Corresponding response characteristics for 60 and 70 °C cases varied from 1.72 – 91.74 – 84.57% and 1.72 – 86.82 – 67.38% for drying time durations of 0 – 240 – 300 min and 0 – 180 – 300 min drying time range, respectively.

For the Jatilao, at 50 °C case, the response varied from 2.4 – 83.25 for drying time enhancement from 0 – 480 min. Corresponding antioxidant activity values for 60 and 70 °C cases refer to variations as 2.4 – 88.91 – 81.23% and 2.4 – 84.15 – 70.19% for variations in drying time as 0 – 360 – 480 min and 0 – 180 – 360 min of drying time, respectively.

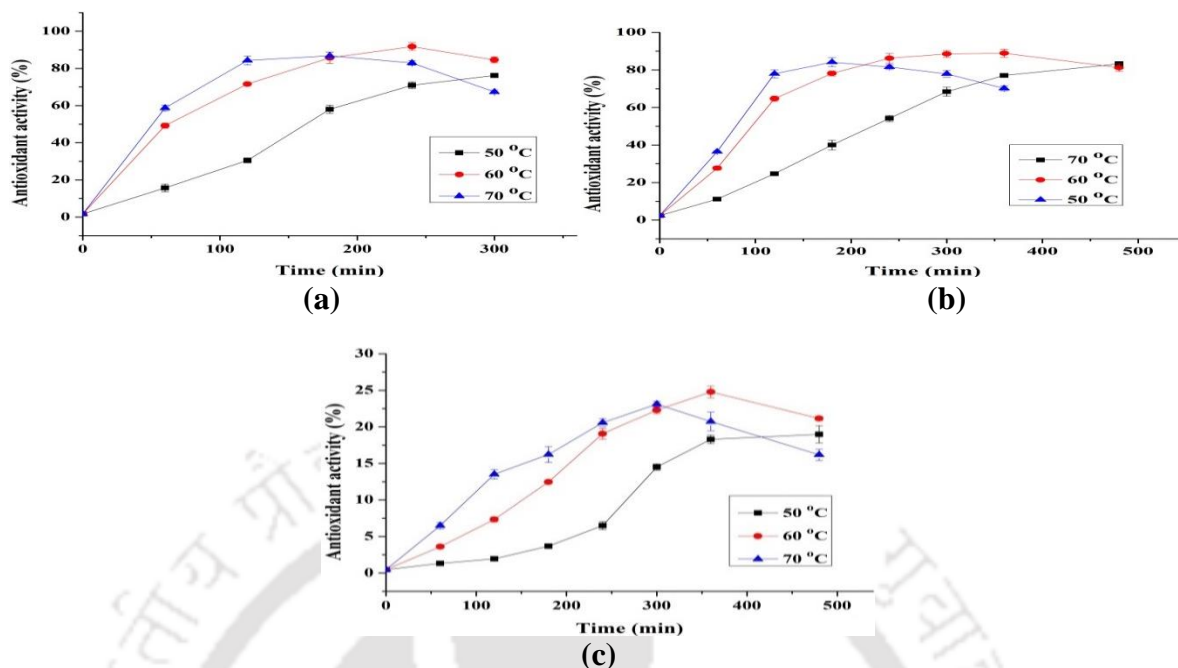


Fig. 4.5: Time dependent variation of antioxidant activity of tray dried (a) Kolmou (b) Jatilao and (c) Pui leafy vegetable systems.

For Pui, the antioxidant activity values for 70 and 60 °C varied as 0.47 – 22.11 – 16.18% and 0.47 – 23.79 – 21.15% for a variation in drying time as 0 – 300 – 480 min and 0 – 360 – 480 min, respectively. On the other hand, at 50 °C, the antioxidant activity varied from 0.47 – 18.98% for a drying time variation from 0 – 480 min.

The possible reason for an increase in antioxidant activity with drying time and temperature is the relative enhancement in dry matter content of the sample and activation of antioxidant compounds. On the contrary, the reduction of antioxidant activity at higher drying temperature and time combination is due to the degradation effect of heat and longer exposure of the heat sensitive constituents. For the antioxidant activity, it is apparent that the response variable has not been sensitively influenced at lower temperature. This is due to the dominance of dry matter enhancement with respect to the detrimental effect of loss of antioxidant

constituents at the lower temperature of 50 °C. Compared to the trends of antioxidant activity at 60 °C, the trend at 70 °C has been significantly detrimental with time in comparison with those being apparent at 60 °C. The peak value of antioxidant activity up to a particular drying time is due to the combination of an increase in dry matter content per gram of sample and activation of antioxidant constituents. The subsequent reduction in antioxidant activity was due to the detrimental effect of longer exposure to heat. Such detrimental effect was more prominent for 70 °C case and therefore reducing trend starts earlier than that being prevalent at 60 °C. Thus, higher temperature and longer exposure to heat are detrimental to retain antioxidant constituents.

4.1.6 Trial and Error Approach based Process Parametric Optimization

For Kolmou, based on drying kinetic studies, the maximum vitamin C (56.14 mg/100g) and antioxidant activity (91.74%) have been obtained for 60 °C and 240 min case. Corresponding moisture content is about 4.36%. On the other hand, for Jatilao, the kinetic studies indicated trial and error based optimal data set as 60 °C, 300 min, 4.36% moisture content, 112.58 mg/100g vitamin C and 88.53% antioxidant activity. Similarly, corresponding values for the tray dried Pui refer to 60 °C, 360 min, 6.04% moisture content, 128.46 mg/100g vitamin C and 23.79% antioxidant activity. The obtained data are comparable to those reported in our earlier work (chapter 3). Further, to identify better tray drying process parameters and develop best fit response variable equations for subsequent mathematical optimization assisted leafy soup formulations, statistical design based studies have been conducted. The findings of such methodology have been detailed in following section.

4.2 Statistical Design based Characterization of Process Parameters and Responses

4.2.1 Response Characteristics

Table 4.3 summarizes experimental data obtained with RSM design based specific combinations of process parameters and measured responses. Both drying temperature and time significantly influenced all response variable values. With increasing drying temperature and time, while moisture content followed a negative trend, both vitamin C and antioxidant activity followed a positive and increasing trend. For Kolmou, for a drying time of 90 min, an increase in drying temperature from 50 to 70 °C fostered reduction in moisture content and enhancement in vitamin C and antioxidant activity from 76.11 – 8.53%, 10.39 – 44.52 mg/100g and 18.13 – 78.37%, respectively. Corresponding range for 300 min drying time refer to 17.89 – 3.67%, 47.47 – 35.25 mg/100g and 76.37 – 66.35%, respectively. However, a contrary trend is apparent for the drying time case of 195 min. For the said case, the vitamin C and antioxidant activity enhanced from 19.01 – 55.33 mg/100g and 39.61 – 88.69%, respectively for an increment in temperature from 46.86 – 60 °C. A further increase in temperature to 74.14 °C affirmed reduction in these parameters to 38.07 and 80.5%, respectively. However, the moisture content was found to reduce from 56.83 – 2.87%. Similar mixed trends (increase followed with a reduction) are apparent at 60 °C. For the case, moisture content, vitamin C and antioxidant activity varied, respectively from 44.85 – 9.34 – 3.48%, 25.54 – 55.33 – 47.97 mg/100g and 44.16 – 88.69 – 74.13% for a variation in drying time from 46.51 – 195 – 343.49 min.

For Jatilao and for 150 min drying time, a variation in drying temperature from 50 - 70 °C fostered the moisture content, vitamin C and antioxidant activity to vary from 46.88 - 5.92%,

34.07 - 90.45 mg/100g and 30.31 - 81.88%. The significant reduction in moisture content with drying temperature is due to the higher moisture diffusivity at a higher temperature. For a higher drying time of 390 min, the corresponding variation of variables were from 16.11 to 4.07%, 85.07 – 65.33 mg/100g and 83.34 – 70.19%, respectively. For a drying time of 270 min, a mixed trend was achieved for vitamin C and antioxidant activity. For the case, the vitamin C varied from 43.55 – 112.29 mg/100g for an increase in drying temperature from 46.86 to 60 °C. Thereafter, it reduced to 69.61 mg/100g at 74.14 °C. Corresponding variation in antioxidant activity was 38.96 – 87.28 – 67.26%. On the other hand, for 270 min drying time duration, the maximum and minimum moisture content have been determined to be 37.69 and 3.28% for 45.86 °C and 74.14 °C, respectively. Similarly, for the intermediate drying temperature of 60 °C, the vitamin C and antioxidant activity increased from 60.68 – 112.29 mg/100g and 61.76 - 87.28% for an increase in drying time from 101.29 - 270 min. Thereafter, the responses reduced to 87.77 mg/100g and 83.07%, respectively for a drying time of 439.71 min. Correspondingly, at 60 °C, the moisture content was obtained as 26.45 and 4.37% for 100.29 and 439.71 min drying time, respectively.

For Pui, the trends were similar for all responses namely moisture, vitamin C and antioxidant activity. Correspondingly, for a variation in temperature from 50 – 70 °C, the response variables varied from 55.28 – 15.93%, 53.65 – 107.6 mg/100g and 6.76 – 17.01% and 7.36 – 4.73%, 115.28 – 95.05 mg/100g and 21.18 – 16.65% for 150 and 390 min drying time cases, respectively. However, at 60 °C, a mixed pattern existed in the drying time range of 202.72 – 330 – 457.28 min. For 150 min and 390 min drying time cases, corresponding response variables varied from 28.62 – 9.61 – 5.14, 82.54 – 129.25 – 116.94 mg/100g and 15.08 – 24.59 – 17.14%, respectively. Similarly, for 330 min drying time case, corresponding variations were

43.21 – 9.61 – 3.82, 69.52 – 129.25 – 104.74 mg/100g and 9.84 – 24.59 – 18.78% for drying range of 46.86 – 60 – 74.14 °C.

In summary, all lower drying time cases confirm enhanced vitamin C content and antioxidant activity with temperature. This is due to increased dry matter content (due to moisture removal from samples) and activated antioxidant constituents at higher temperature. Similar incremental trends also exist for the lower drying temperature of 50 °C and for entire range of investigated drying time. The above mentioned reason can be ascribed for the apparent trend. However, for the higher temperature case of 70 °C and enhanced drying time of 300 (Kolmou), 390 (Jatilao) and 420 min (Pui), both vitamin C and antioxidant activity reduced. This is due to the heat sensitivity of the constituents at higher drying temperature and prolonged exposure to heat. For the 60 °C case, the initial phase of increment for vitamin C and antioxidant activity is due to enhanced dry matter content and activation of few constituents. However, the later phase affirmed a reduction in both response variable values. This is due to the detrimental influence of long term exposure to higher temperature that deteriorates the relevant constituents in the samples. A similar mixed trend is apparent for drying time cases of 195 min (Kolmou), 270 min (Jatilao) and 330 min (Pui) in the drying temperature range of 46.86 – 74.14 °C.

Table 4.3: RSM design based experimental data summary of tray dried leafy vegetable systems.

Run	Temp (°C)	Kolmou				Jatilao				Pui			
		Time (Min)	Moisture (%)	Vit C (mg/100g)	Antioxidant Activity (%)	Time (Min)	Moisture (%)	Vit C (mg/100g)	Antioxidant activity (%)	Time (Min)	Moisture (%)	Vit C (mg/100g)	Antioxidant activity (%)
1	50	300	17.89 ± 0.63	47.47 ± 1.64	76.37 ± 0.85	390	16.11 ± 0.09	85.07 ± 1.82	83.34 ± 0.90	420	15.93 ± 0.35	107.6 ± 0.78	17.01 ± 0.33
2	45.86	195	56.83 ± 1.18	19.01 ± 1.04	39.61 ± 1.46	270	37.69 ± 0.26	43.55 ± 0.58	38.96 ± 0.45	330	43.21 ± 0.56	69.52 ± .72	9.84 ± 0.07
3	60	195	9.87 ± 0.92	55.85 ± 1.26	88.63 ± 1.17	270	6.29 ± 0.67	111.36 ± 3.88	83.62 ± 2.91	330	9.35 ± 1.22	128.39 ± 1.83	23.53 ± 0.83
4	60	195	8.17 ± 0.92	53.34 ± 1.26	88.05 ± 1.17	270	6.31 ± 0.67	114.52 ± 3.88	89.19 ± 2.91	330	8.79 ± 1.22	132.77 ± 1.83	24.55 ± 0.83
5	74.14	195	2.87 ± 0.11	38.07 ± 1.15	80.5 ± 1.15	270	3.28 ± 0.07	69.61 ± 1.38	67.26 ± 0.42	330	3.82 ± 0.14	104.74 ± 0.42	18.78 ± 0.26
6	60	195	9.91 ± 0.92	57.03 ± 1.26	88.27 ± 1.17	270	6.49 ± 0.67	114.23 ± 3.88	87.32 ± 2.91	330	9.06 ± 1.22	128.57 ± 1.83	24.28 ± 0.83
7	60	195	10.12 ± 0.92	55.68 ± 1.26	89.93 ± 1.17	270	7.18 ± 0.67	115.4 ± 3.88	87.67 ± 2.91	330	9.14 ± 1.22	129.19 ± 1.83	25.73 ± 0.83
8	60	195	9.82 ± 0.92	56.15 ± 1.26	87.09 ± 1.17	270	6.08 ± 0.67	110.15 ± 3.88	88.54 ± 2.91	330	12.07 ± 1.22	127.48 ± 1.83	24.04 ± 0.83
9	60	46.51	44.85 ± 0.76	25.54 ± 0.17	44.16 ± 2.02	100.29	26.45 ± 0.52	60.68 ± 0.76	61.76 ± 1.17	202.72	28.62 ± 2.15	82.54 ± 0.55	15.08 ± 0.16
10	70	90	8.53 ± 0.09	44.52 ± 0.28	78.37 ± 1.51	150	5.92 ± 1.10	94.45 ± 2.16	81.88 ± 1.73	240	7.36 ± 0.21	115.28 ± 1.64	21.18 ± 0.47
11	60	195	8.13 ± 0.92	54.92 ± 1.26	90.16 ± 1.17	270	5.12 ± 0.67	108.07 ± 3.88	87.28 ± 2.91	330	9.23 ± 1.22	129.11 ± 1.83	25.38 ± 0.83
12	70	300	3.67 ± 0.08	35.25 ± 0.43	66.35 ± 0.34	390	4.07 ± 0.16	65.33 ± 0.48	70.19 ± 1.06	420	4.73 ± 0.08	95.05 ± 2.10	16.65 ± 0.16
13	60	343.49	3.48 ± 0.13	47.97 ± 0.52	74.13 ± 0.63	439.71	4.37 ± 1.06	87.77 ± 0.63	83.07 ± 2.12	457.28	5.14 ± 0.12	116.94 ± 2.28	17.14 ± 0.18
14	50	90	76.11 ± 2.16	10.39 ± 0.14	18.13 ± 0.26	150	46.88 ± .08	34.07 ± 0.44	30.31 ± 1.09	240	55.28 ± 0.28	53.65 ± 0.31	6.76 ± 0.13

4.2.2 ANOVA based Identification of Best Fit Model

Table 4.4 (a) – (b), Table 4.5 (a) – (b) and Table 4.6 (a) – (b) represent a comparative framework for alternate fitness models and associated statistical parameters obtained during the analysis of variance (ANOVA) for Kolmou, Jatilao and Pui, respectively. For all cases, among various models, quadratic model possessing A , B , AB , A^2 and B^2 significant terms can be inferred to be the best fit model to represent all measured response characteristics. **Table 4.7** summarizes best fit model coefficients being defined with eq. 2.15 in chapter 2. For such cases, model F – values were significantly high. Also, the p values for all cases are low and about $p < 0.0001$. The lack of fit values was insignificant. The lack of fit F values for moisture content, vitamin C and antioxidant activity were 1.87, 2.38, and 2.23 for Kolmou, 2.07, 1.66, and 3.81 for Jatilao and 2.65, 2.84 and 0.64 for Pui. Corresponding p – values were 0.2523, 0.1864, and 0.2029 for Kolmou, 0.2232, 0.2893, and 0.0921 for Jatilao and 0.1602, 0.1454 and 0.6222 for Pui, respectively. For all samples and for all responses, the associated R^2 , $\text{adj } R^2$ and $\text{pred } R^2$ were found to be greater than 0.9286. On the other hand, adequate precision and C.V. (%) values for the responses were all greater than 34.56, 3.73 and 1.96, respectively for Kolmou, Jatilao and Pui samples, respectively. This affirms good fitness of the models.

Table 4.4: Summary of (a) alternate model fitness parameters and (b) ANOVA data of tray dried Kolmou vegetable system.

(a)																
Source	Moisture					Vitamin C					Antioxidant activity					
	Sum of Squares	df	Mean Square	F Value	p-value Prob > F	Sum of Squares	df	Mean Square	F Value	p-value Prob > F	Sum of Squares	df	Mean Square	F Value	p-value Prob > F	
Mean vs Total	5151.75	1	5151.75			25816.39	1	25816.39			72828.22	1	72828.22			
Linear vs Mean	4820.85	2	2410.42	15.44	0.0006	741.46	2	370.73	1.79	0.2123	2440.61	2	1220.3	3.25	0.0778	
2FI vs Linear	659.46	1	659.46	6.24	0.0316	537.08	1	537.08	3.08	0.1096	1234.12	1	1234.12	4.26	0.0660	
Quadratic vs 2FI	1048.53	2	524.26	462.11	< 0.0001	1722.02	2	861.01	358.59	< 0.0001	2881.44	2	1440.72	720.13	< 0.0001	Suggested
Cubic vs Quadratic	2.66	2	1.33	1.24	0.3539	5.09	2	2.55	1.08	0.3968	9.07	2	4.54	3.93	0.0812	Aliased
Residual	6.42	6	1.7			14.12	6	2.35			6.93	6	1.16			
Total	11689.66	14	834.98			28836.15	14	2059.73			79400.39	14	5671.46			

(b)																
Source	Moisture					Vitamin C					Antioxidant activity					
	Sum of Squares	df	Mean Square	F Value	p-value Prob > F	Sum of Squares	df	Mean Square	F Value	p-value Prob > F	Sum of Squares	df	Mean Square	F Value	p-value Prob > F	
Model	6528.84	5	1305.77	1150.97	< 0.0001	3000.56	5	600.11	249.94	< 0.0001	6556.16	5	1311.23	655.4	< 0.0001	significant
A-Temp	3046.33	1	3046.33	2685.18	< 0.0001	298.47	1	298.47	124.31	< 0.0001	1459.27	1	1459.27	729.4	< 0.0001	
B-Time	1774.52	1	1774.52	1564.15	< 0.0001	442.99	1	442.99	184.5	< 0.0001	981.33	1	981.33	490.51	< 0.0001	
AB	659.46	1	659.46	581.28	< 0.0001	537.08	1	537.08	223.68	< 0.0001	1234.12	1	1234.12	616.86	< 0.0001	
A ²	738.15	1	738.15	650.64	< 0.0001	1255.21	1	1255.21	522.77	< 0.0001	1502.78	1	1502.78	751.15	< 0.0001	
B ²	386.33	1	386.33	340.53	< 0.0001	588.89	1	588.89	245.26	< 0.0001	1600.18	1	1600.18	799.83	< 0.0001	
Residual	9.08	8	1.13			19.21	8	2.4			16.01	8	2			
Lack of Fit	4.8	3	1.6	1.87	0.2523	11.29	3	3.76	2.38	0.1864	9.16	3	3.05	2.23	0.2029	not significant
Pure Error	4.28	5	0.86			7.92	5	1.58			6.85	5	1.37			
Cor Total	6537.92	13				3019.77	13				6572.17	13				

Table 4.5: Summary of (a) alternate model fitness parameters and (b) ANOVA data of tray dried Jatilao vegetable system.

(a)															
Source	Moisture					Vitamin C					Antioxidant activity				
	Sum of Squares	df	Mean Square	F Value	p-value Prob > F	Sum of Squares	df	Mean Square	F Value	p-value Prob > F	Sum of Squares	df	Mean Square	F Value	p-value Prob > F
Mean vs Total	2372.24	1	2372.24			104623.52	1	104623.52			77315.1	1	77315.1		
Linear vs Mean	1801.46	2	900.73	13.73	0.0010	1190.24	2	595.12	0.74	0.4997	1407.77	2	703.88	2.39	0.1377
2FI vs Linear	209.09	1	209.09	4.08	0.0710	1448.56	1	1448.56	1.96	0.1922	1047.17	1	1047.17	4.77	0.0540
Quadratic vs 2FI	507.31	2	253.65	405.49	< 0.0001	7322.9	2	3661.45	352.37	< 0.0001	2135.31	2	1067.65	138.44	< 0.0001
Cubic vs Quadratic	2.59	2	1.3	3.23	0.1117	19.32	2	9.66	0.91	0.4522	16.01	2	8.005	1.05	0.4061
Residual	2.41	6	0.4			63.81	6	10.63			45.69	6	7.61		
Total	4895.11	14	349.65			114668.35	14	8190.6			81967.04	14	5854.79		

(b)															
Source	Moisture					Vitamin C					Antioxidant activity				
	Sum of Squares	df	Mean Square	F Value	p-value Prob > F	Sum of Squares	df	Mean Square	F Value	p-value Prob > F	Sum of Squares	df	Mean Square	F Value	p-value Prob > F
Model	2517.86	5	503.57	805	< 0.0001	9961.7	5	1992.34	191.74	< 0.0001	4590.24	5	918.05	119.04	< 0.0001
A-Temp	1291.92	1	1291.92	2065.25	< 0.0001	675.18	1	675.18	64.98	< 0.0001	769.15	1	769.15	99.74	< 0.0001
B-Time	509.54	1	509.54	814.54	< 0.0001	515.06	1	515.06	49.57	0.0001	638.62	1	638.62	82.81	< 0.0001
AB	209.09	1	209.09	334.25	< 0.0001	1448.56	1	1448.56	139.41	< 0.0001	1047.17	1	1047.17	135.79	< 0.0001
A ²	382.22	1	382.22	611.01	< 0.0001	5392.28	1	5392.28	518.94	< 0.0001	1929.21	1	1929.21	250.16	< 0.0001
B ²	160.15	1	160.15	256.01	< 0.0001	2446.02	1	2446.02	235.4	< 0.0001	313.02	1	313.02	40.59	0.0002
Residual	5	8	0.63			83.13	8	10.39			61.7	8	7.71		
Lack of Fit	2.77	3	0.92	2.07	0.2232	41.47	3	13.82	1.66	0.2893	42.91	3	14.3	3.81	0.0921
Pure Error	2.23	5	0.45			41.66	5	8.33			18.78	5	3.76		
Cor Total	2530.63	13				10044.83	13				4651.94	13			
Total															

Table 4.6: Summary of (a) alternate model fitness parameters and (b) ANOVA data of tray dried Pui vegetable system.

(a)															
Source	Moisture					Vitamin C					Antioxidant activity				
	Sum of Squares	df	Mean Square	F Value	p-value Prob > F	Sum of Squares	df	Mean Square	F Value	p-value Prob > F	Sum of Squares	df	Mean Square	F Value	p-value Prob > F
Mean vs Total	3511.73	1	3511.73			165208.85	1	165208.85			5206.37	1	5206.37		
Linear vs Mean	2354.73	2	1177.37	16.03	0.0006	2070.45	2	1035.22	1.88	0.1979	98.45	2	49.22	1.51	0.2630
2FI vs Linear	337.09	1	337.09	7.16	0.0233	1375.67	1	1375.67	2.95	0.1168	54.61	1	54.61	1.80	0.2095
Quadratic vs 2FI	451.71	2	225.86	93.49	< 0.0001	4623.86	2	2311.93	408.67	< 0.0001	298.82	2	149.41	253.06	< 0.0001
Cubic vs Quadratic	11.08	2	5.54	4.03	0.0777	27.93	2	13.96	4.83	0.0562	1.24	2	0.62	1.06	0.4026
Residual	8.25	6	1.37			17.33	6	2.89			3.49	6	0.58		
Total	6674.59	14	476.76			173324.09	14	12380.29			5662.98	14	404.50		

(b)															
Source	Moisture					Vitamin C					Antioxidant activity				
	Sum of Squares	df	Mean Square	F Value	p-value Prob > F	Sum of Squares	df	Mean Square	F Value	p-value Prob > F	Sum of Squares	df	Mean Square	F Value	p-value Prob > F
Model	3143.54	5	628.71	260.24	< 0.0001	8069.98	5	1614.00	285.30	< 0.0001	451.88	5	90.38	153.07	< 0.0001
A-Temp	1648.12	1	1648.12	682.19	< 0.0001	1222.37	1	1222.37	216.08	< 0.0001	89.13	1	89.13	150.97	< 0.0001
B-Time	706.61	1	706.61	292.48	< 0.0001	848.08	1	848.08	149.91	< 0.0001	9.32	1	9.32	15.78	0.0041
AB	337.09	1	337.09	139.53	< 0.0001	1375.67	1	1375.67	243.17	< 0.0001	54.61	1	54.61	92.50	< 0.0001
A ²	373.42	1	373.42	154.57	< 0.0001	3317.64	1	3317.64	586.45	< 0.0001	191.51	1	191.51	324.37	< 0.0001
B ²	106.27	1	106.27	43.99	0.0002	1637.44	1	1637.44	289.45	< 0.0001	129.80	1	129.80	219.85	< 0.0001
Residual	19.33	8	2.42			45.26	8	5.66			4.72	8	0.59		
Lack of Fit	11.87	3	3.96	2.65	0.1602	28.51	3	9.50	2.84	0.1454	1.31	3	0.44	0.64	0.6222
Pure Error	7.46	5	1.49			16.75	5	3.35			3.42	5	0.68		
Cor Total	3162.87	13				8115.24	13				456.61	13			

Table 4.7: Regression coefficients of best fit quadratic model to represent response characteristics of tray dried leafy vegetable systems.

Sample	Response	A_0	M_1	M_2	M_3	M_4	M_5
Kolmou	Moisture	682.02	-16.33	-1.13	1.22×10^{-2}	9.99×10^{-2}	6.56×10^{-4}
	Vitamin C	-624.24	18.41	1.05	-1.1×10^{-2}	-0.13	-8.10×10^{-4}
	Antioxidant activity	-772.97	21.73	1.63	-1.67×10^{-2}	-0.14	-1.34×10^{-3}
Jatilao	Moisture	480.63	-11.53	-0.60	6.02×10^{-3}	7.19×10^{-2}	3.23×10^{-4}
	Vitamin C	-1282.73	37.63	1.70	-1.59×10^{-2}	-0.27	-1.26×10^{-3}
	Antioxidant activity	-824.93	24.02	1.13	-1.35×10^{-2}	-0.16	-4.52×10^{-4}
Pui	Moisture	639.15	-13.33	-1.03	1.02×10^{-2}	7.11×10^{-2}	4.68×10^{-4}
	Vitamin C	-1353.91	33.47	2.56	-2.06×10^{-2}	-0.21	-1.84×10^{-3}
	Antioxidant activity	-320.38	7.80	0.60	-4.11×10^{-3}	-5.09×10^{-2}	-5.18×10^{-4}

4.2.3 Response Surface Characteristics of the Dried Samples

The response surface plots of moisture content, vitamin C and antioxidant activity for Kolmou, Jatilao and Pui have been illustrated in **Fig. 4.6 (a) – (c)**, **Fig. 4.7 (a) – (c)** and **Fig. 4.8 (a) – (c)**, respectively. The moisture content for all vegetable samples followed a negative trend with drying temperature and time. This is due to a higher rate of heat transfer and higher effective moisture diffusivity. Higher drying temperature and time facilitated better moisture evaporation and transport from inner portions to the outer surface of the sample. On the other hand, their interaction and square terms influenced the moisture content positively ($p < 0.0001$). The minimum and maximum moisture content achieved for Kolmou, Jatilao and Pui samples were 2.87 and 74.11%, 3.28 and 46.88% and 3.82 and 55.28%, respectively.

On the other hand, vitamin C and antioxidant activity followed contrary trends. The effect of drying temperature and time were significant and positive for both responses ($p < 0.0001$). A significant but negative affect was observed for the interaction and quadratic terms of independent variables ($p < 0.0001$). These response surfaces are comparable with those being

reported for hot air dried olive leaves (Erbay and Icier 2009), *Artemisia absinthium* leaves (Karimi et al. 2012), torch ginger (Juhari et al. 2012) and mango seed kernel samples (Ekorong et al. 2015). The highest vitamin C and antioxidant activity achieved were 55.33 mg/100g and 88.69%, respectively for Kolmou, 112.29 mg/100g and 87.28% for Jatilao, respectively and 129.25 mg/100g and 24.59%, respectively for Pui.

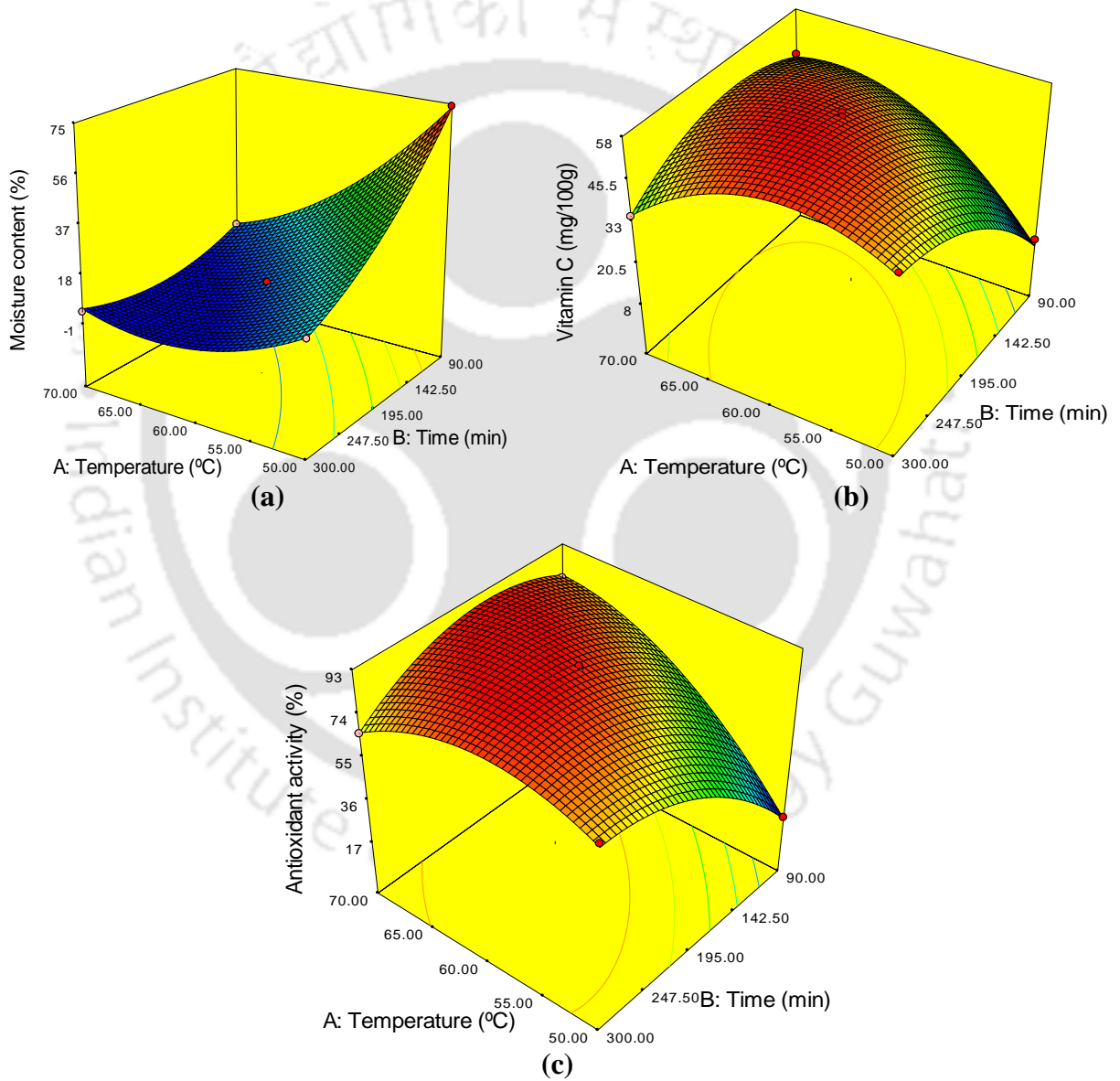


Fig. 4.6: 3-D response surface plots of tray dried Kolmou vegetable system (a) Moisture content (b) Vitamin C content and (c) Antioxidant activity.

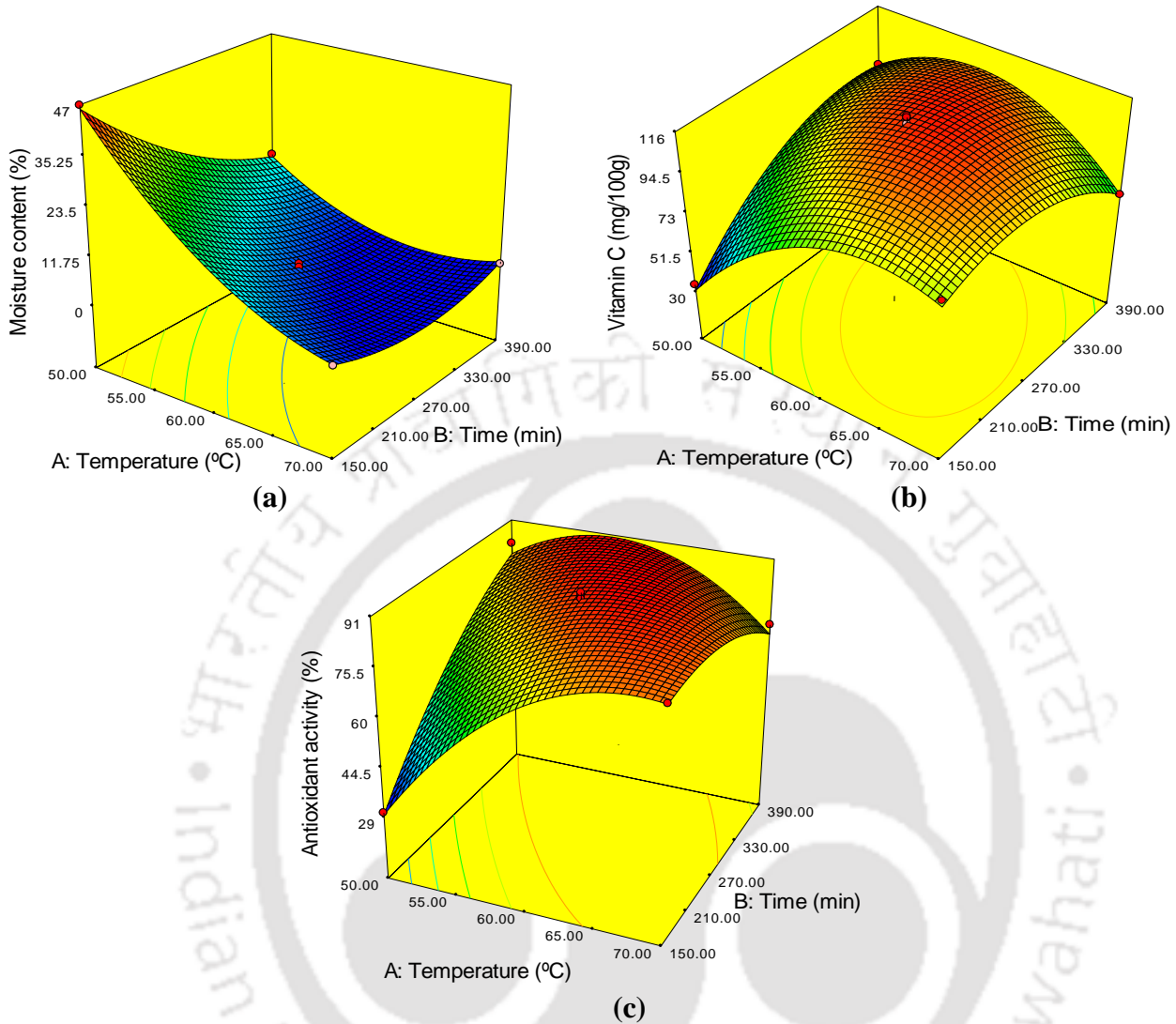


Fig. 4.7: 3-D response surface plots of tray dried Jatilao vegetable system (a) Moisture content (b) Vitamin C content and (c) Antioxidant activity.

The positive effect of drying temperature and time on vitamin C and antioxidant activity is due to enhanced dry matter content and expression of these components. Being volatile, the vitamin C and antioxidant constituents being volatile continuously diminished in due course of drying. Also, higher temperature and longer exposure had a detrimental effect on both variable. However, the same variable on a wet basis enhanced with drying temperature and time as moisture content reduced significantly. The lower values for vitamin C content and antioxidant

activity at a lower temperature have been attributed to the high moisture content of the sample. However, such enhancement only existed up to a certain limit and thereafter negative sensitivity is apparent for higher combinations of temperature and time. On the other hand, due to detrimental effect, higher interaction and quadratic terms reduced vitamin C and antioxidant activity values.

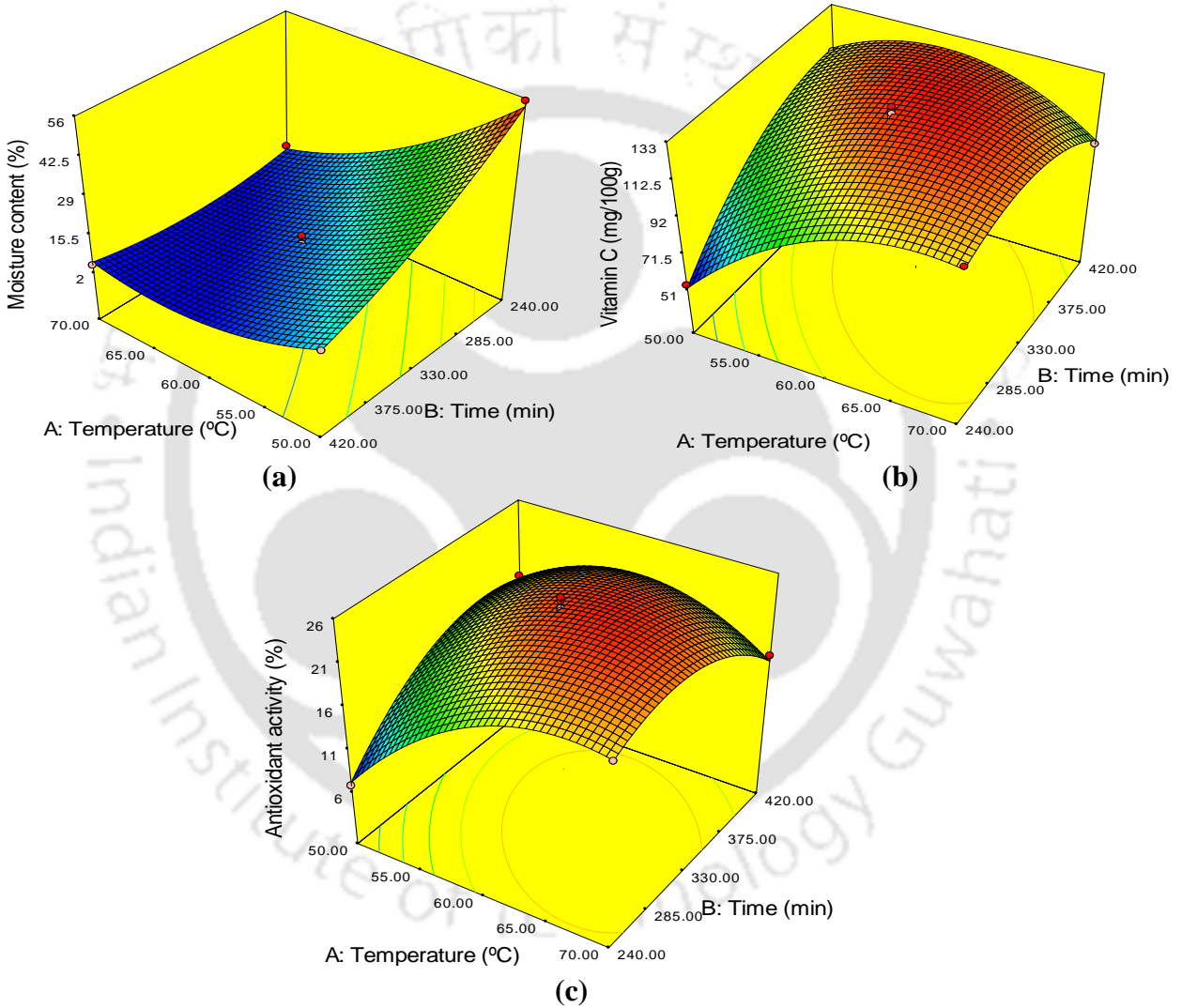


Fig. 4.8: 3-D response surface plots of tray dried Pui vegetable system (a) Moisture content (b) Vitamin C content and (c) Antioxidant activity.

4.2.4 Optimization of Process Parameters

Table 4.8 (a) presents various optimal data sets corresponding to trial and error, RSM experimental and numerical optimization approaches for the said leafy vegetables. Based on RSM based numerical optimization, the optimal tray drying process characteristics for Kolmou were 61.28 °C drying temperature, 236.90 min drying time, 2.87% moisture content, 57.02 mg/100g vitamin C content and 91.36% antioxidant activity. Similarly, for Jatilao, the values were 61.62 °C drying temperature, 292 min drying time, 3.28% moisture, 113.36 mg/100g vitamin C and 89.37% antioxidant activity. On the other hand, for Pui, these values were 63.90 °C, 342.19 min, 4.37%, 130.98 mg/100g and 24.99%, respectively. These optimal data sets are in agreement with those determined experimentally. This is ensured with the lower standard deviation values of 0.61, 2.18 and 1.05 for Kolmou, Jatilao and Pui, respectively.

On the other hand, corresponding RSM experimental based optimal values were 60 °C drying temperature, 195 min drying time, 9.34% moisture, 55.33 mg/100g vitamin C and 88.69% antioxidant activity for Kolmou, 60 °C drying temperature, 270 min drying time, 6.24% moisture, 112.29 mg/100g vitamin C and 87.28% antioxidant activity for Jatilao and 60 °C drying temperature, 330 min drying time, 9.61% moisture, 129.25 mg/100g vitamin C and 24.59% antioxidant activity for Pui samples.

On the other hand, experimental trial and error based optimal values refers to 60 °C drying temperature, 240 min drying time, 4.36% moisture, mg/100g vitamin C content and 91.74% antioxidant activity for Kolmou; 60 °C drying temperature, 300 min drying time, 4.36% moisture, 112.58 mg/100g vitamin C and 88.53% antioxidant activity for Jatilao and 60 °C drying temperature, 360 min drying time, 6.04% moisture, 128.46 mg/100g vitamin C and 23.79% antioxidant activity, respectively for Pui leafy vegetable sample.

Compared with trial and error based optimum values, the RSM based optimal values for vitamin C and antioxidant activity were almost similar. However, the moisture content was lower in comparison to that obtained from RSM experimental and trial and error based data sets. These values are closely related to data obtained at temperature 60 °C and time 240 min for all samples reported in our previous work (chapter 3). In this chapter, response variables have been reported to be about 4.21% moisture content, 56.96 mg/100g vitamin C content and 92.34% antioxidant activity for Kolmou sample; 7.68% moisture content, 112.68 mg/100g vitamin C content and 89.45% antioxidant activity for Jatilao sample and 10.44% moisture content, 123.94 mg/100g vitamin C content and 23.08% antioxidant activity for Pui sample, respectively. For Kolmou and Pui, the obtained values were higher than the data (50 and 107.28 mg/100g) being reported for shadow and oven dried Kolmou (Doka et al. 2014) and Pui (Saha et al. 2015), respectively. The lower values for these cases have been possibly due to the detrimental effect of shadow and oven drying, respectively on the mentioned variable. On the other hand, antioxidant activity for Kolmou has been reported to be 85% in a relevant literature (Dong-Jiann et al. 2005), which is lower than the value obtained in this work. Thereby, the findings confirm upon the superiority of intermittent airflow assisted tray drying system for the drying of chosen leafy vegetables.

In summary, compared to trial and error and RSM experimental optimal values, the RSM based numerically optimized values improved in terms of lower moisture content and higher vitamin C and antioxidant activity. Further, it shall be noted that for chopped Kolmou samples, the optimal drying temperature indicated a reduction in drying time to 195 min but with a significant reduction in vitamin C (47.25 mg/100g) and antioxidant activity (84.16%) characteristics. Similarly, the responses as well reduced significantly for chopped Jatilao and Pui

dried at 61.62 and 63.90 °C, respectively (Table 4.8 (a)). Hence, chopping is not recommended for the said leafy vegetables.

Table 4.8: Experimental, predicted and literature data of the optimal response characteristics of tray dried leafy vegetable systems.

(a)

Sample	Approach/optimal parameter	Drying method	Temp (°C)	Time (Min)	Moisture (%)	Vit C (mg/100g)	Antioxidant activity (%)	Reference
Kolmou	RSM optimized	Tray drying	61.28	236.90	2.87	57.02	91.36	This work
	RSM non- optimized	do	60	195	9.34	55.33	88.69	do
	Kinetics best	do	60	240	4.18	56.14	91.74	do
	Experimental at optimized conditions	do	61.23	237.07	3.44 ± 0.16	57.88 ± 0.62	92.06 ± 1.21	do
Chopped Kolmou	Experimental	do	61.23	195	2.54 ± 0.28	47.25 ± 0.34	84.16 ± 0.93	do
Kolmou	Experimental	do	60	240	4.21	56.96	92.34	Chapter 3
Jatilao	RSM optimized	Tray drying	61.62	292	3.28	113.36	89.37	This work
	RSM non- optimized	do	60	270	6.24	112.29	87.28	do
	Kinetics best	do	60	300	4.36	112.58	88.91	do
	Experimental at optimized conditions	do	61.62	292	3.42 ± 0.1	113.74 ± 1.26	88.93 ± 0.91	do
Chopped Jatilao	Experimental	do	61.62	255	2.38 ± 0.15	102.53 ± 2.17	79.26 ± 1.44	do
Jatilao	Experimental	do	60	240	7.68	112.68	89.45	Chapter 3
Pui	RSM optimized	Tray drying	63.90	342.19	4.37	130.98	24.99	This work
	RSM non- optimized	do	60	330	9.61	129.25	24.59	do
	Kinetics best	do	60	360	6.04	128.46	23.80	do
	Experimental at optimized conditions	do	63.90	342.19	4.19 ± 0.38	132.46 ± 2.18	25.34 ± 0.66	do
Chopped Pui	Experimental	do	63.90	342.19	3.16 ± 0.34	119.64 ± 3.26	17.48 ± 1.13	do
Pui	Experimental	do	60	240	10.44	123.94	23.08	Chapter 3

(b)

Sample	Drying methods	Optimal drying conditions	Optimal drying characteristics						Reference
			Moisture (%)	Carbohydrate (%)	Soluble protein (%)	Fat (%)	Fibre (%)	Ash (%)	
Kolmou	Tray drying	61.28 °C, 236.90 min	2.87	49.82	11.04	4.40	9.37	11.52	This work
	do	60 °C, 240 min	4.21	49.27	10.74	4.26	8.74	10.98	Chapter 3
Jatilao	Tray drying	61.62 °C, 292 min	3.42	41.24	15.30	9.27	12.93	14.38	This work
	do	60 °C, 240 min	4.21	39.61	14.62	8.67	12.15	13.88	Chapter 3
Pui	Tray drying	63.90 °C, 342.19 min	4.37	48.14	10.38	4.17	7.13	16.58	This work
	do	60 °C, 240 min	10.44	46.05	9.91	3.87	6.53	16.32	Chapter 3

4.3 Proximate Characteristics of Optimally Tray Dried Samples

Table 4.8 (b), respectively depict the proximate characteristics (namely carbohydrate, soluble protein, ash, fat and crude fibre) of leafy vegetable samples being subjected to optimal drying conditions. Among these leafy vegetables, Jatilao possessed higher fat (9.27%), soluble protein (15.30%) and crude fibre (12.93%) in comparison with Kolmou and Pui. For these samples, the corresponding response values were 4.40, 11.04 and 9.37% and 4.17, 10.38 and 7.13%, respectively. On the other hand, carbohydrate and ash content were higher in tray dried Kolmou (49.82%) and Pui (16.58%) samples, respectively. The carbohydrate content of dried Jatilao and Pui were 41.24 and 48.14, respectively. Similarly, the corresponding ash content of Kolmou and Jatilao were 11.52% and 14.38, respectively. In conclusion, all leafy vegetables are rich sources of proximate parameters. These findings are in good agreement reasonable with the best data reported in the previous chapter (chapter 3). Accordingly, such data has also been presented in **Table 4.8 (b)**.

4.4 Summary

With no prior art available for the chosen vegetables, this study affirms upon the realization of useful insights related to tray drying of Kolmou, Jatilao and Pui leafy vegetable samples. Firstly, among trial and error based kinetics approach and RSM based design of experiments approach, the later ensured better identification of optimum process variables for all samples (61.28 °C temperature, 236.9 min drying time, 2.87% moisture content, 57.02 mg/100g vitamin C content and 91.36% antioxidant activity for Kolmou, 61.62 °C drying temperature, 292 min drying time, 3.28% moisture, 113.36 mg/100g vitamin C and 89.37% antioxidant activity for Jatilao and 63.90 °C drying temperature, 342.19 min drying time, 4.37% moisture, 130.98 mg/100g vitamin

C and 24.99% antioxidant activity for Pui). Secondly, the response equations obtained for chosen leafy vegetables can be used further to carry out a mature mathematical optimization to achieve near optimal soup formulation with maximum response characteristics. On the other hand, drying kinetics facilitated the determination of moisture diffusivity of the tray drying process (6.36×10^{-13} to 5.16×10^{-12} m²/s, $1.79 - 5.36 \times 10^{-12}$ m²/s and $9.94 \times 10^{-13} - 6.23 \times 10^{-12}$ m²/s for Kolmou, Jatilao and Pui, respectively) and activation energy of the tray dried leafy vegetables (45.21, 25.3 and 43.15 kJ/mol for Kolmou and Pui, respectively).

In summary, the reported values affirm coherence and compatibility of all leafy vegetables for the development of mixed food product. However, parallel processing of the leafy vegetables is not recommended due to significant variations in optimal drying time. Also, since Kolmou and Pui are good source of antioxidants and vitamin C, respectively and Jatilao constitutes good vitamin C and antioxidants, these leafy vegetables are suitable to achieve mixed food products such as vegetable soups. With developed model equations for all response variables for all leafy vegetables can therefore be used to formulate mathematical optimization based leafy vegetable soup product. Thereby, the carried out investigations affirm that the underutilized leafy vegetables of the North-East India can be transformed into value added products such as soups and thereby drive economic competitiveness of the horticultural sector in the North-East India.

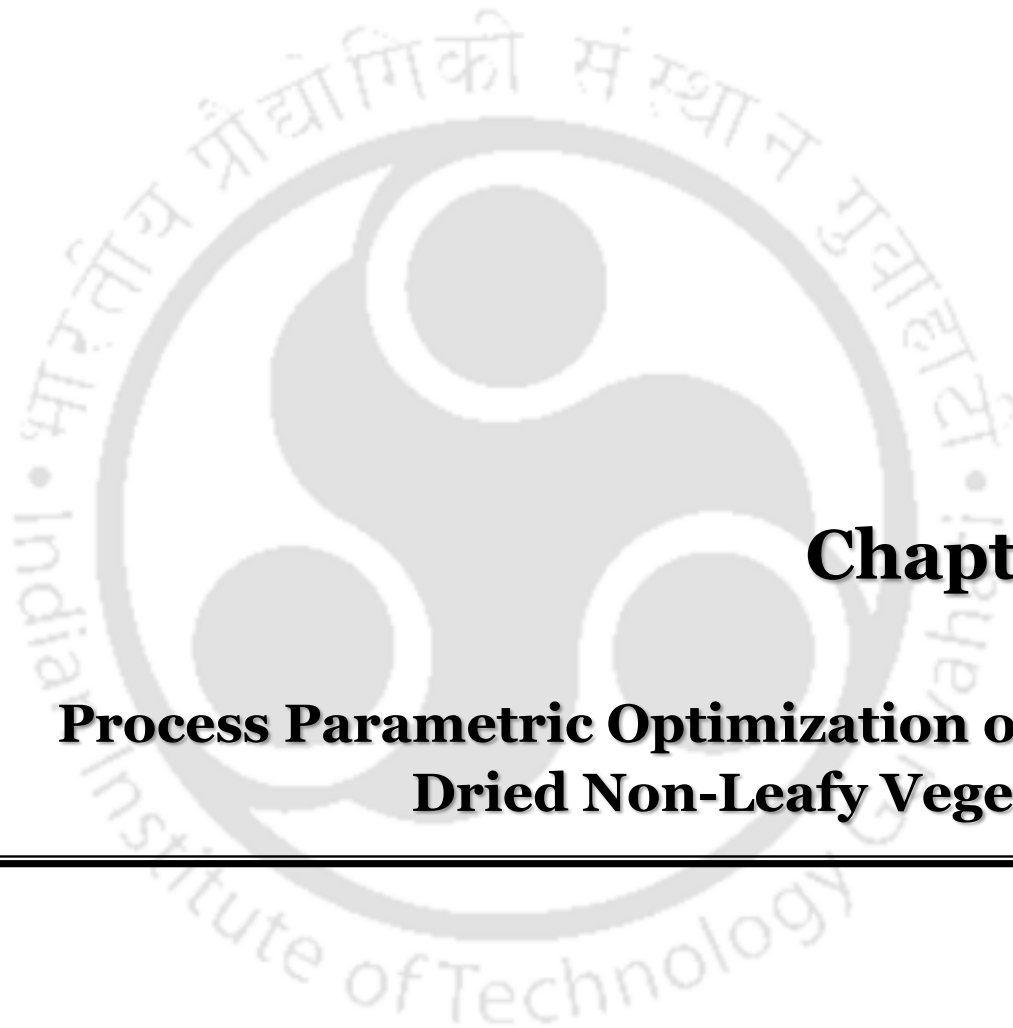
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Chapter 5:

Process Parametric Optimization of Tray Dried Non-Leafy Vegetables



Process Parametric Optimization of Tray Dried Non-Leafy Vegetables

In this chapter, a detailed account of the optimal characteristics of tray dried chosen non-leafy vegetables has been addressed in the context of the associated optimal process parameters. After providing a brief synopsis of the carried out work in the available prior art in section 5.1, the following section addresses trial and error based process-product characteristics such as moisture kinetics, moisture diffusivity and activation energy. Further, vitamin C and antioxidant activity variation with drying temperature and time has also been addressed for the chosen vegetables. In section 5.3, the optimal process-product characteristics have been addressed by targeting response surface methodology based approach. This involved statistical design analysis of alternate models and upon their analysis of variance (ANOVA) data trends. Thereafter, best fit model has been identified to eventually conduct numerical optimization of process variables. Finally, in section 5.4, proximate characteristics of optimally tray dried non-leafy vegetables have been summarized. Following this, a critical summary of the findings has been presented in section 5.5 of the thesis.

5.1 Introduction

The prior art (Jha and Sit 2020; Zahoor and Khan 2019; Ouyang et al. 2020; Gupta et al. 2013; Abano et al. 2014; John et al. 2014) did not accommodate an integral approach of kinetics and RSM based tray during characteristics of the chosen non-leafy vegetables or associated food

products. Secondly, trial and error based optimization addressed in the literature (Jha and Sit 2020; Zahoor and Khan 2019; Ouyang et al. 2020; John et al. 2014) did not consider nutritionally important parameters such as vitamin C and antioxidant activity.

Considering the lacunae, the thesis targets upon the combinational optimization of process parameters based on kinetics and RSM studies. Kinetic studies targeted evaluation of drying kinetics associated to moisture removal, vitamin C and antioxidant activity variability trends. The trial and error based approach involves drying characteristic curves, moisture diffusivity and activation energy evaluation. This enables to deduce useful insights into the compatibility of selected vegetables with other non-leafy vegetables and its coherence towards product formulation. On the other hand, statistical design involves the determination of optimal process parameters by following the statistical experimental design methodology. The analysis of variance (ANOVA) and subsequent optimization was addressed using Design expert 7.0 software. Such methodology facilitates the identification of best process variables and does not necessitate upon a careful selection of independent variables. However, this is not the case for the trial and error based approach. In summary, the integrated methodology involving both approaches ensures useful insights with respect to the parametric sensitivity for the desired characteristics in due course of drying. Such studies provide guidelines for the generalization of studies associated with process-product characteristics and food product development. Finally, for best product-process conditions, proximate parameters were evaluated to assist furthering efforts towards high quality food product formulation.

5.2 Kinetics based Characterization of Process Parameters and Responses

5.2.1 Drying Kinetics

For Kolphul, Kaskal, Komora and Posola, the moisture ratio (MR) variation with drying time have been depicted in **Fig. 5.1 (a), (b), (c) and (d)**, respectively. As shown, for all cases, the moisture content follows an exponentially declining trend with drying time. The illustrated drying characteristic curves affirm two drying phases. Among these, the first phase corresponds to an initial rapid reduction phase that affirms significant MR reduction with drying time.

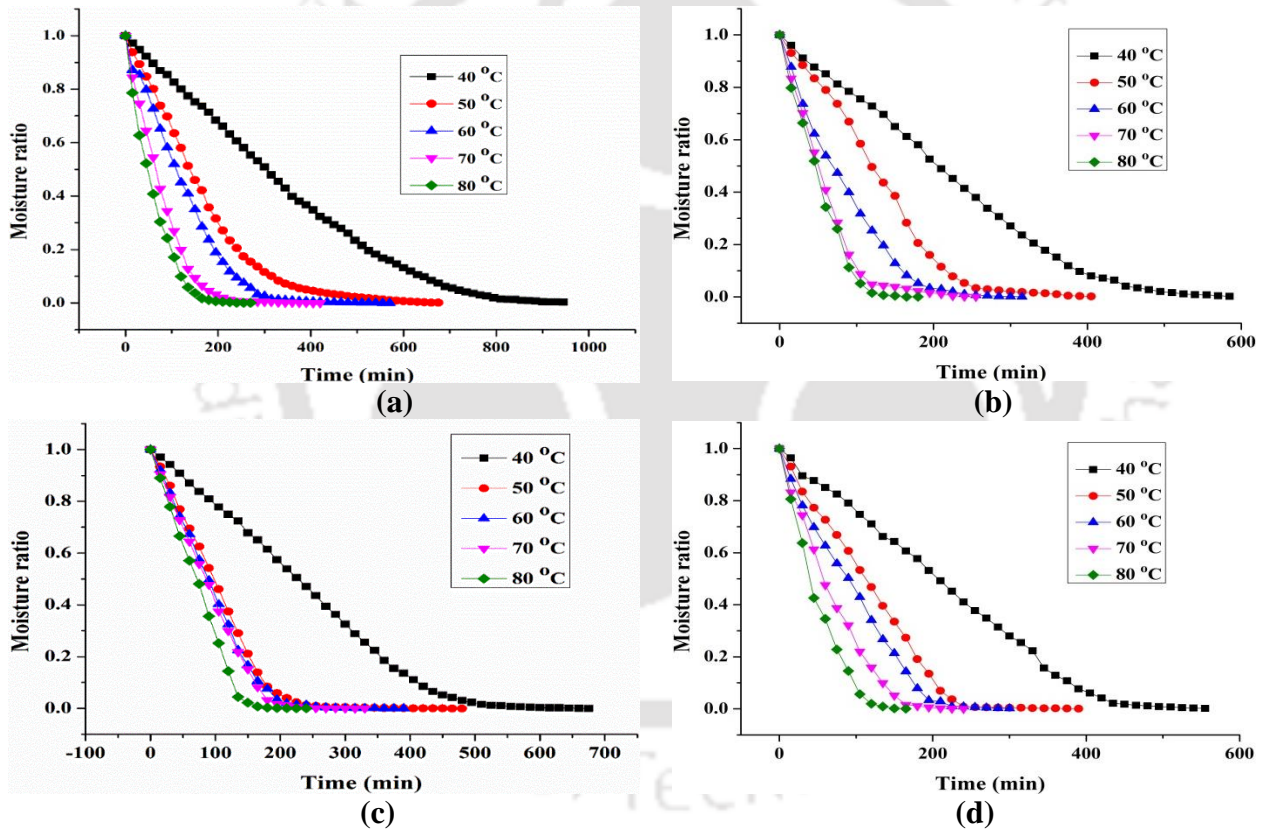


Fig. 5.1: Drying characteristics of tray dried (a) Kolphul (b) Kaskal (c) Komora and (b) Posola vegetables.

For Kolphul, the moisture ratio rapidly reduced from 1 – 0.168, 0.116, 0.117, 0.128 and 0.171 for 40, 50, 60, 70 and 80 °C for a corresponding drying time of 555, 300, 225, 135 and 105

min, respectively. For Kaskal samples, the MR similarly reduced significantly during this phase for a variation in drying time from 0 – 345, 0 – 225, 0 – 180, 0 – 165 and 0 – 105 min at drying temperatures of 40, 50, 60, 70 and 80 °C, respectively. Corresponding MR values varied from 1 – 0.157, 1 – 0.036, 1 – 0.079, 1 – 0.016 and 1 – 0.057, respectively. For Komora, the initial rapid MR reduction was apparent up to 420, 225, 210, 195 and 135 min drying time and the corresponding MR variations were from 0.91 – 0.085, 0.77 – 0.024, 0.74 – 0.02, 0.73 – 0.018 and 0.67 – 0.044, respectively. Similarly, for the Posola case, at various drying temperatures, the corresponding MR varied from 1 – 0.119, 1 – 0.079, 1 – 0.082, 1 – 0.048 and 1 – 0.051 for a drying time variation from 0 – 375, 0 – 225, 0 – 165, 0 – 120 and 0 – 105 min, respectively.

Following the first drying phase, at various drying temperatures, the second phase involved a slower reduction in corresponding MR values with drying time. This is apparent in the corresponding drying time range values of 555 – 795, 300 – 570, 225 – 375, 135 – 255 and 105 – 165 min and 375 – 600, 225 – 420, 165 – 330, 120 – 270, and 105 – 195 min of drying time for Kolphul and Kaskal, respectively. Similarly, for Komora and Posola, the corresponding values were 420 – 675, 225 – 480, 210 – 405, 195 – 330 and 135 – 240 min and 345 – 570, 225 – 405, 180 – 315, 165 – 255 and 105 – 180 min. Thereafter, near equilibrium MR values have been obtained which varied marginally due to uncontrollable variations in drying temperature, ambient humidity etc. This is due to achievement of equilibrium moisture content (EMC). Hence, no further variation in moisture content and weight of the sample has been observed. Therefore, MR value did not vary after reaching the EMC value.

The rapid reduction in MR trend was due to higher moisture diffusivity that was achieved due to availability of sufficient surface moisture in the dried horticultural samples. Contrary to this, the second slower drying phase is attributed to the lower moisture transport from the inner

portions of the sample to their respective surfaces. Such lower moisture transport rate is due to the resistances offered by the adjacent sample entities and hard crust of the samples. The overall moisture kinetics in terms of MR versus drying time followed an exponential reducing trend. The trend is similar to that being reported for hot air dried ripe banana (Doymaz 2010), hot air dried pumpkin (Limpaiboon 2011) and tray dried buttermilk soaked chopped banana blossom (John et al. 2015).

5.2.2 Fitness of Alternate Drying Models

Table 5.1 (a) – (d) present results associated with the fitness of alternate drying models with respect to evaluated drying characteristics. For Kolphul, among all models, the Page model was the best fit model with high R^2 , low RSS and reduced chi-squared values for all cases. However for 40 °C case, Wang and Singh model also affirmed good fitness. Among alternate models except at 40 °C, Wang and Singh model possessed poor fitness indices (relatively lower R^2 , higher RSS and reduced chi-squared values). For Kaskal and at 40, 50 and 70 °C, the Page model was the best fit model with high R^2 , low RSS and reduced chi-squared values. However, for 60 and 80 °C case, the best fit model was the Wang and Singh model. On the other hand, for Posola case, Wang and Singh model possessed the best fitness indices (higher R^2 , lower RSS and reduced chi-squared values) for all temperature cases. For Komora, Page model possessed best fitness parameters to represent evaluated drying data at all temperatures. This was confirmed with highest R^2 and lowest Chi-squared and RSS values. Also, the temperature positively influenced the drying rate constant. Compared to time, the temperature effect was significant for moisture removal from the product and hence drying rate constant enhanced with temperature for all samples.

Table 5.1: Alternate drying model fitness parameters representing tray drying characteristics of (a) Kolphul (b) Kaskal (c) Komora and (d) Posola.

		(a)					
Model	Model equation	Parameters	40 °C	50 °C	60 °C	70 °C	80 °C
Newton	$MR = \exp(-kt)$	k	0.00279	0.00595	0.0078	0.0128	0.01671
		R^2	0.9519	0.97302	0.96943	0.98169	0.98974
		RSS	0.30984	0.11421	0.10793	0.04402	0.01718
		χ^2	0.00492	0.00254	0.00284	0.00157	9.54×10^{-4}
		k	1.55×10^{-4}	6.59×10^{-4}	9.21×10^{-4}	0.00288	0.0079
Page	$MR = \exp(-kt^n)$	n	1.47907	1.41483	1.42098	1.32531	1.17218
		R^2	0.99571	0.99879	0.9956	0.99702	0.9952
		RSS	0.02718	0.005	0.01513	0.0069	0.00758
		χ^2	4.38×10^{-4}	1.14×10^{-4}	4.09×10^{-4}	2.55×10^{-4}	4.46×10^{-4}
		a	1.12321	1.10945	1.08957	1.06456	1.02856
Henderson and Pabis	$MR = a \exp(-kt)$	k	0.00311	0.00653	0.0084	0.01353	0.01714
		R^2	0.96653	0.98227	0.97518	0.98453	0.98998
		RSS	0.21215	0.07336	0.08531	0.03586	0.01583
		χ^2	0.00342	0.00167	0.00231	0.00133	9.31×10^{-4}
		a	1.33934	1.13475	1.11971	1.08122	1.05453
Logarithmic	$MR = a \exp(-kt) + c$	c	-0.28889	-0.05022	-0.05599	-0.03039	-0.04365
		k	0.00183	0.0057	0.00722	0.01239	0.01511
		R^2	0.99354	0.98755	0.98324	0.98809	0.99487
		RSS	0.04032	0.05034	0.05606	0.02659	0.00763
		χ^2	6.61×10^{-4}	0.00117	0.00156	0.00102	4.77×10^{-4}
Wang and Singh	$MR = M_0 + at + bt^2$	M_0	1.03697	0.98717	0.96201	0.89567	0.91872
		a	-0.00215	-0.00397	-0.00482	-0.00651	-0.0094
		b	1.10×10^{-6}	3.88×10^{-6}	5.74×10^{-6}	1.10×10^{-5}	2.31×10^{-5}
		R^2	0.99783	0.98527	0.98423	0.95896	0.9848
		RSS	0.01351	0.05957	0.05273	0.09159	0.0226
Singh et al. (2014)	$MR = \exp(-kt) - akt$	χ^2	2.21×10^{-4}	0.00139	0.00146	0.00352	0.00141
		k	0.00195	0.00544	0.00717	0.01229	0.01006
		a	0.12065	0.01675	0.01507	0.00646	0.01572
		R^2	0.98988	0.98038	0.97783	0.98461	0.99401
		RSS	0.06414	0.08121	0.0762	0.03567	0.00947
Silva et al. (2012)	$MR = \exp(-at + bt^{1/2})$	χ^2	0.00103	0.00185	0.00206	0.00132	5.57×10^{-4}
		a	0.00425	0.00886	0.01128	0.01773	0.02026
		b	-0.0284	-0.03966	-0.04231	-0.04662	-0.02967
		R^2	0.98188	0.99332	0.98631	0.99215	0.99289
		RSS	0.11484	0.02764	0.04706	0.0182	0.01124
χ^2	0.00185	6.28×10^{-4}	0.00127	6.74×10^{-4}	6.61×10^{-4}		

(b)

Model	Model equation	Parameters	40 °C	50 °C	60 °C	70 °C	80 °C
Newton	$MR = \exp(-kt)$	k	0.00425	0.00748	0.01191	0.01711	0.01911
		R^2	0.93377	0.92729	0.98037	0.97346	0.96456
		RSS	0.28473	0.23857	0.04089	0.04721	0.05144
		χ^2	0.28473	0.00884	0.00195	0.00278	0.00429
Page	$MR = \exp(-kt^n)$	k	0.0001	0.00013	0.00343	0.00261	0.00293
		n	1.61062	1.796	1.26707	1.44114	1.4487
		R^2	0.99284	0.99477	0.99414	0.99649	0.99191
		RSS	0.02998	0.01651	0.00162	0.00036	0.01076
Henderson and Pabis	$MR = a \exp(-kt)$	χ^2	0.00078	0.00063	0.00058	0.00588	0.000097
		a	1.12977	1.1471	1.05879	1.07488	1.06675
		k	0.00477	0.00843	0.01254	0.01821	0.0202
		R^2	0.94979	0.94408	0.98293	0.97724	0.96646
Logarithmic	$MR = a \exp(-kt) + c$	RSS	0.21031	0.17669	0.03386	0.0381	0.04462
		χ^2	0.00553	0.0068	0.00169	0.00238	0.00406
		a	1.47809	1.2881	1.10847	1.1046	1.16364
		c	-0.42801	-0.19384	-0.07983	-0.04578	-0.12828
Wang and Singh	$MR = M_0 + at + bt^2$	k	0.0024	0.00572	0.01019	0.01612	0.01509
		R^2	0.98738	0.96864	0.99256	0.98155	0.98299
		RSS	0.05146	0.09526	0.01402	0.02895	0.02057
		χ^2	0.00139	0.0381	0.00073	0.00193	0.00206
Singh et al. (2014)	$MR = \exp(-kt) - akt$	M_0	1.04815	1.08096	0.97055	0.9685	1.00868
		a	-0.00331	-0.0062	-0.0078	-0.01068	-0.01361
		b	2.4×10^{-6}	8.7×10^{-6}	0.000015	0.000028	0.000045
		R^2	0.99221	0.98469	0.99574	0.97603	0.99544
Singh et al. (2014)	$MR = \exp(-kt) - akt$	RSS	0.03179	0.04651	0.00804	0.03763	0.00552
		χ^2	0.00085	0.00186	0.00042	0.00251	0.00055
		k	0.00266	0.00565	0.01061	0.01597	0.01607
		a	0.18616	0.08062	0.0229	0.01209	0.03904
Silva et al. (2012)	$MR = \exp(-at + bt^{1/2})$	R^2	0.98373	0.95884	0.99062	0.97751	0.98072
		RSS	0.06815	0.13005	0.0186	0.03765	0.02565
		χ^2	0.00179	0.005	0.00093	0.00235	0.00233
		a	0.00679	0.01283	0.01586	0.02618	0.02895
Silva et al. (2012)	$MR = \exp(-at + bt^{1/2})$	b	0.03982	0.0646	-0.03865	0.07318	0.0759
		R^2	0.97097	0.97144	0.9897	0.99102	0.98257
		RSS	0.12161	0.09023	0.02044	0.01503	0.02319
		χ^2	0.0032	0.00347	0.00102	0.00093	0.00211

(c)

Model	Model equation	Parameters	40 °C	50 °C	60 °C	70 °C	80 °C
Newton	$MR = \exp(-kt)$	k	0.00399	0.00945	0.01024	0.01069	0.01315
		R^2	0.9255	0.9499	0.9527	0.9486	0.9389
		RSS	0.39015	0.16696	0.13501	0.13042	0.1229
		χ^2	0.00867	0.00522	0.00519	0.00593	0.00768
		k	7.83×10^{-5}	3.57×10^{-4}	5.46×10^{-4}	6.41×10^{-4}	7.01×10^{-4}
Page	$MR = \exp(-kt^n)$	n	1.69	1.67	1.61	1.59	1.65
		R^2	0.9930	0.9960	0.9960	0.9932	0.9895
		RSS	0.03587	0.01293	0.01105	0.01645	0.01972
		χ^2	8.15×10^{-4}	4.13×10^{-4}	4.43×10^{-4}	7.83×10^{-4}	0.00131
		a	1.15	1.14	1.12	1.11	1.11
Henderson and Pabis	$MR = a \exp(-kt)$	k	0.00454	0.01051	0.01129	0.01172	0.01438
		R^2	0.9457	0.9621	0.9632	0.9580	0.9470
		RSS	0.27798	0.12241	0.10079	0.10179	0.09979
		χ^2	0.00632	0.00395	0.00403	0.00485	0.00665
		a	1.41	1.17	1.17	1.20	1.26
Logarithmic	$MR = a \exp(-kt) + c$	c	-0.33039	-0.0587	-0.08302	-0.12709	-0.19221
		k	0.00255	0.00906	0.00921	0.00878	0.00971
		R^2	0.9816	0.9708	0.9756	0.9765	0.9728
		RSS	0.09194	0.09108	0.06435	0.05422	0.04787
		χ^2	0.00214	0.00304	0.00268	0.9765	0.00342
Wang and Singh	$MR = M_0 + at + bt^2$	M_0	1.0732	1.0002	1.0231	1.0386	1.0517
		a	-0.00325	-0.00617	-0.00723	-0.008	-0.01023
		b	2.39×10^{-6}	8.93×10^{-6}	1.23×10^{-5}	1.50×10^{-5}	2.44×10^{-5}
		R^2	0.9901	0.9735	0.9880	0.9929	0.9890
		RSS	0.04979	0.08276	0.3148	0.01641	0.01941
Singh et al. (2014)	$MR = \exp(-kt) - akt$	χ^2	0.00116	0.00276	0.00131	8.20×10^{-4}	0.00139
		k	0.00263	0.00864	0.009	0.00888	0.01015
		a	0.15061	0.01649	0.0259	0.04283	0.0693
		R^2	0.9745	0.9508	0.9663	0.9695	0.9674
		RSS	0.13045	0.15862	0.09255	0.07401	0.06151
Silva et al. (2012)	$MR = \exp(-at + bt^{1/2})$	χ^2	0.00296	0.00512	0.0037	0.00352	0.0041
		a	0.00664	0.01572	0.01664	0.01711	0.02167
		b	-0.04344	-0.06873	-0.06737	-0.06605	-0.07861
		R^2	0.9693	0.9822	0.9829	0.9782	0.9718
		RSS	0.165716	0.05744	0.04694	0.05289	0.0532
		χ^2	0.00357	0.00185	0.00188	0.00252	0.00355

(d)

Model	Model equation	Parameters	40 °C	50 °C	60 °C	70 °C	80 °C
Newton	$MR = \exp(-kt)$	k	0.00429	0.00823	0.01008	0.01408	0.01981
		R^2	0.92484	0.93628	0.95553	0.97222	0.97608
		RSS	0.31078	0.193	0.09767	0.04872	0.03127
		χ^2	0.0084	0.00742	0.00488	0.00304	0.00284
Page	$MR = \exp(-kt^n)$	k	0.00012	0.00036	0.00133	0.00289	0.00482
		n	1.62726	1.62437	1.42071	1.35335	1.34204
		R^2	0.98769	0.98754	0.98584	0.9939	0.99495
		RSS	0.04951	0.03629	0.02955	0.01002	0.006
Henderson and Pabis	$MR = a \exp(-kt)$	χ^2	0.00138	0.00145	0.00156	0.00066	0.0006
		a	1.12882	1.11986	1.07777	1.06584	1.0581
		k	0.00482	0.00908	0.01078	0.01491	0.02083
		R^2	0.94075	0.947	0.95983	0.97518	0.97792
Logarithmic	$MR = a \exp(-kt) + c$	RSS	0.23837	0.15436	0.08382	0.04081	0.02625
		χ^2	0.00662	0.00617	0.00441	0.00272	0.00262
		a	1.64546	1.25065	1.23602	1.15429	1.15392
		c	-0.60587	-0.18287	-0.21127	-0.12486	-0.12642
Wang and Singh	$MR = M_0 + at + bt^2$	k	0.00204	0.00619	0.00698	0.01114	0.01561
		R^2	0.98741	0.9725	0.98522	0.99075	0.99225
		RSS	0.04925	0.07688	0.02921	0.0142	0.00829
		χ^2	0.00141	0.0032	0.00162	0.00101	0.00092
Singh et al. (2014)	$MR = \exp(-kt) - akt$	M_0	1.04067	1.05056	1.0097	0.99583	0.99833
		a	-0.00322	-0.00639	-0.0073	-0.00984	-0.01394
		b	2.26×10^{-6}	9.53×10^{-6}	0.000013	0.000024	0.000048
		R^2	0.99062	0.98864	0.99414	0.99823	0.9974
Singh et al. (2012)	$MR = \exp(-at + bt^{1/2})$	RSS	0.03669	0.03177	0.01158	0.00272	0.00278
		χ^2	0.00105	0.00132	0.00064	0.00019	0.0003
		k	0.00243	0.0064	0.00779	0.01188	0.01668
		a	0.25924	0.06651	0.06978	0.03854	0.03943
Silva et al. (2012)	$MR = \exp(-at + bt^{1/2})$	R^2	0.98498	0.96624	0.98342	0.98865	0.99069
		RSS	0.06041	0.09831	0.0346	0.01866	0.01107
		χ^2	0.00168	0.00393	0.00182	0.00124	0.00111
		a	0.00688	0.01308	0.01443	0.01999	0.02899
Silva et al. (2012)	$MR = \exp(-at + bt^{1/2})$	b	0.0401	0.05679	0.04604	0.05286	0.0687
		R^2	0.96285	0.9674	0.9729	0.98654	0.99065
		RSS	0.14944	0.09495	0.05655	0.02212	0.01111
		χ^2	0.00415	0.0038	0.00298	0.00147	0.00111

5.2.3 Moisture Diffusivity and Activation Energy

For various drying temperature cases, the moisture diffusivity of the tray dried vegetables have been evaluated using linear fitness plots of $\ln MR$ vs t (**Fig 5.2 (a) – (d)**). The obtained diffusivity values and literature data have been summarized in Table 5.2 (a) – (d) and affirm enhanced moisture diffusivity at higher temperature. For a variation in drying temperature from 40 – 80 °C, the moisture diffusivity of Kolphul, Kaskal, Komora and Posola in the tray drying system enhanced from $9.64 \times 10^{-12} - 5 \times 10^{-11} \text{ m}^2/\text{s}$, $1.56 \times 10^{-11} - 7.59 \times 10^{-11} \text{ m}^2/\text{s}$, $1.78 \times 10^{-11} - 6.02 \times 10^{-11} \text{ m}^2/\text{s}$ and $1.79 \times 10^{-12} - 7.35 \times 10^{-12} \text{ m}^2/\text{s}$, respectively. Thus, with higher moisture diffusivity, greater moisture removal rate can be achieved at higher drying temperature. Similar trends have also been reported for various food materials (Limpaiboon 2011; John et al. 2015; Doymaz 2010). The obtained data are in agreement with those being reported for various food materials (Zogzas et al. 1996).

Thereafter, Arrhenius plots have been prepared for the moisture diffusivity data (**Fig. 5.3 (c) – (d)**). Using the slope of the linear fitness plots of Arrhenius diagrams, the activation energies of Kolphul, Kaskal, Komora and Posola have been determined as 37.38, 34.10, 26.56 and 30 kJ/mol, respectively. These are reasonable and moderately higher in comparison with the general range of activation energy values being reported for various food materials (10 – 110 kJ/mol) (Mwithiga and Olwal 2005). The obtained activation energies have also been comparable with those reported for hot air dried pumpkin (Limpaiboon 2011), tray dried buttermilk soaked chopped banana blossom (John et al. 2015) and hot air dried ripe banana (Doymaz 2010).

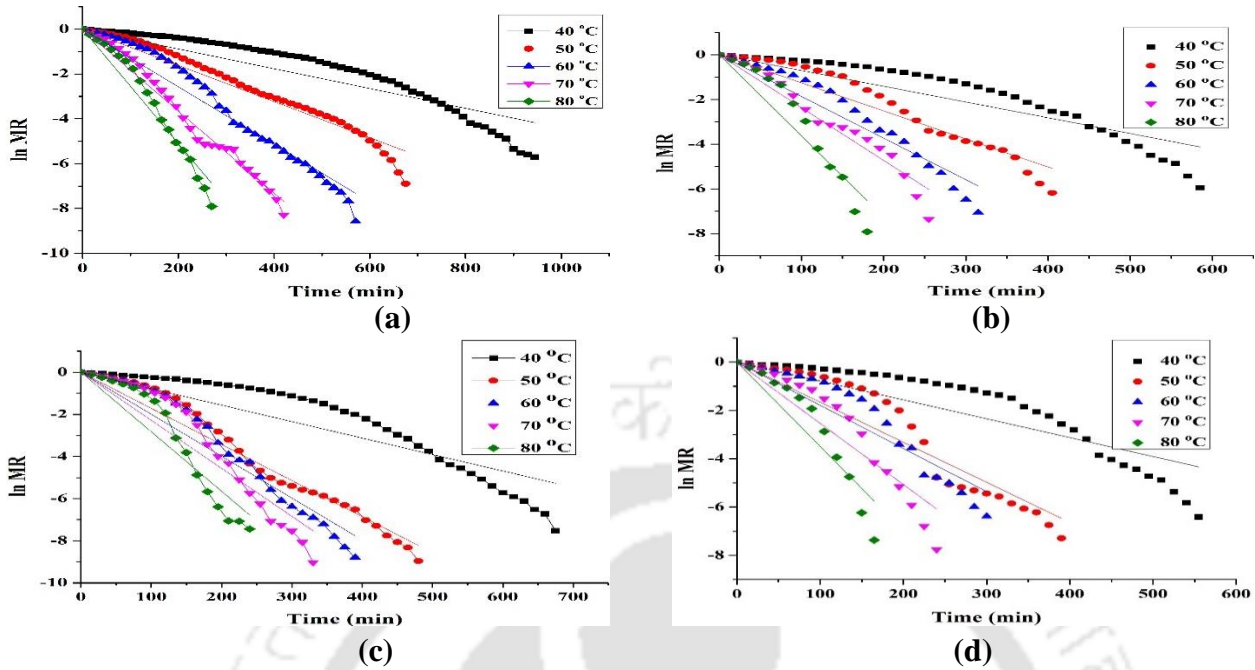


Fig. 5.2: ln MR versus drying plots of tray dried (a) Kolphul (b) Kaskal (c) Komora and (d) Posola vegetable systems.

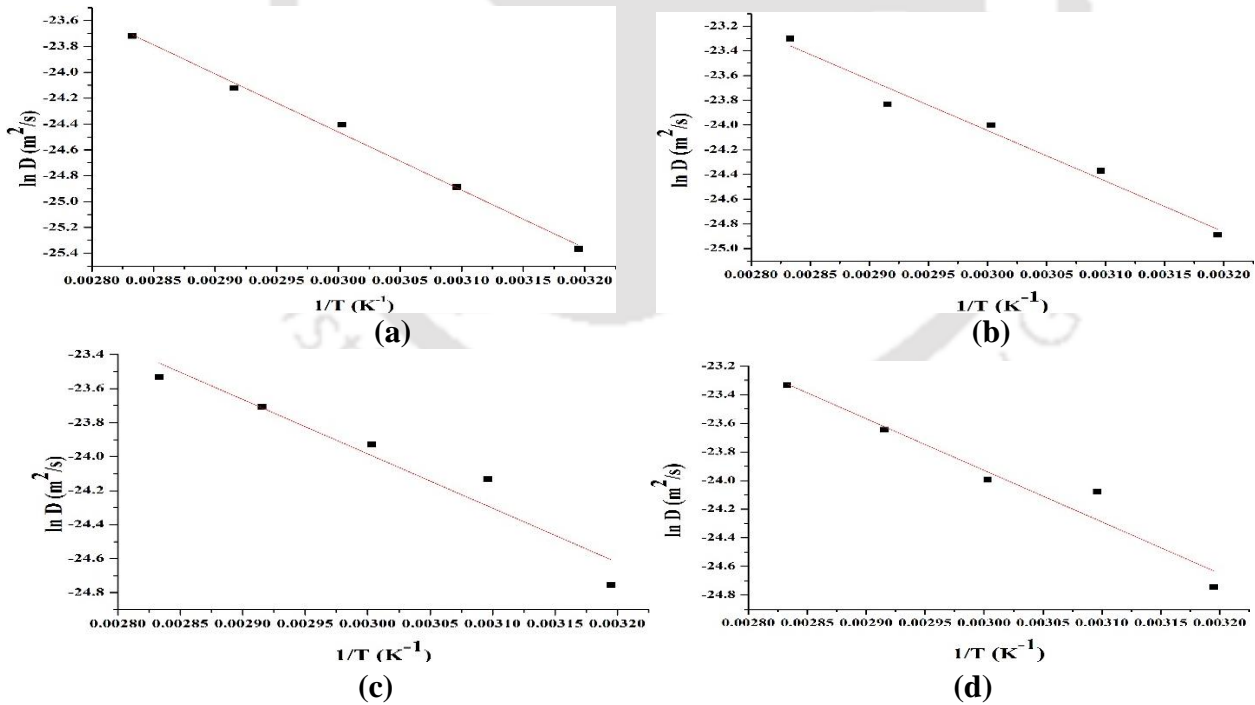


Fig. 5.3: Arrhenius plots for tray dried (a) Kolphul (b) Kaskal (c) Komora and (d) Posola vegetable systems.

Table 5.2: Moisture diffusivity and activation energy data summary of tray dried non-leafy vegetable systems.

Sample	Drying method	Temperature (°C)	Diffusivity (m ² /s)	Activation energy (kJ/mol)	Reference
Kolphul	Intermittent airflow assisted tray drying	40	9.635×10^{-12}	37.38	This work
		50	1.555×10^{-11}		do
		60	2.519×10^{-11}		do
		70	3.347×10^{-11}		do
Buttermilk soaked chopped banana blossom	Tray drying	40 -60	$5.45 - 8.09 \times 10^{-9}$	50.06	John et al. (2014)
		80	5.004×10^{-11}	do	
Kaskal	Intermittent airflow assisted tray drying	40	1.56×10^{-11}	34.10	This work
		50	2.60×10^{-11}		do
		60	3.77×10^{-11}		do
		70	4.46×10^{-11}		do
Ripe banana	Hot air drying	50 -80	$7.374 \times 10^{-11} - 2.148 \times 10^{-10}$	32.65	Doymaz (2010)
		80	7.59×10^{-11}	do	
Komora	Intermittent airflow assisted tray drying	40	1.78×10^{-11}	26.56	This work
		50	3.31×10^{-11}		do
		60	4.06×10^{-11}		do
		70	5.07×10^{-11}		do
Pumpkin	Hot air drying	55 -65	$1.36 - 5.30 \times 10^{-10}$	27.84-37.84	Limpaiboon (2011)
		80	6.02×10^{-11}	do	
Posola	Intermittent airflow assisted tray drying	40	1.79×10^{-11}	30	This work
		50	3.50×10^{-11}		do
		60	3.80×10^{-11}		do
		70	5.38×10^{-11}		do
Ripe banana	Hot air drying	50 -80	$7.374 \times 10^{-11} - 2.148 \times 10^{-10}$	32.65	Doymaz (2010)
		80	7.35×10^{-11}	do	

5.2.4 Vitamin C and Antioxidant Activity Characteristics

For all chosen tray dried non-leafy vegetables, mixed trends were apparent for vitamin C and antioxidant activity i.e., the vitamin C and antioxidant activity values increased up to a moderate combination of drying time and temperature and thereafter reduced with further increase in both drying time and temperature. For all vegetables, appropriate graphical representations of vitamin C and antioxidant activity characteristics have been depicted in **Fig. 5.4 (a) – (d)** and **Fig. 5.5 (a)**

– (d), respectively. For Kolphul and at 50 °C drying temperature, the vitamin C and antioxidant activity increased from 4.53 – 82.23 and 2.27 – 84.79% for drying time variation from 0 – 720 min. This is due to accelerated moisture removal that enhances dry matter content in the sample. At drying temperatures of 60 and 70°C, the vitamin C and antioxidant activity enhanced from 4.53 – 85.24 and 4.53 – 82.26 mg/100g for a corresponding variation in drying time from 0 – 600 min and 0 – 360 min, respectively. Similarly, corresponding variations in antioxidant activity were from 2.27 – 86.82% and 2.27 – 85.42%. On the other hand, the variable values followed an opposite trend from 600 – 720 and 360 – 720 min of drying time to reach the lower value of 81.46 and 60.25 mg/100g, respectively for 60 and 70 °C. Similarly, the antioxidant activity variable reduced from 86.82 – 78.28% and 85.42 – 65.36%, respectively.

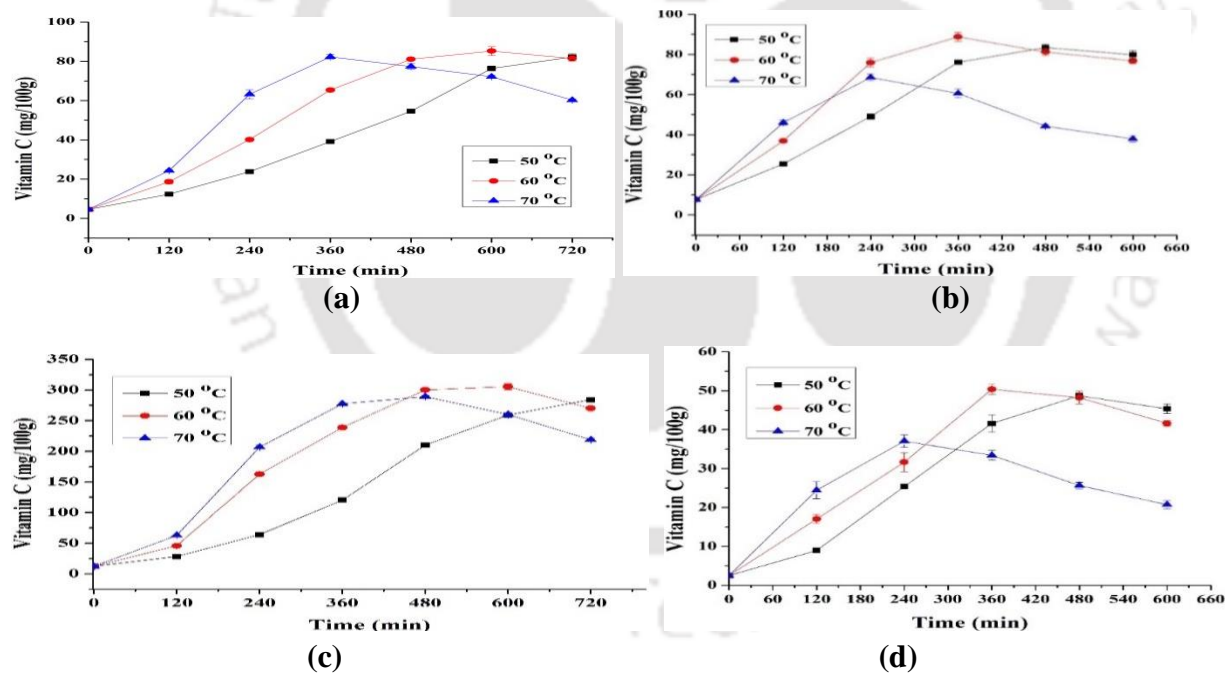


Fig. 5.4: Vitamin C characteristic plots of (a) Kolphul (b) Kaskal (c) Komora and (d) Posola vegetable systems.

At 50 °C drying temperature and for Kaskal, the vitamin C varied as 7.66 – 83.42 – 79.84 mg/100g for 0 – 480 – 600 min drying time variation. Similarly, for antioxidant activity, the

corresponding values, respectively varied of 2.18 – 68.24 – 62.79%. On the other hand, at 60 °C, and for drying time variation as 0 – 360 – 600 min, the vitamin C varied as 7.66 – 88.79 – 76.82 mg/100g. Corresponding trends for antioxidant activity refers to 2.18 – 71.85 – 59.3%. Similarly, at the higher drying temperature of 70 °C, the corresponding trends of vitamin C values varied as 7.66 – 68.57 – 37.94 mg/100g for a drying time variation of 0 – 240 – 600 min. Corresponding antioxidant activity varied as 2.18 – 58.02 – 35.18%.

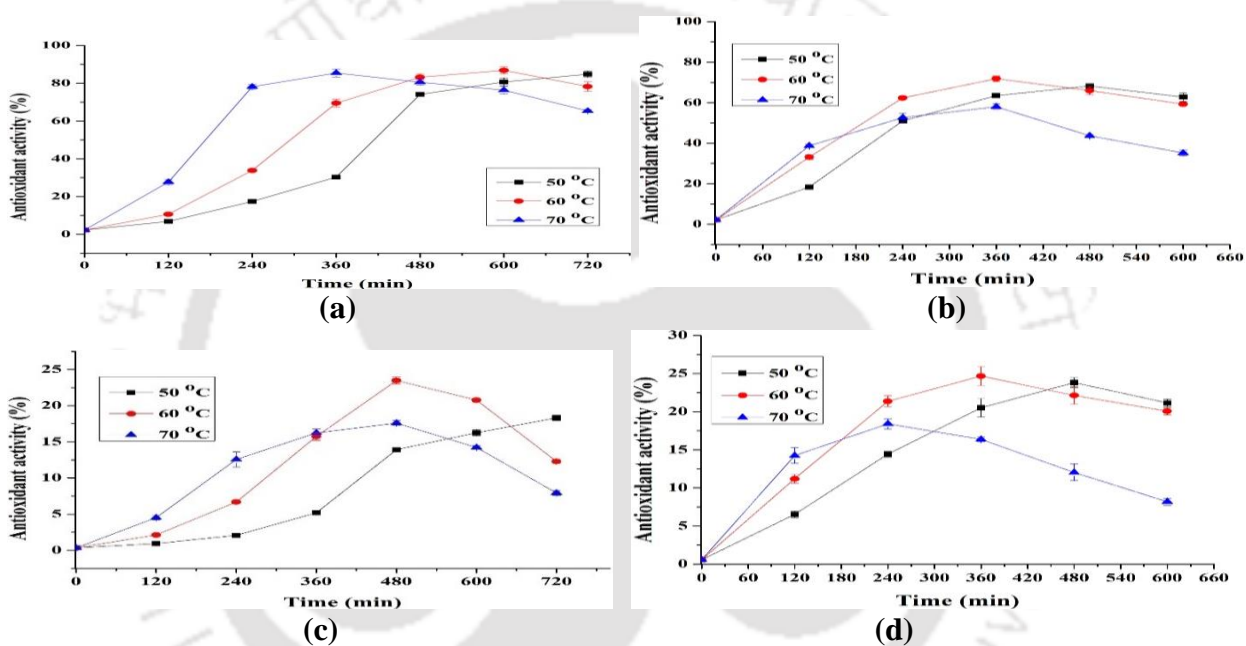


Fig. 5.5: Antioxidant activity characteristic plots of (a) Kolphul (b) Kaskal (c) Komora and (d) Posola vegetable systems.

For Komora and at 50 °C, the vitamin C and antioxidant activity enhanced from 12.18 – 284.32 mg/100g and 0.34 – 18.31%, respectively for a drying time variation up to 720 min. However, at higher drying temperatures, a mixed trend (increasing followed with decreasing trend) was apparent at higher drying temperature. At 60 °C, the vitamin C and antioxidant activity varied from 12.18 – 305.41 – 270.15 mg/100g and 0.34 – 23.48– 12.29%, respectively for a variation in drying time from 0 – 600 – 720 min. Similarly, at 70 °C, the parameter values

varied from 12.18 – 289.14 – 218.76 mg/100g and 0.34 – 17.6 – 7.93%, respectively for a variation in drying time as 0 – 480 – 720 min.

At 50 °C drying temperature and for Posola, the vitamin C and antioxidant activity varied as 2.47 – 48.72 – 45.36 mg/100g and 0.58 – 23.81 – 21.14% for a drying time variation as 0 – 480 – 600 min. On the other hand, at 60 °C, for a drying time variation as 0 – 360 – 600 min, the vitamin C and antioxidant activity varied as 2.47 – 50.38 – 41.61 mg/100g and 0.58 – 24.67 – 20.06%, respectively. Similarly, at the higher drying temperature of 70 °C, the corresponding trends were 2.47 – 37.06 – 20.7 mg/100g and 0.58 – 18.4 – 8.17% for a drying time variation as 0 – 240 – 600 min for vitamin C and antioxidant activity, respectively.

For the higher drying temperature case, the vitamin C value reduction after reaching a maximum value is due to the prolonged drying time that resulted in the deterioration of vitamin C content. Vitamin C constituents in the vegetable samples are well known to be heat sensitive. Hence, with prolonged exposure to heat, they do undergo degradation (Santos and Silva 2008). This is not the case for shorter drying time durations. For the case, the detrimental effect of heat is not predominant in comparison with the enhanced dry matter content of the sample achieved due to effective removal of the moisture. Also, the reduction in vitamin C is very likely due to the oxidation of active compounds to dehydro-ascorbic acid and subsequent hydrolysis to 2, 3-diketogolonic acid and further oxidation and polymerization of these components to generate various inactive compounds (Thankitsunthorn et al. 2009).

At higher drying temperature, the antioxidant activity trend followed a reduced profile than those being depicted at a lower temperature. This is due to the heat sensitivity of antioxidant constituents for prolong exposure to heat. At lower temperature, long drying time was required to

overcome the ever increasing reduction in the variable values. The observed mixed response variations for both measured characteristics has been due to higher dry matter content and expression of inactive constituents for increasing trend (up to moderate combinations of drying time and temperature) and loss of heat sensitive compounds that affirm subsequent reducing trends (for higher drying time and temperature ranges).

5.2.5 Trial and Error Approach based Process Parametric Optimization

Thus, based on the trial and error facilitated drying temperature and time variation, the optimal set of process variables refers to 60 °C drying temperature, 600 min drying time, 85.24 mg/100g vitamin C and 86.82% antioxidant activity for Kolphul, 60 °C drying temperature, 360 min drying time, 6.14% moisture content, 88.79 mg/100g vitamin C content and 71.85% antioxidant activity for Kaskal. Corresponding optimal values for Komora and Posola refer to 60 °C, 480 min, 10.13%, 300.18 mg/100g and 23.48% and 60 °C, 360 min, 3.82%, 50.38 mg/100g and 24.67%, respectively. These values are comparable to those obtained for tray dried Kolphul, Kaskal, Komora and Posola at 60 °C and 600 min of drying time (Chapter 3).

5.3 Statistical Design based Characterization of Process Parameters

Based on the CCD based RSM, **Table 5.3** summarizes experimental findings associated to variant combinations of process parameters and evaluated response variables. In the table, each data set corresponds to average value of triplicate runs and its standard deviation. With low standard deviation values, consistency of the measured data assures confidence to proceed further towards the ANOVA.

Table 5.3: CCD (RSM) based experimental data of tray dried non-leafy vegetables.

Sample	Run	Temp (°C)	Kolphul			
			Time (Min)	Moisture (%)	Vit C (mg/100g)	Antioxidant Activity (%)
Kolphul	1	45.86	555	36.36 ± 1.07	43.52 ± 0.82	56.47 ± 0.68
	2	60	555	8.22 ± 0.80	86.24 ± 1.59	87.47 ± 1.56
	3	60	555	7.68 ± 0.80	87.61 ± 1.59	86.24 ± 1.56
	4	60	555	8.62 ± 0.80	86.26 ± 1.59	88.12 ± 1.56
	5	60	788.35	5.11 ± 0.37	78.92 ± 0.96	76.55 ± 3.11
	6	70	390	5.95 ± 0.24	76.34 ± 0.82	80.67 ± 1.84
	7	60	555	7.11 ± 0.80	84.41 ± 1.59	88.38 ± 1.56
	8	50	390	46.71 ± 2.15	32.54 ± 1.86	43.49 ± 0.63
	9	60	321.65	25.47 ± 0.71	58.7 ± 1.33	60.02 ± 2.04
	10	60	555	6.54 ± 0.80	88.74 ± 1.59	90.45 ± 1.56
	11	60	555	8.36 ± 0.80	85.08 ± 1.59	89.92 ± 1.56
	12	70	720	4.48 ± 0.08	59.44 ± 2.18	64.77 ± 2.38
	13	74.14	555	3.27 ± 0.14	67.61 ± 1.68	72.51 ± 0.74
	14	50	720	13.49 ± 0.40	81.07 ± 2.46	85.52 ± 1.81
Kaskal	1	60	196.51	16.07 ± 1.08	68.42 ± 1.38	58.56 ± 3.14
	2	70	240	4.38 ± 0.27	70.24 ± 2.15	55.83 ± 0.72
	3	60	345	5.71 ± 0.37	89.97 ± 1.40	71.98 ± 0.56
	4	60	345	5.92 ± 0.37	90.07 ± 1.40	72.24 ± 0.56
	5	70	450	3.52 ± 0.22	48.34 ± 1.59	46.09 ± 1.20
	6	74.14	345	2.17 ± 0.41	45.25 ± 2.23	42.73 ± 1.58
	7	60	345	6.48 ± 0.37	89.12 ± 1.40	72.37 ± 0.56
	8	50	240	27.36 ± 1.47	52.39 ± 0.84	50.74 ± 2.21
	9	60	345	6.6 ± 0.37	88.18 ± 1.40	71.61 ± 0.56
	10	60	345	6.08 ± 0.37	86.94 ± 1.40	73.29 ± 0.56
	11	60	345	6.52 ± 0.37	90.73 ± 1.40	72.42 ± 0.56
	12	50	450	8.71 ± 1.14	86.15 ± 1.75	65.39 ± 0.94
	13	45.86	345	21.99 ± 2.11	59.7 ± 2.09	52.47 ± 1.26
	14	60	493.49	4.54 ± 0.35	82.61 ± 1.86	64.26 ± 0.68
Komora	1	60	685.92	4.69 ± 0.42	281.68 ± 2.36	14.53 ± 0.37
	2	60	495	10.53 ± 0.61	300.49 ± 2.19	23.16 ± 0.62
	3	60	495	9.23 ± 0.61	302.57 ± 2.19	22.75 ± 0.62
	4	60	304.08	26.34 ± 0.53	198.14 ± 3.04	10.24 ± 0.18
	5	70	360	8.33 ± 0.27	276.35 ± 1.66	15.12 ± 0.26
	6	60	495	8.98 ± 0.61	303.04 ± 2.19	22.68 ± 0.62
	7	50	630	13.77 ± 0.36	266.96 ± 1.29	15.86 ± 1.14
	8	60	495	8.86 ± 0.61	297.82 ± 2.19	23.48 ± 0.62
	9	60	495	9.06 ± 0.61	301.24 ± 2.19	21.84 ± 0.62
	10	70	630	3.85 ± 0.08	250.42 ± 0.96	11.3 ± 0.19
	11	50	360	45.18 ± 1.24	118.65 ± 2.14	4.92 ± 0.08
	12	60	495	9.47 ± 0.61	303.93 ± 2.19	21.65 ± 0.62
	13	45.86	495	36.15 ± 0.71	160.08 ± 1.83	8.33 ± 0.43
	14	74.14	495	4.17 ± 0.15	258.12 ± 2.4	12.42 ± 0.22
Posola	1	60	202.72	17.48 ± 1.45	34.8 ± 2.04	16.49 ± 0.53
	2	70	240	3.55 ± 0.56	36.51 ± 1.22	15.93 ± 0.48
	3	60	330	3.9 ± 0.53	52.07 ± 1.62	24.32 ± 0.91
	4	60	330	4.79 ± 0.53	53.38 ± 1.62	24.91 ± 0.91
	5	70	420	2.3 ± 0.29	26.54 ± 0.51	10.56 ± 1.16
	6	74.14	330	2.03 ± 0.18	22.34 ± 1.32	8.14 ± 0.35
	7	60	330	4.17 ± 0.53	50.62 ± 1.62	25.75 ± 0.91
	8	50	240	30.45 ± 2.16	24.38 ± 0.72	12.07 ± 1.08
	9	60	330	4.28 ± 0.53	53.24 ± 1.62	24.59 ± 0.91
	10	60	330	3.68 ± 0.53	49.36 ± 1.62	26.83 ± 0.91
	11	60	330	5.06 ± 0.53	52.21 ± 1.62	25.33 ± 0.91
	12	50	420	6.68 ± 0.36	51.72 ± 0.84	22.46 ± 1.26
	13	45.86	330	23.12 ± 0.63	32.25 ± 1.44	15.94 ± 0.60
	14	60	457.28	2.42 ± 0.30	44.79 ± 1.13	21.61 ± 0.45

5.3.1 Response Characteristics

An analysis of data presented in **Table 5.3** affirms that both drying time and temperature detrimentally influence moisture content for all vegetable samples.

For Kolphul, as depicted from **Table 5.3**, the drying temperature had a detrimental influence on the moisture content of the sample. At 390 min, the moisture content, vitamin C and antioxidant activity varied from 46.71 – 5.95%, 32.54 – 76.34 mg/100g and 43.49 – 80.67%, respectively for an increase in drying temperature from 50 – 70 °C. This further varied from 13.49 – 4.48%, 81.07 – 59.44 mg/100g and 85.52 – 64.77% at higher drying time of 720 min and similar temperature increment. However, at the central point of drying time, the moisture content reduced from 36.36 – 3.27% for a temperature variation of 45.86 – 74.14 °C. Similarly, the trend was decreasing (25.47 – 5.11%) for a drying time variation from 321.65 – 788.35 min at 60 °C. On the other hand, at the central drying time point (330 min), the vitamin C and antioxidant activity variable increased from 43.52 – 86.39 mg/100g and 56.47 – 88.43% for a variation in temperature from 45.86 – 60 °C and reduced to 67.61 mg/100g and 72.51% at 74.14 °C, respectively. Similarly, at 60 °C and for a variation of drying time from 321.65 – 555 min, the variables correspondingly enhanced from 58.7 – 86.39 mg/ 100g and 60.02 – 88.43% and subsequently reduced to 78.92 mg/100g and 76.55%, respectively for a higher drying time of 788.35 min.

For the Kaskal vegetable and for a drying temperature of 240 and 450 min, a variation in drying temperature from 50 – 70 °C enabled a significant reduction in moisture content from 27.36 – 4.38% and 8.71 – 3.52%, respectively. However, for an enhancement in drying temperature from 45.86 – 74.14 °C, the moisture content reduced from 21.99 – 2.17% for 345 min drying time. On the other hand, the vitamin C content and antioxidant activity followed a

contrary trend with drying time and temperature. For 240 min drying time case, the enhancement in drying temperature from 50 – 70 °C steadily enhanced the response variables from 52.39 – 70.24 mg/100g and 50.74 – 55.83% for vitamin C and antioxidant activity, respectively. However, a contrary trend was apparent for the corresponding drying temperature variation and for a higher drying time of 450 min. Accordingly, the vitamin C content and antioxidant activity reduced from 86.15 – 48.34 mg/100g and 65.39 – 46.09%, respectively. On the other hand, for 345 min drying time, the drying temperature enhancement in the order of 45.86 – 60 – 74.14 °C enabled to achieve mixed trends of vitamin C content and antioxidant activity (59.7 – 89.17 – 45.25 mg/100g and 52.47 – 72.28 – 42.73%, respectively). Similar trend is apparent at 60 °C drying temperature that indicated corresponding enhancement in the response variables from 68.42 – 89.17 mg/100g and 58.56 – 72.28%, respectively for a variation in drying time from 196.51 – 345 min. Following this, the corresponding variable value reduced to 82.61 mg/100g and 54.26%, respectively for a higher drying time of 493.49 min.

For the Komora sample, the moisture content of the sample reduced significantly with increasing drying temperature. For an increase in drying temperature from 50 – 70 °C and 360 min drying time, the moisture content, vitamin C and antioxidant activity reduced from 45.18% - 8.33%, 118.65 – 276.35 mg/100g and 4.92 – 15.12%, respectively. On the other hand, these variables reduced from 13.77 – 3.85% 266.96 – 250.42 mg/100g and 15.86 – 11.3%, respectively for a higher drying time of 630 min. At the central point of drying time, the moisture content varied from 36.15 – 4.17% in the temperature range of 45.86 – 74.14 °C. At 60 °C, the moisture content reduced from 26.34 – 4.69% for an increase in drying time from 304.08 – 685.92 min. this is due to the higher dry matter content of the sample fostered with greater removal of moisture per gram. On the other hand, for vitamin C and antioxidant activity, a mixed trend was

observed at the central point of drying time and temperature enhancement from 45.86 - 60 - 74.14 °C. The corresponding variables varied from 160.08 – 301.52 – 258.12 mg/100g and 8.33 – 22.94 – 12.42%, respectively. However, for the intermediate temperature of 60 °C, the parameter variation was mixed with enhancement from 198.14 – 301.52 mg/100g and 10.24 – 22.94% for an increase in drying time from 304.08 – 495 min and subsequent reduction to 281.68 mg/100g and 14.53%, respectively for an even drying time of 685.92 min.

For the Posola vegetable, the moisture content, vitamin C and antioxidant activity variations were (30.45 – 3.55% and 6.68 – 2.30%), (24.38 – 36.51 mg/100g and 51.72 – 26.54 mg/100g) and (12.07 – 15.93% and 22.46 – 10.56%) for 240 and 420 min drying time, respectively. On the other hand, for 330 min drying time case, the drying temperature variation as 45.86 – 60 – 74.14 °C facilitated the responses to vary as 23.12 – 2.03% (moisture content), 32.25 – 51.78 – 22.34 mg/100g (vitamin C) and 15.94 – 25.29 – 8.03% (antioxidant activity). Similarly, at the drying temperature of 60 °C, the variation in drying time as 202.72 – 330 – 457.28 min enabled vitamin C and antioxidant activity to vary as 38.8 – 51.78 – 44.79 mg/100 g and 16.49 – 25.29 – 21.61%, respectively for the Posola vegetable.

The reduction in moisture content of the sample was due to enhanced moisture removal driven with higher moisture diffusivity at higher temperature and prolonged exposure to heat. Thus, significant moisture content reduction at higher temperature was due to higher moisture diffusivity. This was due to difference between the water vapour pressure in the environment and that of the sample. On the other hand, vitamin C and antioxidant activity trends were contrary with increasing temperature. These are due to higher dry matter per gram sample and dominance of the active components during the increasing trend of the response variable characteristics. However, it is evident that the dry matter enhancement contributed only to a certain extent for

the parameter following which a reduction in the constituents contributed towards a reduced value range for both vitamin C and antioxidant activity responses. However, for the contrary reducing variable characteristics, it is apparent that heat sensitivity of vitamin C and antioxidants lead to their degradation at higher drying temperatures and prolonged drying time values. Such a hypothesis is also supported with the analysis presented in the literature that indicated irreversible conversion of vitamin C constituents to inactive compounds through the respective combinations of oxidation, hydrolysis and polymerization reactions (Thankitsunthorn et al. 2009). On the other hand, the simultaneous effect of higher temperature and prolonged exposure to heat proved to be detrimental to the vitamin C and antioxidant activity. This is due to the loss of constituents contributing towards these parameters in the dried vegetable product.

5.3.2 ANOVA based Identification of Best Fit Model

Table 5.4 – 5.7 (a – b) depict the summary of data associated to RSM based model fitness studies and relevant ANOVA (analysis of variance) analysis of tray drying characteristics of Kolphul, Kaskal, Komora and Posola, respectively. For all cases, all response variables (moisture content, vitamin C and antioxidant activity) could be most appropriately represented using quadratic models. For all responses, all terms such as A , B , AB , A^2 and B^2 have been identified to be significant terms ($p < 0.05$). Quadratic model coefficient data for all responses (defined with eq. 2.15 in chapter 2) has been presented in **Table 5.8**. For the quadratic model with significant terms, higher model F-values (162.32 – 274.87, 166.10 – 1339.49, 188.44 – 1539.66 and 164.05 – 253.60 for Kolphul, Kaskal, Komora and Posola, respectively) have been obtained. Also, for all cases, the p values were lower than 0.0001. The lack of fit values were not significant (F values varied from 2.16 – 2.83, 1.33 – 3.39, 0.42 – 3.35 and 0.76 – 3.06 for Kolphul, Kaskal, Komora and Posola, respectively). The adequate precision values for the responses were greater

than 38.06, 43.95, 50.10 and 26.71 for Kolphul, Kaskal, Komora and Posola, respectively. Due to these values being significantly greater than 4, the best fit model terms indicate their adequacy in terms of desired attributes of good model fitness.



Table 5.4: (a) Model fitness and (b) analysis of variance (ANOVA) data of moisture, vitamin C and antioxidant activity responses for Kolphul vegetable system.

(a)															
Source	Moisture					Vitamin C					Antioxidant activity				
	Sum of Squares	df	Mean Square	F Value	p-value Prob > F	Sum of Squares	df	Mean Square	F Value	p-value Prob > F	Sum of Squares	df	Mean Square	F Value	p-value Prob > F
Mean vs Total	2507.68	1	2507.68			73802.26	1	73802.26			81867.25	1	81867.25		
Linear vs Mean	1669.4	2	834.70	14.96	0.0007	848.73	2	424.37	1.43	0.2813	497.61	2	248.80	1.15	0.3524
2FI vs Linear	252.02	1	252.02	6.97	0.0248	1070.27	1	1070.27	4.86	0.0520	838.97	1	838.97	5.43	0.0420
Quadratic vs 2FI	353.12	2	176.56	162.32	< 0.0001	2170.50	2	1085.25	274.87	< 0.0001	1516.34	2	758.17	217.75	< 0.0001
Cubic vs Quadratic	5.45	2	2.73	5.03	0.0521	18.85	2	9.42	4.44	0.0656	5.84	2	2.92	0.80	0.4939
Residual	3.25	6	0.54			12.74	6	2.12			22.02	6	3.67		
Total	4790.92	14	342.21			77923.34	14	5565.95			84748.02	14	6053.43		

(b)															
Source	Moisture					Vitamin C					Antioxidant activity				
	Sum of Squares	df	Mean Square	F Value	p-value Prob > F	Sum of Squares	df	Mean Square	F Value	p-value Prob > F	Sum of Squares	df	Mean Square	F Value	p-value Prob > F
Model	2274.54	5	454.91	418.21	< 0.0001	4089.50	5	817.90	207.16	< 0.0001	2852.92	5	570.58	163.88	< 0.0001
A-Temp	1165.63	1	1165.63	1071.6	< 0.0001	395.34	1	395.34	100.13	< 0.0001	191.24	1	191.24	54.92	< 0.0001
B-Time	503.77	1	503.77	463.13	< 0.0001	453.39	1	453.39	114.83	< 0.0001	306.37	1	306.37	87.99	< 0.0001
AB	252.02	1	252.02	231.69	< 0.0001	1070.27	1	1070.27	271.08	< 0.0001	838.97	1	838.97	240.96	< 0.0001
A ²	270.85	1	270.85	249.00	< 0.0001	1745.09	1	1745.09	442.00	< 0.0001	962.02	1	962.021	276.30	< 0.0001
B ²	106.28	1	106.28	97.71	< 0.0001	565.38	1	565.38	143.20	< 0.0001	668.74	1	668.74	192.07	< 0.0001
Residual	8.70	8	1.09			31.59	8	3.95			27.85	8	3.48		
Lack of Fit	5.47	3	1.82	2.83	0.1462	18.90	3	6.30	2.48	0.1756	15.74	3	5.25	2.16	0.2106
Pure Error	3.23	5	0.65			12.69	5	2.54			12.12	5	2.42		
Cor Total	2283.24	13				4121.09	13				2880.77	13			

Table 5.5: (a) Model fitness and (b) analysis of variance (ANOVA) data of moisture, vitamin C and antioxidant activity responses for Kaskal vegetable system.

(a)															
Source	Moisture					Vitamin C					Antioxidant activity				
	Sum of Squares	df	Mean Square	F Value	p-value Prob > F	Sum of Squares	df	Mean Square	F Value	p-value Prob > F	Sum of Squares	df	Mean Square	F Value	p-value Prob > F
Mean vs Total	1134.90	1	1134.90			78466.76	1	78466.76			54061.80	1	54061.80		
Linear vs Mean	555.15	2	277.57	18.42	0.0003	331.40	2	165.70	0.52	0.6057	118.92	2	59.46	0.46	0.6444
2FI vs Linear	79.12	1	79.12	9.13	0.0129	774.51	1	774.51	2.87	0.1210	148.72	1	148.72	1.16	0.3066
Quadratic vs 2FI	84.60	2	42.30	166.10	< 0.0001	2678.99	2	1339.49	580.79	< 0.0001	1278.19	2	639.10	1797.47	< 0.0001
Cubic vs Quadratic	1.29	2	0.64	5.13	0.0502	8.45	2	4.22	2.53	0.1593	1.26	2	0.63	2.40	0.1713
Residual	0.75	6	0.13			10.00	6	1.67			1.58	6	0.26		
Total	1855.81	14	132.56			82270.10	14	5876.44			55610.48	14	3972.18		

(b)															
Source	Moisture					Vitamin C					Antioxidant activity				
	Sum of Squares	df	Mean Square	F Value	p-value Prob > F	Sum of Squares	df	Mean Square	F Value	p-value Prob > F	Sum of Squares	df	Mean Square	F Value	p-value Prob > F
Model	718.87	5	143.77	564.53	< 0.0001	3784.89	5	756.98	328.22	< 0.0001	1545.83	5	309.17	869.54	< 0.0001
A-Temp	394.80	1	394.80	1550.18	< 0.0001	203.97	1	203.97	88.44	< 0.0001	97.89	1	97.89	275.32	< 0.0001
B-Time	160.35	1	160.35	629.60	< 0.0001	127.42	1	127.42	55.25	< 0.0001	21.03	1	21.03	59.15	< 0.0001
AB	79.12	1	79.12	310.67	< 0.0001	774.51	1	774.51	335.82	< 0.0001	148.72	1	148.72	418.27	< 0.0001
A ²	61.29	1	61.29	240.64	< 0.0001	2466.39	1	2466.39	1069.41	< 0.0001	1127.65	1	1127.65	3171.54	< 0.0001
B ²	29.34	1	29.34	115.21	< 0.0001	337.00	1	337.00	146.12	< 0.0001	219.53	1	219.53	617.42	< 0.0001
Residual	2.04	8	0.25			18.45	8	2.31			2.84	8	0.36		
Lack of Fit	1.37	3	0.46	3.39	0.1110	8.61	3	2.87	1.46	0.3315	1.26	3	0.42	1.33	0.3619
Pure Error	0.67	5	0.13			9.84	5	1.97			1.58	5	0.32		
Cor Total	720.91	13				3803.34	13				1548.68	13			

Table 5.6: (a) Model fitness and (b) analysis of variance (ANOVA) data of moisture, vitamin C and antioxidant activity responses for Komora vegetable system.

(a)															
Source	Moisture					Vitamin C					Antioxidant activity				
	Sum of Squares	df	Mean Square	F Value	p-value Prob > F	Sum of Squares	df	Mean Square	F Value	p-value Prob > F	Sum of Squares	df	Mean Square	F Value	p-value Prob > F
Mean vs Total	2817.57	1	2817.57			935764.85	1	935764.85			3791.07	1	3791.07		
Linear vs Mean	1610.83	2	805.41	19.53	0.0002	17018.11	2	8509.05	3.34	0.0733	38.05	2	19.03	0.42	0.6695
2FI vs Linear	181.31	1	181.31	6.66	0.0274	7589.89	1	7589.89	3.72	0.0826	54.46	1	54.46	1.21	0.2962
Quadratic vs 2FI	266.67	2	133.34	188.44	< 0.0001	20345.7	2	10172.85	1539.66	< 0.0001	445.98	2	222.99	730.59	< 0.0001
Cubic vs Quadratic	3.77	2	1.89	5.99	0.0371	3.03	2	1.52	0.18	0.8376	0.14	2	0.07	0.18	0.8364
Residual	1.89	6	0.31			49.83	6	8.3			2.3	6	0.38		
Total	4882.04	14	348.72			980771.41	14	70055.1			4332	14	309.43		

(b)															
Source	Moisture					Vitamin C					Antioxidant activity				
	Sum of Squares	df	Mean Square	F Value	p-value Prob > F	Sum of Squares	df	Mean Square	F Value	p-value Prob > F	Sum of Squares	df	Mean Square	F Value	p-value Prob > F
Model	2058.81	5	411.76	581.93	< 0.0001	44953.7	5	8990.74	1360.74	< 0.0001	538.49	5	107.7	352.86	< 0.0001
A-Temp	1057.92	1	1057.92	1495.14	< 0.0001	9786.67	1	9786.67	1481.21	< 0.0001	16.31	1	16.31	53.45	< 0.0001
B-Time	552.91	1	552.91	781.42	< 0.0001	7231.44	1	7231.44	1094.47	< 0.0001	21.74	1	21.74	71.22	< 0.0001
AB	181.31	1	181.31	256.24	< 0.0001	7589.89	1	7589.89	1148.73	< 0.0001	54.46	1	54.46	178.44	< 0.0001
A ²	214.44	1	214.44	303.06	< 0.0001	15160.59	1	15160.59	2294.55	< 0.0001	281.96	1	281.96	923.8	< 0.0001
B ²	69.43	1	69.43	98.12	< 0.0001	6604.13	1	6604.13	999.53	< 0.0001	197.7	1	197.7	647.74	< 0.0001
Residual	5.66	8	0.71			52.86	8	6.61			2.44	8	0.31		
Lack of Fit	3.78	3	1.26	3.35	0.1133	28.81	3	9.6	2	0.2331	0.49	3	0.16	0.42	0.7452
Pure Error	1.88	5	0.38			24.05	5	4.81			1.95	5	0.39		
Cor Total	2064.47	13				45006.56	13				540.93	13			

Table 5.7: (a) Model fitness and (b) analysis of variance (ANOVA) data of moisture, vitamin C and antioxidant activity responses for Posola vegetable system.

(a)																
Source	Moisture					Vitamin C					Antioxidant activity					
	Sum of Squares	df	Mean Square	F Value	p-value Prob > F	Sum of Squares	df	Mean Square	F Value	p-value Prob > F	Sum of Squares	df	Mean Square	F Value	p-value Prob > F	
Mean vs Total	926.82	1	926.82			24713.64	1	24713.64			5399.04	1	5399.04			
Linear vs Mean	734.91	2	367.45	13.89	0.0010	175.03	2	87.52	0.61	0.5628	64.25	2	32.13	0.78	0.4818	
2FI vs Linear	126.79	1	126.79	7.72	0.0195	348.01	1	348.01	2.81	0.1249	62.09	1	62.09	1.59	0.2359	
Quadratic vs 2FI	160.36	2	80.18	164.05	< 0.0001	1218.33	2	609.16	218.65	< 0.0001	384.38	2	192.19	253.60	< 0.0001	Suggested
Cubic vs Quadratic	2.00	2	1.00	3.13	0.1173	10.02	2	5.01	2.45	0.1670	1.73	2	0.87	1.20	0.3638	Aliased
Residual	1.91	6	0.32			12.27	6	2.05			4.33	6	0.72			
Total	1952.79	14	139.48			26477.30	14	1891.24			5915.83	14	422.56			

(b)																
Source	Moisture					Vitamin C					Antioxidant activity					
	Sum of Squares	df	Mean Square	F Value	p-value Prob > F	Sum of Squares	df	Mean Square	F Value	p-value Prob > F	Sum of Squares	df	Mean Square	F Value	p-value Prob > F	
Model	1022.06	5	204.41	418.24	< 0.0001	1741.37	5	348.27	125.01	< 0.0001	510.73	5	102.15	134.78	< 0.0001	significant
A-Temp	466.74	1	466.74	954.99	< 0.0001	91.56	1	91.56	32.86	0.0004	45.46	1	45.46	59.99	< 0.0001	
B-Time	268.17	1	268.17	548.70	< 0.0001	83.47	1	83.47	29.96	0.0006	18.79	1	18.79	24.79	0.0011	
AB	126.79	1	126.79	259.42	< 0.0001	348.01	1	348.01	124.91	< 0.0001	62.09	1	62.09	81.93	< 0.0001	
A ²	118.24	1	118.24	241.93	< 0.0001	1098.86	1	1098.86	394.41	< 0.0001	331.17	1	331.17	436.97	< 0.0001	
B ²	53.39	1	53.39	109.25	< 0.0001	180.83	1	180.83	64.91	< 0.0001	75.23	1	75.23	99.26	< 0.0001	
Residual	3.91	8	0.49			22.29	8	2.79			6.06	8	0.76			
Lack of Fit	2.53	3	0.84	3.06	0.1299	10.13	3	3.38	1.39	0.3481	1.90	3	0.63	0.76	0.5618	not significant
Pure Error	1.38	5	0.28			12.16	5	2.43			4.16	5	0.83			
Cor Total	1025.97	13				1763.66	13				516.79	13				

Table 5.8: Best fit (quadratic model) regression model coefficients representing tray drying characteristics of all chosen non-leafy vegetable systems.

Sample	Response	A_0	M_1	M_2	M_3	M_4	M_5
Kolphul	Moisture	528.01	-11.14	-0.49	0.0048	0.0606	0.00014
	Vitamin C	-963.64	24.65	1	-0.0099	-0.1537	-0.00032
	Antioxidant activity	-772.57	19.06	0.95	-0.0088	-0.1141	-0.00035
Kaskal	Moisture	275.99	-5.62	-0.42	0.0042	0.0288	0.00018
	Vitamin C	-898.82	26	1.26	-0.0133	-0.1828	-0.00061
	Antioxidant activity	-535.95	16.48	0.71	-0.0058	-0.1236	-0.00049
Komora	Moisture	415.75	-9.33	-0.33	0.0035	0.0539	0.000081
	Vitamin C	-2471.98	68.93	2.27	-0.0223	-0.4531	-0.00079
	Antioxidant activity	-301.8	8.49	0.26	-0.0019	-0.0618	-0.00014
Posola	Moisture	375.44	-7.63	-0.66	0.0063	0.04	0.00033
	Vitamin C	-650.61	17.72	1.06	-0.0104	-0.122	-0.00061
	Antioxidant activity	-336.7	9.24	0.54	-0.0044	-0.067	-0.00039

5.3.3 Response Surface Characteristics of the Dried Samples

For all chosen vegetable samples, response surface plots (**Fig. 5.6 – 5.9**) moisture content indicate negative influence of drying temperature and time ($p < 0.0001$). This is due to higher combinations of moisture diffusivity and heat transfer at higher combinations of drying time and temperature. On the other hand, the effect is contrary for vitamin C and antioxidant activity ($p < 0.05$). This is due to the higher dry matter and dominance of active compounds up to moderate operating conditions. However, the pertinent positive effect was up to a certain limit and subsequent reduction. Such reduced later trend has been due to the detrimental effect of higher temperature and drying time.

Further, the quadratic terms of both independent variables confirm a positive influence on moisture content but negative influence for vitamin C and antioxidant activity for all vegetables.

The p value of such positive influence was lower than 0.0001. Similarly, the moisture content

followed a positive trend with respect to interaction terms. However, for vitamin C and antioxidant activity, the influence was negative for all cases ($p < 0.0001$).

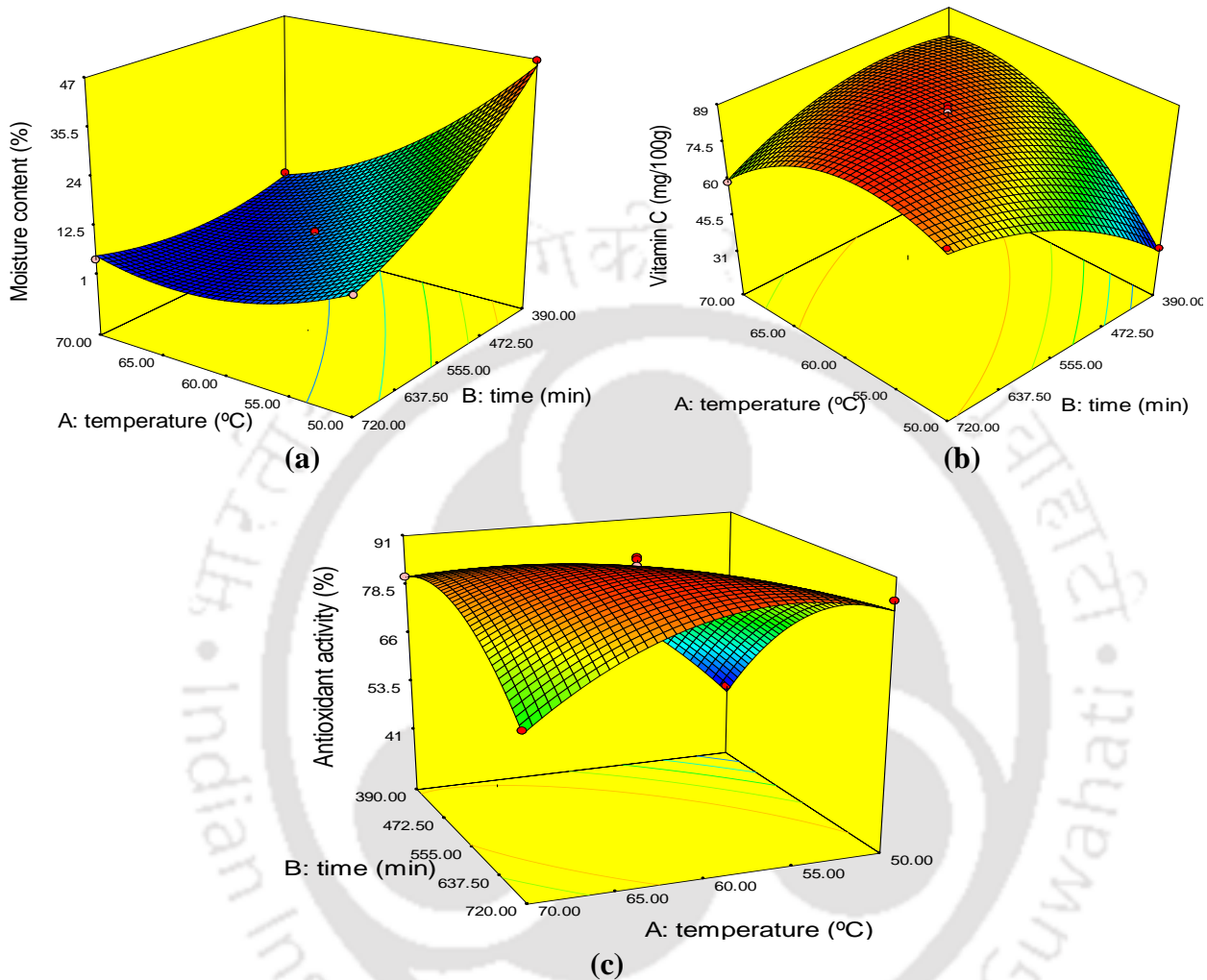


Fig. 5.6: 3-D response surface plots of Kolphul vegetable system responses (a) Moisture (b) Vitamin C and (c) Antioxidant activity.

In summary, increasing trends of vitamin C and antioxidant activity for various cases are attributed to higher drying matter content in the samples and dominance of their active constituents with respect to simultaneous degradation effect. The negative effect of various terms is due to degradation of vitamin C and antioxidants at higher drying time and temperature. Such response characteristics were also reported for systems such as hot air dried mango seed kernels

(Ekorong et al. 2015), torch ginger (Juhari et al. 2012), olive leaves (Erbay and Icier 2009). Also, similar trends do exist for tray dried leafy vegetable samples (Chapter 4).

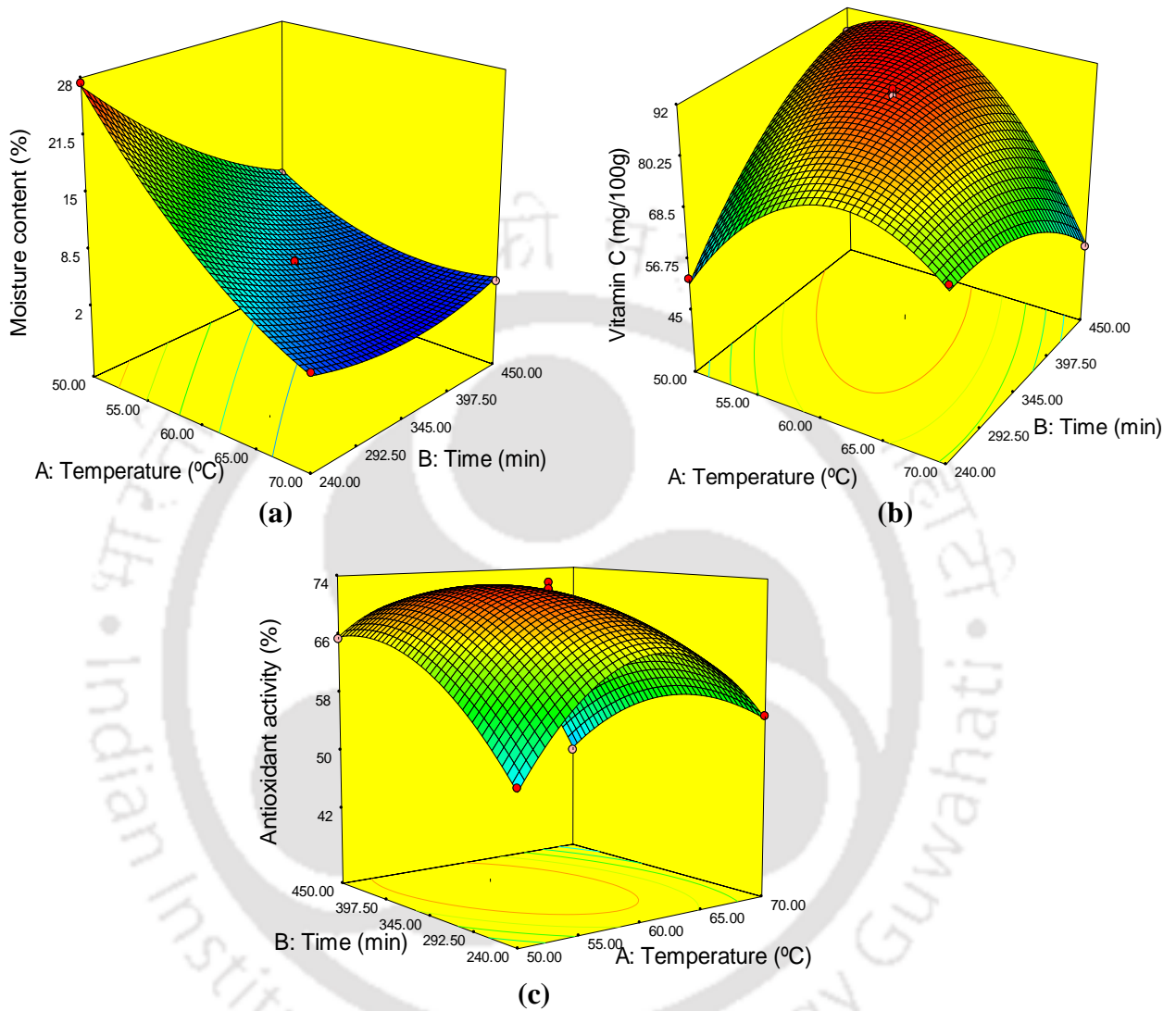


Fig. 5.7: 3-D response surface plots of Kaskal vegetable system responses (a) Moisture (b) Vitamin C and (c) Antioxidant activity.

For Kolphul sample, vitamin C and antioxidant activity increased with temperature and time up to a certain range and thereafter reduced with a further increment in the operating parameter values. The maximum vitamin C content (86.39 mg/100g) and antioxidant activity

(88.43%) was found at 60 °C and 555 min. Minimum value for the responses were obtained as 32.54 mg/100g and 43.49% at 50 °C and 390 min.

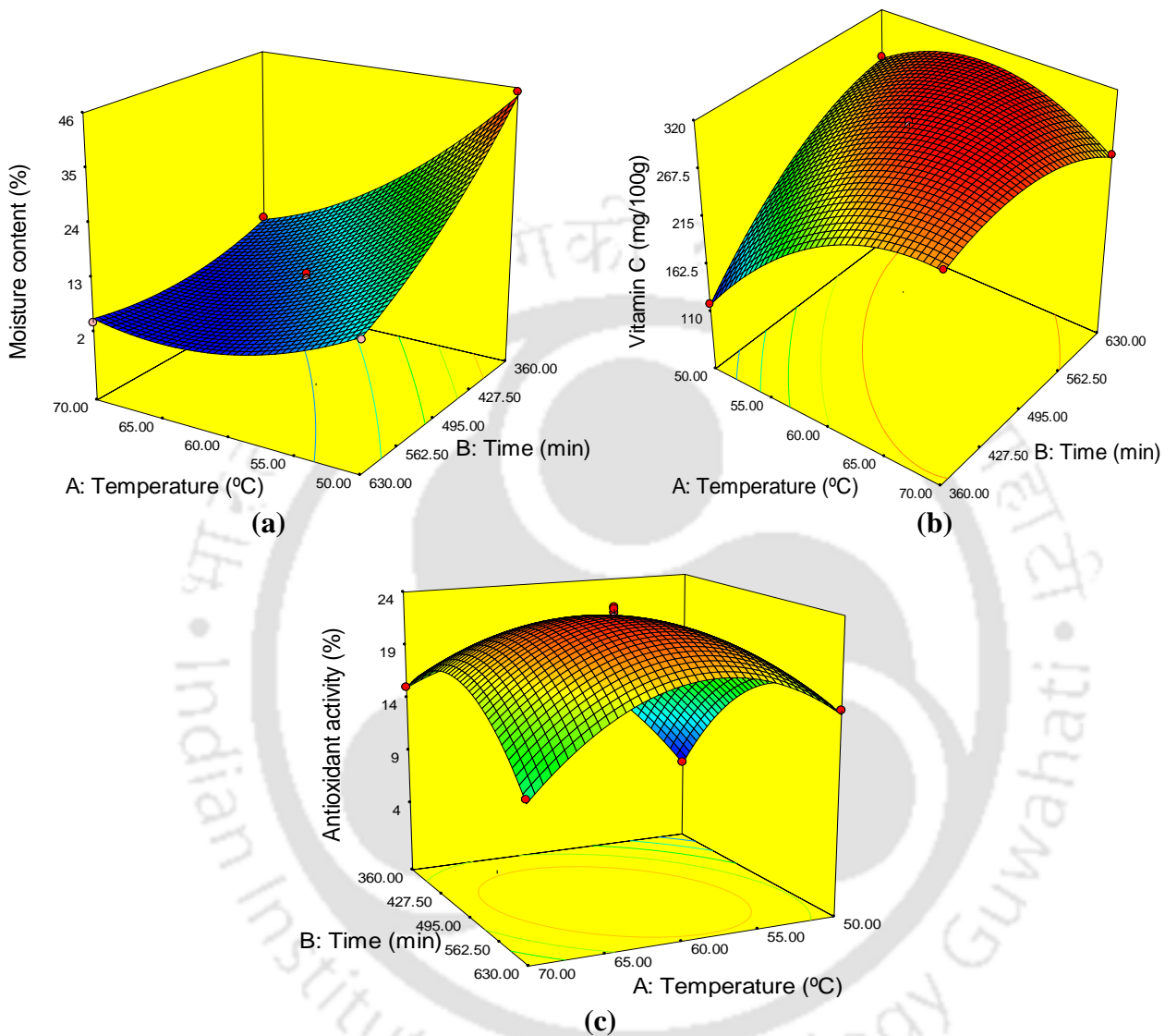


Fig. 5.8: 3-D response surface plots of Komora vegetable system responses (a) Moisture (b) Vitamin C and (c) Antioxidant activity.

For Komora, the maximum values obtained for vitamin C and antioxidant activity were 89.17 mg/100g and 72.28%, respectively. These were obtained at 60 °C drying temperature and 345 min drying time. Similarly, for Komora and Posola, corresponding maximum values for

vitamin C (301.52 and 50.38 mg/100g) and antioxidant activity (22.94 and 24.67%) were achieved at 60 °C drying temperature and 495 and 330 min drying time, respectively.

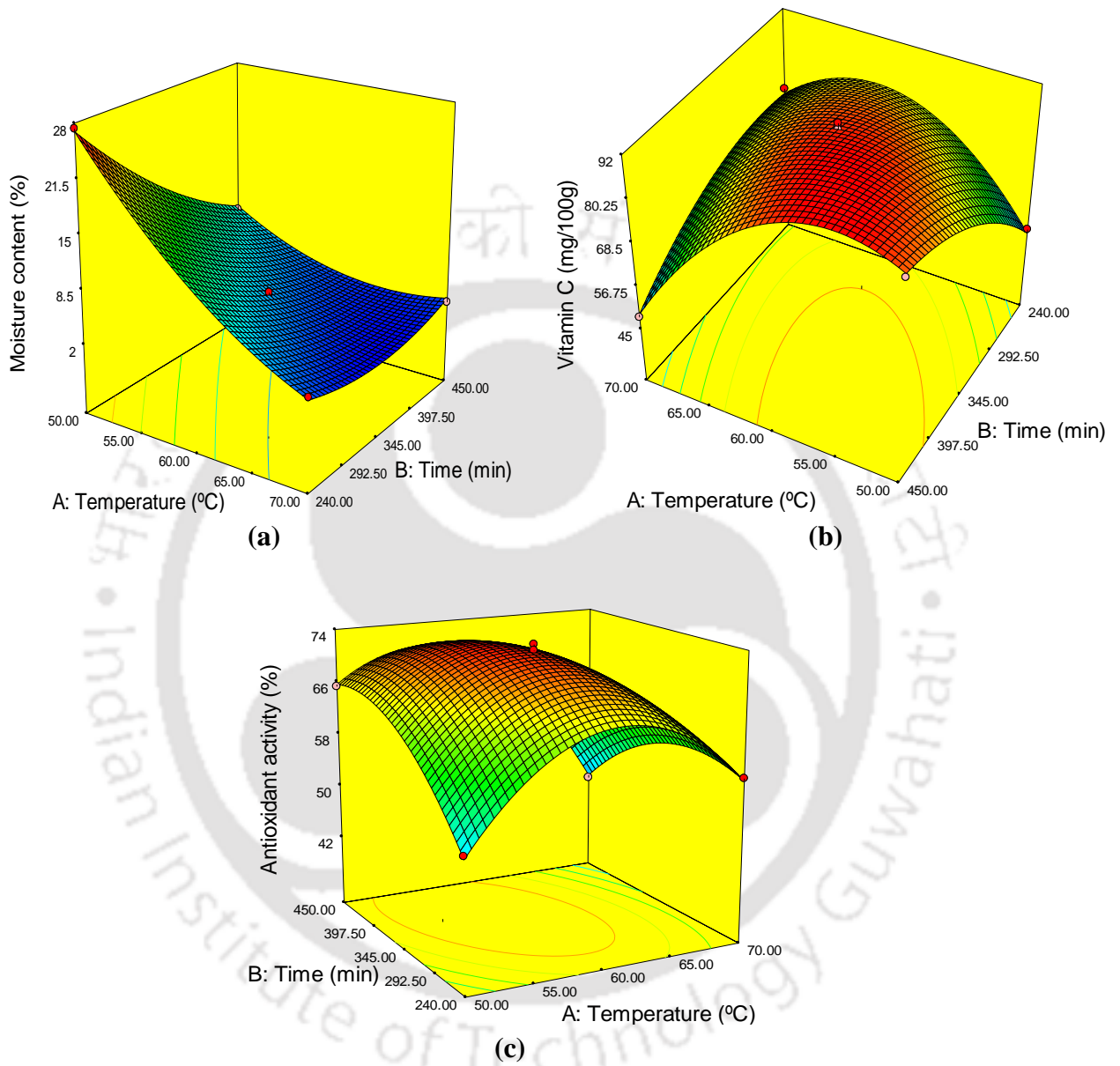


Fig. 5.9: 3-D response surface plots of Posola vegetable system responses (a) Moisture (b) Vitamin C and (c) Antioxidant activity.

5.3.4 Optimization of Process Parameters

Based on diverse mentioned methodologies, optimal values of process parameters as well as responses can be obtained for three alternate scenarios namely trial and error based experimentally optimal data, RSM method based experimentally optimal data and RSM based numerical optimization based data. For these cases, Table 5.9 summarizes alternate sets of optimal process parameters and response values. The same table also mentions the most relevant and best literature data for all cases. A useful analysis of three data sets indicates that the RSM based numerically optimal data sets are the best. This corresponds to marginally higher values for vitamin C and antioxidant activity and marginally lower moisture content with respect to the other cases. Various optimal data sets can be summarized as follows:

- For Kolphul, the trial and error, RSM experimental and RSM optimization based approaches affirmed optimal data sets as 60 °C, 600 min, 6.96% (moisture) 85.24 mg/100g (vitamin C) and 86.82% (antioxidant activity); 60 °C, 555 min, 7.76% (moisture) 86.39 mg/100g (vitamin C) and 88.43% (antioxidant activity) and 61.82 °C, 611.52 min, 3.98% (moisture) 87.69 mg/100g (vitamin C) and 89.04% (antioxidant activity), respectively. Similar results were also obtained for previous data set summarized for the vegetable using tray drying at 60 °C and 600 min (Chapter 3). Among various sets of optimum values, RSM based optimum process parameters were the best. The most relevant literature affirms that for chopped and buttermilk soaked banana blossom samples, 9% moisture exists for processed samples at a 60°C and 195 min of drying in tray dryer (John et al. 2014).
- For Kaskal, corresponding optimal values corresponds to 60 °C drying temperature, 360 min drying time, 6.14% moisture content, 88.79 mg/100g vitamin C content and 71.85%

antioxidant activity; 60 °C drying temperature, 345 min drying time, 6.14% moisture content, 89.17 mg/100g vitamin C content and 72.28% antioxidant activity and 58.66 °C drying temperature, 400.31 min drying time, 5.09% moisture content, 90.73 mg/100g vitamin C content and 72.34% antioxidant activity, respectively.

Table 5.9: Experimental, predicted and literature data of the response characteristics of tray dried non-leafy vegetable systems.

Sample	Approach/optimal parameter	Drying method	Temp (°C)	Time (Min)	Moisture (%)	Vit C (mg/100g)	Antioxidant activity (%)	Reference
Kolphul	RSM optimized	Tray drying	61.82	611.52	3.98	87.69	89.04	This work
	RSM non- optimized	do	60	555	7.76	86.39	88.43	do
	Kinetics best	do	60	600	6.96	85.24	86.82	do
	Experimental at optimized conditions	do	61.82	611.52	4.72 ± 0.12	87.28 ± 2.26	90.16 ± 1.74	do
		do	60	600	7.34	84.51	86.38	Chapter 3
Kaskal	RSM optimized	Tray drying	58.66	400.31	5.09	90.73	72.34	This work
	RSM non- optimized	do	60	345	6.23	89.17	72.32	do
	Kinetics best	do	60	360	6.14	88.79	71.85	do
	Experimental at optimized conditions	do	58.66	400.31	4.68 ± 0.37	91.60 ± 2.18	71.06 ± 1.42	do
		do	60	600	4.37	78.87	60.24	Chapter 3
Komora	RSM optimized	Tray drying	62.77	530.83	5.09	310.40	22.67	This work
	RSM non- optimized	do	60	495	9.36	301.52	22.94	do
	Kinetics best	do	60	480	10.13	300.18	23.48	do
	Experimental at optimized conditions	do	62.77	530.83	6.42 ± 0.24	312.47 ± 1.98	23.08 ± 0.35	do
		do	60	600	7.30	304.37	21.36	Chapter 3
Posola	RSM optimized	Tray drying	57.59	389.42	2.84	53.38	25.72	This work
	RSM non- optimized	do	60	330	4.31	51.78	25.29	do
	Kinetics best	do	60	360	3.82	50.38	24.67	do
	Experimental at optimized conditions	do	57.59	389.42	3.26 ± 0.28	51.34 ± 1.53	26.47 ± 1.15	do
		do	60	600	1.56	42.25	21.68	Chapter 3

- For Komora, the optimal data set refers to 60 °C drying temperature, 480 min drying time, 10.13% moisture, 300.18 mg/100g vitamin C, 23.48% antioxidant activity based on trial and error approach, 60 °C drying temperature, 495 min drying time, 9.36% moisture, 301.52 mg/100g, 22.94% antioxidant activity based on RSM experimental approach and 62.77 °C drying temperature, 530.83 min drying time, 5.09% moisture, 310.40 mg/100g and 22.67% antioxidant activity based on numerical optimization (RSM) approach.

- Similarly, for the Posola, the best data corresponds to 57.59 °C drying temperature, 389.42 min drying time, 2.84% moisture content, 53.38 mg/100g vitamin C content and 25.72% antioxidant activity (RSM numerical optimization approach). Corresponding trial and error and RSM experimental based optimal values refer to 60 °C, 330 min, 3.82%, 50.38 mg/100g and 24.67% and 60 °C, 360 min, 3.82%, 51.78 mg/100g and 25.29%, respectively.

In summary, RSM numerical optimized data sets have been proven to be the best among all sets of optimal data for all cases. Also, predicted response variable values using RSM based numerical optimization are very close to those obtained through additional experimental investigations that were carried out using RSM optimized data set. For such data, the standard deviations were lower than 0.8, 1.72, 1.46 and 0.84 for Kolphul, Kaskal, Komora and Posola, respectively. However, the values are comparatively higher than the tray dried Kolphul, Kaskal, Komora and Posola samples at 60 °C and 600 min (reported in Chapter 3 of the thesis).

5.4 Proximate Characteristics of Optimally Tray Dried Samples

Table 5.10 summarizes data obtained from experimental investigation of samples processed at optimal parametric conditions indicated by RSM numerical optimization approach. For Kolphul, among evaluated parameters, carbohydrate content (50.94%) and soluble protein (8.83%) were the highest and lowest, respectively. Corresponding crude fibre, ash and fat content values were 13.26, 11.82 and 9.04%, respectively.

For Komora, the highest and lowest values were obtained for carbohydrate (62.88%) and fat content (3.21%), respectively. The dried vegetable is also a good source of crude fibre (12.36%). Corresponding soluble protein and ash content were 6.19 and 8.41%, respectively. All

such characteristics are in good agreement with the data summarized for tray dried Komora sample at 60 °C and 600 (Chapter 3).

Table 5.10: Proximate characteristics of optimally tray dried non-leafy vegetable systems.

Sample	Drying methods	Optimal drying conditions	Optimal drying characteristics					Reference
			Carbohydrate (%)	Soluble protein (%)	Fat (%)	Fibre (%)	Ash (%)	
Kolphul	Tray drying	61.82 °C, 611.52 min	50.94	8.83	9.04	13.26	10.82	This work
	do	60 °C, 600 min	50.52	8.26	8.57	12.35	10.84	Chapter 3
Kaskal	Tray drying	58.66 °C, 400.31 min	81.13	3.42	3.12	1.17	4.29	This work
	do	60 °C, 600 min	81.37	2.99	2.93	1.08	4.55	Chapter 3
Komora	Tray drying	62.77 °C, 530.83 min	62.88	6.19	3.21	12.36	8.41	This work
	do	60 °C, 600 min	62.35	5.98	3.03	12.04	8.22	Chapter 3
Posola	Tray drying	57.59 °C, 389.42 min	60.57	4.18	4.79	16.04	12.5	This work
	do	60 °C, 600 min	61.19	3.74	4.93	15.67	12.42	Chapter 3

On the other hand, for Kaskal and Posola, among various proximate parameters, highest value was obtained for carbohydrate (81.13 and 60.57% for Kaskal and Posola, respectively) content of the vegetables. On the other hand, lowest value was obtained with respect to fat content (3.12%) for Kaskal and soluble protein content (4.18%) for Posola, respectively. The soluble protein content for Kaskal was found to be 3.42%. Fat content of Posola was about 4.79%. Also, Posola was found to be rich in crude fibre (16.04%) and ash content (12.5%). On the other hand, for the Kaskal case, the crude fibre and ash contents were 1.17% and 4.29%, respectively. The proximate values for both tray dried vegetable cases are comparable with those reported in Chapter 3 for tray dried Kaskal and Posola at 60 °C drying temperature and 600 min drying time. Due to the proximate parameters being highly insensitive towards heat, no significant difference can be found between the obtained and reported data in Chapter 3.

5.5 Summary

Several useful insights can be gained from the experimental investigations addressed in this work. Firstly, the coherence and symphony of statistical design and trial and error approaches is evident with respect to the identification of best combinations of process parameters and response variables for tray dried Kolphul, Kaskal, Komora and Posola. Among these approaches, while the former approach provided optimal precision of the said parameters and variables, the later approach enabled insights related to drying characteristics, moisture diffusivity and activation energy. Both approaches affirmed that the drying time and temperature have profound influence on the desired combinations of response variable characteristics (associated sensitivities have been analyzed in Appendix F). Moisture diffusivity of tray dried Kolphul, Kaskal, Komora and Posola have been evaluated to be about $9.64 \times 10^{-12} - 5 \times 10^{-11} \text{ m}^2/\text{s}$, $1.56 \times 10^{-11} - 7.59 \times 10^{-11} \text{ m}^2/\text{s}$, $1.78 \times 10^{-11} - 6.02 \times 10^{-11} \text{ m}^2/\text{s}$ and $1.79 \times 10^{-12} - 7.35 \times 10^{-12} \text{ m}^2/\text{s}$, respectively. Corresponding activation energies were also similar (with a variation of $\pm 10.82 \text{ kJ/mol}$) and this indicated that vegetables have compatibility in terms of drying behavior. In other words, Kolphul, Kaskal, Komora and Posola can be deployed together for the development of value added products such as ready to consume soups.

On the other hand, the optimal process parameters and response variables correspond to $61.82 \text{ }^\circ\text{C}$ drying temperature, 611.52 min drying time, 3.98% moisture, 87.69 mg/100g vitamin C and 89.04% antioxidant activity for Kolphul sample; $58.66 \text{ }^\circ\text{C}$ drying temperature, 400.31 min drying time, 5.09% moisture content, 90.73 mg/100g vitamin C content and 72.34% antioxidant content for Kaskal; $62.77 \text{ }^\circ\text{C}$ drying temperature, 530.83 min drying time, 5.09% moisture, 310.40 mg/100g and 22.67% antioxidant activity for Komora and $57.59 \text{ }^\circ\text{C}$ drying temperature, 389.42 min drying time, 2.84% moisture content, 53.38 mg/100g vitamin C content and 25.72%

antioxidant content for Posola. Thus, soup formulations of Kolphul, Kaskal, Komora and Posola can be prepared with dominant constitution of Komora and Kolphul due to enhanced antioxidant and vitamin C characteristics. Thereby, these insights are anticipated to widen the applicability of tray dried Kolphul, Kaskal, Komora and Posola vegetables in terms of translational ready to consume food products such as soups and thereby promote entrepreneurship opportunities for the rural community of developing countries such as India.

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Chapter 6:
**Formulation, Optimization and
Characterization of Leafy Vegetable Soup Mix
Recipe**



Formulation, Optimization and Characterization of Leafy Vegetable Soup Mix Recipe

After providing relevant orientation towards the targeted objectives of this chapter in section 6.1, the following section (section 6.2) targets upon the mathematical optimization based optimal chosen leafy vegetable mix constitution by following either individual and co-drying approaches. Thereafter, section 6.3 elaborates with respect to the findings related to sensory characterization based optimal soup mix formulations. For composition purposes, control formulations have also been considered to judge upon the optimization of evaluated sensory characteristics. All such formulations have been based on varied ratios of dry optimal leafy vegetable mix corn flour. Following this, rheological characteristics of optimal reconstituted soup formulate is addressed in section 6.4. These include steady state, time dependent and dynamic flow behavioural characteristics. Finally, a summary of the findings has been presented in section 6.5.

6.1 Introduction

A critical analysis of available prior-art confirms the following research gaps. Firstly, there are very few articles targeted upon the optimization of leafy vegetable based soup formulations (Alozie and Ene-Obong 2018; Kokani et al. 2019; Niththiya et al. 2014; Sudarsan et al. 2017). In all such endeavours, the vegetable composition is not optimized and is chosen based on a trial and error methodology. Secondly, compared to these research methodologies outlined in the literature, a mature methodology involving mathematical optimization based vegetable mix formulation would be beneficial to retain desired

nutritional characteristics and thereby facilitate investigations associated to the optimization of thickening agent and spice mix. In other words, nutritional characteristics can better evolve sensory characteristics of the soup formulations. In this work, a mature research methodology has been adopted for the chosen leafy vegetables. The specific novelty refers to a two-step methodology that emphasize upon mathematical optimization as a first step to determine nutritionally potent vegetable mixture formulation and subsequent guide the trial and error based sensory evaluations of varied vegetable mix to thickening agent ratio. For such evaluations, fixed choice of water to solids ratio was considered. The mathematical non-linear programming model was solved using MS-Excel solver. The model was developed using RSM based ANOVA analysis of measured drying characteristics and subsequent identification of best fit model to represent the said characteristics for the chosen vegetables. The MS-Excel based mathematical optimization targeted either co-drying or individual drying approaches using an intermittent air flow assisted tray drying technology.

In summary, to ensure better acceptability and feasibility of the product, trial and error based sensory experiments were conducted for variant thickening agent (corn flour) to vegetable mix ratio and for fixed ratio of solids to water ratio (1 g: 20 mL). The sensory analysis was based on a 9-pointer hedonic scale. Further, steady state, time dependent and dynamic rheological studies have been conducted for the optimal soup formulation being prepared with optimal combinations of leafy vegetable mix and thickening agents. For all such investigations, the spice constitution was fixed to specific values based on earlier literature (Srivastava et al. 2019; Chaudhari and Solanke 2016; Sugumar and Guha 2020; Dhiman et al. 2017) and few preliminary trials.

6.2 Evaluation of Optimal Dry Leafy Vegetable Mix Formulation

6.2.1 Individual Drying

The non-linear programming model being summarized in eq. 2.16 – 2.29 (chapter 2) was solved using MS-Excel. Thereby, the optimal dry leafy vegetable mix was obtained as 10:74.95:15.05 for Kolmou, Jatlaho and Pui, respectively. For the case, the highest objective function value of 1250.98 was achieved. **Table 6.1** presents the predicted responses variable characteristics for each leafy vegetable and their optimum mix during individual drying. For the mixture, optimal moisture content, vitamin C and antioxidant were obtained as 3.21%, 110.38 mg/100g and 80%, respectively. The standard deviation between experimental and predicted values was less than 2%. The corresponding optimum values for individual leafy vegetables were found to (1.98%, 56.72 mg/100g, 91.71%), (3.02%, 113.30 mg/100g, 89.50%) and (5%, 131.51 mg/100g, 24.92%) for Kolmou, Jatlaho and Pui, respectively. Corresponding optimal drying temperature and time were (62.39 °C, 231.97 min), (61.65 °C, 295.59 min) and (62.52 °C, 350.89 min), respectively. These data are comparable with the findings being reported in chapter 3 of the thesis. The vitamin C and antioxidant activity values were comparatively similar to the data reported in chapter 3. However, marginal enhancement in these values was obtained for both Jatlaho and Pui. On the other hand, the moisture content of the samples was significantly reduced in due course of optimization. Since each vegetable is likely to have its own characteristics of optimal drying time and temperature, individual vegetable drying and subsequent mixing of dried vegetables together enabled the achievement of optimal nutritional characteristics of the leafy vegetable mix and hence its optimal constitution. Optimal data for Jatlaho are similar to those being achieved with the RSM experimental research data reported in chapter 4 of the thesis. All values were very close to the experimental data that were obtained for same drying conditions (standard deviation varying from 0.38 – 1.74, 1.05 – 3.18 and 0.66 – 2.14, respectively).

Table 6.1: Experimental and predicted optimal parameters-responses data sets for alternate leafy vegetable systems and individual drying case.

		Drying method	Optimized temp (°C)	Optimized time (Min)	Moisture (%)	Vit C (mg/100g)	Antioxidant activity (%)	Reference
Kolmou	Predicted	Tray drying with intermittent airflow	62.39	231.97	1.98	56.72	91.71	This work
	Experimental	do	62.39	231.97	2.35 ± 0.21	57.62 ± 1.18	89.51 ± 1.46	do
	Experimental	do	60	240	4.21	56.96	92.34	Chapter 3
Jatilao	Predicted	Tray drying with intermittent airflow	61.65	295.59	3.02	113.30	89.50	This work
	Experimental	do	61.65	295.59	3.24 ± 0.18	110.07 ± 2.06	88.14 ± 1.63	do
	Experimental	do	60	240	7.68	112.68	89.45	Chapter 3
Pui	Predicted	Tray drying with intermittent airflow	62.52	350.89	5	131.51	24.92	This work
	Experimental	do	62.52	347.49	4.62 ± 0.38	129.43 ± 3.14	25.51 ± 1.35	do
	Experimental	do	60	240	10.44	123.94	23.08	Chapter 3
Mix (10:75.95:1 4.05)	Predicted	-	-	-	3.21	110.38	80	This work
	Experimental	-	-	-	2.94 ± 0.26	112.16 ± 2.35	81.62 ± 2.17	do
	Literature	Oven	-	-	-	-	-	-

Compared to the other two vegetables, the optimal soup mix constitution has higher Jatilao proportion. This is due to its higher vitamin C and antioxidant activity constituents. On the other hand, Kolmou and Pui, respectively contain high antioxidant activity and vitamin C. However, due to significantly lower vitamin C and antioxidant activity for Kolmou and Pui, respectively, lower content has been indicated for these two vegetables in the optimal vegetable mix formulation. Similarly, the literature affirmed that for an optimal vegetable mix formulation being targeted for higher antioxidant activity, the constitution referred to the vegetable possessing higher antioxidant activity value (Larrosa et al. 2015). Therefore, the mixed leafy vegetable soup formulation with higher constitution of Jatilao has been anticipated to provide a nutritionally optimal soup product in terms of enriched vitamin C content and antioxidant activity. Such a product would provide nutritionally enriched constitution for obvious health beneficial effect on the health.

6.2.2 Co-drying

Table 6.2 summarizes predicted values of the responses for the chosen individual leafy vegetables and their optimal mix formulation in due course of co-drying. Compared to the individual drying case, inferior solution has been obtained for the case. The maximum objective function value was 1236.18 which is marginally lower than that obtained for individual drying (1250.98). This is due to better retention of vitamin C and antioxidant activity and better removal of moisture from the samples. It is obvious that for a particular vegetable, a specific temperature-time combination is needed to achieve better retention of nutritional properties. For co-drying, since same drying temperature and time is applicable for all vegetable samples, it is apparent that the Kolmou sample dried relatively for longer drying time (298.28 min). Hence, this affirms towards over drying of the sample and degradation of

nutritional constituents. On the other hand, due to insufficient moisture removal (lower dry matter content), nutritional properties have not been well pronounced in the final product.

The optimum proportion of Kolmou, Jatilao and Pui for the case was 10:80:10 with the proportion of all leafy vegetables reaching either lower or upper bound values. This conveys that a further relaxation in the bounds will further reduce Kolmou and Pui and simultaneously enhances Jatilao contribution in the final mix. Therefore, further improvement in formulation ultimately refers to Jatilao based soup formulation only. To achieve a mixed formulation with the chosen leafy vegetables, the mentioned proportion was considered as an optimum value for the co-drying case. For the optimal soup mix, the predicted moisture content, vitamin C and antioxidant activity were 3.43%, 108.48 mg/100g and 82.52%, respectively. These values are in agreement with experimental data (standard deviations of 1.12%, 0.83% and 2.17%, respectively). On the other hand, optimum response variable values corresponding to individual vegetables (namely Kolmou, Jatilao and Pui) were (0.81%, 52.86 mg/100g and 84.29%), (2.86%, 113.20 mg/100g and 89.58%) and (10.59%, 126.37 mg/100g and 24.33%), respectively. The optimum drying temperature and time were 61.71 °C and 298.28 min, respectively. The experimentally determined values were similar to the predicted values (with standard deviation values lower than 1.8%).

It is evident that due to over drying of Kolmou and under drying of Pui, both vitamin C and antioxidant activity were reduced in comparison with individual drying. This is due to loss of certain components in due course of drying. Such constituents are sensitive towards applied conditions of heat and duration of drying (Santos and Silva 2008). The positive effect of drying temperature and time on vitamin C and antioxidant activity has been analyzed to be due to enhanced dry matter content and component expression. However, such enhancement only existed up to a certain limit and thereafter negative sensitivity has been observed for

higher combinations of temperature and time. Also, for the vitamin C content case, the reduction is possibly due to the oxidation of active compounds to dehydro-ascorbic acid and eventual hydrolysis to 2, 3-diketogolonic acid and further oxidation and polymerization of such components to various inactive compounds. On the other hand, the moisture content was high for Pui and was marginally low for Kolmou. These values are also lower than those obtained after drying at 60 °C and 240 min drying time (chapter 3). For Jatilao, the optimal data is similar to that being reported in chapter 4.

In summary, co-drying is not a promising drying method due to loss of nutritional parameters. This is primarily due to over or under drying of some leafy vegetables. For the co-drying case, the concentration of Jatilao is even higher than that being obtained for the individual drying case. Since the drying conditions did not favour greater constitution of Kolmou and Pui samples for the co-drying cases, the corresponding response variables have lower values. Hence, obtained optimal mix formulation has lower proportions of these vegetables. Therefore, drying all leafy vegetables at same temperature-time combination is not promising as some vegetables may undergo poor or over drying with loss of nutritional properties for fixed choice of the process parameters for all cases. Also, for Jatilao, the optimal drying conditions achieved for the co-drying case are similar to those obtained from individual drying case (and findings in chapter 4). This affirms the definite achievement of optimal process variables for both cases for Jatilao. With high constitution of vitamin C and antioxidant activity, the Jatilao concentration is apparently higher in the near optimal soup formulation. The lower proportions of Kolmou and Pui vegetable samples in the mix formulation are due to their lower vitamin C and antioxidant activity values, respectively. Thereby, the optimization did not favour a greater constitution of these vegetables in the mix. Similar inferences can be drawn from Larrosa et al. (2015), who affirmed that the ingredients with higher antioxidant activity constituted significantly in the optimal vegetable mix.

Table 6.2: Experimental and predicted optimal parameters-responses data sets for alternate leafy vegetable systems and co-drying case.

		Drying method	Optimized temp (°C)	Optimized time (Min)	Moisture (%)	Vit C (mg/100g)	Antioxidant activity (%)	Reference
Kolmou	Predicted	Tray drying with intermittent airflow	61.71	298.28	0.81	52.86	84.29	This work
	Experimental	do	61.71	298.28	1.67 ± 0.18	50.72 ± 0.94	83.13 ± 2.31	do
	Experimental	do	60	240	4.21	56.96	92.34	Chapter 3
Jatilao	Predicted	Tray drying with intermittent airflow	61.71	298.28	2.86	113.20	89.58	This work
	Experimental	do	61.71	298.28	2.59 ± 0.34	112.09 ± 2.38	90.26 ± 2.12	do
	Experimental	do	60	240	7.68	112.68	89.45	Chapter 3
Pui	Predicted	Tray drying with intermittent airflow	61.71	298.28	10.59	126.37	24.33	This work
	Experimental	do	61.71	298.28	12.16 ± 0.4	124.65 ± 2.7	22.93 ± 0.83	do
	Experimental	do	60	240	10.44	123.94	23.08	Chapter 3
Mix (10:80:10)	Predicted	-	-	-	3.43	108.48	82.52	This work
	Experimental	-	-	-	3.86 ± 0.33	105.68 ± 1.99	80.94 ± 1.62	do
	Literature	-	-	-	-	-	-	-

6.2.3 Optimal Vegetable Mix Formulation

Among individual and co-drying approaches, the former is most suitable to achieve desired formulation due to its optimal response variable characteristics. Therefore, the optimum dry leafy vegetable mix of Kolmou, Jatilao and Pui can be considered as 10:74.95:15.05 with 3.21% moisture content, 110.38 mg/100g vitamin C and 80% antioxidant activity. With this proportion of leafy vegetables, formulation F_M and other control formulations (F_K , F_J and F_P) have been prepared. Further, the soup formulation was optimized based on formulations F_1 , F_2 , F_3 and F_4 . These have been deduced based on variant percentage ratios of 20, 30, 40 and 50% of vegetable mix to corn flour on a dry basis. All formulations have been summarized in **Table 6.3** that indicate fixed choice of spice constitution. The dry soup mix was thereafter reconstituted through 20 g dry mix with 400 mL water and subsequent boiling for 10 min. such reconstitution was based on previous literature (Srivastava et al. 2019; Chaudhari and Solanke 2016; Sugumar and Guha 2020; Dhiman et al. 2017) and few preliminary investigations. Finally, all formulations as reconstituted soups were subjected to sensory analysis. Adopted methodology for sensory analysis has been mentioned in section 2.5.3 of the Ph.D. thesis.

Table 6.3: Constitution of other ingredients in alternate leafy vegetable based soup formulations.

Ingredients/Formulation	F_K	F_J	F_P	F_M	F_1	F_2	F_3	F_4
Kolmou powder (g)	5	0	0	0.5	0.2	0.3	0.4	0.5
Jatilao powder (g)	0	5	0	3.75	1.5	2.25	3	3.75
Pui powder (g)	0	0	5	0.75	0.3	0.45	0.6	0.75
Corn flour (g)	5	5	5	5	8	7	6	5
Ginger powder (g)	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5
Onion powder (g)	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5
Garlic powder (g)	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5
Milk powder (g)	1	1	1	1	1	1	1	1
Salt (g)	3	3	3	3	3	3	3	3
Black pepper (g)	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5
Vegetable oil (mL)	2	2	2	2	2	2	2	2
Carrot flakes (g)	2	2	2	2	2	2	2	2

6.3 Sensory Characteristics of Reconstituted Soup

6.3.1 Optimization with respect to Control Samples

Sensory characteristics are highly important for a developed food product towards formal market acceptance. Sensory characteristics ensure that a developed food product is viable and feasible due to the acceptability of consumers based on sensory fitness.

The sensory characteristics of alternate formulations F_P , F_K , F_J and F_M have been depicted in **Fig. 6.1 (a) – (d)**, respectively. These were based on the average scores obtained through sensory reports of 10 trained experts. Among the said formulations, F_J and F_M possessed similar scores. Further, marginally higher scores were obtained for F_M . Highest scores for taste (7.9), texture (8), flavour (7.6), consistency (8.4), mouth feel (7.8), after feel (7.7) appearance (7.7) and overall acceptability (7.9) were obtained for formulation F_M . Corresponding range of sensory scores for control formulations were 7.2 – 7.8, 7.3 – 7.8, 7.4 – 7.6, 7.7 – 8.2, 7.5 – 7.8, 7.4 – 7.5, 7.4 – 7.6 and 7.3 – 7.8 for taste, texture, flavor, consistency, mouth feel, after feel, appearance and overall acceptability, respectively. Among control formulations, superior average sensory values were obtained for F_J (7.8 taste, 7.8 texture, 7.4 flavour, 8.2 consistency, 7.8 mouth feel, 7.5 after feel, 7.6 appearance and 7.8 overall acceptability). However, after feel (7.4 – 7.5) and appearance (7.4 – 7.6) remained almost similar for all cases with marginally higher for mixed formulation F_M (7.7 each for both after feel and appearance).

For all cases, the appearance is similar due to predominant greenish colour constitution of the prepared soups. On the other hand, Kolmou based formulation F_K possessed lowest sensory scores for all sensory parameters. The higher scores for the formulation F_M is likely due to interaction of all leafy vegetables to rigorously enhance all evaluated sensory properties of the product. Therefore, it is herewith inferred that the

combination of all chosen leafy vegetables might have also improvised the sensory characterized. The complex biochemical interactions of the constituents of various leafy vegetables are very likely to have contributed towards the synergy and collation of favourable sensory characteristics. Moreover, higher scores for F_J (Jatilao based soup formulation) justified the realization of higher quality parameters for F_M (mix formulation). This is due to the formulation comprising maximum proportion (75%) of Jatilao in the mix. On the other hand, the lower sensory values for Kolmou and Pui based control soup formulations affirmed their partial or non-compatibility of the vegetables for soup formulation in comparison with the Jatilao based soup formulation. Therefore, lower constitutions of these dried vegetables (10 and 15%, respectively for Kolmou and Pui) are desirable in the mixed leafy vegetable soup formulation to eventually justify their mutual characteristics of lower sensory qualities and mathematical programming based lower constitutions in the mixed formulation. The lower sensory scores for these control soup formulations have been attributed to the immature constitution and development of soup sensory characteristics.

Among control soup formulations, since F_J possessed highest sensory characteristics, the concentration of Jatilao can be expected to increase in the mix formulation F_M to achieve better formulation with desired characteristics. However, since F_M has highest sensory scores among all four formulations along with its high concentration of Jatilao (75%), F_M is herewith concluded to be the optimum formulation. Henceforth, further optimization of the formulation can be targeted based on F_M .

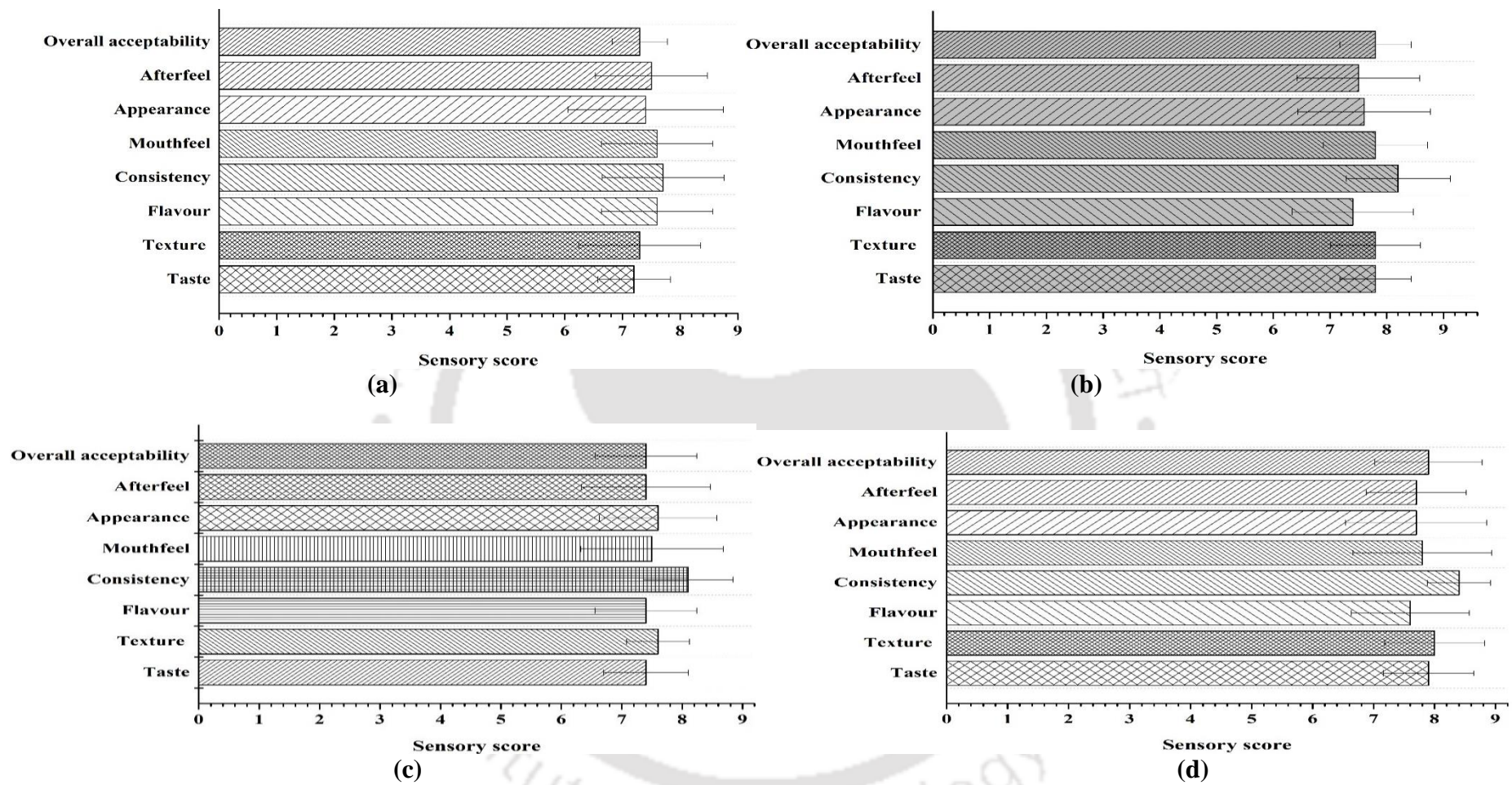


Fig. 6.1: Sensory characteristics of alternate leafy vegetable soup formulations - (a) F_K (control), (b) F_J (control), (c) F_P (control) and (d) F_M (mixed) formulations.

6.3.2 Optimization of Vegetable Mix to Corn Flour Ratio

Fig. 6.2 (a) – (d) illustrate the sensory characteristics (scores) of alternate formulations being developed with variant dry vegetable mix (optimal) to corn flour ratio. The sensory characteristics were found to be significantly influenced with variation in the ratio of dry vegetables mix (optimal) to corn flour. Among F_1 , F_2 , F_3 and F_4 , F_3 has been evaluated to possess highest sensory scores of taste (8.2), texture (8), flavour (8.2), consistency (8.5), mouth feel (8.1), after feel (7.9), appearance (7.8) and overall acceptability (8.4). Similar results have been reported for soy-mushroom-moringa soup (Farzana et al. 2017). Also, the overall acceptability was higher than the leafy vegetable soup formulation being reported in the literature in terms of trial and error based formulation constituting radish, turnip, carrot and cauliflower leaves using trial and error approach (Joshi et al. 2020). For this case, the overall acceptability has been reported to be 7.07 and 7.62 for the radish and turnip leaves based formulations, respectively. Combinatorial high sensory characterizations for this case are possibly due to optimal synergy and interactions between various constituents of the formulation. This leads to best combination of flavour and consistency quality characteristics. Further, due to maximum synergy in F_3 formulation, it is very likely that the sensory buds have been activated and satisfied to the best possible extent to thereby indicate best quality characteristics.

On the other hand, lowest sensory scores were obtained for F_2 . Corresponding values for F_2 were 7.8 (taste), 7.9 (texture), 8.1 (flavour), 7.9 (consistency), 7.8 (mouth feel), 7.5 (after feel), 7.7 (appearance) and 7.7 (overall acceptability). However, compared to the F_3 , appearance was better for F_1 and F_2 . This is due to light green colour of soup samples that was imparted due to higher proportion of the corn flour. On the contrary, other sensory characteristics of F_1 , F_2 and F_4 were lower. This is probably due to higher constitution of corn in F_1 and F_2 that significantly enhances higher viscosity and unusual characteristics of the

soup. All these are very likely to have not been appreciated by the panel experts. On the other hand, higher leafy vegetable concentration in F₄ contributed to unnecessary herbal flavour, taste, texture, appearance and overall acceptability of the product.

In summary, based on mathematical nutritional optimization followed with trial and error based sensory evaluation, the optimal soup formulation refers to 0.4 g Kolmou, 3 g Jatilao, 0.6 g Pui, 6 g corn flour, 0.5 g ginger powder, 0.5 g onion powder, 0.5 g garlic powder, 1 g milk powder, 0.5 g black pepper, 2 mL vegetable oil, 2 g carrot flakes and 3 g salt on a dry basis and reconstitution of 20 g mix with 400 mL water.



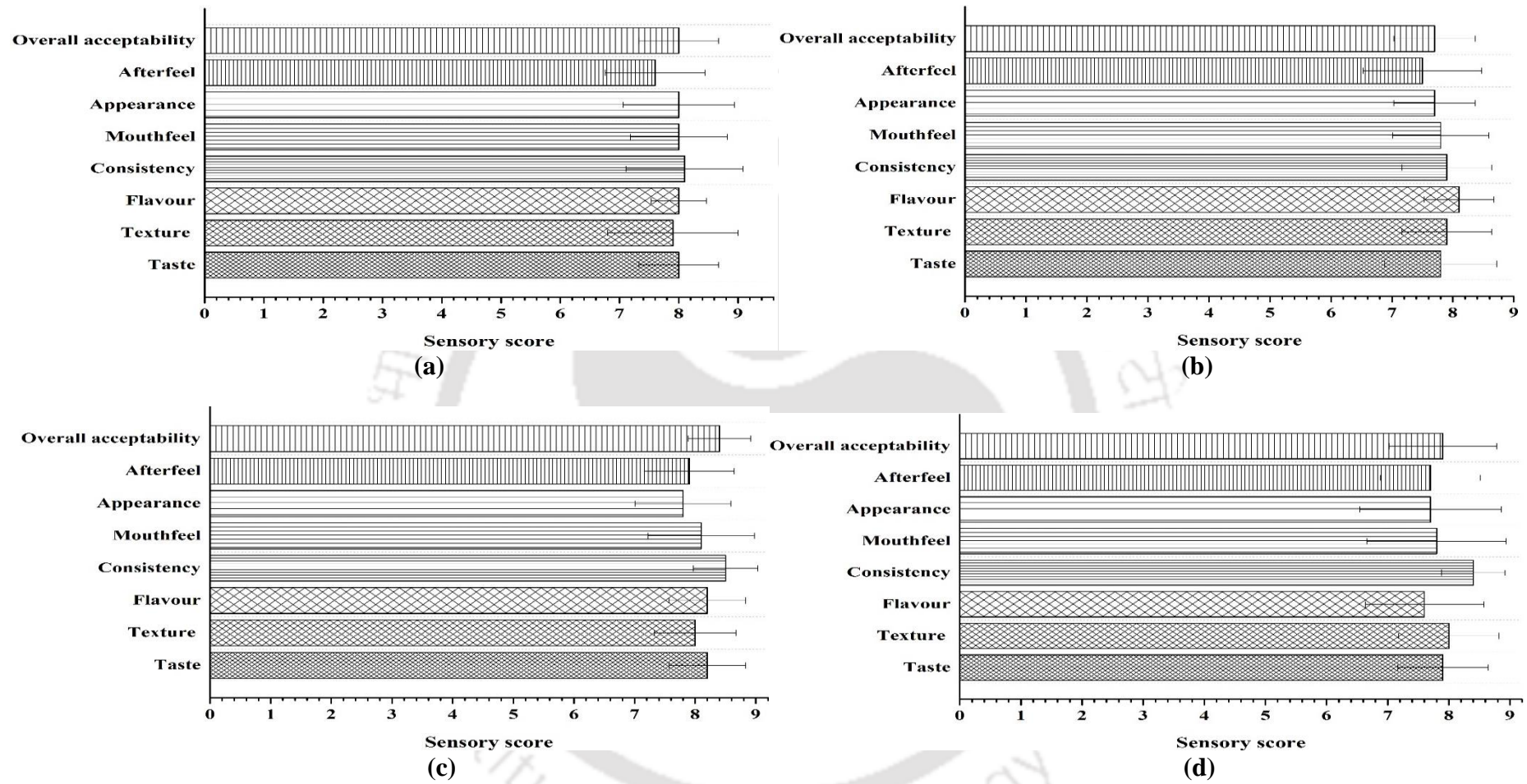


Fig. 6.2: Sensory characteristics of mixed leafy vegetable soup formulations and variant dry vegetable mix to corn flour ratios - (a) F₁ (b) F₂ (c) F₃ (d) F₄ formulations.

6.4 Rheological Characteristics of Optimal Soup Recipe

6.4.1 Steady State Rheological Characteristics

Fig. 6.3 (a) and **(b)** depicts the variation of shear stress and viscosity with shear rate at various temperatures. The steady state rheological study of F₃ (sensory and nutritionally optimal reconstituted soup formulation) affirmed that the shear stress increased with shear rate. At 30, 40 and 50 °C, the shear stress varied from 11.60 – 26.30, 7.46 – 19.70 and 4.04 – 15.30 Pa for a shear rate variation of 0.1 – 100 s⁻¹, respectively. On the other hand, the system viscosity reduced correspondingly from 125 – 0.28, 80.70 – 0.20 and 43.80 – 0.15 Pa.s. The viscosity reduction with temperature is due to reduced velocity gradient between the two consecutive layers of the fluid. For the high temperature case, a weak dragging force exist between the two fluid layers to thereby confirm upon the shear thinning behaviour of the soup sample.

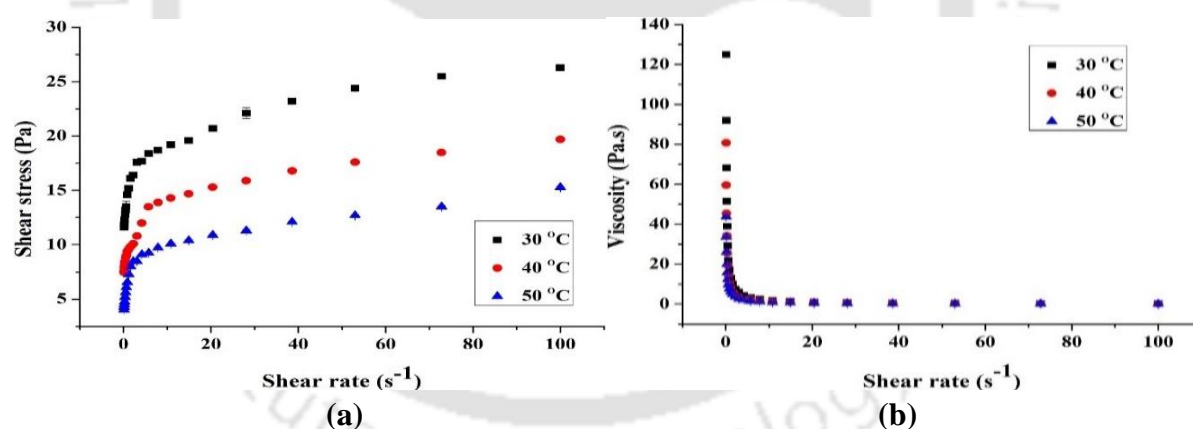


Fig. 6.3: Influence of shear rate on **(a)** shear stress and **(b)** viscosity of optimal leafy vegetable soup formulation.

The shear stress enhancement with shear rate was rapid initially. Thereafter, the variable enhanced gradually. During the initial phase, the pertinent abrupt deformation of maximum numbers of soup constituent particles led to the maximum displacement of the

particles. Thereby, the variable varied rapidly during this phase. Also, the viscosity reduced at a higher rate and this is due to greater reduction of velocity gradient between two consecutive liquid layers. On the other hand, at a later stage, the reduced variation of shear stress and viscosity was due to minimum liquid particle displacement or deformation due to applied shear rate. This was due to partial restoration of few particles or applied shear rate did not facilitate restoration of few particles to the original state. Further, the influence of temperature was found to be profound at the higher value. This is due to the development of a weak dragging force between two consecutive liquid layers and subsequent enhanced greater displacement of the constituents.

The obtained shear rate versus shear stress data has been evaluated to fit well with both Power and Herschel-Bulkley models ($R^2 = 0.9740 - 0.9867$ and $0.9867 - 0.9922$, respectively). The model fitness plot and associated parameters of shear stress versus shear rate has been depicted in **Fig. 6.4** and **Table 6.4**, respectively. The flow behaviour index (n) was found to be less than 1 for both models (Power and Hershel – Bulkley model). This confirms pseudo-plastic shear thinning behaviour of the sample. The F_3 soup rheological characteristics are comparable to that of tomato soup (Chavan et al. 2015; Yilmaz et al. 2010; Jayasinghe et al. 2016; Abdel-Haleem and Omran 2014). In the power law model, the consistency index (K) reduced from 14.82 – 6.63 for an increase in temperature from 30 – 50 °C. However, a mixed variation of consistency index is evident in the Herschel-Bulkley model. Hence, Power law model can be concluded to better describe the flow behaviour of the sample. Such inferences are in good agreement with those being reported for tomato soup (Chavan et al. 2015) and mango jam (Basu et al. 2013).

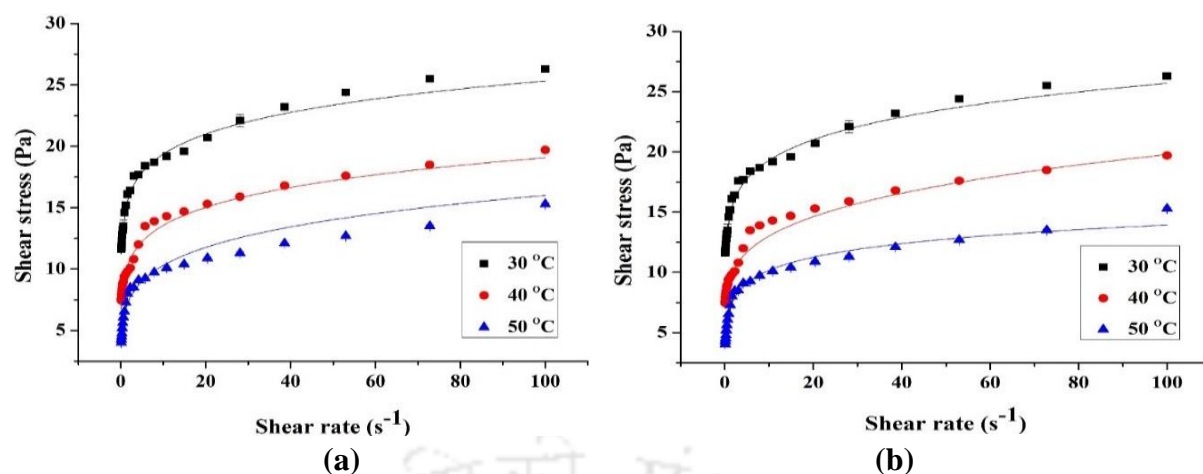


Fig. 6.4: Fitness plots associated to alternate models representing shear rate versus shear stress data of optimal leafy vegetable soup formulation (a) Power law and (b) Herschel-Bulkley model.

Table 6.4: Fitness parameters of Power and Herschel-Bulkley models associated to shear rate-shear stress data of optimal leafy vegetable soup formulation.

Model/Temp.	Parameters	30 °C	40 °C	50 °C
Power law	K	14.82096	9.64223	6.62635
	n	0.11629	0.14814	0.19158
	R^2	0.98671	0.97398	0.97588
Herschel-Bulkley	τ_0	3.1659	5.32388	-18.98775
	K	11.57227	4.09567	25.83691
	n	0.14464	0.27387	0.05264
	R^2	0.98666	0.99225	0.99011

6.4.2 Time dependent rheological characteristics

With non-Newtonian flow behaviour of the reconstituted soup of F₃ formulation, the shear stress-time plot (**Fig. 6.5**) exhibited a few marginal variations in its shape for parametric choices of 10 s⁻¹ shear rate, 300 sec time of shearing and 30 °C temperature. The viscosity and shear stress reduced initially and thereby reached an equilibrium state. These values varied initially from 2.04 – 1.86 Pa (viscosity) and 20.30 – 18.60 Pa.s (shear stress) for a variation in time from 0 – 30 sec. The equilibrium condition was achieved after 30 sec of applied shear rate.

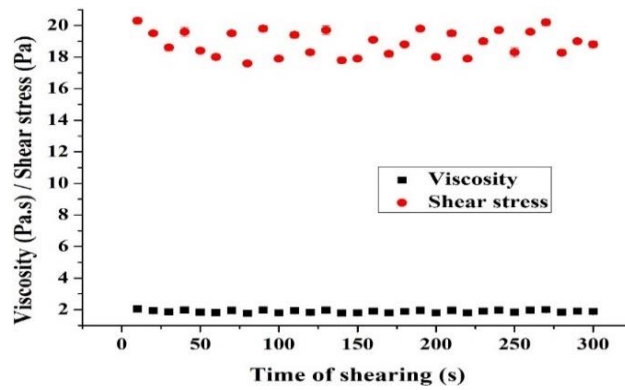


Fig. 6.5: Influence of shear time on viscosity and shear stress of optimal leafy vegetable soup formulation.

The formalization of a near equilibrium state can be attributed to the elastic behaviour of the fluid. In due course of the release of the applied forces, the reconstituted soup sample regained to its original state. Thus, a negligible variation in shear stress and viscosity has been observed for time dependent rheological property of the soup sample. However, marginal variations were due to the fact that as the fluid tries to regain its original state after applying a constant shear rate, a partial restoration of its state may have existed at some point. However, greater restoration is possible for other data points. Therefore, a fluctuation in viscosity and shear stress is apparent. The initial reduction of these variables is due to structural changes in the fluid that are being induced by applied shear rate. Similar behaviour has been reported for the tomato soup system (Chavan et al. 2015).

6.4.3 Dynamic rheological characteristics

Fig. 6.6 depicts the variation of storage modulus (G'), loss modulus (G'') and loss tangent (G'/G'') with sweep frequency (from 0.01 – 10 Hz). It can be observed that former two dynamic characteristics enhanced with increasing frequency. Corresponding variations for G' and G'' have been from $6.32 \times 10^2 - 1.48 \times 10^3$ and $7.12 \times 10^1 - 2.29 \times 10^2$, respectively. The G' and G'' reductions followed a rapid reduction during the initial phase followed with gradual

and slower reductions. On the other hand, the loss tangent (G'/G'') reduced from 8.88 – 7.12 for frequency enhancement from 0.01 – 0.1 Hz. After 0.1 Hz, almost constant values have been achieved. For all frequencies, G' dominated over G'' . This affirms elastic but not viscous behaviour of the soup. On the other hand, higher initial values for G'/G'' confirm upon a significant and strong elastic behavior of the soup sample.

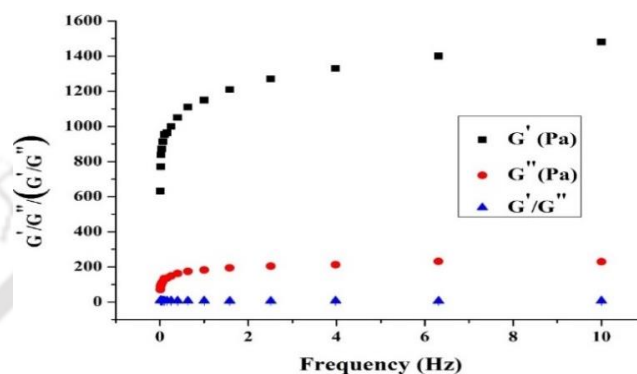


Fig. 6.6: Effect of frequency on the storage modulus, loss modulus and loss factor of optimal leafy vegetable soup formulation.

The elastic behaviour of reconstituted soup sample is due to the restoration of initial state of sample after each applied external force. For the given applied shear rate, the sample could regain its original structure in due course of the external force being released. For this reason, G' dominated over G'' for the soup sample. A complex interaction of ingredients (particularly leafy vegetable powder and corn flour with water) is anticipated to form a weak gel structure that enhances its elasticity. Similar results were reported for tomato soup (Chavan et al. 2015), Korean traditional sea mustard soup and Pollock soup (Kim et al. 2014).

6.6 Summary

The findings of the chapter demonstrated a mature methodology for realization of the optimal development of nutritionally rich and sensory based best fit leafy vegetable soup mix product.

A concise framework for the integration of mathematical, sensory and rheological approaches has been presented and demonstrated. The mathematical optimization was duly facilitated through the RSM based ANOVA analysis of individual leafy vegetable tray drying characteristics. Thereby, non-linear programming based models have been developed and applied for either individual or co-drying cases to achieve desired set of nutritional characteristics. The subsequent step of sensory analysis involved a trial and error based approach to optimize the thickening agent to vegetable mix ratio. Finally, rheological evaluation has been considered for the optimal soup mix.

Based on such hierarchical vegetable soup mix optimization studies, the optimum dry mix soup formulation refers to 0.4 g Kolmou, 3 g Jatilao, 0.6 g Pui, 6 g corn flour, 0.5 g ginger powder, 0.5 g onion powder, 0.5 g garlic powder, 1 g milk powder, 0.5 g black pepper, 2 mL vegetable oil, 2 g carrot flakes and 3 g salt on a dry basis with reconstitution of 20g mix and 400 mL water. The achieved soup formulation is nutritionally superior with high vitamin C (110.38 mg/100g) and antioxidant activity (80%). The overall acceptability for the reconstituted soup was found to be 8.4 for 9-point hedonic scale. On the other hand, reconstituted soup sample could be best described as a pseudo-plastic shear thinning fluid. Dynamic rheological investigations affirmed elastic behaviour of the sample. In summary, the article provided a useful and practical methodology for the due consideration of nutritional characteristics in subsequent sensory analysis based vegetable soup product development. Such investigations are anticipated to serve as useful resources to develop value added food products from underutilized endemic horticultural produces of NE India.

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Chapter 7:
**Formulation, Optimization and
Characterization of Non-leafy Vegetable Soup
Mix Recipe**



Formulation, Optimization and Characterization of Non-leafy Vegetable Soup mix Recipe

In this Chapter, the first section presents the state-of-the-art based pedagogy being adopted to achieve the optimal non-leafy vegetable soup mix recipe. Following this, section 6.2 details upon the results obtained with MS-Excel based solver for the resolution of non-linear programming model being formulated to represent proportional contributions of non-leafy vegetables to the soup mix characteristics. Thereby, both individual and co-drying cases have been considered to determine the optimal vegetable only mix formulations. Section 6.3 presents the critical findings associated with the trial and error based sensory analysis of optimal vegetable mix based alternate soup formulations. This involved determination of control as well as trial and error based variations in dry non-leafy vegetable mix to corn flour ratios in the considered formulations. Following this, section 6.4 details upon the findings associated to the optimally reconstituted soup mix recipe in terms of its rheological characteristics such as steady state, time dependent and dynamic flow behavioural characteristics of the soup. Finally, a summary of the best findings of the chapter has been presented in section 6.5.

7.1 Introduction

The available prior-art in the specific field of non-leafy soup product development indicates relevant applications of either trial and error or RSM based approaches (Jayasinghe et al. 2016; Abdel-Haleem and Omran 2014; Chavan et al. 2015b). Among these, the former

methodology involved consideration of several alternate formulations with variant ingredient constitution. On the other hand, the later methodology addressed ingredient variation in the soup recipe based on a statistical experimental design approach. Thereby, either sensory or nutritional or both sensory and nutritional bias was being set to determine the optimal non-leafy soup mix recipe constitution. Thus, based on the critical analysis of the available prior art, a mature rational approach involving mathematical optimization is lacking in the product development philosophy. Such considerations would facilitate prior optimization of nutritional characteristics with respect to sensory and rheological characterizations. Further, for the chosen non-leafy vegetables, no literature exists in the context of soup product development.

Based on the above mentioned lacunae, the thesis novelty refers to the application of a mature mathematical approach that targeted non-linear programming based optimization of the non-leafy vegetable mix based soup constitution. The vegetables that have been deployed for the soup include all mentioned non-leafy vegetables including Kolphul (*Musa balbisiana* Colla.), Kaskal (*Musa splendida*), Komora (*Benesica hispida*) and Posola (*Musa balbisiana* Colla.).

The non-linear programming model being formulated in terms of appropriate proportions of non-leafy vegetable constitution in the vegetable soup mix was solved using Microsoft Excel Solver. The model was being built on the RSM based non-linear response expressions that were determined using methodology outlined in the previous chapters of the Ph.D. thesis. Thereby, best combinations of nutritional parameters have been obtained for the optimal non-leafy vegetable constitution in the vegetable only soup mix formulation. Thereafter, fixed choices of alternate ingredients have been considered with respect to optimized vegetable only soup mix recipe for the subsequent optimization of sensory

characteristics and rheological analyses. Henceforth, the mature research methodology fostered towards best combinations of nutritional, sensory and rheological characteristics. For this, 9-pointer hedonic scale was adopted to carry out sensory analyses of alternate soup formulations. Such investigations were facilitated through a two-stage approach. The first stage involved the sensory analyses of optimal vegetable mix recipe and other control recipes being formulated with only one non-leafy vegetable for a specific recipe. Following this, the second stage investigations considered alteration of recipes based on systematic variations in optimal vegetable mix to corn flour ratio. For all such cases, soup reconstitution was targeted through a dry soup mix to water ratio of 20 g: 400 mL (Srivastava et al. 2019; Chaudhari and Solanke 2016; Sugumar and Guha 2020; Dhiman et al. 2017). Eventually, the best soup formulation with all ingredients was subjected to rheological characterizations. Such analyses define palatability, swelling issues and product consistency of the optimal soup mix formulation and provide useful insights with respect to product consistency. Thereby, the developed formulation can be analyzed from the acceptability perspective of the value added food product.

7.2 Evaluation of Optimal Dry Non-Leafy Vegetable Mix Formulation

7.2.1 Individual Drying

Based on MS-Excel solver based mathematical optimization, individual drying has been proven to be superior in comparison with co-drying case. Thereby, optimal recipe for the former case referred to higher combinations of response variables (vitamin C and antioxidant activity) and lower moisture content than those being obtained with the latter case. This is due to the fact that each vegetable has its own specific combination of optimal combination of drying temperature and time at which optimal response characteristics could be obtained. For the individual drying case, both predicted and experimental data sets have been

summarized in **Table 7.1**. The best data set corresponds to an optimal proportion of 29.09:15:45.91:10 for Kolphul, Kaskal, Komora and Posola, respectively with an optimal objective function value of 1949.79. Corresponding optimal response values were 5.29% moisture content, 187.09 mg/100g vitamin C and 50% antioxidant activity. These findings are comparable and similar to those being obtained from subsequently conducted experimental investigations. For these cases, the standard deviations of the predicted responses with respect to the experimental data set were 0.53, 2.47 and 0.61 for moisture content, vitamin C and antioxidant activity, respectively. The soup mix indicated higher constitution of Komora due to its higher vitamin C content (187.09 mg/100g) and lower constitution of Posola due to its poor nutritional characteristics. The lower constitutions of Kaskal and Posola in the soup mix have been attributed to their poorer combination of vitamin C and antioxidant activity characteristics. It is well known that the solution of a NLP formulation involves an assignment of lower values for the vegetables with relatively poorer constitution of the mentioned variables. On the other hand, the vegetable with higher vitamin C and antioxidant activity constituted higher contribution towards the optimal non-leafy vegetable mix. Therefore, the constitutions of Kolphul and Komora have been higher in the mix. Also, it is evident that the concentration of Komora is significantly high in comparison with that of other non-leafy vegetables. This is due to significantly higher content of vitamin C content (310.34 mg/100g) for Komora in comparison with Kolphul (87.97 mg/100g), Kaskal (91.1 mg/100g) and Posola (53.55 mg/100g) vegetable samples. Similar inferences have been reported in the literature with respect to suggested vegetable mix formulation (Larrosa et al. 2015). The authors affirmed that the vegetable with higher desired characteristics (such as antioxidant activity) were significant in their constitution in the soup mix recipe. Also, similar inferences have been deducted for the leafy vegetable mix as those being presented in the previous (chapter 6). The higher constitution (75%) of leafy vegetable

namely Jatilao has been achieved due to its higher constitution of vitamin C and antioxidant activity values in comparison with the soup mix lower constitutions of Kolmou (10%) and Pui (15%).

On the other hand, the optimal values for individual vegetable samples refer to 60.50 °C drying temperature, 610.02 min drying time, 5.08% moisture content, 87.97 mg/100g vitamin C and 89.41% antioxidant activity for Kolphul; 57.90 °C drying temperature, 389.32 min drying time, 6% moisture content, 91.10 mg/100g vitamin C and 72.81% antioxidant activity for Kaskal; 62.07 °C drying temperature, 532.35 min drying time, 5.53% moisture content, 310.34 mg/100g vitamin C and 22.82% antioxidant activity for Komora and 56.99 °C drying temperature, 378.79 min drying time, 3.71% moisture content, 53.55 mg/100g vitamin C and 25.93% antioxidant activity for Posola. The model based response data are comparable with the corresponding experimental data obtained at the optimal combination of drying temperature and time for each vegetable (standard deviations \leq 1.28, 1.65, 1.93 and 1.10 for Kolphul, Kaskal, Komora and Posola, respectively). The optimal data sets of non-leafy vegetable samples are in good agreement with those reported in the earlier chapters of the Ph.D. thesis Chapter 5).

Table 7.1: Experimental and predicted process and response variable data for non-leafy vegetable mix soup system and individual tray drying case.

		Drying method	Optimized temp (°C)	Optimized time (Min)	Moisture (%)	Vit C (mg/100g)	Antioxidant activity (%)	Reference
Kolphul	Predicted	Tray drying	60.50	610.02	5.08	87.97	89.41	This work
	Experimental	do	60.50	610.02	6.13 ± 0.47	86.16 ± 2.04	88.29 ± 1.72	do
	Experimental	do	60	600	7.34	84.51	86.38	Chapter 3
Kaskal	Predicted	Tray drying	57.90	386.32	6	91.1	72.81	This work
	Experimental	do	57.90	386.32	5.35 ± 0.39	88.76 ± 2.42	72.16 ± 0.97	do
	Experimental	do	60	600	7.34	84.51	86.38	Chapter 3
Komora	Predicted	Tray drying	62.07	532.35	5.53	310.34	22.82	This work
	Experimental	do	62.07	532.35	6.31 ± 0.38	313.07 ± 2.64	21.30 ± 0.63	do
	Experimental	do	60	600	7.34	84.51	86.38	Chapter 3
Posola	Predicted	Tray drying	56.99	378.79	3.71	53.55	25.93	This work
	Experimental	do	56.99	378.79	4.55 ± 0.4	55.17 ± 1.84	23.38 ± 1.34	do
	Experimental	do	60	600	7.34	84.51	86.38	Chapter 3
Mix (29.09:15:45.91:10)	Predicted	Tray drying	-	-	5.29	187.09	50	This work
	Experimental	-	-	-	4.58 ± 0.36	190.52 ± 3.08	49.14 ± 1.74	do
	Literature	-	-	-	-	-	-	-
Mix (25.33:20:44.67:10)	Predicted	Tray drying	-	-	5.33	184.48	50	This work
	Experimental	-	-	-	5.63 ± 0.52	183.25 ± 2.33	51.26 ± 0.86	do
	Literature	-	-	-	-	-	-	-
Mix (21.58:25:43.42:10)	Predicted	Tray drying	-	-	5.37	181.86	50	This work
	Experimental	-	-	-	4.72 ± 0.32	184.38 ± 2.94	47.83 ± 2.14	do
	Literature	-	-	-	-	-	-	-
Mix (17.83:30:42.17:10)	Predicted	Tray drying	-	-	5.41	179.24	50	This work
	Experimental	-	-	-	5.08 ± 0.66	178.71 ± 3.22	48.26 ± 1.28	do
	Literature	-	-	-	-	-	-	-

7.2.2 Co-drying

A summary of results obtained for co-drying case has been presented in **Table 7.2**. The table affirms comparatively poorer data set for the co-drying case in comparison with the individual drying case. The optimal objective value of 1749.32 was achieved for response characteristics of 6.44% moisture content, 188.41 mg/100g vitamin C and 50% antioxidant activity. The data set is similar to those being obtained in subsequent experimental investigations (standard deviation ≤ 2.02). The comparatively lower values for all response variables during co-drying is due to the over drying of Kaskal, Komora and Posola vegetable samples. Such over drying was detrimental for the retention of heat sensitive nutritional components. With prolonged exposure to heat, these responses are very likely to get degraded (Santos and Silva 2008). However, the same was not the case for shorter exposure to heat. For the case, the detrimental effect of heat is not predominant in comparison with the enhanced dry matter content of the sample being achieved due to effective removal of the moisture. Also, for the vitamin C content case, the reduction has been possibly due to the oxidation and polymerization of active compounds to various inactive compounds (Thankitsunthorn et al. 2009). On the other hand, insufficient drying of Kolphul did not facilitate nutritional parameters to dominate. This is due to low dry matter content in the sample. The corresponding optimal proportion of various non-leafy vegetable samples has been obtained as 34.88:15:40.12:10 for Kolphul, Kaskal, Komora and Posola, respectively. Higher constitution of Komora and Kolphul samples in the optimal vegetable mix is due to higher content of the desired response characteristics (i.e. higher vitamin C and antioxidant activity). On the other hand, lower proportions of dried Kaskal and Posola powder in the optimal mix is due to their poorer constitutions of antioxidant activity and vitamin C content. Similar constitution has been indicated in the literature cited vegetable soup recipe (Larrosa et al. 2015). The author inferred higher constitution of the dried vegetable with higher

antioxidant activity value. Such an inference has also been reported in the previous chapter (chapter 6).

Further, the optimal set of drying temperature and time for all vegetables during co-drying refers to 61.41 °C and 504.98 min, respectively. Corresponding co-drying process based optimal response characteristics were 8.59% moisture content, 84.69 mg/100g vitamin C and 86.76% antioxidant activity for Kolphul; 4.05% moisture content, 75.50 mg/100g vitamin C and 60.08% antioxidant activity for Kaskal; 7.31% moisture content, 307.15 mg/100g vitamin C and 23.08% antioxidant activity for Komora and 3.77% moisture content, 36.11 mg/100g vitamin C and 14.65% antioxidant activity for Posola. Subsequent experimental investigations at optimal drying temperature and time data indicated best fitness of the responses with lower standard deviations ($\leq 1.18, 2.45, 1.48$ and 2.78 for Kolphul, Kaskal, Komora and Posola, respectively) between predicted and experimental data sets for all cases. The obtained response characteristics are comparable to those being reported previously in the Ph.D. thesis (chapters 3 and 5).

In summary, the co-drying affirmed poorer process characteristics with significantly lower objective function in comparison with the individual drying case. The co-drying based optimal data set indicated moderately higher moisture content for Kolphul and Komora and hence insufficient drying time. Also, due to lower dry matter content for such case, vitamin C and antioxidant activity values are also comparatively lower. On the other hand, due to over drying of Kaskal and Posola, the mentioned response variables reduced significantly. Such reduction is due to the detrimental influence of heat on the evaluated nutritional responses. On the other hand, for both Kaskal and Posola samples, moisture content remained almost similar to those obtained with individual drying. This is due to the onset of equilibrium drying state in due course of the large drying time being set for both samples.

Table 7.2: Experimental and predicted process and response variable data for non-leafy vegetable mix soup system and co-drying case.

		Drying method	Optimized temp (°C)	Optimized time (Min)	Moisture (%)	Vit C (mg/100g)	Antioxidant activity (%)	Reference
Kolphul	Predicted	Tray drying with intermittent airflow	61.41	504.98	7.31	84.69	86.76	This work
	Experimental	do	61.41	504.98	8.24 ± 0.40	84.19 ± 2.13	89.03 ± 1.86	do
	Experimental	do	60	600	7.34	84.51	86.38	Chapter 3
Kaskal	Predicted	Tray drying with intermittent airflow	61.41	504.98	4.09	75.50	60.08	This work
	Experimental	do	61.41	504.98	4.75 ± 0.52	77.82 ± 1.66	63.06 ± 1.38	do
	Experimental	do	60	600	7.34	84.51	86.38	Chapter 3
Komora	Predicted	Tray drying with intermittent airflow	61.41	504.98	7.31	307.15	23.08	This work
	Experimental	do	61.41	504.98	6.74 ± 0.38	310.57 ± 2.70	21.56 ± 1.28	do
	Experimental	do	60	600	7.34	84.51	86.38	Chapter 3
Posola	Predicted	Tray drying with intermittent airflow	61.41	504.98	3.77	36.11	14.65	This work
	Experimental	do	61.41	504.98	4.16 ± 0.29	39.18 ± 1.24	17.06 ± 0.83	do
	Experimental	do	60	600	7.34	84.51	86.38	Chapter 3
Mix (37.28:10:47.72:5)	Predicted	-	-	-	6.44	188.41	50	This work
	Experimental	-	-	-	7.82 ± 1.15	191.27 ± 2.36	51.48 ± 0.93	do
	Literature	-	-	-	-	-	-	-

7.2.3 Optimal Vegetable Mix Formulation

Since individual drying provided best response characteristics, the optimal non-leafy vegetable only composition thus obtained (29.09:15:45.91:10 for Kolphul, Kaskal, Komora and Posola, respectively) was chosen as the near optimum dry soup mix. Corresponding optimal response characteristics were 5.29% moisture content, 187.09 mg/100g vitamin C and 50% antioxidant activity.

Table 7.3: Ingredient compositions in alternate non-leafy vegetable soup mix formulations.

Ingredients/Formulation	F _A	F _B	F _C	F _D	F _{M1}	F _{M2}	F _{M3}	F _{M4}	F ₅	F ₆	F ₇	F ₈
Kolphul powder (g)	5	0	0	0	1.45	1.27	1.08	0.89	0.43	0.65	0.86	1.08
Kaskal powder (g)	0	5	0	0	0.75	1.00	1.25	1.50	0.50	0.75	1.00	1.25
Komora powder (g)	0	0	5	0	2.30	2.23	2.17	2.11	0.87	1.30	1.74	2.17
Posola powder (g)	0	0	0	5	0.50	0.50	0.50	0.50	0.20	0.30	0.40	0.50
Corn flour (g)	5	5	5	5	5	5	5	5	8	7	6	5
Ginger powder (g)	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5
Onion powder (g)	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5
Garlic powder (g)	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5
Milk powder (g)	1	1	1	1	1	1	1	1	1	1	1	1
Salt (g)	3	3	3	3	3	3	3	3	3	3	3	3
Black pepper (g)	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5
Vegetable oil (mL)	2	2	2	2	2	2	2	2	2	2	2	2
Carrot flakes (g)	2	2	2	2	2	2	2	2	2	2	2	2

Considering the optimal combination of all non-leafy vegetables, a mix formulation F_{M1} was prepared. Further, other four formulations were also prepared using only one of the vegetables i.e., either of Kolphul (F_A), Kaskal (F_B), Komora (F_C) and Posola (F_D) vegetable samples. Thus, F_{A-D} were deployed as control samples to further augment upon the sensory characteristics of excel-solver based optimal vegetable only recipe. Various soup formulations with other fixed choices of ingredients have been summarized in **Table 7.3**. Fixed choices of various spices were based on few preliminary investigations and literature data (Srivastava et al. 2019; Chaudhari and Solanke 2016; Sugumar and Guha 2020; Dhiman

et al. 2017). For recipes F_{A-D} , and F_{M1} , the corn flour to vegetable dry mix was chosen as 50:50. This was based on available literature data (Srivastava et al. 2019; Chaudhari and Solanke 2016; Sugumar and Guha 2020; Dhiman et al. 2017). Further, sensory analysis was carried out with varied corn flour to vegetable dry mix as 20:80 (F_5), 30:70 (F_6); 40:60 (F_7) and 50:50 (F_8). For all samples, soups were realized through reconstitution with water at a ratio of 20 g of dry mix to 400 mL of water. Thereafter, soups were prepared through 10 min boiling and were provided to trained experts for sensory analysis. The reconstitution ratio was based on few preliminary investigations and available literature data (Srivastava et al. 2019; Chaudhari and Solanke 2016; Sugumar and Guha 2020; Dhiman et al. 2017). Also, it is to be noted that additional formulations F_{M2-M4} reported in **Table 7.3** will be delineated in the following sections and sub-sections of the Ph.D. thesis.

7.3 Sensory Characteristics of Reconstituted Soup

7.3.1 Optimization with respect to Control Samples

The sensory data of alternate mix and control soup formulations have been summarized in **Table 7.4**. The sensory scores for mix formulation F_{M1} refer to 7.7 taste, 7.8 texture, 7.6 flavour, 7.6 consistency, 7.6 mouth feel, 7.8 after feel, 7.8 appearance and 7.9 overall acceptability. Among various control soup formulations, the highest sensory scores were obtained for Kaskal based soup formulation (F_B). These refer to 8.2 taste, 8.3 texture, 7.9 flavour, 8.3 consistency, 8 mouth feel, 8.1 after feel, 7.9 appearance and 8.4 overall acceptability. Also, marginally lower sensory scores were obtained for Komora based soup formulation F_C (7.8 taste, 8 texture, 7.7 flavour, 8.4 consistency, 7.9 mouth feel, 7.9 after feel, 7.8 appearance and 8.1 overall acceptability). For the F_B case, highest sensory scores are possibly due to optimal interaction and compatibility of various ingredients and vegetables in their constituent proportions. On the other hand, corresponding sensory data for Kolphul (F_C)

and Posola (F_D) based soup formulations were 7.8 taste, 7.7 texture, 8.1 flavour, 7.7 consistency, 7.8 mouth feel, 7.6 after feel, 8 appearances and 7.7 overall acceptability and 7.0 taste, 6.8 texture, 6.7 flavour, 6.8 consistency, 6.6 mouth feel, 7.3 after feel, 6.9 appearance and 6.7 overall acceptability, respectively.

Table 7.4: Sensory characteristics of control formulations of non-leafy vegetable soup mix system.

Characterization/formulation	F_A	F_B	F_C	F_D	F_{M1}
Taste	7.8 ± 0.79	8.2 ± 0.79	7.8 ± 0.63	7 ± 0.67	7.7 ± 0.67
texture	7.7 ± 0.48	8.3 ± 0.67	8 ± 0.67	6.8 ± 0.79	7.8 ± 0.42
Flavour	8.1 ± 0.74	7.9 ± 0.57	7.7 ± 0.67	6.7 ± 0.67	7.6 ± 0.70
Consistency	7.7 ± 0.82	8.3 ± 0.67	8.4 ± 0.52	6.8 ± 0.79	7.6 ± 0.97
Mouth-feel	7.8 ± 0.63	8 ± 0.67	7.9 ± 0.57	6.6 ± 0.52	7.6 ± 0.52
After-feel	8 ± 0.47	7.9 ± 0.57	7.8 ± 0.63	6.9 ± 0.57	7.8 ± 0.42
Appearance	7.6 ± 0.52	8.1 ± 0.74	7.9 ± 0.74	7.3 ± 0.67	7.8 ± 0.79
Overall acceptability	7.7 ± 0.48	8.4 ± 0.70	8.1 ± 0.32	6.7 ± 0.82	7.9 ± 0.32

Thus it is apparent that the Kaskal based soup formulation has highest overall sensory scores for many parameters and marginally lower values for some other parameters. On the other hand, significant lower values have been achieved for Posola based soup formulation (F_D). Thus, it can be concluded that Kaskal vegetable proportion shall be high in the mix formulation F_{M1} to ensure upon its overall acceptability. On the other hand, lowest sensory characteristics for F_D formulation indicates that the sensory characteristics of the mix formulation F_{M1} may be improved by further reducing the amount of Posola sample in the mix formulation F_{M1} . However, since, the concentration of Posola sample in the mix formulation F_{M1} was only 10% of total vegetable powder mix, further reduction in its value

could not be allowed. Further, with relatively lower sensory scores for F_A formulation, Kolphul content may be further reduced marginally to enhance sensory characteristics of the mix sample. Thus, lower values for sensory scores and NLP based proportional contribution of Posola towards optimal non-leafy vegetable mix justified one other. On the other hand, since the response characteristics namely vitamin C (75.50 mg/100g) and antioxidant activity (60.08%) has been close to the overall response characteristics of the optimal vegetable mix, a further enhancement in the Kaskal constitution of would have negligible effect on the overall desired characteristics of the optimal non-leafy vegetable mix. Accordingly, the constitutions of other vegetables such Kolphul and Komora would be adjusted by solving the NLP model and obtaining a solution that has marginal variation in terms of the quality characteristics of the mix. Further details with respect to the same have been presented as follows.

In summary, Kaskal and Kolphul content may be, respectively enhanced and reduced in the mix formulation F_{M1} to enhance its sensory characteristics without compromising significantly upon the desired optimal nutritional characteristics. To do so, additional formulations (F_{M2} , F_{M3} and F_{M4} with higher Kaskal percentage of 20%, 25% and 30%, respectively in comparison with the lower Kaskal content of 15% in F_{M1}) have been targeted. For these formulations, it shall be noted that the optimization of other vegetable content was not fixed and was determined from the resolution of non-linear programming model being supplemented with the fixed choice of the above mentioned Kaskal content. In other words, the total content of Kolphul, Komora and Posola in F_{M2} , F_{M3} and F_{M4} in the optimized non-leafy vegetable dry mix refers to 80%, 75% and 70%, respectively. Thereafter, F_{M2-M4} have been optimized using MS-Excel solver and modified non-linear programming model with fixed choice of Kaskal content. Accordingly, the solver based composition of F_{M2} , F_{M3} and

F_{M4} have been obtained as 25.33:20:44.67:10, 21.58:25:43.42:10 and 17.83:30:42.17:10 (Kolphul:Kaskal:Komora:Posola), respectively.

Table 7.5 summarizes the sensory scores obtained for the above mentioned alternate vegetable mix formulations. The table affirms that maximum sensory scores have been obtained for the F_{M3} formulation (with Kaskal content of 25% in the vegetable only mix). For the mix, the objective function value of the non-linear programming model was 1897.14. Corresponding response characteristics refer to 5.37%, vitamin C 181.86 mg/100g and 50% antioxidant activity. The sensory scores for the formulation refer to 8.2 taste, 8.3 texture, 7.9 flavour, 8.3 consistency, 7.9 mouth feel, 8.1 after feel, 8.2 appearance and 8.4 overall acceptability. The highest scores for the F_{M3} formulation are due to the optimal interaction and compatibility of the constituents in their respective proportions including optimal expression of Kaskal in the vegetable mix. On the other hand, formulations with lower (F_{M2}) and higher (F_{M4}) Kaskal content were not appreciated by the experts. This is due to lesser and over expression of Kaskal constituents in corresponding soup mix samples.

Table 7.5: Sensory characteristics of non-leafy soup mix formulations with variant Kaskal constitution.

Characterization/formulation	F_{M1}	F_{M2}	F_{M3}	F_{M4}
Taste	7.7 ± 0.67	7.8 ± 0.63	8.2 ± 0.79	7.5 ± 0.71
texture	7.8 ± 0.42	7.8 ± 0.63	8.3 ± 0.48	7.6 ± 0.97
Flavour	7.6 ± 0.70	7.9 ± 1.10	7.9 ± 0.74	7.5 ± 0.85
Consistency	7.6 ± 0.97	7.8 ± 0.63	8.3 ± 0.48	7.8 ± 0.79
Mouth-feel	7.6 ± 0.52	7.9 ± 0.32	7.9 ± 0.74	7.6 ± 0.70
After-feel	7.8 ± 0.42	7.8 ± 0.79	8.2 ± 0.63	7.3 ± 0.48
Appearance	7.8 ± 0.79	7.7 ± 0.48	8.1 ± 0.57	7.7 ± 0.95
Overall acceptability	7.9 ± 0.32	8 ± 0.47	8.4 ± 0.52	7.6 ± 0.52

In summary, compared to the mentioned control and other samples, F_{M3} soup with the vegetable constitution of Kolphul:Kaskal:Komora:Posola as 21.58:25:43.42:10 was the best and exhibited an overall acceptability sensory score of 8.4 out of 9. Compared to the original F_{M1} sample formulation, the overall acceptability sensory is significantly high. For the best soup recipe, the predicted response characteristics were 5.37% moisture content, 181.86 mg/100 g vitamin C and 50% antioxidant activity. The experimental investigations confirmed thereafter lower standard deviation of the responses (standard deviation < 1.6 for all responses).

7.3.2 Optimization of Vegetable Mix to Corn Flour Ratio

The sensory characteristics of alternate soup formulations being prepared with variant vegetable mix to corn flour ratios have been summarized in **Table 7.6**. The corn flour content in a soup mix profoundly influences its sensory characteristics. Among various formulations, F₇ (40:60 dry vegetable mix to corn flour ratio) soup recipe performed the best with highest sensory scores for all considered parameters (taste 8.4, texture 8.3, flavour 8.2, consistency 8.4, mouth feel 8.3, after feel 8.3, appearance 8 and overall acceptability 8.6). Also, similar but marginally lower sensory scores were obtained for the F₈ formulation (dry vegetable mix to corn flour ratio 50:50). For the formulation, corresponding sensory scores were 8.2 taste, 8.3 texture, 7.9 flavour, 8.3 consistency, 7.9 mouth feel, 8.1 after feel, 8.2 appearance and 8.4 overall acceptability. The highest sensory scores for these systems are due to expression of various non-leafy vegetables, other ingredients and consistency of their optimal mixture towards sensory mouth buds of the experts. Similar inference was drawn for the leafy vegetable mix based soup product formulation identified in chapter 6 of the Ph.D. thesis.

Table 7.6: Sensory characteristics of non-leafy soup mix formulations with variant dry soup mix to corn ratios.

Characterization/formulation	F₅	F₆	F₇	F₈
Taste	7.1 ± 0.88	7.3 ± 0.63	8.4 ± 0.52	8.2 ± 0.79
Texture	7.2 ± 0.63	7.5 ± 0.63	8.3 ± 0.67	8.3 ± 0.48
Flavour	7.4 ± 0.70	7.5 ± 1.10	8.2 ± 0.63	7.9 ± 0.74
Consistency	7.3 ± 0.82	7.8 ± 0.63	8.4 ± 0.70	8.3 ± 0.48
Mouth-feel	7.5 ± 0.53	7.6 ± 0.32	8.3 ± 0.67	7.9 ± 0.74
After-feel	6.9 ± 0.88	7.2 ± 0.79	8.3 ± 0.48	8.2 ± 0.63
Appearance	7.5 ± 0.85	7.5 ± 0.48	8.0 ± 0.67	8.1 ± 0.57
Overall acceptability	7.3 ± 0.67	7.7 ± 0.47	8.6 ± 0.52	8.4 ± 0.52

On the other hand, lowest sensory scores were obtained for the F₅ formulation (taste 7.1, texture 7.2, flavour 7.4, consistency 7.3, mouth feel 7.5, after feel 6.9, appearance 7.5 and overall acceptability 7.3). This is due to higher corn content in the soup mix that translated into a relatively thicker soup and imparted undesired characteristics in terms of the best sensory evaluation by the experts. Moreover, the soup formulation with higher corn flour concentration has been anticipated to provide flavour characteristics and texture of soup being prepared with corn flour and other fixed spices. Similar trends were apparent for the leafy vegetable soup formulations reported for leafy vegetable soup formulations in chapter 6 of the Ph.D. thesis.

Thus, in summary, the optimal soup formulation refers to that of F₇ with dry vegetable mix to corn flour ratio of 40:60. Corresponding sensory characteristics of the reconstituted soup (20g dry soup mix to 400 mL water) refer to 8.4 taste, 8.3 texture, 8.2 flavour, 8.4 consistency, 8.3 mouth feel, 8.3 after feel, 8 appearance and 8.6 overall acceptability. The sensory characteristics have been marginally higher in comparison with the leafy vegetable

mix soup formulation being reported in the previous chapter (chapter 6). These characteristics are comparable with those being reported in the available literature for several vegetable soup products (Farzana et al. 2017; Abdel-Haleem and Omran 2014; Jayasinghe et al. 2016).

7.5 Rheological Characteristics of Optimal Soup Recipe

7.5.1 Steady State Rheological Characteristics

The steady state rheological characteristics were conducted for the best reconstituted F₃ soup formulation. Relevant characteristics in terms of shear stress versus shear rate and viscosity versus shear rate data have been depicted in **Fig. 7.1 (a)** and **(b)**, respectively. The figures clearly depict increasing shear stress trend with shear rate and essentially constitutes two phases. The first phase corresponds to a rapid enhancement in shear stress with shear rate (rapid increase in shear stress from 6.88 – 14.1, 4.97 – 11.5 and 1.64 – 6.1 Pa for a variation in shear rate from 0.1 – 10.8 s⁻¹ at 30, 40 and 50 °C, respectively). Following this phase, the second phase involves a gradual enhancement in shear stress with shear rate (corresponding variation from 14.1 – 18.2, 11.5 – 14.7 and 6.1 – 9.48 Pa for a shear rate variation from 10.8 – 100 s⁻¹ at 30, 40 and 50 °C, respectively). On the contrary, the viscosity trend indicated exponential reduction from 74.5 – 0.18, 53.8 – 0.15 and 17.8 – 0.095 Pa.s for a shear variation from 0.1 – 100 s⁻¹ at 30, 40 and 50 °C, respectively.

The rapid enhancement in shear stress is due to abrupt displacement of maximum number of constituent particles of the soup sample. In other words, the velocity gradient between two adjacent fluid layers reduced drastically and hence viscosity reduced abruptly during the initial phase of shear rate. On the other hand, reduced variations of shear stress and viscosity are due to the minimum displacement of particles. Thereby, the application of shear rate does not facilitate restoration of all particles. Hence, minimum particle displacement

occurs in this regime. Further, the partially restored particles are also very likely to have not been displaced beyond a certain extent. In summary, minimal fluid particle deformation occurred at the later stage of applied shear rate. On the other hand, higher temperature profoundly influenced both shear stress and viscosity. This is due to greater displacement of constituent particles being accomplished due to the development of weak dragging force between consecutive fluid layers.

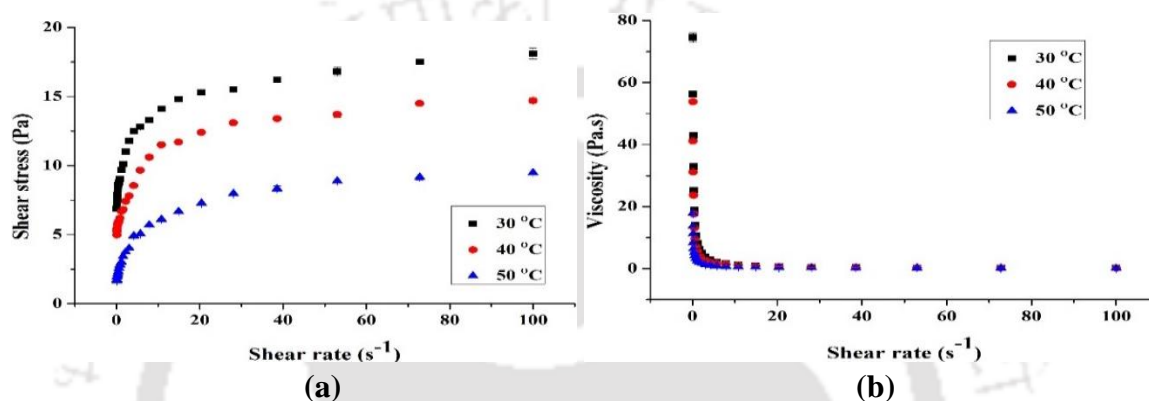


Fig. 7.1: Influence of shear rate on (a) shear stress and (b) viscosity for optimal non-leafy vegetable mix soup formulation.

The measured shear stress versus shear rate data were subjected to fitness studies using both Power and Herschel-Bulkley models that possessed flow behaviour index (n) value lower than 1. The model fitness plots and associated parameters have been presented in **Fig. 7.2 (a) – (b)** and **Table 7.7**, respectively. Both the models have been well fitted to the obtained data with high R² values (0.9943 and 0.9940, respectively for Power and Herschel-Bulkley model). The figures and table affirm that the reconstituted soup sample followed a pseudo-plastic shear thinning flow behaviour. Such flow behavioural characteristics have been reported in relevant prior art for various soup products (Chavan et al. 2015a; Yilmaz et al. 2010; Jayasinghe et al. 2016; Abdel-Haleem and Omran 2014). For the models, the consistency index (k) reduced from 9.66 – 3.02 and 9.55 – 2.89, respectively for Power and Herschel-Bulkley model and for an increase in temperature from 30 – 50 °C. Such a trend

was reported for tomato soup (Chavan et al. 2015a), mango jam (Basu et al. 2013). Similar behavior characteristics were analyzed for the leafy vegetable soup reported in the former chapter of the Ph.D. thesis (chapter 6).

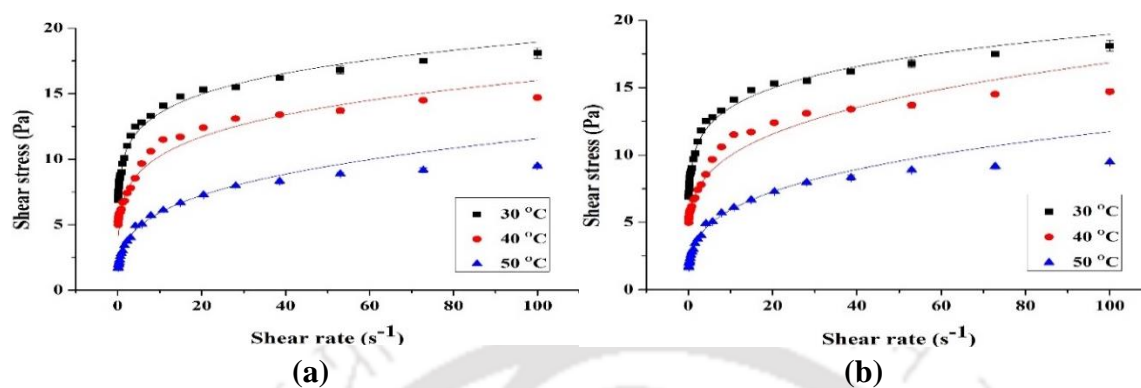


Fig. 7.2: Fitness plots to represent alternate models predicting shear rate versus shear stress data of optimal non-leafy vegetable mix soup system (a) Power law (b) Herschel-Bulkley model.

Table 7.7: Fitness parameters of Power and Herschel-Bulkley models and optimal non-leafy vegetable mix soup system.

Model/Temp.	Parameters	30 °C	40 °C	50 °C
Power law	k	9.6626	6.54921	3.0239
	n	0.14598	0.19389	0.29148
	R ²	0.99432	0.98306	0.99273
Herschel-Bulkley	τ_0	0.1079	3.14283	0.12466
	k	9.55094	3.38448	2.89149
	n	0.1475	0.30373	0.30177
	R ²	0.99403	0.98946	0.99247

7.5.2 Time Dependent Rheological Characteristics

Fig. 7.3 depicts the time dependent variation of shear stress and viscosity for a fixed shear rate choice of the best reconstituted soup formulation. The figure clearly depicts constant trends for both variables along with few marginal fluctuations. At constant shear rate of 10 s^{-1} , the shear stress and viscosity values varied marginally from 12.40 – 15.10 Pa and 1.25 –

1.50 Pa.s, respectively for a variation in time from 0 – 300 sec. Thereby, the trends affirm a negligible variation in the evaluated responses with time. In other words, time dependent flow behavior was not prevalent. This is due to restoration of initial fluid state in due course of time at the applied constant shear rate. However, minor variations are apparent. These are attributed to the partial restoration of initial state at few but not at all time data sets. The time dependent rheological characteristics of the soup sample have been in good agreement with those being evaluated for the leafy vegetable soup (chapter 6) and tomato soup (Chavan et al. 2015a).

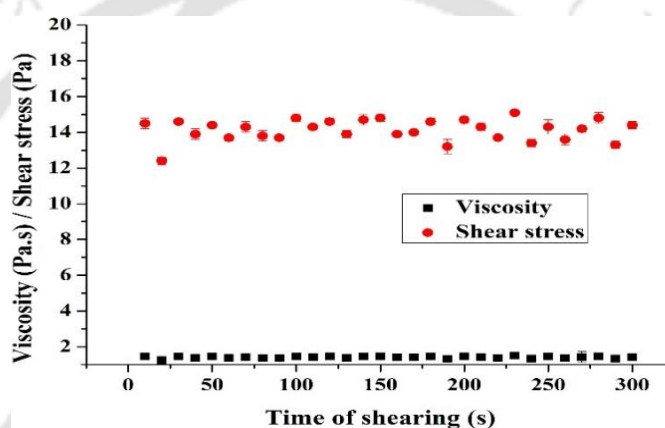


Fig. 7.3: Influence of shearing time on the viscosity and shear stress of optimal non-leafy vegetable mix soup system.

7.5.3 Dynamic Rheological Characteristics

Variations in dynamic flow parameters such as storage modulus (G'), loss modulus (G'') and loss tangent (G'/G'') with respect to variations in frequency have been investigated. Relevant trends have been depicted in **Fig. 7.4**. For frequency variation from 0.01 – 10 Hz, both storage and loss moduli enhanced gradually from $5.9 \times 10^2 - 1.34 \times 10^3$ and $6.86 \times 10^2 - 2.17 \times 10^2$, respectively. For the case, storage modulus (G') was dominant in comparison with the loss modulus (G'') in due course of the applied frequency range (0.01 – 10 Hz) and for constant strain condition of 0.05 at 30 °C. Such dominant characteristic of G' over G''

confirms elastic behaviour of the soup sample. This is due to regaining of its original state after each stroke of external force was applied. Such an effect always induced higher G' in comparison with the G'' value. Complex interactions of all ingredients are likely to foster the formation of a weak gel structure of the soup sample. Such weak gel structure is anticipated to induce elastic behavior of the sample during dynamic rheological investigations.

On the other hand, for a variation in frequency from 0.1 to 10 Hz, the loss tangent (G'/G'') varied from 8.60 – 6.14. Overall, the evaluated dynamic flow characteristics of the soup sample were in good agreement with those being depicted for the leafy vegetable soup (chapter 6 of the Ph.D. thesis), tomato soup (Chavan et al. 2015a), Korean traditional sea mustard soup and Pollock soup (Kim et al. 2014).

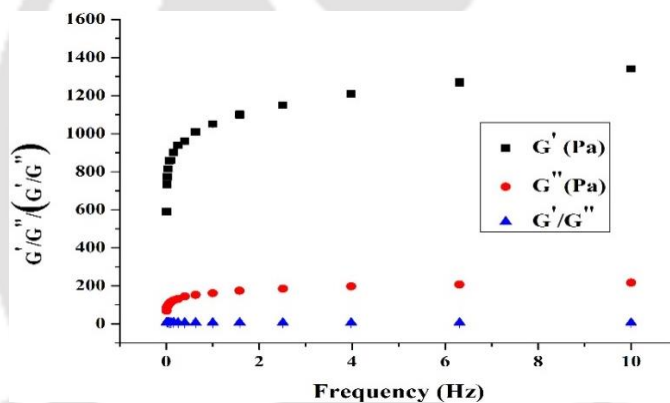


Fig. 7.4: Influence of frequency upon storage modulus, loss modulus and loss factor of optimal non-leafy vegetable mix soup system.

7.6 Summary

In this chapter, the research findings related to the non-leafy soup product development witnessed several novel outcomes. Firstly, a mature mathematical optimization approach was supplemented to the trial and error based sensory evaluation of alternate soup formulations. The optimization model was based on the RSM design based best fit non-linear model

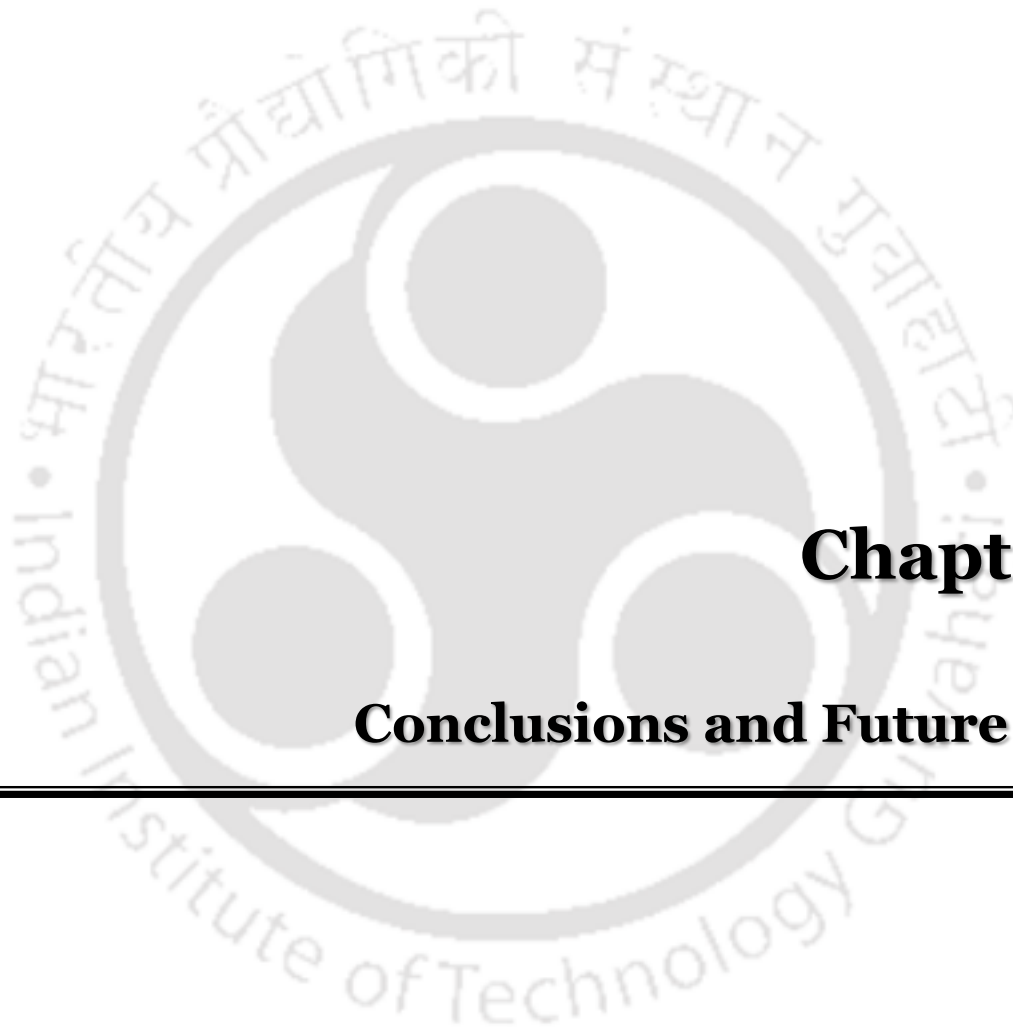
expressions identified to represent nutritional characteristics of individual vegetable that were considered in the thesis. Compared to co-drying, individual drying allowed better achievement of set objective function and indicated programming based prediction of optimal response characteristics as 5.37% moisture content, 181.86 mg/100 g vitamin C and 50% antioxidant activity. Corresponding optimal constitution of non-leafy vegetable only mix referred to 21.58:25:43.42:10 for Kolphul, Kaskal, Komora and Posola, respectively. Secondly, mixed formulations indicated domination of Kaskal among all chosen vegetables to improvise upon the compositions. Thirdly, trial and error based sensory evaluation affirmed optimization of 40:60 ratio for dried vegetable mix to corn flour ratio. Accordingly, the best performing formulation referred to the F₇ recipe with an overall sensory acceptability of 8.6 out of 9 for a reconstituted 20 g dry soup mix with 400 mL water. In summary, the best non-leafy vegetable soup formulation refers to 0.86 g Kolphul, 1 g Kaskal, 1.74 g Komora, 0.4 g Posola, 6 g corn flour, 0.5 g ginger powder, 0.5 g onion powder, 0.5 g garlic powder, 1 g milk powder, 0.5 g black pepper, 2 mL vegetable oil, 2 g carrot flakes and 3 g salt on a dry basis. Fourthly, the best reconstituted soup formulation exhibited pseudo-plastic shear thinning rheological flow behaviour. In conclusion, the key research findings of the chapter contribute towards value added product development of underutilized non-leafy vegetables of the North-east India and thereby promote diversification of food product portfolio. Thereby, the research can catalyze sustainability of rural economy through the promotion of food processing sector in the region well known for its bio-diversity of wild and endemic horticultural produces.

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Chapter 8:

Conclusions and Future Work



Conclusions and Future Work

In section 8.1, the Chapter details upon important conclusions being deduced from the research addressed in the Ph.D. thesis. Following this, section 8.2 delineates upon the possible scope for future work in the chosen field of research.

8.1 Conclusions

Experimental and theoretical investigations being addressed in the Ph.D. thesis have been effective to provide useful conclusions in the broad field of vegetable soup formulation using underutilized and endemic horticultural produces. A brief account of the same has been presented in the following sub-sections.

8.1.1 Optimization of alternate drying methods for dried leafy and non-leafy vegetables

- Among oven and tray drying processes, for a combination of drying temperature and time of 60 °C and 4 h for leafy vegetables (Kolmou, Jatilao and Pui leafy vegetables) and 10 h for non-leafy vegetables (Kolphul, Kaskal, Komora and Posola non-leafy vegetables), the tray dried vegetable samples possessed superior combinations of proximate (carbohydrate, protein, ash, crude fibre and fat contents), vitamin C and antioxidant activity characteristics.
- Among the chosen leafy and non-leafy vegetables, highest and lowest vitamin C was obtained for tray dried Pui (123.94 mg/100g) and Komora (304.37 mg/100g),

respectively. Correspondingly, antioxidant activity was highest and lowest for tray dried Kolmou (92.34%) and Kolphul (86.38%) vegetable samples.

- All chosen leafy and non-leafy vegetables have been evaluated to be very good sources of carbohydrate, crude fibre and ash content. Corresponding highest parametric values were obtained for Kaskal (81.37%), Posola (15.86%) and Pui (16.32%), respectively.

In summary, among oven and tray drying processes, the later has been proven to be best to achieve good retention of nutritional characteristics of the chosen leafy and non-leafy vegetable samples. Therefore, such investigations would facilitate and promote the wider applicability of tray drying system towards drying of wider horticultural produces of the country due to its ability to effectively retain nutritional content in dried samples. This would promote industrial application to develop various value added ready to eat/cook food products from various abundant bio-resources of India. Further, this work involves a systematic approach and set forth a baseline to proceed further with the optimization of tray drying process parameters to maximize the nutritional contents of the final dried products. The same has been addressed in the mentioned chapters.

8.1.2 Optimization of process parameters for tray dried leafy vegetables

- The moisture reduction trend with time followed an exponential decline trend, which could be best represented with Page model for most cases. Significant influence of drying temperature and time were apparent for all evaluated responses including moisture content, vitamin C and antioxidant activity.
- Studies conducted towards evaluation of drying kinetics affirmed moisture diffusivities to vary about 6.36×10^{-13} – 5.16×10^{-12} m²/s, 1.79 – 5.36×10^{-12} m²/s and 9.94×10^{-13} –

$6.23 \times 10^{-12} \text{ m}^2/\text{s}$ for Kolmou, Jatilao and Pui leafy vegetables. Corresponding activation energies were 45.21, 25.3 and 43.15 kJ/mol.

- Both vitamin C and antioxidant activity followed a mixed trend indicating an initial increase followed with consistent reduction.
- Statistical design based studies affirmed best fitness of quadratic models to represent response characteristics of all tray dried leafy vegetables.
- For all leafy vegetable samples, among drying kinetics and statistical design based approaches, best process parameters were obtained for the statistical design case.
- Optimal process parametric data set of chosen leafy vegetables can be summarized as follows:
 - a) Kolmou: 61.28 °C temperature, 236.9 min drying time, 2.87% moisture content, 57.02 mg/100g vitamin C content and 91.36% antioxidant activity.
 - b) Jatilao: 61.62 °C drying temperature, 292 min drying time, 3.28% moisture, 113.36 mg/100g vitamin C and 89.37% antioxidant activity.
 - c) Pui: 63.90 °C, 342.19 min, 4.37% moisture, 130.98 mg/100g vitamin C and 24.99% antioxidant activity.

With high vitamin C and antioxidant activity content, the chosen leafy vegetables can be deployed to prepare value added products such as ready to eat/cook soup products. Mathematical optimization can be conveniently targeted to achieve best constitution of leafy vegetable soup mix recipe. In summary, the thesis affirmed RSM-CCD based design approach to be highly promising to yield useful insights into optimal tray drying process parameters and response characteristics of the chosen leafy vegetables. The best fit quadratic response characteristics models can be conveniently deployed to develop and solve a non-

linear programming based optimization model for the simultaneous maximization of vitamin C and antioxidant activity content and minimization of moisture content. Thereby, nutritionally rich leafy vegetable soup mix recipe can be identified.

In summary, this investigation affirms coherence and compatibility of all leafy vegetables for the development of mixed food product. However, parallel processing of the leafy vegetables is not recommended due to significant variations in optimal drying time and temperature. Thus, relevant guideline has been set in this work for the development of various mix food formulations. Also, with Jatilao being a good source of vitamin C and antioxidants while Kolmou and Pui being a good source of antioxidant activity and vitamin C characteristics, they can be deployed to achieve mixed food products such as vegetable soups. In summary, the mentioned approach can be replicated for other food material systems to develop mix product formulations. Thereby, the carried out investigations affirm that the underutilized leafy vegetables of the NE region can be transformed into value added products such as soups and thereby drive economic competitiveness of the horticultural sector in the region.

8.1.3 Optimization of process parameters for tray dried non-leafy vegetables

- For all chosen non-leafy vegetables and for most temperature cases, the Page model best represented the pertinent drying characteristics of the tray drying process.
- For the chosen non-leafy vegetable samples, the moisture diffusivity varied from $9.64 \times 10^{-12} - 5 \times 10^{-11} \text{ m}^2/\text{s}$, $1.56 \times 10^{-11} - 7.59 \times 10^{-11} \text{ m}^2/\text{s}$, $1.78 \times 10^{-11} - 6.02 \times 10^{-11} \text{ m}^2/\text{s}$ and $1.79 \times 10^{-12} - 7.35 \times 10^{-12} \text{ m}^2/\text{s}$ for Kolphul, Kaskal, Komora and Posola, respectively. Corresponding activation energy values were 37.38, 34.10, 26.56 and 30 kJ/mol, respectively.

- The variation of vitamin C and antioxidant activity with temperature followed a mixed trend that indicated an intermittent higher value followed with a steady decrease.
- For all tray dried non-leafy vegetable samples, the CCD-RSM statistical design based experimental data set was best represented using quadratic models. Among trial and error and RSM design approaches, best response characteristics were obtained for the statistical design case.
- The best data sets corresponding to process parameters and response characteristics of the tray dried chosen vegetable samples have been obtained as follows:
 - a) Kolphul: 61.82 °C drying temperature, 611.52 min drying time, 3.98% moisture 87.69 mg/100g vitamin C and 89.04% antioxidant activity.
 - b) Kaskal: 58.66 °C drying temperature, 400.31 min drying time, 5.09% moisture content, 90.73 mg/100g vitamin C and 72.34% antioxidant activity.
 - c) Komora: 62.77 °C drying temperature, 530.83 min drying time, 5.09% moisture, 310.40 mg/100g vitamin C and 22.67% antioxidant activity.
 - d) Posola: 57.59 °C drying temperature, 389.42 min drying time, 2.84% moisture content, 53.38 mg/100g vitamin C and 25.72% antioxidant activity.
- With desired nutritional characteristics, the chosen non-leafy vegetables can be utilized to achieve nutritionally rich ready to eat/cook food products such as soups.

In summary, the Ph.D. thesis affirmed useful insights with respect to product-process characteristics of the mentioned underutilized non-leafy vegetables. Hence, ready to cook/eat food products such as soups can be developed using the mentioned ingredients. The response quadratic models can be conveniently expressed as a non-linear programming model. Such modelling efforts facilitate the determination of best nutritional characteristics of the soup mix

being deduced with the chosen vegetables and their optimal constitution. Thereby, nutritionally rich recipe with near optimal constitution of the chosen non-leafy vegetables can be obtained, which can be further optimized to achieve a soup recipe with the best sensory and rheological characteristics.

Thus, the obtained data will be beneficial to identify coherence and compatible non-leafy vegetables for mixed product formulations. With all important characterization, underutilized non-leafy vegetables can be explored commercially for the cost effective realization food product formulations such as ready to cook or eat food products. Soup formulations of chosen vegetables can be prepared with dominant constitution of Kolphul and Komora, moderate constitution of Kaskal and lower constitution of Posola due to high combination of antioxidant and vitamin C for Kolphul, significant higher content of vitamin C for Komora, moderate constitution of vitamin C and antioxidant activity characteristics of Kaskal and lower combination of these variable characteristics for Posola. Thereby, these insights have been anticipated to widen the applicability of tray dried non-leafy vegetables in terms of translational ready to consume food products such as soups and thereby promote entrepreneurship opportunities for the rural community of developing economies such as India.

8.1.4 Formulation and characterization of leafy vegetables based mix soup recipe

- The carried out investigations affirmed the need for mature and rational mathematical optimization to supplement the trial and error based sensory characterization of leafy vegetable mix soup formulations.

- Among two drying methodologies, individual drying is preferred with respect to co-drying due to achievement of higher vitamin C (110.38 mg/100g) and antioxidant activity (80 %) and lower moisture content (3.21 %) in the leafy vegetable only soup mix recipe.
- Based on Excel solver based mathematical optimization, the optimal constitution of leafy vegetable soup mix refers to a composition of 10:75.95:14.05 for Kolmou, Jatilao and Pui.
- Among various control soup formulations (prepared with only one of the leafy vegetables), Jatilao based soup formulation possessed highest sensory scores. This is in agreement with the optimal soup recipe indicating 76% of Jatilao in the vegetable mix only recipe.
- Further, based on sensory analysis of alternate formulations prepared with variant dry vegetable mix to corn flour ratio, the optimal soup formulation refer to F₃ (40:60) with an overall acceptability of 8.4/9 for the reconstituted soup system with solids to water ratio of 20 g: 400 mL.
- The rheological characteristics of the best identified soup mix reconstituted recipe refers to pseudo-plastic shear thinning behaviour and elastic behaviour (G' dominated over G'') during steady state and dynamic analyses, respectively.

Thus, a Microsoft-Excel solver based mathematical optimization based on non-linear programming has been targeted to ensure maximum retention of desired nutritional parameters for the formulation of vegetable mixed soup product. Further, sensory and rheological based optimization ensures gastronomically enhanced soup product with higher consumer acceptance. Such product facilitates to meet the demanding factors such as enhanced health awareness and tasty ready to cook food product. Further, the two tier approach can be replicated for other mix

food formulations with rich nutritional and functional food products. Thereby, the methodology would diversify the process-product portfolio for the ready to eat/cook food products of commercial importance. In summary, the work identified a nutritionally and sensory based optimal recipe using under-utilized leafy vegetables such as Kolmou, Jatilao and Pui. The adopted methodology involved a two-step procedure that considered a rigorous mathematical optimization of the vegetable only recipe followed with trial and error based vegetable to corn flour ratio optimization. The identified recipe is anticipated to instil confidence in the diversification of ready to cook/eat soup products in the region and contributes towards sustainable rural economy of North-east India through the promotion of adequate food processing sectors.

8.1.5 Formulation and characterization of non-leafy vegetables based mix soup recipe

- An optimal non-leafy vegetable based mix soup formulation was successfully achieved in terms of best combinations of nutritional and sensory characteristics. A step approach was duly followed to optimize first nutritional content of the vegetable mix using mathematical optimization tools and thereafter consideration of trial and error approaches to identify recipe with best fit characteristics of sensory analysis.
- Among individual and co-drying cases, the former indicated best characteristics in terms of lower moisture content (5.29%) and higher combinations of vitamin C (187.09 mg/100g) and antioxidant activity (50%) contents. The optimal vegetable only mix soup recipe referred to a constitution of 21.58:25:43.42:10 for Kolphul, Kaskal, Komora and Posola, respectively.

- Among alternate formulations, the F₃ soup formulation being prepared with 40:60 dry vegetable mix to corn flour ratio performed the best in terms of the overall acceptability of 8.6/9 for the reconstituted dry soup mix to water ratio of 20 g to 400 mL.
- The optimal reconstituted soup exhibited a pseudo-plastic shear thinning behaviour during steady state analysis. Dynamic rheological investigations affirmed elastic behaviour (G' dominated over G'') of reconstituted optimal soup.

In summary, with higher desired combinations of response and sensory characteristics, an optimal non-leafy soup formulation has been successfully identified with the chosen underutilized and endemic vegetables and the mentioned two tier approach. Further, the soup formulation is anticipated to foster diversification of product development portfolio for soup products and other similar value added food products with commercial prominence. Thus, the addressed research can profusely contribute towards development of various nutritionally rich and functional food products with higher consumer acceptability from wider bio-resources available in North-East India. Further, this would provide a better sustenance for the farmers and ensure improvement in the rural economy of the region. Thereby, it would promote rural entrepreneurship and food processing sectors in North-East India.

The overall conclusions of the Ph.D. thesis can be presented as follows:

- a) Compared to sun, shade, oven and other expensive techniques such as freeze, microwave, pulse electric drying etc., the intermittent airflow assisted tray system is a promising processing technique to process the chosen leafy and non-leafy vegetables. Such technology would be highly relevant for the translational research associated to abundant yet abandoned quantities of horticultural produces.

- b) The intermittent airflow assisted tray drying system facilitates chosen dried horticultural produces to retain combinations of good nutritional characteristics (high vitamin C and antioxidant activity) and shelf life parameters (low moisture content). The CCD-RSM based experimental design methodology further affirmed on such product-process characteristics.
- c) Nutritionally and sensory based best fit recipes have been developed to produce leafy and non-leafy vegetable soups. The integration of mathematical and sensory approaches has been outlined in the thesis.
- d) Based on rigorous mathematical optimization, the vegetable only soup mix recipe was obtained for individual drying with the weight based constitution of 10:75.95:14.05 for Kolmou, Jatilao and pui, respectively and the leafy vegetable soup and 21.58:25:43.42:10 for Kolphul, Kaskal, Komora and Posola, respectively and for non-leafy vegetables.
- e) The optimal soup leafy and non-leafy based mix soup formulations refer to F3 (40:60 dry vegetable mix to corn flour ratio) formulation.
- f) For a solid to water reconstitution ratio of 20g to 400 mL, the overall acceptability of the optimally reconstituted leafy and non-leafy vegetable mix based soup recipes were 8.4/9 and 8.6/9, respectively. For both cases, the soups exhibited pseudo-plastic shear thinning behavior during steady state rheological analysis and elastic behavior during time dependent analysis.
- g) Finally, the thesis demonstrated possible avenues for translational research in the product-process optimization and product development based on abundant horticultural produces of North-east India. Thereby, the thesis can serve and inspire as a useful resource to promote value added food product related food processing in the region.

8.2 Future work

Various possible extensions of the research methodology and product design palette indicated in the Ph.D. thesis are as follows:

- Other underutilized leafy and non-leafy vegetables can be targeted for the development of alternate vegetable soup formulations. Thereby, further diversification of value added product portfolio can be targeted.
- The nutritional characteristics of chosen leafy and non-leafy vegetables can be targeted from the perspective of other significant response characteristics such as phenolic content, flavonoids, vitamins and other phytochemicals.
- Other inexpensive drying techniques such as vacuum, microwave drying can be targeted for their efficacy towards value added product development using chosen and additional leafy and non-leafy vegetable sources. Similar technologies can be targeted to develop customized soup products by following the integrated two step methodology of vegetable soup research.
- The optimization of other ingredients in the leafy and non-leafy soup formulations can be targeted using either trial and error or RSM design based approaches and for the chosen vegetables. Similar studies can be extended towards other underutilized yet abundant vegetables.
- Alternate soup formulations addressed in the work can be subjected to rheological investigations to gain useful insights with respect to the rheological profile of the soup formulations. Thereby, useful research dividends can be conveniently obtained with respect to further optimization of swelling and consistency characteristics of the vegetable soup systems.

- Storage and shelf life studies of the developed dry leafy and non-leafy vegetable mix based soup recipes can be attempted. The shelf life of dried vegetables are often quantified through the measurement of its water activity. Since relevant instrumentation is not available at IIT Guwahati, the moisture content of the samples has been measured. However, future work needs to address such measurements in due course of shelf life analysis.
- Packaging studies associated to soup formulations for the maximization of shelf life and product longevity will be a viable strategy.
- It is well known that Vitamin C is susceptible to oxidation in an adverse environment. Hence, effective packaging materials need to be used and the shelf life studies in due course of long term storage of the dried food products need to be evaluated in laboratory environment in terms of the Vitamin C degradation. The same shall be addressed in the future research work that greatly targets upon the onfield application of the developed non-leafy and leafy vegetable mix soup products. Needless to convey, storage materials such as pouches and their cost has an influence on the Vitamin C content of the stored products. Thus, associated tradeoffs need to be considered in this regard.
- Additional characterization of the soup samples in terms of physical (colour), mechanical (texture, caking characterization etc.) and microbial characteristics can be targeted so as to ensure safe consumption of the products.
- The adopted two tier methodology for soup formulation can be conveniently applied to alternate food processes and product formulations.

In summary, the research work provides a systematic approach for the development of mixed vegetable soup formulation. It widens the scope for the application of other drying

methods such as vacuum, fluidized bed, microwave drying etc. towards relevant retention of prominent nutritional and phytochemical characteristics in the optimally formulated mix food products. Hence, it further diversifies the process-product portfolio for the development of nutritional and functional food products from largely underutilized leafy and non-leafy vegetables. Further, the two tier product development research methodology associated with the optimal vegetable soup formulation can be extended towards the development of alternate product formulations and associated food processing schemes.

On the other hand, the carried out research would be beneficial from the perspective to widen and assure upon a mature rational methodology being applied towards various food product development investigations. Hence, this would promote the food processing sectors and widen the product diversity of commercial importance. Further, maximum utilization of underutilized bio-resources can be entertained towards the successful translation as various food products. This would meet the nutritional demand in the market and as well mitigate the malnutrition of the weaker section of the country. Also, for such noble causes, more rural entrepreneurs would be created to eventually enhance the realization of a sustainable rural economy of the country.





Publications



List of Publications

Published articles in international refereed journals

- [1] **Mondal, I. H.**, Rangan, L., & Uppaluri, R. V. (2020d). A robust and novel methodology for the optimal targeting of leafy vegetable mix soup formulations. *LWT-Food Science and Technology*, 134, 110152.
- [2] **Mondal, I. H.**, Rangan, L., & Uppaluri, R. V. (2020c). Process-product characteristics of tray-dried *Benincasa hispida*. *Journal of Food Processing and Preservation*, 44(9), e14697.
- [3] **Mondal, I. H.**, Rangan, L., & Uppaluri, R. V. (2020b). Symphony of kinetics and statistical design approaches for response analysis during tray drying of *Lagenaria siceraria* leaves. *Journal of Thermal Analysis and Calorimetry*, doi: 10.1007/s10973-020-09782-w.
- [4] **Mondal, I. H.**, Rangan, L., & Uppaluri, R. V. S. (2020a). Parametric optimality of tray dried *Musa balbisiana* Colla blossom. *Journal of Food Science and Technology*, 57, 4599-4612.
- [5] **Mondal, I. H.**, Rangan, L., & Uppaluri, R. V. (2019). Effect of oven and intermittent airflow assisted tray drying methods on nutritional parameters of few leafy and non-leafy vegetables of North-East India. *Heliyon*, 5(11), e02934.

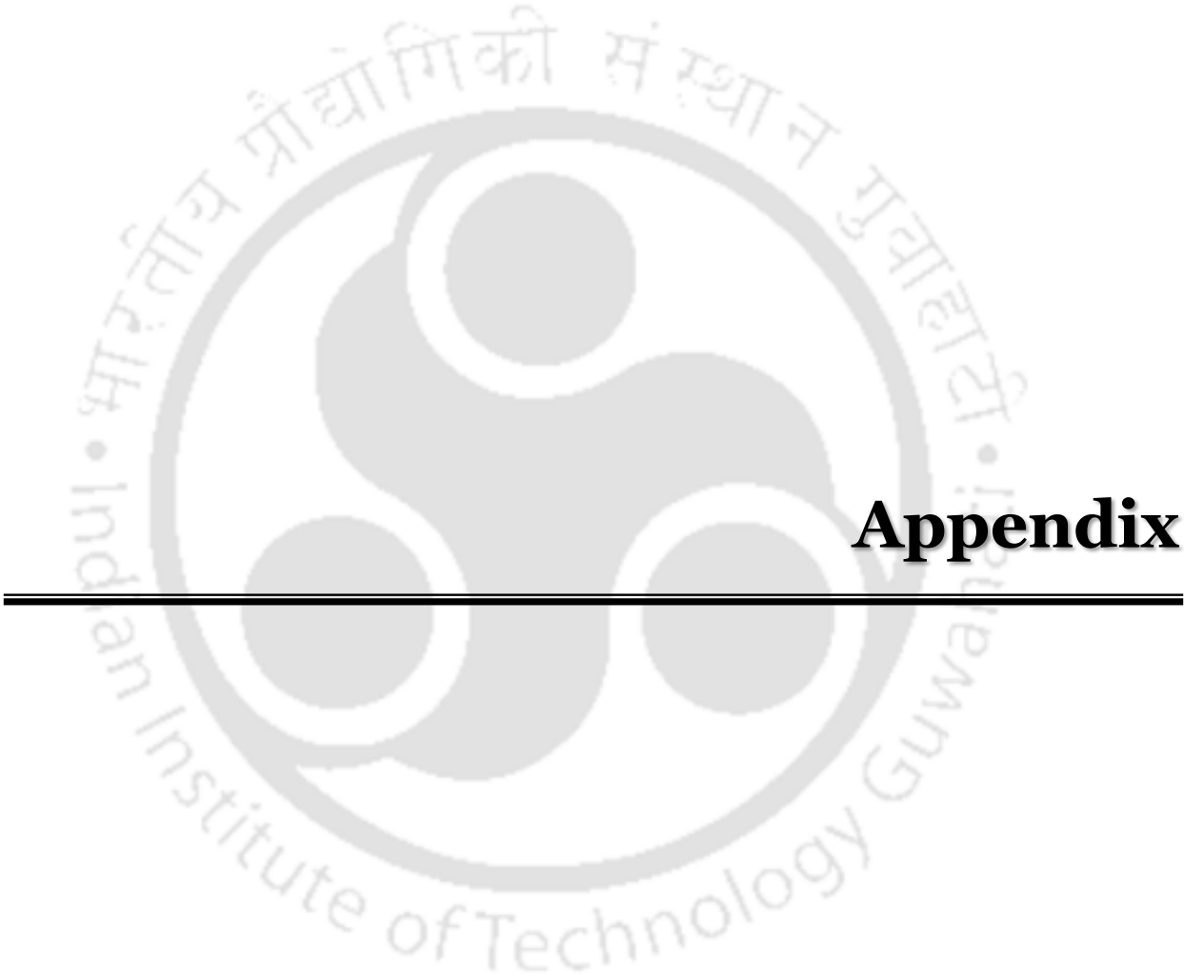
Communicated/Under preparation

- [1] **Mondal, I. H.**, Rangan, L., & Uppaluri, R. V. Tray drying characteristics of *Musa splendida* and *Musa balbisiana* Colla psuedo-stem. (Submitted)

- [2] **Mondal, I. H.**, Rangan, L., & Uppaluri, R. V. (2020). A robust and novel methodology for the optimal targeting of non-leafy vegetable mix soup formulations. (To be submitted)

Conference Presentations (National and International)

- [1] **Mondal, I. H.**, Rangan, L., & Uppaluri, R. V (2020). Trial and error and statistical design based parametric optimality of tray dried *Musa splendida*. Bioengineering 2020, 10 – 11th Dec, 2020 organized by NIT Rourkela.
- [2] **Mondal, I. H.**, Uppaluri, R. V., & Rangan, L. (2020). Combinational trial and error and statistical design based optimality of tray dried *Musa balbisiana* Colla. pseudo-stem. CHEMCON 2020, 27 – 29th Dec, 2020 organized by IChE.
- [3] **Mondal, I. H.**, Rangan, L., & Uppaluri, R. V (2020). Process parametric and variable characteristics of tray dried *Ipomoea aquatica* Forssk and *Basella alba* leaves. North-East Green Summit, fifth edition 16 – 18th Nov, 2020 organized by VIBGYOR N.E. Foundation and IIT Guwahati.





Appendix A: Calibration Curve for The Determination of soluble protein concentration

Bradford method was deployed to determine the soluble protein content of the tray dried leafy and non-leafy vegetable samples. The methodology involves calibration curve preparation through the dilution of standard bovine serum albumin solution (BSA) (100 ppm). Thereafter, the diluted solutions with 20, 40, 60, 80 and 100 ppm solutions were prepared. Subsequently, 2 mL of Bradford reagent was added to each of these solutions and after thorough mixing, their absorbance was measured a UV-visible spectrophotometer (Model No.: UV-2600, Make: Shimadzu, Singapore). Thereby, the absorbance versus concentration plot was prepared as a calibration curve to express the soluble protein content of analyzed sample as an equivalent of the BSA. The obtained calibration curve is depicted in **Fig. A1**.

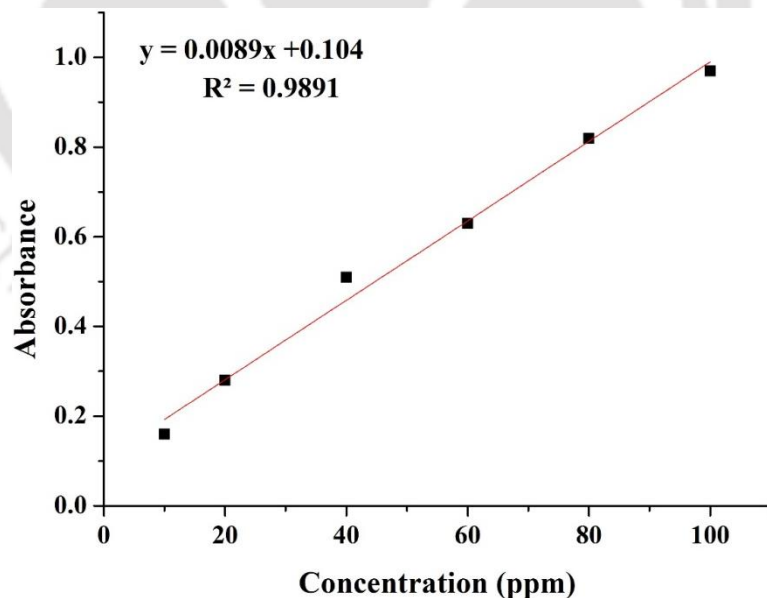


Fig. A1: Calibration curve for the determination of soluble protein.

Appendix B: Sample Calculations for the Estimation of Proximate and Nutritional Characteristics

In this section, a sample calculation has been presented for the determination of proximate and nutritional characteristics of tray dried or fresh chosen vegetable samples.

a) Yield: Weight of fresh sample taken (W_f): 8.95 g; weight of tray dried sample (W_s): 1.27 g

$$\text{Using eq. (2.1), \% Yield} = \frac{1.27}{8.95} \times 100 = 14.19\%$$

b) Moisture content: Weight of sample taken (W_s): 0.3025 g; weight of oven dried (105 °C for 12 h) sample (W_a): 0.2848 g

$$\text{Using eq. (2.2), \% Moisture} = \frac{0.3025 - 0.2848}{W_s} \times 100 = 5.85\%$$

c) Ash content: Weight of sample taken (W_s): 0.3047 g; weight of residue obtained after incineration at 600 °C for 6 h (W_a): g

$$\text{Using eq. (2.3), \% Ash} = \frac{0.031}{0.3047} \times 100 = 10.17\%$$

d) Crude fibre content: Weight of fresh sample taken (W_s): 1 g; weight loss during ignition (W_i): 0.095 g

$$\text{Using eq. (2.4), \% Crude fibre} = \frac{0.095}{1} \times 100 = 9.5\%$$

e) Fat content: Weight of fresh sample taken (W_s): 6 g; weight of extract obtained (W_f): 0.23 g

$$\text{Using eq. (2.5), \% Fat} = \frac{0.23}{6} \times 100 = 3.83\%$$

f) Carbohydrate content: Absorbance of diluted sample (A_s): 0.571 g; Absorbance of standard sample (A_{std}): 0.282 g

$$\text{Using eq. (2.6), \% Carbohydrate} = \frac{0.571}{0.282} \times 25 = 50.62\%$$

g) Using eq. (2.7) % Crude protein = $100 - (5.85 + 50.62 + 3.83 + 9.50 + 10.17) = 20.03\%$

h) Vitamin C content: Volume of ascorbic acid consumed (V_1): 3.55 mL; volume of sample extract consumed (V_2): 0.16 mL; weight of sample taken (W_s): 0.1 g.

$$\text{Using eq. (2.8), Vitamin C (mg/100g)} = \frac{0.5 \times 0.16 \times 10 \times 100}{3.55 \times 5 \times 0.1} = 45.70 \text{ mg/100g}$$

i) Antioxidant activity: Absorbance of control sample (A_c): 0.337; Absorbance of experimental sample (A_s): 0.049

$$\text{Using eq. (2.9), \% Antioxidant activity} = \frac{0.337 - 0.049}{0.337} \times 100 = 85.46\%$$

Appendix C: Nutritional Characteristics of Fresh Leafy and Non-leafy Vegetables

For baseline measurement, a paste sample was prepared with fresh vegetable samples. A mortar-pestle was used for the purpose. Thereafter, the paste sample was subjected to nutritional analyses such as moisture content, carbohydrate, soluble protein, ash, crude fibre, fat and crude protein content.

Table C1: Nutritional characteristics of fresh (a) leafy (b) non-leafy vegetables

(a)				
Parameter (%)/sample	Kolmou	Pui	Jatilao	
Moisture	86.65 ± 1.86	89.78 ± 1.73	87.02 ± 1.54	
Carbohydrate	6.87 ± 0.62	5.25 ± 0.38	5.57 ± 0.53	
Crude protein	3.14 ± 0.32	1.92 ± 0.15	2.53 ± 0.22	
Fat	0.59 ± 0.16	0.44 ± 0.13	1.22 ± 0.27	
Crude fibre	1.22 ± 0.08	0.75 ± 0.14	1.71 ± 0.11	
Ash	1.53 ± 0.09	1.86 ± 0.17	1.95 ± 0.14	
Vitamin C (mg/100g)	2.68 ± 0.28	8.23 ± 0.6	5.07 ± 1.02	
Antioxidant activity	1.72 ± 0.25	0.47 ± 0.18	2.40 ± 0.15	

(b)				
Parameter (%)/sample	Kolphul	Kaskal	Komora	Posola
Moisture	90.72 ± 1.58	84.09 ± 1.84	96.05 ± 1.26	94.49 ± 2.15
Carbohydrate	5.06 ± 0.47	13.54 ± 0.62	2.66 ± 0.28	3.41 ± 0.32
Crude protein	1.04 ± 0.14	0.95 ± 0.16	0.30 ± 0.05	0.24 ± 0.07
Fat	0.86 ± 0.06	0.49 ± 0.12	0.13 ± 0.04	0.28 ± 0.04
Crude fibre	1.24 ± 0.05	0.18 ± 0.06	0.51 ± 0.11	0.87 ± 0.13
Ash	1.09 ± 0.07	0.76 ± 0.08	0.35 ± 0.04	0.69 ± 0.08
Vitamin C (mg/100g)	4.53 ± 0.42	7.66 ± 1.03	12.18 ± 1.23	2.47 ± 0.38
Antioxidant activity	2.27 ± 0.35	2.18 ± 0.16	0.34 ± 0.12	0.58 ± 0.17

Appendix D: Sample Calculations for the Determination of Moisture Diffusivity and Activation Energy

In this section, a sample calculation has been presented to determine moisture diffusivity and activation energy values of the tray dried leafy and non-leafy vegetables. The calculation procedure is as follows:

a) Moisture diffusivity: Thickness of sample ($2L$): 0.27 mm; π : 3.14.

Slope (MR versus t curve) = - 0.0145

$$D \text{ (} \frac{\text{m}^2}{\text{s}} \text{)} = - \frac{\text{Slope} \times 4 \times L^2}{\pi^2} = - \frac{(-0.0145) \times 4 \times 0.135^2}{3.14^2 \times 60 \times 10^{-6}} = 1.79 \times 10^{-12} \text{ m}^2 / \text{s}$$

b) Activation energy: Universal gas constant: 8.314 J/mol K.

Slope (MR versus t curve) = - 3119.2

$$E_a \text{ (} \frac{\text{kJ}}{\text{mol}} \text{)} = - \frac{\text{Slope} \times R}{1000} = - \frac{(-3119.2) \times 8.314}{1000} = 25.93$$

Appendix E: Sample Calculation for the evaluation of formulation soup mix nutritional characteristics

In this section, a sample calculation has been presented to delineate upon various parameters being computed in due course of MS Excel-solver based mathematical optimization of non-linear programming model. Thereby, optimal leafy/non-leafy vegetable soup mix recipe's nutritional characteristics and objective function values have been determined. These are as follows:

a) Moisture content of individual vegetable (e.g. Kolmou): Drying temperature (T_k): 62.39 °C; drying time (t_k): 231.97 min.

Using equation 2.15 and table 4.7 (coefficients of quadratic equations),

$$\begin{aligned} \% \text{ Moisture content} &= 682.02 - 16.33 \times 62.39 - 1.13 \times 231.97 + 1.22 \times 10^{-2} \times 62.39 \times 231.97 \\ &+ 9.99 \times 10^{-2} \times 62.39^2 + 6.56 \times 10^{-4} \times 231.97^2 = 1.98 \end{aligned}$$

Vitamin C content of individual vegetable (e.g. Kolmou): Using equation 2.15 and table 4.5 5 (coefficients of quadratic equations),

$$\begin{aligned} \text{Vitamin C (mg/100g)} &= -624.24 + 18.41 \times 62.39 + 1.05 \times 231.97 - 1.1 \times 10^{-2} \times 62.39 \times 231.97 \\ &- 0.13 \times 62.39^2 - 8.10 \times 10^{-4} \times 231.97^2 = 56.72 \end{aligned}$$

Antioxidant activity of individual vegetable (e.g. Kolmou): Using equation 2.15 and table 4.5 5 (coefficients of quadratic equations),

$$\begin{aligned} \% \text{ Antioxidant activity} &= -772.97 + 21.73 \times 62.39 + 1.63 \times 231.97 - 1.67 \times 10^{-2} \times 62.39 \times 231.97 \\ &- 0.14 \times 62.39^2 - 1.34 \times 10^{-3} \times 231.97^2 = 91.71 \end{aligned}$$

b) Moisture content of optimal vegetable mix: % weight of dried Kolmou in the mix (w_k): 10; % weight of dried Jatilao in the mix (w_j): 74.95; % weight of dried Pui in the mix (w_p): 14.05; moisture content of Kolmou: 1.98%; moisture content of Jatilao: 3.02%; moisture content of Pui: 5%.

Using equation 2.19,

$$\text{Moisture content of mix (G)} = \frac{10 \times 1.98 + 74.95 \times 3.02 + 14.05 \times 5}{10 + 74.95 + 14.05} = 3.21\%$$

c) Vitamin C content of optimal vegetable mix: % weight of dried Kolmou in the mix (w_k): 10; % weight of dried Jatilao in the mix (w_j): 74.95; % weight of dried Pui in the mix (w_p): 14.05; vitamin C content of Kolmou: 56.72 mg/100g; vitamin C content of Jatilao: 113.30 mg/100g; vitamin C content of Pui: 131.51 mg/100g.

Using equation 2.17,

$$\text{Vitamin C content of mix (E)} = \frac{10 \times 56.72 + 74.95 \times 113.30 + 14.05 \times 131.51}{10 + 74.95 + 14.05} = 110.38 \text{ mg/100g}$$

d) Antioxidant activity of optimal vegetable mix: % weight of dried Kolmou in the mix (w_k): 10; % weight of dried Jatilao in the mix (w_j): 74.95; % weight of dried Pui in the mix (w_p): 14.05; antioxidant activity of Kolmou: 91.71%; antioxidant activity of Jatilao: 89.50%; antioxidant activity of Pui: 24.92%.

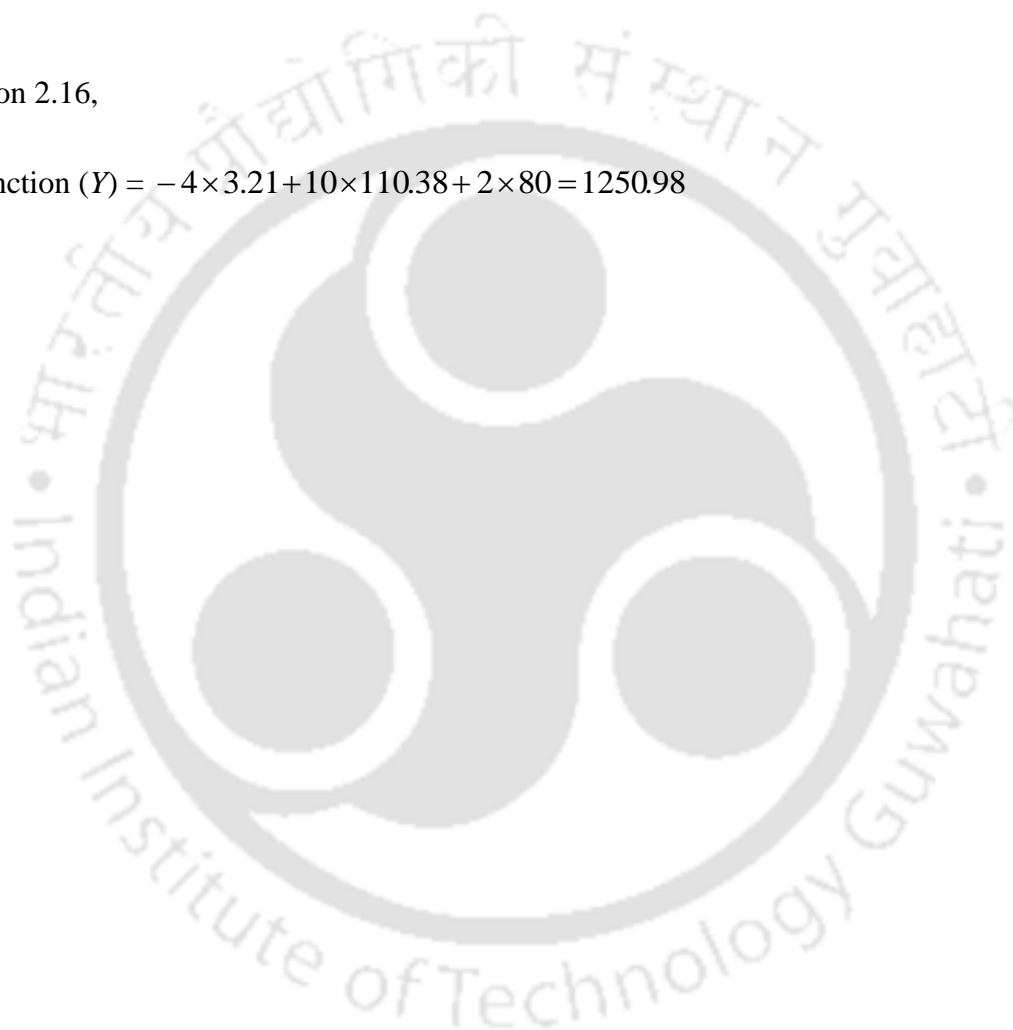
Using equation 2.18,

$$\text{Antioxidant activity of mix (F)} = \frac{10 \times 91.71 + 74.95 \times 89.50 + 14.05 \times 24.92}{10 + 74.95 + 14.05} = 80\%$$

e) **Objective function:** Weight factor associated to moisture content (w_1): - 4; weight factor associated to vitamin C content (w_2): 10; weight factor associated to antioxidant activity (w_3): 2

Using equation 2.16,

$$\text{Objective function (Y)} = -4 \times 3.21 + 10 \times 110.38 + 2 \times 80 = 1250.98$$



Appendix F: Analysis of Variance (ANOVA) Data for Tray Dried Leafy and Non-leafy Vegetable Mix Soup Formulations

In this section, the ANOVA data being obtained for various leafy and non-leafy vegetable formulations has been presented. These indicate significant variations between the alternate soup formulations ($p < 0.05$).

Table F1: Analysis of variance (ANOVA) data summary of leafy vegetable based control and mix soup formulations.

	DF	Sum of Squares	Mean Square	F Value	Prob>F
Model	3	0.86594	0.28865	5.48403	0.0043
Error	28	1.47375	0.05263		
Total	31	2.33969			

Table F2: Analysis of variance (ANOVA) data summary of mix soup formulations with variant ratios of dry leafy vegetable mix to corn flour ratio.

	DF	Sum of Squares	Mean Square	F Value	Prob>F
Model	3	0.50344	0.16781	3.87127	0.0196
Error	28	1.21375	0.04335		
Total	31	1.71719			

Table F3: Analysis of variance (ANOVA) data summary of non-leafy vegetable based control and mix soup formulations.

	DF	Sum of Squares	Mean Square	F Value	Prob>F
Model	4	7.894	1.9735	55.98582	9.99×10^{-15}
Error	35	1.23375	0.03525		
Total	39	9.12775			

Table F4: Analysis of variance (ANOVA) data summary of non-leafy vegetable mix soup formulations with variant Kaskal proportions.

	DF	Sum of Squares	Mean Square	F Value	Prob>F
Model	3	1.4925	0.4975	25.44292	3.77×10^{-08}
Error	28	0.5475	0.01955		
Total	31	2.04			

Table F5: Analysis of variance (ANOVA) data summary of mix soup formulations with variant dry non-leafy vegetable mix to corn flour ratio.

	DF	Sum of Squares	Mean Square	F Value	Prob>F
Model	3	6.01094	2.00365	55.47796	6.65×10^{-12}
Error	28	1.01125	0.03612		
Total	31	7.02219			

Appendix G: Sensitivity of Responses with respect to Drying Process Parameters

Sensitivity refers to the variation of a particular response in due course of the variation in drying process parameters. Henceforth, the highest or lowest value being achieved for each sample is important. In this regard, it is interesting to note that for a significant reduction in moisture content, correspondingly significant enhancement in vitamin C and antioxidant activity has been evaluated. The sensitivity under those circumstances refers to the proportional or marginal enhancement of nutritional characteristics with variations in moisture content. For example, if a 100% reduction in moisture content translates into 100% enhancement in either Vitamin C or antioxidant activity, it indicates the negligible influence of drying process parameters to detriment or increment the evaluated response. Based on all evaluated response characteristics of leafy and non-leafy vegetables, the following table summarizes the associated sensitivity parameters:

Table G1: Summary of response variable sensitivity towards tray drying process.

Sample	Response	Fresh sample	Max/min value	% increase/decrease
Kolmou	Moisture content	86.65	2.87	2919.16
	Vitamin C	2.68	56.14	1994.78
	Vitamin C (mg/absolute dry of 100g sample)	20.07	57.80	187.92
	Antioxidant activity	1.72	91.74	5233.72
	Antioxidant activity (% per absolute dry sample)	1.29	9.45	633.09
Jatilao	Moisture content	87.02	3.28	2553.05
	Vitamin C	5.07	112.58	2120.51
	Vitamin C (mg/absolute dry of 100g sample)	39.06	116.40	198.00
	Antioxidant activity	2.4	88.91	3604.58
	Antioxidant activity (% per absolute dry sample)	1.85	9.19	397.16

Sample	Response	Fresh sample	Max/min value	% increase/decrease
Pui	Moisture content	89.78	3.82	2250.26
	Vitamin C	8.23	130.46	1485.18
	Vitamin C (mg/absolute dry of 100g sample)	80.53	135.64	68.44
	Antioxidant activity	0.47	24.79	5174.47
	Antioxidant activity (% per absolute dry sample)	0.46	2.58	460.46
Kolphul	Moisture content	90.72	3.27	2674.31
	Vitamin C	4.53	85.24	1781.68
	Vitamin C (mg/absolute dry of 100g sample)	48.81	88.12	80.52
	Antioxidant activity	2.27	86.82	3724.67
	Antioxidant activity (% per absolute dry sample)	2.45	8.98	266.93
Kaskal	Moisture content	84.09	2.17	3775.12
	Vitamin C	7.66	88.79	1059.14
	Vitamin C (mg/absolute dry of 100g sample)	48.15	90.76	88.51
	Antioxidant activity	2.18	71.85	3195.87
	Antioxidant activity (% per absolute dry sample)	1.37	7.34	436.00
Komora	Moisture content	96.05	3.85	2394.81
	Vitamin C	12.18	305.41	2407.47
	Vitamin C (mg/absolute dry of 100g sample)	308.35	317.64	3.01
	Antioxidant activity	0.34	23.48	6805.88
	Antioxidant activity (% per absolute dry sample)	0.86	2.44	183.70
Posola	Moisture content	94.49	2.03	4554.68
	Vitamin C	2.47	50.38	1939.68
	Vitamin C (mg/absolute dry of 100g sample)	50.38	51.42	14.71
	Antioxidant activity	0.58	24.67	4153.45
	Antioxidant activity (% per absolute dry sample)	1.05	2.52	139.22

Thus, the following inferences can be deduced from the above table:

- For leafy vegetables, compared to fresh samples, reduction in moisture content was similar (2250.26 – 2919.16%) for all cases. On the other hand, vitamin C and antioxidant activity reduced by 1485.18 – 2120.51% and 3604.58 – 5233.72% respectively. Highest

- enhancement in vitamin C and antioxidant activity were apparent for Jatilao (2120.51%) and Kolmou (5233.72%) cases respectively.
- b) Among all leafy vegetables, compared to reduction moisture content (2553.05%), optimal enhancement in vitamin C content (2120.51%) and antioxidant activity (3604.58%) was observed for Jatilao sample.
- c) For non-leafy vegetables, moisture content reduction varied from 2394.81 – 4553.45% while vitamin C and antioxidant activity reduced by 1059.14 – 2407.47% and 3195.87 – 6805.88% respectively.
- d) Highest moisture content reduction (4153.45%) was observed for Posola. On the other hand, maximum enhancement in vitamin C and antioxidant activity was achieved for Komora (2407.47% and 6805.88% respectively) against its moisture reduction of 2394.81%.
- e) Since, compared to moisture reduction, optimal enhancement in response variables were achieved for Jatilao and Komora samples among leafy and non-leafy vegetables respectively, therefore, maximum proportion of these vegetables has been obtained for leafy and non-leafy vegetables based soup formulations respectively.

Appendix H: Images of Alternate Samples Studied in the Ph.D. Thesis

(a)

Oven Drier



Tray Drier



(b)

Fresh Sample

Tray Dried Sample



Komou

Jatilao

Pui

Kolmou

Jatilao

Pui



Kolphul

Kaskal

Posola

Komora

Kolphul

Kaskal

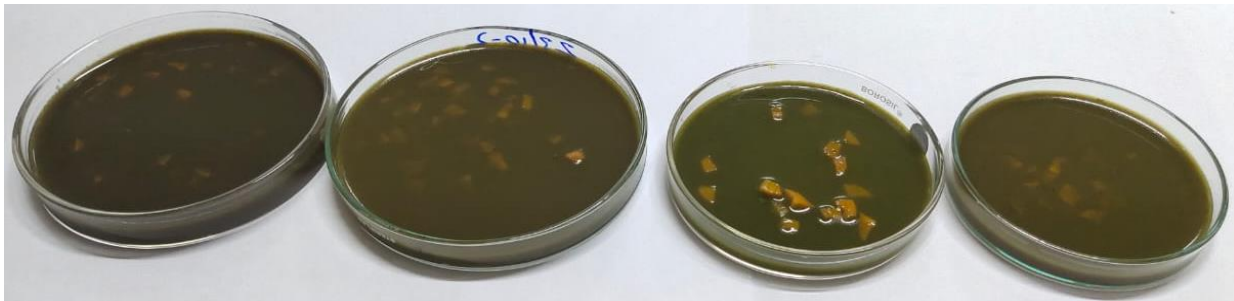
Posola

Komora

(c)

Reconstituted Vegetable Soup Mix Formulation

Leafy Vegetable based Formulations

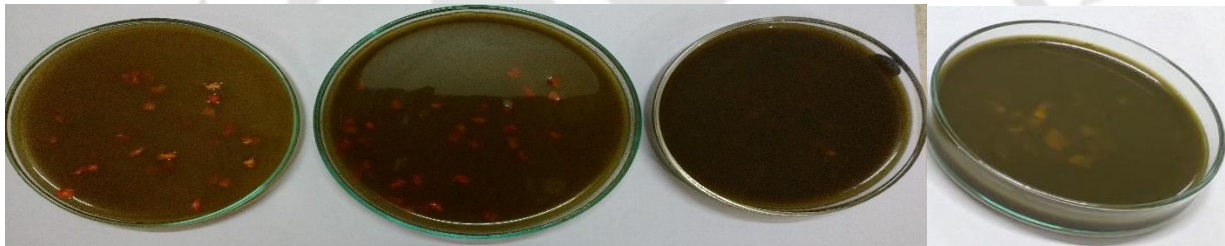


FK

FJ

FP

FM



F1

F2

F3

F4

(d)

Reconstituted Non-leafy Vegetable Soup Mix Formulation

Non-leafy Vegetable based Formulations

