Provenance Study of the Archaeological Pottery of Northeast India

A Thesis Submitted
in Partial Fulfillment of the Requirements
for the Degree of

Doctor of Philosophy

by
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In the loving memory of my Mother

Late Smt. Damyantee Singh

In expression of my thankfulness to my

Father
Shri Indra Pati Singh

Brother
Mr. Krishna Kumar Singh

Sister
Geeta Singh

and

Sister in Law
Smt. Sunita Singh

For their continuous support and encouragement

Declaration

I do hereby declare that this thesis ***“Provenance Study of the Archaeological Pottery of Northeast India”*** is the result of research carried out by me in the Department of Humanities and Social Sciences, Indian Institute of Technology Guwahati, India under the supervision of Dr. Sukanya Sharma.

In keeping with the general practice of reporting scientific observations, due acknowledgement has been made wherever work described is based on findings of other investigators.

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Certificate

It is certified that the matter contained in this thesis entitled, “***“Provenance Study of the Archaeological Pottery of Northeast India***” is original. The work has been carried out by Pankaj Singh for the Degree of Doctor of Philosophy under my supervision and the same has not been submitted elsewhere for a degree.

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Acknowledgements

This thesis is a result of love, good wishes and encouragement of numerous people who have selflessly helped me to complete my research work.

Firstly, I wish to deeply thank Dr. Sukanya Sharma, Supervisor who is my tutor, mentor, inspiration and provided me opportunity to carry out this research work. She always helped me out whenever possible, and answered countless queries.

I am highly indebted to members of my Doctoral Committee—Professor Sukumar Nandi, Professor Alika Khare, Professor Gopal Das and Dr. Shakuntala Mahanta for their continuous suggestions and encouragement throughout my research work.

I am grateful to a few individuals and organizations for their help during research work.

First and foremost our sincere thanks to Dr. S. S. Gupta, Superintending Archeologist, Archaeological Survey of India, Guwahati Circle, and Mr. Sanjay Panda, Asst. Archaeologist, Guwahati Circle for providing us pottery samples from their collection. I am also thankful to Archaeological Survey of India, Guwahati circle and for providing me kind permission to explore and collect pottery samples from various archaeological sites of Sibsagar and Sonitpur (Tezpur) District, Assam. I am thankful to Mr. B.C. Das, BK Nath, Joy Bora, Sunil, Bharat and Yadav Archaeological Survey of India, Guwahati circle, who helped me a lot during exploration in Sibsagar and Tezpur.

I also acknowledge CIF (Central Instruments Facility, IIT Guwahati) for allowing me to use TGA/DSC, Raman Spectroscopy, SEM-EDX and FESEM instruments.

My acknowledgements go to Dr. N. Suresh and Mr. N. K. Juyal, Wadia Institute of Himalayan Geology, Dehradun for helping me to use available facilities such as Optical Luminescence Dating (OSL) and Scanning Electron Microscope and Energy Dispersive X Ray spectroscopy.

I am thankful to Dr. Pankaj Biswas, Department of Mechanical Engineering for his blessing and inspiration. I am also grateful to Dr. Sidananda Sarma (Material Science Laboratory) Department of Physics for Powder XRD and Analytical Equipment Laboratory Department of Chemistry, IIT Guwahati for FTIR Spectroscopy. I am also thankful to The Head, Department of Chemistry, IIT Guwahati for allowing to conduct experiments in departmental laboratory. I am thankful to Dr. S. Kanakraj, Department of Mechanical Engineering, IIT Guwahati for giving me permission to avail instrument facilities in Material Science Laboratory. I acknowledge Dr. Sachin D. Kore for his valuation suggestions. My sincere thanks go to Dr. Ajay Kalamdhad, Department of Civil Engineering, IIT Guwahati for allowing me to the avail facilities in Environmental Engineering Laboratory.

I wish to thank Professor Alika Khare, Department of Physics, IIT Guwahati for her suggestions and support while conducting LIBS and Raman Spectroscopy experiments. I am thankful to Professor Gopal Das, Head of Centre, Centre for Environment, IIT Guwahati for permitting me to use Atomic absorption spectrophotometer and

Trinocular Laboratory Microscope in Laboratory. I also acknowledge Mr. Indrajit and Ms. Ishita Mal in helping me for extracting and analyzing data from LIBS and Raman Spectroscopy.

I am grateful to Department of Humanities and Social Sciences, IIT Guwahati, for providing me necessary infrastructure to complete this work. Lakshminath Bezbaroa Central Library (IIT Guwahati), departmental library of Department of Ancient Indian History, Culture and Archaeology, University of Allahabad and Centre for Archaeological Studies & Training, Eastern India, Kolkata for providing me vital resources for this thesis.

I heartily acknowledge to Dr. J. J. Laskar, Department of Geological Science, Gauhati University for helping me to complete Thin Section Petrographic analysis. It would be almost impossible for me to complete this part of my thesis without his support and contribution.

I wish to deeply thank Professor Prakash Sinha, Department of Ancient Indian History, Culture and Archaeology, University of Allahabad for his enormous encouragement and moral support. I am grateful to the Experimental Archaeology Laboratory for avail facilities of phytolith and microscopic analysis of samples. I am also thankful to Professor Vinod Nautiyal, HNB University, Garhwal for valuable suggestions and discussions. I also acknowledge the support and suggestions of Professor K. Krishnan, MS University, Vadodara.

I am much indebted to Dr. Okram Kumar Singh, D. M. College of Commerce, Manipur, for giving pottery samples from his collection. I heartily thankful Dr. Tia Toshi Jamir, Department of History and Archaeology, Nagaland University for providing me pottery samples from his collections and in time response to our quarries. I am thankful to Professor Abdul Ali Ashraf, Department of Anthropology, Gauhati University, for giving me pottery from his collection. My thanks go also to Dr. Marco Mitri, Department of History, Union Christian College, Meghalaya for providing me pottery samples from his collection from various sites of Meghalaya.

I extremely appreciate help of Sukhamoy, Nazbul, Prasanta, Arghya, Satya and Bappadity throughout my research work. They were part of daily horizon, shared thoughts, laughs, terrible coffees and teas and made my research easy and joyful. I am thankful to Kishor for helping me to analyze Micro Hardness data. I am also grateful to the villagers of Assam who helped me a lot during fieldwork.

I am thankful to Gaurav, Krishnanjan, Lavish and Maharshi for improving and correcting thesis and help me to formatting the same.

I am thankful to all my Allahabadi friends Shailesh, Chandramani, Amit, Awadh, Tulika, Ashutosh, Shailesh, Darshan, Ankur and Saurabh for love, laughter and friendship and words do not suffice to express gratitude to them.

I am thankful to Dr. Naresh Singh, Arya Kanya PG Mahila College, Varanasi for his valuable suggestions and information about ethnography and ethnoarchaeology.

I am obliged to my seniors Madhuri, Tejasha, Rashmi and Parag. Thanks also to Sangita, Bandana, Gunjan, Jimmy, Madhulika, Minakshi, Monmi, Nirmala, Rupan, Pranti and Violeena and all other research scholars from Department of Humanities and Social Sciences for their joyful and lovely company. I am highly grateful to my juniors Priyanka, Momi, Swagata and Jitendra for their love and support.

I wish to thank from the bottom of my heart – Anamika Madam, Aditya Sir, Anupam Sir, Arindam Sir, Kishor, Anand, Subrata, Basant, Mukul, Shyam, Vinod, Shatrughan, Siddesh, Rakhi, Mitali, Trishna, Maharshi, Geetimukta, Abhrodeep, Vinay and Rajendra. My special thanks go to Mr. Aarohan for his precious and sweet talks and making scale. I am heartily thankful to Swapnali madam for her delicious food and her frequent parties. Special thanks go to N K Das Sir, Arun, Gaurav, Subrata, Ashutosh, Shiv and Nand Kishor for doing parties together. Special thanks goes to Dibang Hostel Cricket teammates – Krishna Mohan, Harshad, Arun, Rajeev, Vivek, Hemant, Ravindra, Amar, Amit, Ashwani, Niranjan, Nitin, Ankit, Mirza, Naveen, Punit and Manoj.

Many thanks go at last my family for their understanding, encouragement and patience in my every endeavor of mine. I am highly indebted and obliged to my brothers and sisters Anju didi, Bhupendra, Bindu, and Babita. I am thankful to all my nephews and nieces Vishal, Suraj, Sameer, Sapna, Shanu, Sahil, Anushka and Kittu. This thesis would not have seen the light of this day without their great wishes, immense patience and great sacrifice.

Pankaj Singh
2017

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Abstract

The thesis entitled ***"Provenance Study of the Archaeological Pottery of Northeast India"*** deals with application of scientific methods in Archaeological science. Study of ancient pottery provides information on many aspects of past, which includes the chronology, trade and technology and it helps to identify the different cultural groups and their influence on one another. Traditionally, pottery can be studied from different aspects — artistic, esthetic, archeological, historical, classificatory, mechanical, mineralogical and chemical. Classificatory studies are based on typology of pottery, and it compares similar shape, size and decoration of vessels or sherds which belongs to a specific culture at a specific time. Study of decorative ideas and styles of pottery such as paintings, plastic decoration — incising, molding and applique reveals information about artistic and conceptual perception of the people. Emerging methods of pottery study are archaeometric or scientific analysis, which belongs to mineralogical, chemical and physical properties of pottery.

The main objective of this thesis will be to analyze a representative assembly of pottery from archaeological sites of Northeast India of Neolithic and historical period. Mineral characterization of clay, identification of tempering material, and ascertaining firing range are some of highlights of study. This will help in reconstructing manufacturing techniques of pottery samples studied. This work is part of study that aims to build a broad regional sequence in Northeast India of pottery traditions in prehistoric and historical period.

In present study, scientific techniques have been used to investigate microstructure of selected prehistoric (Neolithic period) and historical pottery samples from various archaeological sites of North East India. Neolithic pottery samples are selected from various sites such as Daojali Hading– Assam; Nongpok Keithelmanbi and Napachik – Manipur; Barapani, Sophet, Sawmer, Law Nongthroh and Gawak Abri – Meghalaya and Ranyak Khen – Nagaland. Historical pottery samples are selected from Ambari, Sri Sri Suryapahar and Bamuni Hills – Assam and Kangla Fort – Manipur. Modern pottery samples are selected from Hirapara, Khehenipara – Kamrup district and Kumargaon-Dergaon, Kumargaon-Dhekial – Golaghat district, Assam.

Various analytical techniques have been used in present study to achieve these objectives: Powder X-Ray Diffraction (XRD), Thin Section Petrography, micro-Raman Spectroscopy, Scanning Electron Microscope (SEM)-coupled with Energy-dispersive X-ray spectroscopy (EDX), Micro Hardness Tester (MHT), Mohs' Scale and Laser Induced Breakdown Spectroscopy (LIBS).

The content of this thesis entitled ***"Provenance Study of the Archaeological Pottery of Northeast India"*** have been divided into five chapters based on results of experimental work carried out during research period as follows –

Chapter 1: Introduction

First chapter is introductory chapter. This chapter will discuss conceptual details of study of pottery in archaeology. Scope of interdisciplinary approach and application of scientific methods or techniques in study of archaeological ceramics is also discussed. Physical, Mechanical, and Thermal properties of ceramic and clay mineralogy will be discussed in this chapter. Pottery production methods and potters' accessories will be discussed in brief. Apart from this, previous scientific studies done by Indian scholars on archaeological pottery from India will be discussed. Lastly, this part will discuss objective of present study and methodological framework of present work.

Chapter 2 Mineralogical and Physicochemical analysis of Neolithic pottery

Present chapter attempts to examine mineralogical and chemical compositions of ceramics using several analytical techniques. Two pottery samples, one from Daojali Hading and other from Gawak Abri, have been dated using OSL method.

Chapter 3 Mineralogical and Physicochemical analysis of Historical Pottery of Assam

A selection of 49 potsherds from Ambari (AMB) Kamrup, Shri Shri Suryapahar (SPR) Goalpara, Bamuni Hill (BMH) - Sonitpur district, Assam dated from 7-12th century AD and Kangla fort (KGF) - Imphal Manipur are studied applying scientific techniques. This chapter will investigate mineralogical and chemical properties of pottery samples from different archaeological sites of Assam from historical period such as – Ambari, Suryapahar, Bamuni Hills, and Kangla Fort from Manipur.

Chapter 4 Ethnographical Investigation of Pottery Tradition in Assam

This chapter presents an ethnographic account of Kumar and Hira potters of Assam on basis of the fieldwork undertaken during course of study. Kumar potters make potteries using wheel while Hira's make pottery by hand. Main objective of this chapter is to compare provenance of archaeological pottery and contemporary pottery, to understand evolution of craft of pottery making in Assam. This will indicate changes and development that has happened over years in pottery manufacturing in Northeast India in general and particularly in Assam.

Chapter 5 Conclusion

Concluding chapter will be summing up basic findings of present research work. By this study we will be able to draw attention of future researchers to way in which studies of this type can provide results which is valuable and vital for correct interpretation of archaeological pottery.

“*When you hold a pot in your hands, when you go over its walls with your fingers, you feel the hands of the potter, his touch. You may not know who he was or what he looked like, but, handling the pot, be it hundreds or thousands of years old, you can still feel the imprint of his hands. It is this fact about a pot that makes it so endearing, so very personal. It makes the physical handing of a pot such an important part that of its appreciation, as important as its visual impact, and at times even more so*”.

‘Ceramics’ Natzler Otto, 1968, pp. 38-39

Chapter 1
Introduction

1.1 Introduction

The past does not come to us in a readily contoured form but has to be reconstructed from the varieties of the evidence left behind by it. The remains of the human past have to be recognized, categorized, investigated and assessed to interrogate and draw conclusions about it. The disciplines of history and archaeology are central to this endeavour.

Archaeology is the science in which we study the past cultures through the material remains in the context of time and space which is left behind by the people. These materials may be either small artefacts, such as micro-blade, or big structures, such as pyramids. Anything that people create, alter or use can be part of the archaeological record.

An archaeologist study remains, which is left behind by human beings. This study helps to understand the relations of physical culture with the natural, social, economic and political atmosphere and recreate all facets of past culture, from day-to-day lives of common people to the Kings (Griffiths, 1999).

The study of past human cultures by archaeologist involves the determination of chronological framework and analysis of the biological characteristics of man, his natural surroundings, his means of survival and his physical culture (Tite, 1972, p. 1). From this data, the archaeologist can recreate economic, political, social and ideological systems of the human culture. To accomplish this objective, archaeologists require extracting the maximum possible amount of information from the available materials and adopting a wide-range of multidisciplinary approach to human culture (Tite, 1972, pp. 1-2).

In recent decades, there has been growing application of scientific methods in the field of archaeology. The interdisciplinary approach of archaeology has been significantly encouraged by the archaeologists and is now considered as an integral part of archaeological studies.

Archaeologists are able to extract valuable information about the use and origin of archaeological objects and materials that were used in analysing the ancient artefacts or material remains with the help of analytical techniques. For example, the chemical and mineralogical analysis of pottery from a particular location can give information regarding the origin or source that allows scholars to draw conclusions regarding the communication, trade and partnership with other locations. It provides information regarding the available techniques in processing materials and subsequently in obtaining the final product.

Archaeological Pottery throws extensive light on nature, culture and the development of society. The study of pottery in Archaeology provides information on chronology, trade and technology etc. and it helps to identify the different cultural groups and their influence on one another.

1.2 Definition of Ceramics

Pottery was the first man made synthetic object. It combines the four basic elements – earth, water, fire, and air. The term ‘ceramic’ is derived from the Greek word ‘keramos’ which means – “burned stuff” or “earthenware.” Kingery *et al.*(1976, p. 3) defined ceramic as – “the art and science of making and using solid articles that have as their essential component and are composed in large part of, inorganic non-metallic materials.”

The word ‘ceramic’ has two sets of meaning, which complicates its precise definition and usage.(Rice, 1987, p. 4)

- First is common in material science
- Second can be employed in art and archaeology

In the context of material science, ceramic refers to composite of metals and non-metals or sometimes to materials made from silicate like clay and hardened by applying heat. The term also incorporates ceramic science, ceramic engineering, and ceramic industries. Within overall ceramic fields, pottery is one of the specific productions that include low-fired tableware and utensils made by clay (Rice, 1987, p. 4).

In archaeology, the term ceramic refers to cooking and serving utensils and other objects that are made of clay. The term ceramic refers to high-fired, usually glazed and vitrified objects of clay, while pottery refers to low-fired, un-vitrified objects such as cooking and storage vessels (Rice, 1987, p. 4).

1.3 Importance of Pottery in Archaeology

Pottery has a long history of the process of manufacture and use. Traditionally, pottery can be studied from different aspects – artistic, aesthetic, archaeological, historical, classificatory, mechanical, mineralogical, and chemical (Rice, 1987, p. 24).

Pottery is one of the most important sources of information for the archaeologists, because, once clay is fired to a sufficient temperature and for a sufficient length of time, clay is indestructible (Gibson & Woods, 1990, p. 6). Pottery is regarded as the most visible sign of the presence of old settlement in an area. Pottery is found profoundly in archaeological sites from the Neolithic period throughout the world and its various functions, its transportability, and its infinitive forms and decorations make pottery a very complex tool for defining stylistic changes through time and for tracing cultural relations. Archaeologists use pottery to decode how various areas within the archaeological site was used. For example, one would expect to find high density of pottery in a food preparation or cooking area, but would not expect to find large number of it in sleeping area.

Pottery can be used to determine a person's social status, gender, or even possibly their connection to others in the group. According to Rice (1987, p. 24), pottery is restricted to the habitations and burial area of the upper stratum of society. Certain kind of pottery may be restricted to the elite or may be used only for mortuary usage.

The study of manufacturing techniques of pottery is useful for archaeologists because pottery making is an additive procedure in which the sequential stages are noted in the finished products. Selection of raw materials, mould, decoration, style, composition and manufacturing techniques of pottery and its usage reveals information about human behaviour (Rice, 1987, p. 25).

According to Rice (1987), one of the archaeologist's traditional aims in excavating an ancient site is to determine the date, and pottery is a significant object in determining chronology. Pottery enables us to reconstruct both types of dates – relative and absolute. Relative dating methods are based on the typology of ceramics such as types, shapes, size, decoration and design, for instance; Northern Black Polished Ware (NBPW) presents a strong base for pre-NBP cultural sequences of northern India. On the basis of this ware can be used to determine the cultural sequences of the later period of NBP. According to Mortimer Wheeler, "NBP is as special in Indian sites as in Europe 'Terra sigillata' has in the coastal area of Mediterranean Sea".

Absolute or Chronometric methods yield dates in absolute time or calendar year. Absolute dating methods are – radiocarbon, hydration, dendrochronology, electron spin resonance, archaeomagnetic, thermo-luminescence and potassium-organ dating. Among these radiocarbon, electron spin resonance, archaeomagnetic, thermo-luminescence and optical stimulation luminescence dating is useful for ceramic materials.

There are three aspects of archaeological pottery's studies: classification, decoration, and composition (Rice, 1987, pp. 25-26).

Classificatory studies of pottery are based on the typology of pottery and compare similar shape, size and decoration of vessels or sherds which belong to a specific culture at a specific time. These typological studies are the bases of relative dating.

Analysis of the decorative ideas and styles of pottery, whether expressed in painting or plastic decoration-incising, moulding, applique, has always yielded insights into the lifeways of a people as well as their artistic perceptions and conceptual structures.

The third and increasing aspect of pottery study is the scientific analysis, which focuses on the paste or composition of ceramics and the properties discussed by that composition.

The legacy of the developments of scientific methods and statistical techniques in recent years can be traced to the technical analysis of pottery. Scientific methods have showed a significant role in the study of the manufacture of archaeological pottery. For instance, thin-section can be useful to know the technical differences such as hand-made versus wheel-throwing (Hodges, 1962). Thermal expansion is useful to estimate firing temperature (Roberts, 1963) and scanning electron microscope is useful to short out the wide range of technical questions (Tite & Maniatis, 1975). Scientific techniques can be used in three areas of ceramic studies – dating, sourcing (provenance studies) and the study of function.

The provenance studies belong to the geographical or geological origin or source of archaeological artifacts. For this purpose, chemical and mineralogical analysis can be carried out to determine the provenance of the pottery that is more useful than other objects.

Functional studies belong to the study of function. Four ways to make progress have been suggested (Orton, *et al.* 1993):

i. To examine the associations of pottery types with the stratigraphic features in which they are found (Millett, 1979)

ii. To examine the residues of original contents or surface treatments.

iii. To examine the physical properties of pottery fabrics to assess their suitability for various functions, such as cooking (Steponaitis, 1983; Bronitsky & Hamer, 1986).

iv. To examine wear marks on pots (Griffiths D., 1978; Hally, 1983).

1.4 The role of interdisciplinary approach in the study of pottery in Archaeological Science

The multidisciplinary or more precisely interdisciplinary approach is the knowledge coming from the study of integrating work through the contribution of different disciplines in a certain project (Ramirez, 2008). In each case, results are assessed, associated, and conferred to obtain, produce and create it as collective knowledge. This study is an effective progress headed for considerate results that are overcome in a particular time and place. For applying an interdisciplinary approach to the particular study, the key objectives should be clear and suitable for the project requiring the involvement of different disciplines. All disciplines will achieve its specific points, without deviating from the key objective of the project.

One of the key objectives of Archaeology is to understand the means of human societies, processes of change in cultures, communication with their surroundings and with other human societies. In addition to this, archaeologists must encourage the interdisciplinary approach to realizing this objective (Ramirez, 2008). For instance, Geography helps to understand the settlement patterns and resources that ancient societies used in the past. The geophysical and geochemical methods help to rediscover the archaeological sites. The ancient societies consumed the types of plant and animals and this can be known by applying botanical and zoological techniques respectively. Physical and Chemical Sciences is also helpful to determine absolute or chronometric dating of the artefacts and sites under investigation.

The interdisciplinary approach in the study of ancient pottery is playing a significant role in Archaeological Sciences. The usefulness of ancient pottery in the archaeological study always attracts archaeologists and other discipline experts to analyze the pottery through multidisciplinary or interdisciplinary approaches. Archaeological methodology is thoroughly taken from other sciences and disciplines especially from physics, chemistry and earth sciences. The use of technology in study of pottery can be divided into two groups –

I. Scientific techniques
II. Non-scientific techniques

The study of pottery and its description is divided into four aspects: physical properties, composition of materials, technique and style. Individually or separately these aspects cannot described the features of pottery. Because potter's techniques and materials affect the physical properties directly and also use of manufacturing techniques are affected by the nature of the material and technique affect the style of pottery (Shepard, 1956, p. 95).

1.5 Applying Scientific Techniques in the study of archaeological pottery

Scientific techniques or methods have been borrowed especially from physics and chemistry for investigating the physical and chemical properties of pottery. Traditionally, pottery can be studied from different aspects – artistic, aesthetic, archaeological, historical, classificatory, mechanical, mineralogical and chemical. Figure 1 shows the diagram of classification of scientific techniques for the study of archaeological ceramics.

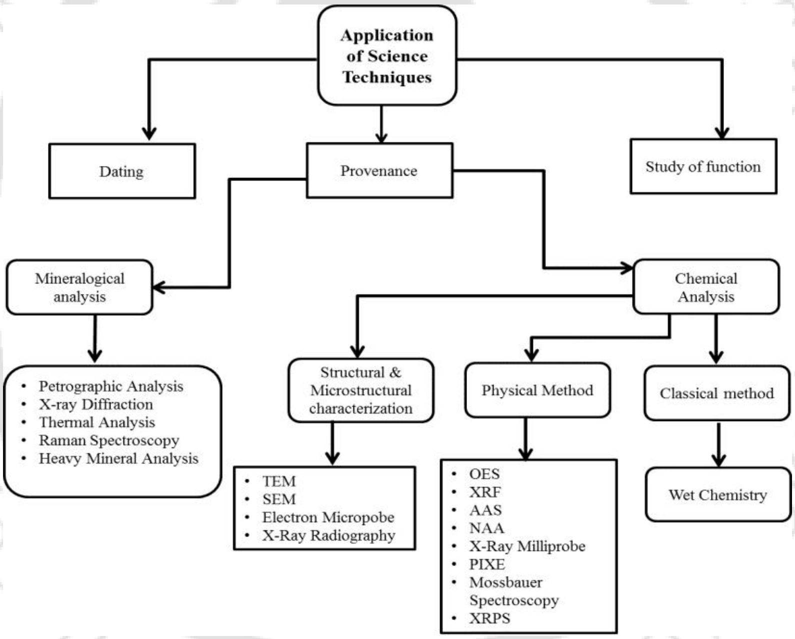

Figure 1.1 Diagram of classification of scientific techniques for the study of archaeological ceramics.

The key methods for scientific study of pottery are -

- ***Technological studies*** of ancient pottery that is based on the methods, by which clays were prepared, shaped, finished and fired.
- ***Scientific Characterization*** of pottery deals with the examination of the properties of ceramics and it involves in their mineralogical composition, chemical composition, microstructure, surface traits and provenance studies (Peacock, 1970, pp. 375-76).

In Archaeology, the essential objectives of scientific studies of artifacts are to reveal two main types of information (Gibson & Woods, 1990, p. 17):

- The composition of the material of artifacts and its *provenance*.
- The technology employed in artifacts.

The ancient pottery has more potential to yield both types of information than any other archaeological artifacts.

1.6 Ceramic Materials: their composition, sources and properties

The manufacturing techniques or throwing or modelling pottery or terracotta figurine are simple, but it is laborious and time-taking procedure. Generally, the raw materials of a pottery production are clay, water, non-plastic inclusions, and fuel. The procedures of pottery production are clay preparation, shaping, decorating and firing. Rice (1987, p. 31) discussed that among all the materials and procedures involved in manufacturing pottery, clay, and its handling processes are the most important. Hence, it is appropriate that discussion on pottery production processes must start with the raw materials: clay and their origin, composition, and properties.

1.6.1 Clays

Rice defined clay as a fine-grained, earthy material that contains particular groups of substances viz. rocks and soil and having certain particle size. The main property of clay is its plasticity when moistened (Rice, 1987, p. 36).

According to Shepard (1956, p. 6), clay is a fine-grained, earthy material that grows plasticity when mixed with water. Main chemical constituents of clay are silica, alumina, and water. Also there is significant quantity of iron, alkalis, and alkaline earth.

As defined in the fields of geology, soil science, agriculture, branches of engineering and the construction industry, “clay” refers to a specific particle-size range; that is, particle smaller than 2 micrometers, or two–thousands of a millimeter (0.002 mm) in diameter.

Figure 2 given below, show the clay is extracted from rocks and rocks are derived from rock-forming minerals. Silicate is the most general rock-forming minerals and silica is key minerals in its composition. The percentage of elements in the composition of silicates minerals are feldspar with slightly higher percentage of 39%, quartz 28%, Ferro-magnesium silicate 2%, Clay minerals and mica 18% and Carbonates 9% (Rice, 1987).

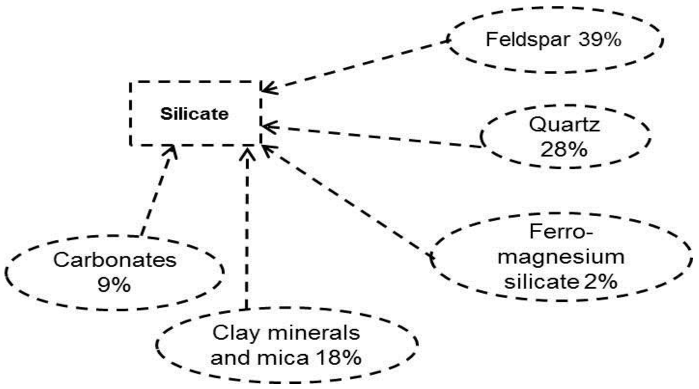

Figure 1.2 Flow chart of rock-forming minerals of clay

1.6.2 Physical Properties of Clay

a. Texture

Texture can be defined as follows-

i. The quantity, size, shape and alignment features of particles in clay materials.
ii. The general character of a fired fabric as viewed in cross-section, defined about particle size (fine vs. coarse), physiognomies of crack (friable, blocky), and density (dense vs. porous) of a pot.
iii. The surface quality of a pottery.

The fineness of texture effects clays' workability and firing behavior and also has the utmost effect on plasticity and fusibility.

b. The colloidal state

The colloidal state is defined as a very fine particle either removed or spread in another substance (Rice, 1987, p. 76). Usually, the diameter of colloid is 1 µm or less (Shepard, 1956, p. 13). There are two type of colloidal substance which are important in understanding clay deposits: first is inorganic colloid that represented by very fine clay mineral particles and the second is organic colloid that affects plasticity, dry strength and color of fired clay piece.

c. Particle shape

Simply, clay particle shape is defined as the size of clay. Rice (1987) mentioned that clay particle size range is smaller than two µm. The shape of clay particles affects the process in which they will pack. The hexagonal, coarse crystal of kaolinite has been identified, and it is also visible through electron microscope and shape is retained even in very fine clays (Shepard, 1956, p. 14). Furthermore, the clay particle can be identified in thin sections of pottery which is not fired at sufficient temperature that can terminate the crystalline structure of the clay minerals.

d. Plasticity

Plasticity is the property of clay that permits it to be moulded when moist and to grip this shape when the shaping forces are removed. During the drying process, the property of plasticity is lost. But the shape will be retained (Rice, 1987, p. 58).

There are numerous factors affecting plasticity:

i. Clay particle size
ii. Clay particle shape
iii. Surface tension of the water
iv. Adsorbed ions
v. Clay mineral component
vi. Clay deposit location
vii. Organic content
viii. Non-clay mineral component of the material
ix. Temperature

e. Base Exchange capacity

The Base Exchange capacity can be defined as the maximum ability of clay to absorb ions which affect the scattering of clay particles. Base Exchange capacity is measured as milli equivalents/100 grams of clay (Shepard, 1956, p. 15).

f. Color

The organic materials and iron composite affect the color of raw clays. Firing processes also affects the color of clay. In addition, if clays are moderately free from contaminations, it will be white. On the other hand, the organic compound and its mass and condition make clay gray to blackish. The presence of hematite, hydrated forms of ferric oxide, goethite, and limonite, makes the color of clay red, brown, buff, and yellow (Shepard, 1956, p. 16).

g. Slaking

Slaking is the rate of disintegration of clay which is absorbed in water. Clays is significantly change by slaking time and affects the plastic state (Shepard, 1956, p. 17).

h. Drying properties

Drying property can be measured by alteration in either length or volume of clay. Due to excessive drying shrinkage can crack the pottery surface (Shepard, 1956, p. 18).

1.6.3 Impurities of Clay

The inclusions and impurities naturally occur in the deposition of clay. Clay commonly have quartz, feldspar, mica, carbonate, iron oxide, titanium and numerous rare minerals and sometime the presence of soluble salts, organic material and calcareous shells in clays as impurities affect the properties of clay and particularly its response to temperature.

1.6.4 Effects of Heat on Clay

Plasticity of clay is lost due to heat. It loses water due to drying in air, and it continues under increasing temperature. Due to heating, clay becomes hardened, and clay mineral structure changes. Continuously, increasing temperature affects the clay properties and water system causes permanent physical modifications (Rice, 1987, p. 80).

1.6.5 Stages of firing

There are three phases occurring in clay properties or ceramic body during firing – dehydration, oxidation, and vitrification. These stages occur at a particular order and each stage starts after the previous one ends completely and firing continues. A brief description of all 3 three stages are below: (Shepard, 1956, p. 20).

The first stage of firing is dehydration. In this stage, the dehydration of clay minerals occurs. Therefore, the porosity of ceramic body increases and plasticity is lost. During dehydration impurities like limonite and goethite are also get dehydrated.

The second stage of firing is oxidation. In this stage the oxidation of impurities like carbon, iron and sulfur occurs. The carbon has maximum affinity towards oxygen. Therefore, the carbon gets oxidized rapidly followed by iron and other impurities. This is chemical change stage unlike the preceding one (Shepard, 1956, p. 21).

The presence of carbonaceous materials in the clay composition increases the temperature of the samples as it reacts with oxygen to form carbon monoxide and dioxide, which are released in the form of gases. The over-firing is possible if the higher amount of carbonaceous materials present in the samples and the ratio of combustion are unconstrained.

Some types of carbonaceous constituents start to oxidize around 225 °C and carbon deposited by smudging oxidizes gradually below 500 °C. Above temperature range of 700-800 °C, carbon may be transformed to a denser form which is not easy to oxidize. Additionally, above this range, vitrification may begin, which obstructs the passage of oxygen.

1.6.6 Effect of firing on the physical properties of pottery

The modifications in the physical properties of pottery which take place throughout the firing process are closely associated with the chemical and mineralogical changes. Porosity is increasing with the formation of gas, when ingredients are dehydrated or disintegrated. Moreover, the reduction of porosity occurs due to the sintering and fluxing and cause shrinkage as elements are formed together and bonded. The process of vitrification is another reason for the reduction of porosity and materials melt and fills pore spaces and forms finer constituents in the matrix (Shepard, 1956, p. 23).

The color of ceramic body may be affected by several factors as discussed by Shepard (1956, p. 24) –

- If carbonaceous materials are not completely oxidized, the ceramic body will be gray or a greyish tone of brown or buff.
- At the temperature of 800 °C, most of the iron bearing minerals can be completely oxidized and achieve its optimum color in the oxidation range.
- Magnetic iron oxide is formed due to oxidizing of ferric oxide at 1000 °C and it persists up to 1300 °C, though in oxidizing condition.
- With the starting of vitrification during firing process, clays begin to lose their optimal color of ferric oxide.
- Additional alumina in some circumstances gives to the formation of a buff or yellow color.
- Presence of lime also affects the color of ferric oxide and it may be red to buff when the lime initiates to have a softening impact on iron-bearing clay, as it becomes well oxidized and then sintered.
- Vitrified calcareous clays usually have a distinct olive or greenish buff color (Shepard, 1956, p. 24).

1.7 Physical, Mechanical and Thermal properties of Pottery

Pottery study focuses on the properties which affect the original usage of the vessels – its capability to carry liquids, to allow loads, to endure unexpected heating and cooling, and to bear impact. The ceramic properties such as physical, mechanical, and thermal give information about use, manufacture techniques and the origin of the raw materials (Rice, 1987, p. 347). These properties can be easily studied by using comparatively simple and fewer equipment procedures. But, others need specific techniques and tools of physical and materials sciences/engineering. These methods allow us to measure and provide the detail reports of significant physiognomies of a ceramic by accurate and reproducible standards. These physical characteristics are: hardness, strength, microstructure, porosity, and thermal properties (Bronitsky, 1986, pp. 209-16). The brief descriptions of these properties are discussed below.

1.7.1 Microstructure

According to Rice (1987, p. 348), the usages of ceramic properties are functions of its inner structure and it has two aspects, which are significant in the investigation of the physical, material and thermal properties of ceramics:

i. The arrangement of atoms within the constituents of a pottery.
ii. The arrangements of these constituents which relate to each other.

In engineering terms, pottery can be explained as a substance containing more than one phase. Kingery (1960, p. 248) defined that ‘a phase may be any part of a structure that is homogenous in nature and confined by a surface which is mechanically divisible from other part of the structure’. In ceramics, phase comprise separate grains, glassy material, and pores. The connections between these phases are essential because during firing and cooling they are continuously altering with chemical and physical properties of ceramic. Throughout these steps, the system adjusts to attain minimum free energy and approach thermodynamic equilibrium at expected temperature, pressure and arrangement. Phase diagrams (Levin, *et al.* 1964) are graphic demonstration of the composition of all phases, their quantity with respect to temperature variations. The complex arrangements of phases contain crystalline and amorphous material, voids, and the borders between them, is called the microstructure of the materials (ceramics).

The microstructure in the low-fired ceramics is determined by its raw materials, firing processes and sintering. However in case of high-fired ceramics the equilibrium between various phases, alterations due to the vitrification and high-temperature mineral formation are the determining aspects for microstructures (Rice, 1987, p. 348).

1.7.2 Porosity

Porosity is one of the key properties of ceramics. Typically, all ceramics ingredients encompass pores or voids, spaces present between or within the dense specks.

The porosity can be defined as the "proportion of the quantity of pore space to the total volume of the section. The size and shape of pores affect density, strength, penetrability, surface area for catalytic reactions, degree of resistance, range of staining by fluids, destructive action and ratio of efflorescence of solvable salts, and resistance to thermal shock" (Rice, 1987, pp. 350-51). Characterization of pores depends on their size, shape, and place in the ceramic surface.

1. *Open pores* may be classified into:
    a. *pocket pores* which have a tinny hole or neck,
    b. *micro pores* which are unable to contain liquid under average pressure,
2. *Loop pores*- two correlated pores
3. *Channel pores* - link two pores or a pore and the surface.
4. *Continuous pores*- expanded from one surface to the other;
5. *Blind-alley pores* - enlarged from the surface without other links (Grimshaw, 1971, pp. 417-419).
6. *Closed or sealed pores:* it occurs naturally the body of pottery either without any outer connection or developed during firing due to opening are sealing due to shrinkages or vitrification (Rice, 1987, p. 350).

1.7.3 Hardness and Strength

Hardness is one of the features which can help to judge the serviceability of ceramics. Hardness has several meanings such as resistance to penetration, abrasion, scratching, crushing, and resilience or elasticity (Shepard, 1956, p. 113).

The hardness of fired clay/ceramic is affected by several factors such as type of impurities, microstructural characteristics, surface treatment, and firing condition. Hardness test can be measured by penetration, abrasion, elasticity, magnetic and electric properties. The most common test is penetration or scratch type done be applying mechanical processes. Commonly, the Mohs' scale is used by mineralogists and archaeologists in the field test.

The term *'Strength* refers to the capability of the materials or ceramic to tolerate several stresses without breaking, cracking, collapsing, or scraping. Strength is studied in terms of resistance to the different mechanical strains (stress = nomal force per unit area) which is applied on materials (Rice, 1987, p. 357).

1.7.4 Thermal properties and stresses

Properties of clay materials are significantly and continuously changed, when it is heated at uniform temperatures or in changing firing atmosphere.

The thermal activity of a ceramic may correlate in two conditions: in the original firing of clay and in the usage of fired pottery during cooking. The expansion of ceramic or changes occurred in distinct constituent or phases are due to these situations and cooling. It is also cause of occurrence of stresses within the body of ceramic (Rice, 1987, p. 363). The thermal stress can be classified into four main categories as following –

I. *Intrinsic Properties:* Integrated as variables into the calculation for guessing thermal shock resistance. These are – modulus of elasticity, strength, thermal conductivity, and thermal expansion coefficient.
II. *Temperature:* The ratio of change in temperature hugely affects vulnerability to thermal stress. Sudden change in temperature is the reason of severe reactions known as thermal shocking. Gradual change in temperature reduces the thermal stresses in the body and permit for slow transmission of temperature without unnecessary strain.
III. *Microstructure:* It can be controlled during pottery production. The key properties are particle size, porosity and hardness.
IV. *Shape and design:* Thermal stress resistance affected by the design, form, dimension, width and surface finish (evenness and glaze) of ceramics.

1.8 Pottery making techniques

Pottery production is a timeless occupation, and the best of pots through the ages have a quality of timelessness about them that transcend chronological and cultural boundaries.

Sankaliya (1970) discussed following methods or techniques in the making of pottery:

1. Handmade

    - (i) Moulded in
        - (a) A basket
        - (b) A pot
    - (ii) Coiled
2. Partly handmade and partly wheel made
3. Made on a turn table
4. Wheel made

The pottery production stages are clay preparation, forming, finishing, decoration, and firing.

1.8.1 Wheel-Throwing

A. Clay Processing

Primarily, potter's clays are sediments extracted from river beds, lakes or ponds. Clay is also formed due to the decomposition of rocks, such as decomposed granite make Kaolin or Chinese clay.

In the geology, clay is sedimentary deposit divided into two types – primary or residual and secondary or transported clay. Primary clay is formed in situ and remains in their origin location in which they are developed. Secondary clay is formed in deposits or beds which have travelled for some distance from its source of origin. The clay can be carried by waves, tides, streams, wind, glaciation, erosion etc.

There are three techniques of clay processing:

- ***Cleaning:*** Eliminating the pebbles, fine gravels, roots and other ingredients from the fully dried and crushed clay is called cleaning. After cleaning, clay is set for successive procedure of mixing and kneading.
- **Mixing:** Selection of clay is important, because all kinds of clay are not appropriate for pottery production. Sometimes, to make clay appropriate for pottery production, two or more types of clay are added in required ratio. Tempering materials such as sand, ash, and cattle-dung is added as inclusions to increase quality of clay.
- ***Kneading:*** Kneading is the last stage of clay preparation. The large quantity of clay is bundled into lumps and carefully squeezed by hand and foot. Sometime, long and cylindrical wooden bat is used for the same.

B. Throwing

In throwing, a chunk of prepared clay is positioned on the rotating wheel-head. Then, the potter chunks by his fingers cautiously to giving it a hollow shape. There are eleven successive stages in throwing:

1. Centring
2. Coning
3. Raising up
4. Plunging
5. Drawing up
6. Thinning the wall
7. Forming
8. Collaring
9. Smoothing
10. Cutting off
11. Removing

The completion timing of all stages of throwing depend upon several human and mechanical factors, such as – speed of the wheel, strength of potter, quality of clay and size of pot.

C. Beating and Enlarging

In beating techniques, potters use beater (generally wooden) on leather-hard surface of vessel in order to modify shape, size, surface features and compress the paste. The paddle and anvil methods are the most common techniques of beating. In paddle techniques, a flat or concave beater is used on leather-hard surface of vessel. In anvil techniques a convex or circular objects made of stone or clay is used on the surface of pottery and usually it is used in interior surface of pottery. Both techniques significantly affect the final characteristics of ceramics. The beating techniques is useful for bonding of segments, eliminate coil marks or irregularities, thinning walls, even the surface and modify or expand outlines of vessel.

1.8.2 Hand-Moulding

Hand-moulding may be categorized into three main types as follows:

a. Pressing

In pressing method, a chunk of clay is pressed in hand using thumb and fingers. The pressing technique is considered as earliest method of moulding ceramics now being applied in current pottery art in the name of press work (Saraswati, 1979, p. 6).

b. Moulding

Moulding technique is instigated from pressing; a lump of clay is pressed deliberately into shape. The shape of vessels may be convex when clay employed on the exterior part of the vessel body or it may be concave when clay applied to interior part of vessel body.

c. Strip and Coiling Methods

In this technique every circuit of a pottery is shaped by a distinct strip of clay extended to merge one upon another.

Coiling is one of the most common pottery making methods, in which rolls of clay are coiled one above other to manufacturing pottery. The word coiling may refer to ring building, segmental coiling or spiral coiling (Rice, 1987, pp. 126-127):

- In ring building separate rings of clay are placed in distinct sequences atop one another.
- In segmental or composite coiling, every ring shaped course is made of numerous sections instead of a single coil of clay which measures complete boundary of pot.
- In spiral coiling, pots are spiralling coil of clay.

1.8.3 Finishing and Decoration

Throwing or hand moulding may give only form to the vessel. Additionally, further treatment is required to give final shape to pot. There are numerous processes of manufacturing like shaving, polishing, painting, engraving, stamping and inlay which are applied for finishing pot.

Shaving: The process shaving is done on leather-hard surface of a pot whenever it is required. It is specially done for the small pots/cups and smoking pipes. It is done either on wheel or without a wheel.

Slipping: Slipping is done on dried pottery and it gives to vessel either bright red or black color depending on the firing methods. The slip preparation technique differs with kind of ochre. The objective of slipping is to cover excess pores in pot and provide lustre to vessel.

Polishing: The key objective of polishing vessel is to brighten the body. Polishing is done using pieces of cloth, pebbles, glass beads etc. which is made for polishing purpose.

Painting: The process of painting can be done before or after firing. If painting is done before firing, any inorganic color like ochre or natural earthy materials is used. After firing, potters have freedom to use both inorganic and organic color to paint vessel.

Stamping and Inlay: Stamping and inlay may be employed on both handmade and wheel-thrown pottery. The imprints are obtained on soft body before drying by direct pressing stamps.

Engraving: Similar to stamping, engraving is also done before drying or moist body of vessel by using numerous sharp and pointed tools when the surface of pottery is leather-hard.

Cut Facets: Like engraving, cut facet is done using sharp edged tools such as knife or blade on half dried pottery. This method is same to one applied in incising ivory or wood.

Perforation: Perforation is done using sharp needle on a vessel when body is stable to touch.

Relief Design: Additional clay is used for making relief designs on exterior surface of pottery.

Drying: The finished products should be dry before firing. Entire drying process takes several days, weeks or one month depending on weather. The vessels may be dried in air, sun-light or in heated sheds.

1.8.4 Methods of Firing

The key objectives of firing are to harden clay, increase workability and durability of clay objects. When clay is fired above 550-600 °C, the initial chemical and physical alterations begin in clay (Rice, 1987, pp. 90-93). Skibo and Schiffer (1987, p. 85) stated that the pottery which are fired at temperature of 550-600 °C will ultimately disintegrate when dipped in water. There are three main types of pottery firing techniques:

1. **Open Firing:** Open firing is also known as clamp or bonfire firing. In open firing methods, fuel is in direct contact of vessel. It is piled on the ground or in a shallow dugout. In open firing method vessels pile over a stratum of fuel and place more fire wood among, above and around the pots. Then, entire pile is covered with more fuel. The most noteworthy characteristic of open firing is that temperature increases fast during initial

stage and duration is short. According to Saraswati (1979, pp. 8-11), open firing is having three sub-types:

(a) ***Open-firing with apical fire-channel:*** In this process of firing, pots are stacked with their mouths reversed in spherical manner in three or four level and this stack looks like a dome. During piling, a hollow is left in the middle, and it forms vertical channel which opens at top of dome. Then, fuel applied among and in between layers of pots and it is joined with apical channel. Then, heap of pots is covered with straw and husks. It is plastered with mud. But the top hole is kept open to kindle fire.

(b) ***Open-firing with horizontal fire-channel:*** In this process of firing, pots are piled in circular fashion and make rows at a foot within a rounded base keeping a narrow passage in between every two rows which act as a fire-channel and it runs straight across diametrically opposite direction. On both sides of fire-channel fuel-pit are made by old and broken-vessels. Then, fire is lightened through all fuel-pit instantaneously by one or two potters.

(c) ***Open-firing without any fire-channel:*** This is the simplest open-firing techniques without preparing any fire-channel. In this process of firing method, vessels are fired on an even or dugout. In first stage, bottom of the dugout is filled with cattle dung or straw and then vessels are piled in circular manner. After piling of pots, it is covered with wood, bamboo-split, wasted saplings, potsherds and then mud paste to protect from cool air to prevent heat properly. The stack is ignited from all sides at once.

2. **Oven firing:** In this firing method, a permanent or temporary oven is made of bricks, clay or stone and the pots are fired in it. The piling starts from hole in centre to final rows. The pots are placed in inverted fashion. Some clay props are made at equal distance temporarily, near stack-hole to protect vessels from coming into direct contact with heat. The vessels are kept on and around these piles.
3. **Kiln Firing:** In this method of firing, pottery and fuel are separate. In kiln firing technique, pots are stacked in a compartment. The wall of compartment is made by clay and bricks in spherical fashion and fired usually on a perforated floor which defends pots from straight contact with flame. In kiln, drought can be control and temperature is familiarized by means of muffling.

Usually, in open firing, rapid-flammable fuels such as, straw, cattle-dung, husks, dry leaves or brushwood is used for baking pottery. Saraswati (1979, pp. 10-11) stated that all methods of open firing differ from approximately 400-700 °C. While, in oven firing technique, temperature is achieved in range of 700-800 °C. Generally, kiln firing is better than open and oven firing because ability of heat conservation is better and higher temperature can be achieved. As making a kiln is expensive and as using it is also equally expensive, kiln firing technique could not attract majority of potter groups in India. Above mentioned firing techniques are performed in an oxidizing atmosphere. In oxidizing atmosphere, oxygen or air supplied throughout the firing process in such a fashion, that complete combustion/oxidation of metal present in clay takes place. This change gives color to a pot either red or grey, depending on the kind of slips used (Saraswati, 1979, pp. 11-12). But, black color of pottery is achieved in reducing atmosphere. In reducing atmosphere, gases burn in such manner that incomplete combustion or smudging takes place in a fire kiln, which gives black color to pot. The presence of carbon has effect of reducing oxides to their metal forms. In reduction method, separate kiln is not required. The piling of pottery in reduction method is as same as in oxidation method. In reduction method, after igniting the fire for certain time, stack and all stack-holes are fully covered by thick clay paste to prevent smoke within kiln.

1.8.5 Potter's Accessories

The potter's wheel: The potter's wheel may be divided into two types:

1. **Single wheel:** The single-wheel may be divided into two sub-groups: socketed and pivoted wheel. Again, these sub-groups may be categorized into two sections: block and spoked wheel. Consequently, all types of single-wheel are grouped into four main varieties:
    a. ***Socked Block-wheel:*** The disc is manufactured of stone, baked or unbaked clay. The socket is fixed at the middle of inner part of wheel, and wheel rests and rotates on an axis embedded discretely in ground or on a platform manufactured of stone, wood or clay.
    b. ***Socketed spoked wheel:*** It has three key parts: nave, felly and attaching spokes. The stone socket is fixed at middle in lower part of nave, and the axis is implant discretely in the ground.
    c. ***Pivoted spoked wheel:*** It has wooden nave, felly and attaching spokes. The construction of pivoted spoked wheel is precisely same to socketed spoked wheel. Only different is that axis is connected to middle of nave underneath.

d. ***Pivoted block-wheel:*** The solid disc is made of wood, baked or unbaked clay. The pivot is attached at lower part of it. While, socket is made separately in ground.

2. **Double wheel:** Sometimes, double wheel is called foot-wheel and also widely known as kick wheel. It has two wooden discs which are associated by a long stick and its tenon rests and rotates in a socket situated at bottom of trench where entire wheel is properly befitted.

Anvil: On the basis of materials and forms anvil can be classified into two main types as following:

i. *Anvil made of baked-clay with a knob* which helps to potters to grip properly in between fingers and thumb.
ii. *Stone anvil without a knob*: generally, shape is spherical or oval. It is definitely held in a clenched hand.

The Beating Base: Usually, beater is made of wood. The beating base can be classified into three main forms as following:

i. Made of baked clay or wooden beater gives firm and strong base for pottery.
ii. Sometimes, vessels are beaten simply on mat, or even on surface assisted by potters' legs; or in other term no such base are used.
iii. A spherical trench covered with grass or ash with a coating of jute bag may assist as vessel-rest. Sometimes, a tinny bamboo strap made and attached to a jute bag and positioned on trench.

1.9 Scientific study of archaeological pottery in India

The application of scientific methods of pottery compositional investigation has flourished in recent decades. In recent times, the application of scientific techniques has been widely used in analysis of archaeological pottery. During last four decades, advancement of instruments specially in sampling handling, accuracy and enhancements in data reduction is the key reason of successful application of analytical techniques in archaeological ceramic studies (Tykot, 2004, p. 408). According to Peacock (1970), technological study and characterization of ceramic are two key approaches for scientific study of pottery in archaeological science. In addition, technological studies of ancient pottery are based on the methods by which clays were prepared, shaped, finished and fired. While, scientific characterization of pottery deals with examination of properties of ceramics, and it involves in their mineralogical composition, chemical composition, microstructure, surface traits and provenance studies (Singh & Sharma, 2016, p. 557). Therefore, mineralogical and physicochemical composition and reconstruction of ceramic production techniques of archaeological pottery can be investigated by using both technological study and scientific characterization.

In India, many scholars have used analytical techniques to investigate archaeological pottery. Most of investigations have been carried out on Harappan pottery. Gupta, (1968-69) investigated technological aspects of handmade and wheel turned pottery produced by potters from Chirag Dilli using microscopic study. The study demonstrated that coarser clay which contained coarse grains of silica and other materials used by potters. Gogte (1982) studied different types of ancient pottery by using chemical analysis and classified these potsherds. The classification of pottery was done using ferrous/ferric ratio measurements and detects elemental carbon present into composition of them. Later, Gogte (1997) investigated the provenance of early historic roulted pottery recovered from Chnadraketugarh-Tamluk, Bengal region by X-Ray Diffraction analysis. He concluded that fine roulted ware groups were manufactured in the Ganga-Brahmaputra delta, India only. Selvakumar (2011) studied relationship between Southeast Asia and India on basis of ceramic and boat tradition. The ceramics were dispersed as items of trade (table ware) and media (storage jar) which can be easily carried by merchandises, personal collection of tourist and rituals vessels. The main types of Indian ware reported from Southeast Asia are fine roulted ware (dish, stamped bowl), various forms in identical fabrics and knobbed vessels. The carved marked ware type is only pottery which has Southeast Asian impacts reported from the India.

Some significant research using physicochemical and mineralogical analysis on technological aspects of Harappan pottery and other types of pottery have been done by Krishnan (Krishnan, 1982a, 1986b, 1992c; Krishnan & Hegde, 1988). The results revealed that presence of minerals and non-plastic inclusions in composition of pottery reflects the local clay deposits. The mixture of clay and crushed manganiferous ore was used for colouring and decorating pottery. Krishnan (1992d) investigated chemical analysis of decorative pigments and slips of Harappan pottery reported from Gujarat region by using energy dispersive X-ray (EDX). Microanalysis has been carried out using scanning electron microscope. The EDX results revealed that manganese quantity is very less and chocolate slip is same as iron rich black pigments. Krishnan and Coningham (1995) studied rouletted ware and other pottery reported from Anuradhapur, Sri Lanka using petrographic techniques. The results demonstrated that evidence of imported Hellenistic pottery which has rouletted patterns and this ware has direct connection with Grey Ware in terms of physiognomies in fabric and forms and it has South Asian origin. Krishnan *et al.*(2005e) studied glaze slip of fine pottery belonging to Harappan period using X-Ray Diffraction. The results revealed that both black and pale slip have hercynite, mullite and quartz in their composition. The elemental analysis indicates that different types of clays have been used to manufacture ceramic body and slip. Krishnan and Zulkernine (2012) investigated the pottery from Wari-Bateswar, Bangladesh using typological and petrographic analysis. The pottery has been classified into several fabric groups on basis of their mineralogical composition. The results revealed that raw clay which has been used to produce these potsherds comes from two different regions. According to Krishnan and Shah (2005), initially, scientific analysis is started to understand the composition of the materials used in clay, slip, pigments and the technological atmosphere under which the final product is manufactured. Later, scientific analysis is applied to examine cultural issues with high rate of accomplishment. In India, application of scientific techniques for investigating archaeological pottery is an emerging trend.

Venkatachalapathy and Ramaswamy (1991) investigated the ancient pottery largely reported from Narayanakuppam, Sambhuvarayar, Tamil Nadu using thin section analysis. The results indicate that angular grains of quartz, orthoclase, oligoclase, pyroxine, hornblende, biotite and iron ore are present in various sizes. The clay is composed of fine clayey materials (45%) and iron (12%) of total volume present in composition of samples. According to Duraiswamy *et al.* (1991), infrared spectroscopy is significantly valuable for the study of clay composition of archaeological pottery. The presence of hydroxyl water and thermal behaviour of pottery is easily detected due to spectra around 3700-3400 cm^{-1} and at 1600 cm^{-1}. The thermal transformation changes the intensities of these bands. On the basis of variant infrared spectra detected in pottery samples are helpful to determine lower firing temperature. Venkatachalapathy *et al.* (2002) studied pottery from Theriruveli, Tamil Nadu, India, excavated in 2000 which belongs to 700-800 AD. They used FTIR, X – Ray Diffraction, and Mossbauer for investigating type of clay minerals in pottery, their origin, oxidation state of ion bearing minerals, firing temperature and method of firing adopted by potters.

Manoharan *et al.* (2007) studied archaeological potteries which belonged to 300 BC collected from Maligaimedu, Tamilnadu, India, using FTIR, X-Ray Diffraction, and Mossbauer spectroscopy, trying to characterize clay minerals, their origin, firing temperature and method of firing adopted by potters.

Musthafa *et al.* (2010) studied pottery which was collected from Tandikkudi in Dindugul district of Tamilnadu, India, belonging to the 4th century AD. They used SEM-EDX and chemical analysis to know raw materials and firing behaviour, and chemical elements.

Velraj *et al.* (2010) studied fragmented pottery samples which were collected from archaeological site of Salamankuppam near the Mahabalipuram in Chennai, Tamilnadu, India of 150 BC. They used SEM-EDX and chemical analytical techniques to know ceramics composition. The result provided information about chemical composition of the potteries, firing atmosphere and firing temperature. Mineral magnetic studies also were performed and proved that all samples were highly magnetically enhanced materials and Paleo-intensity measurements by Thellier method yielded the field intensity value of 42.15 ± 2.0 μT.

Ravisankar *et al.* (2013) studied pottery coming from Kottapalayam, Karur, Tamilnadu by using FTIR, Powder X-ray diffraction, thermal gravimetric analysis and scanning electron microscopy-energy dispersive X-ray spectrometer ((SEM-EDX). The results indicate that minerals have been identified by using FTIR and XRD. The constituent of minerals suggest that pottery samples were fired in between 800-850 °C.

1.10 Pottery studies in Northeast India

First pioneer work on pottery reported from northeast India has carried out by Dr. T. C. Sharma. Sharma (1967) explained a stratified cultural deposit from Daojali Hading (North Cachar Hill). Before site was destroyed by road-builders he carried out a salvage operation and collected large quantities of potsherds and stone implements. It was first time that sherds have been found in this area, association with stone axes. Technologically sherds can be divided into four groups of ware as follows: (i) Cord-marked pottery (ii) Incised pottery (iii) Stamped pottery and (iv) Plain fine red pottery.

Goswami and Sharma (1963) described art of making Neolithic pottery of Daojali Hading. The paste was very course and shape was obtained by lining paste along a basket or bamboo vessels were sun dried. The color of vessels is between varying shades of grey.

Roy (1977, unpublished Ph. D. thesis) in his research work titled ‘A Study of Ceramics from Neolithic to Medieval Period of Assam: An Ethno-Archaeological Approach’ studied ceramics from Daojali Hading and Ambari. He used Hira and Kumar potter groups to draw ethno-archaeological conclusions.

Medhi (1990, pp. 37-44) pointed out after careful examination of excavated finds from Daojali Hading and Sarutaru that cord-marked pottery has close affinity to those found in Southeast Asia. Medhi also mentioned kaolin pottery discovered from Ambari, historical site in Guwahati, Assam.

Ashraf (1990) in his book titled ‘Prehistoric Arunachal: a report on archaeological exploration and excavation at Kamla Valley with reference to Parsi Parlo of Lower Subansiri District’ described the Neolithic pottery found in Phase 2.

Singh (1998-99) discussed about cord impressed pottery reported from Napachik and other sites and made a short note on pottery from Manipur (2008). Various types of pottery have been reported from numerous archaeological sites from Northeast Indian. For example, Sawmer, Sanmer, Low Longthrough and Barapani reported by Dr. Marco Mitri; Ranyak Khen reported by Dr. Tia Toshi Jamir; Napachik and Nongpokkeithelmonbi by Dr. O K Singh. None of these scholars had used scientific techniques for analysing pottery. All these studies classified pottery based on design and colour. Only through ethno-archaeological studies technique was reconstructed. The chemical composition of ceramics of Assam is not known neither place of origin, specific technique employed is known. No attempt was made to place pottery types in a broader chronological sequence. Stratigraphical sequencing was not done. As a result of this ceramic tradition of Assam is still a missing chapter in history and archaeology books of India and world. The present study attempts to fill these gaps in pottery studies of Assam.

1.11 The Present Study

Material characteristics of ceramics may depend on many complex factors: technological, regional, cultural and functional ones. Present study intends to deal mainly with regional factors, comparing local sediments with material of early ceramics from Northeast India.

The main focus of present study is to understand provenance of prehistoric and historical pottery of Northeast India. Identification of elements, tempering material, and firing range are some of highlights of the study. This work aims to build a broad regional sequence in

Northeast India, particularly in Assam of pottery traditions in different periods of history. The study also includes reconstructing technique of production.

Our planned analysis is aimed at a systematic study of regional factor, as one aspect of problems connected with archaeological pottery production. By this, we hope to get closer to solving archaeometric, petrological and historical problems emerging in study of pottery. Various analytical techniques have been used in present study to achieve desired objectives.

1.12 Methodology

Generally, scientific investigation of ancient pottery is started with explanation and classification of ceramic and its properties recognised as characterization. To understand manufacturing technique, provenance and use of archaeological pottery and technological aspects of ethnographic pottery, mineralogical and physicochemical characterization has been broadly used. The key methods chosen to achieve objectives of present study are both mineralogical and physicochemical methods. These are Furrier Transform Infrared (FTIR), X-Ray Diffraction (XRD), Thin Section Petrography, Raman Spectroscopy, Scanning Electron Microscope (SEM), Energy-dispersive X-ray spectroscopy (EDX), Micro Hardness Tester (MHT), Mohs' Scale and Laser Induced Breakdown Spectroscopy (LIBS). Ethnoarchaeological and scientific techniques have been also used to understand production techniques of ethnographic pottery. The application of these sophisticated analytical techniques and data interpretation will provide enormously important information about analysed ancient pottery of studied region. It is also very useful to reconstruct technology of studied pottery, to determine provenance of ancient pottery and understand production technology of modern pottery. The brief description of application of scientific techniques for present study is described below.

1.12.1 Thin Section Petrography

Petrographic methods have been thoroughly borrowed from Geological Science and it is used to explain and classify rock – minerals/rock forming minerals through analysis with a special or polarize microscope. Petrography deals with source, occurrence, structure and history of rocks and includes chemical and optical characterizations. The study of ceramic provenance started with observation and identification of composition, fabric and characterization of ceramic in terms of raw materials and their technology (Singh P. , 2015).

A thin section is approximately 30 µm thick slice of pottery or other artefacts which is fixed on a glass slide. Then this prepared slide is observed in either petrographic or polarizing light microscope or optical microscope.

According to Riederer (2004, p. 143), studies of ceramic thin sections give three types of information regarding their paste and raw materials, which is helpful to know technology of pottery manufacturing.

i. First, *mineralogical composition of coarse grained temper*, which differs significantly from place to place, depending on both regional geology and potters' habits and experience.
ii. Second, *determination of percentage of temper in ceramics* - grain size, grain size distribution and number, size, shape of pores and it is helpful to know potters' technique of preparing and shaping clay.
iii. Third, sometimes helpful *to determine and estimate firing temperature*, firing length and firing atmosphere by detecting changes of minerals during firing ceramics.

Thin-section petrography investigation was carried out on Leica Petrological Microscope. Thin section slide has been used to capture thin section photomicrograph.

Thin section petrographic analysis is a destructive technique. First step of thin section slide preparation is to cut pottery in selected direction to make a small chip which is large enough to hold with finger. Sometimes, pottery sample is very fragile or porous; it should be saturated with resin before sectioning to make sure that it will not break or disintegrate. Then, sawing surface is polished on one side till we get a smooth surface body of pottery. Polishing can be done either by hand or with grinding machine. Polishing should be done using abrasive powder i.e. carborundum powder of above 1000 mesh (grit). After that, polished side of vessel is attached to a glass microscope slide using adhesive such as thermosetting epoxy or Araldite. Then, other side of pottery should be grind and polished to get standard thickness i.e. 20-30 µm (0.03 mm.). At this thickness of thin section slide of sample different minerals shows identifiable diverse interference colours under polarizing microscope. The polarizing microscope is equipped with two polarizing foils or polarizers. One polarizing foil is placed below thin section which helps light waves to oscillate only in one direction. Other polarizer is placed above sample and it is oriented in crossed position.

1.12.2 X – Ray Diffraction (XRD)

Application of X-ray diffraction method is useful for characterizations of minerals by their crystalline structure (Zussman, X-Ray Diffraction, 1977). This method was first applied for mineral characterizations in 1912 by a German physicist, Von Laue. In X-ray diffraction analysis, X-rays are produced by bombarding of electrons on a target made of various elements (copper, molybdenum, iron, chromium). It is a powerful tool to identify minerals in soil based on d-spacing and relative peak intensities.

The XRD Spectra was recorded in 2θ range from 10 to 80° on TTRA III Riguku X-ray Diffractometer using CuKα radiation at a wavelength of 1.5406 Å. The specific wavelengths are characteristic of target materials such as Cu, Fe, Cr and Mo, but Copper (Cu) is most common target materials for XRD, with CuKα radiation at 1.5406 Å.

X-ray diffractometers consists of three basic elements:

1. An X-ray tube: is generated in a cathode ray by heating a filament to produce electrons.
2. A sample holder
3. An X-ray detector

The pottery sample was crushed in an agate mortar and a fine grained powder for recording XRD spectra was prepared. Mortars of other types like iron, steel etc also may be use. But, contamination of sample should be avoided by using iron mortar or any other surface which can cause contamination. For XRD analysis powder less than 10 µm (or 200-mesh) in size is ideal. After crushing pottery sample carefully into powder, it is placed in sample holder and upper surface is smeared with glass slide to make it even.

For data analysis Origin 6.0 has been used. After that, each of peaks obtained from XRD data has been analysed to get 2θ values using Origin 6.0 software. The higher versions of Origin software such as OriginPro 8 or OriginPro 9 are also available. Then, each of this 2θ degree was converted by applying Bragg's law ($n \lambda = 2d \times \sin\theta$) to getting d values. After that these d values have been compared with literature, and available online sources such as International Centre for Diffraction Data as Powder Diffraction File (ICDD-PDF), American Mineralogist Crystal Structure Database (AMCSD), mindat.org and webmineral.com to determine distance of lattice planes which provide mineralogical framework of studied samples.

1.12.3 Fourier transform infrared spectroscopy (FTIR)

The FTIR technique has been used to study type of clay mineral, their structural characteristic and lower limit of firing temperature during manufacturing. The FTIR examination of archaeological pottery may provide very important information, such as identification of unknown materials; determine quality or consistency of a sample and amount of components in a mixture.

The FTIR spectra of samples embedded in KBr pellet were recorded using Perkin-Elmer Spectrum One FTIR spectrometer with 5 scan modes in wavelength region 4000–400 cm^{-1}.

The sample must be translucent to Infrared radiation. Due to this, certain salts such as NaCl or KBr can be used to prepared pellet.

The potassium bromide (KBr) has been dried in hot air oven ICT (30-300 °C) in temperature of 60-100 °C for 12 hours to avoid moisture or humid. To prepare KBr pellet, proportion of sample in KBr should be in range of 0.2% to 1%. Then, powder of sample and KBr grind properly in clean mortar (agate). After that, finely crushed mixture of sample and KBr is pressed under very high pressure KBr Press (Maximum press up to 280 Kg/cm2) using maximum weight of 50-55 kg/ cm^2 in Evacuable pellet die. Usually, diameter of pellet is 1 cm and thickness 1-2 mm. Evacuable pellet die consists of piston, die body, anvil, basc, o-ring and vacuum.

1.12.4 Micro hardness Tester (MHT) and Mohs' Scale

The term Micro hardness Test usually refers to static indentations made by loads exceeding one kgf. The indenter is either Vickers diamond pyramid or Knoop elongated diamond pyramid. The surface being tested generally requires a metallographic finish (MVH user's manual guide).

Micro hardness test or more precisely microindentation hardness can be defined as calculation of size of a microindentation made by a diamond indenter of definite geometric impressed into a polished surface of workpiece by a known load (Gramaticu, *et al.*, 2010, p. 10). According to Gramaticu *et al.* (2010), the micro hardness test is useful indicator of physical and mechanical properties of archaeological ceramics. It is vital to determine hardness of various types of archaeological ceramics (Gramaticu M. *et al*., p. 5, 2010).

Mohs' scale is used by mineralogists and archaeologists in field test that were first proposed by Friedrich Mohs in 1882. Scratch test or Mohs' scale is a very rough test that do not shows any homogeneity in shape of point or in force applied in making scratch. Mohs' scale has a series of mineral from 1-10 where 1 is referring to talc, softest mineral and 10 is refer to diamond, hardest mineral. According to Shepard (1956, p. 114), use of scratch test (Mohs' scale) to measuring hardness of a pottery gives clue about surface of potsherds that can be indicative of how well it will withstand abrasion. Pottery that has hardness of 2.5 to 3 unit in Mohs' scale has not had high luster and bright color. 5 to 6 unit hardness for pottery is considered good for service ware but pottery with mohs' hardness 7 or more is considered as the most technologically sound.

The hardness of pottery has been measured using Micro Hardness Tester: Make-Omni Tech. Pottery samples (1x1 cm^2 approx.) are used for micro hardness test. Thickness of workpieces depends on thickness of pottery. For analysis, workpieces are polished by using abrasive papers of 200, 400, 500, 800 and 1200 grit on slow moving wheel with rounding speed about maximum 20 rpm. At 6 different locations on each sample, sherds are measured and 500 gf loads is applied on each point of workpieces. Then, absolute hardness value (HV) has been determined by standard deviation of values of these six measured locations.

1.12.5 Thermal Analysis

Clay minerals can be characterized by different types of scientific methods that focus on thermal behaviour such as thermal expansion analysis, differential thermo-gravimetric analysis (DTA), thermo-gravimetric analysis (TGA) and evolved gas analysis. When clay minerals are heated or cooled at controlled rates (Ware, 1971, pp. 273-305), specific elements react differently and characteristically. TGA is a crucial tool for determination of firing temperature of ancient pottery or ceramic materials. The pottery samples undergo control heating and detector of instruments detect signal as enthalpy change.

Differential thermal analysis (DTA) is based on measurement of alterations of clays heated to temperatures of 1000-1300 °C. This change can be classified as either released (exothermic reactions) or absorbed (endothermic reactions).

In *Thermo-gravimetric analysis* (TGA), when a sample is heated around 1000 °C, changes in weight can be measured. In firing temperature of 400°-650 °C dehydroxylation occurs due to loss of weight.

Differential thermo-gravimetric or dynamic differential calorimetric analysis can measure changes in weight per unit time.

Thermal expansion analysis is characterized by dimensional changes of clay based on minerals from heating temperature 100-200 °C to 500-800 °C.

The FTIR results indicate that firing temperature range is in between 500 to 900 °C of all analysed pottery in present study (including Neolithic, Historical and Modern pottery). In addition, upper limit of heating temperature 900 °C has been selected.

Sample preparation is not required. Only, 5-8 μg. powder of pottery samples has been used to take TG-DSC analysis. Thermogravimetric analysis (TGA) is generally based on high degree accuracy of three measurements of changes in mass, temperature and change in temperature. TGA and DSC analysis were carried out up to 900 °C at 30 °C/min in a static nitrogen atmosphere using NETZSCHSTA449F3 Thermal Analyzer.

1.12.6 Laser Induced Breakdown Spectroscopy (LIBS)

LIBS is a quantitative analytical technique which focuses on absolute value for individual elements and suitable for specific compositional classification of materials. It is basically a non-destructive, quick elemental investigation technique with applicability for use in situ. Furthermore, LIBS may be applicable to examine element content of various types of materials such as ceramics, icons, jewellery, metal, polychrome, sculpture, glass, painted artworks and stone artefacts (Melessanaki *et al.* p. 157, 2002; Giakoumaki *et al*. p. 749, 2006; Anglos D., 2001). Samples preparation is not required. LIBS spectrum was recorded using Make- StellerNet (BLACK-Comet, C-SR-25 Spectrometer) in range of 200 to 600 nm wavelength. The Litron Lasers Model –LPY7864-10G were used.

1.12.7 Raman spectroscopy

The use of Raman spectroscopy for identifying and studying archaeological materials has flourished in recent years. Raman spectroscopy is based on inelastic scattering of monochromatic light, commonly from a laser source in visible range or near infrared range or near UV range. This method is extensively used to analyze chemical and physical properties. This is also helpful to analyze pigment, glaze, and other mineralogical compositions of pottery and to determine firing temperature. Main limitation of this method is contamination of Raman spectra due to fluorescence (Smith & Dent, 2005, pp. 16-20).

Raman spectroscopy is a suitable analytical technique to characterize archaeological ceramics and this is non-invasive and non-destructive method. Furthermore, Raman spectroscopy yields information about structural physiognomies and minerals present in ceramics. Moreover, it gives information on molecular constituent which permits to differentiate between various mineral polymorphs, for instance, anatase and rutile (TiO_2) or calcite and aragonite ($CaCO_3$) (Olivares, *et al.*, p. 1544, 2010).

Raman spectroscopy has been carried out with Horiba Jobin Vyon, Model LabRam HR. The wavelengths during experiment are 488, 514 and 633 nm with Raman spectra in between 100-2000 cm^{-1}. Collection time is varying from 5 to 100 seconds and laser power is varying. The powder and solid pottery sample from 16.3 to 51 mW were used to record μ-Raman spectra. Detail of experiment specification has been tabulated in Table 1.1 to 1.3. High fluorescence of pottery sample prevents recording of explainable Raman spectra. Therefore, Raman spectroscopy has been used to identify minerals present in composition of pottery.

1.12.8 Scanning electron microscope (SEM) and Energy Dispersive X-Ray (EDX)

In SEM (Thornton, 1968) focused ray of electrons scan a particular sample and analyse surface area of sample. A thin film of gold or carbon or platinum is required to make surface of samples conductive. SEM is widely used to reconstruct manufacturing technology, identify microscopic structure of ceramics materials including slips. SEM is also used to identify structural changes in fabric that occurred due to sintering, which are greatly helpful to determine firing temperature. The characteristic X-rays are produced when target is bombarded with electrons, giving information about element present in composition of samples. SEM combined with EDX yield information not only structural characteristics of samples but also produce information about elements present in sample.

SEM images of all pottery samples have been taken using Make-ZEISS EVO 40 EP-SEM. EDX have been taken under Make-Bruker LN2 Free X Flash 4010 SDD Detector (Software Quantax 200). The solid and powder (fresh fracture surface and fresh grinded powder of pottery) pottery samples were coated with thin layer of carbon. For this, double side adhesive Carbon Conductive tab (Maximum diameter 12 mm) has been used. The carbon tab (approx. 6 mm in size or 1/4th of Carbon Conductive tab) is stuck on SEM stub (maximum diameter of stub is 12 mm) and then pottery sample (solid and powder) has been paste. Then this stub is placed in coating machine and coated with carbon. Usually for coating, gold and platinum can also be used.

Table 1.1 Experimental specification of each Neolithic pottery during recording Raman spectra

Si. No.	Sample name	Wavelength (nm)	Power (mW)	Time (second)
1	DJL	514	50	50
2	GWKC	514	16.6	20
3	GWKF	514	16.6	20
4	BRP-01	514	16.6	5
5	BRP-02	514	16.6	10
6	KM-01	514	33	30
7	KM-02	514	50	10
8	KM-03	514	50	10
9	NPC-01	633	16.6	5
10	NPC-02	633	16.6	5
11	NPC-03	633	16.6	30
12	NPC-04	488	16.6	30
13	NPC-05	633	16.6	10
14	RYK-01	488	16.6	20
15	RYK-02	488	16.6	5
16	RYK-03	488	16.6	30
17	RYK-04	488	16.6	30
18	RYK-05	488	16.6	30
19	SPT-01	514	50	50
20	SPT-02	514	50	50
21	SMR-01	514	16.6	10
22	SMR-02	514	50	10
23	SMR-03	633	16.6	20
24	SMR-04	633	16.6	5
25	SMR-05	488	16.6	30
26	SMR-06	488	16.6	10
27	SMR-07	488	16.6	5
28	SMR-08	488	16.6	30
29	SMR-09	488	16.6	25
30	LNGH-01	514	50	50
31	LNGH-02	514	50	50
32	LNGH-03	514	50	50

Table 1.2 Experimental specification of each Historical pottery (Ambari) during recording Raman spectra

Si. No.	**Sample name**	**Wavelength (nm)**	**Power (mW)**	**Time (second)**
1	AMB-01	488	16.6	20
2	AMB-02	488	16.6	5
3	AMB-03	488	16.6	30
4	AMB-04	488	16.6	20
5	AMB-05	488	16.6	10
6	AMB-06	488	16.6	20
7	AMB-07	488	16.6	20
8	AMB-08	488	16.6	10
9	AMB-09	488	16.6	10
10	AMB-10	488	16.6	10
11	AMB-11	488	16.6	10
12	AMB-12	488	16.6	5
13	AMB-13	488	16.6	5
14	AMB-14	488	16.6	5
15	AMB-15	488	16.6	20
16	AMB-16	488	16.6	30
17	AMB-17	488	16.6	15
18	AMB-18	488	16.6	30
19	AMB-19	488	16.6	30
20	AMB-20	488	16.6	30
21	AMB-21	488	16.6	10
22	AMB-22	488	50	50
23	AMB-23	488	16.6	10
24	AMB-24	488	16.6	10
25	AMB-25	488	16.6	10
26	AMB-26	488	16.6	10
27	AMB-27	488	16.6	10
28	AMB-28	488	16.6	10
29	AMB-29	488	16.6	10
30	AMB-30	488	16.6	10
31	AMB-31	488	16.6	10
32	AMB-32	488	16.6	10
33	AMB-33	488	16.6	10

Table 1.3 Experimental specification of each Historical pottery (Sri Sri Suryapahar, Bamuni Hill and Kangla Fort) during recording Raman spectra

Si. No.	Sample name	Wavelength (nm)	Power (mW)	Time (second)
01	SPR-01	488	16.6	10
02	SPR-02	488	16.6	10
03	SPR-03	488	16.6	10
04	SPR-04	488	16.6	10
05	SPR-05	488	16.6	10
06	SPR-06	488	16.6	10
07	SPR-07	488	16.6	10
09	SPR-08	488	16.6	10
09	BMH-01	488	16.6	10
10	BMH-02	514	30	10
11	BMH-03	514	30	10
12	BMH-04	514	50	10
13	BMH-05	514	50	10
14	KGF-01	514	50	100
15	KGF-02	514	50	100
16	KGF-03	514	50	100

1.12.9 Ethnoarchaeological Method

According to Costin (2000), for archaeologists, ethnoarchaeological approaches are useful to interpreting and tracking pottery production using comparative data, descriptive frameworks, and hypotheses of source of ceramics. The significant aim of ethnoarchaeology approach is to provide an explanation of archaeological artefacts/materials/ceramics through analysing ethnographic data. The ethnoarchaeological approach may be useful to reveal how clay procurement and preparation reflects elemental composition of pottery and behaviour through investigating materials from living potter communities.

In present study, fieldwork has been conducted in Assam for year of 2014-2015. The field works have been conducted in Hirapara, Khehnipara – Kamrup District and Kumargaon-Dergaon, Kumargaon-Dhekial – Golaghat, Assam. In field work, we have documented the collection of the clay and firing process of pottery. In the field work, we have documented entire process from collection of clay to firing techniques of pottery. For this we have taken interviews of respective potters, capturing photographs and shooting videos of their manufacturing processes.

1.13 Conclusion

Application of scientific techniques or archaeometric techniques or more precisely analytical methods in studying archaeological pottery to understand its manufacture, use and provenance is developed as a methodology in archaeological studies today. The mineralogical and physicochemical studies of pottery are greatly enlightening to scholars. The ethnographic and ethnoarchaeological studies of modern pottery and potter communities have greatly helped researchers to understand technological aspects of ancient pottery production techniques. For example, hand moulding, throwing, decorating, colouring pot, firing processes, and marketing of finished product and usage of pottery in societies. However, compositional study of materials from prehistoric reference has developed as a significant module of most systematically oriented archaeological analysis.

In present study, scientific techniques have been applied to reconstruct technology of selected prehistoric and historical pottery samples from various archaeological sites of North East India. Ethnographical and scientific techniques have been applied on selected modern pottery to understand production techniques. It is also useful to understand ancient manufacturing techniques of pottery. Moreover, present study is able to draw attention of scholar to way in which studies of this type can provide facts which is valuable and vital for understanding pottery. As highlighted by present study, application of archaeometric methods provided here will expose new paths for analysis of ceramic technology and archaeology of Northeast India.

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Chapter 2
Mineralogical and Physicochemical analysis of Neolithic Pottery

2.1 Introduction

The distinctive cultural features of Northeast Indian Neolithic period is cord marked pottery and double shouldered Celt which was recovered for first time in region from Daojali Hading, Dima Hasao district, Assam. The excavation was carried out by M. C. Goswami and T. C. Sharma in 1961. Later, same context was found in another Neolithic site Sarutaru, excavated in 1971-74 in vicinity of Guwahati city. This is supposed to be an eastern Asiatic Neolithic tradition. Geographically and culturally, Northeast India is a land bridge between India and Southeast Asia and with China. There is a hypothesis that Celt manufacturing techniques entered India through Assam-Burma (Myanmar) corridor (Worman, 1949, p. 199).

The first reported Neolithic implement from region is a jadeite Neolithic axe. It was found by Sir John Lubbock in 1867 and published in 'Athaneum' from London. Later, In 1870 E. H. Steel discovered some Neolithic tools made of jadeite from villages of Namsang Nagas. Coggin Brown wrote an article on stored findings in Indian Museum, Kolkata in *Journal of the Asiatic Society of Bengal* in 1914 and also included them in his *Catalogue of Prehistoric Antiquities in the Indian Museum*, Calcutta, published in 1917. Dasgupta (1913) published an article on these findings in same journal. Collection of 385 stone specimens from different parts of Northeast India, are kept in Pitt Rivers museum contributed by several persons like J. H. Hutton, J.P. Mills. G. D. Walker, J. H. Grace etc. Hutton explained these findings in his article *'The Prehistory of Assam'* published in 1928 and later it was studied by Prof. T. C Sharma (1966a).

A. H. Dani (Dacca Museum) added a chapter on 'Neolithic Cultures of Assam' in his thesis from Institute of Archaeology, London. Later, this thesis was published as a book in 1960. According to Dani (1960), main tool types are facetted tool, shouldered tool, splayed axe, rounded butt axe, axe with broad cutting edge, tanged axe and wedge-blades, grooved hammer stones. Dani explained typological framework of Neolithic tools from Northeast India for the very first time. He divided Neolithic Cultures of Assam (now Northeast India) into six different zones:

1. Cachar Hills Zone
2. Sadiya Frontier Zone
3. Naga Hills Zone
4. Khasi Hills Zone
5. Garo Hills Zone, and
6. Brahmaputra Valley Zone.

The next major work was by Prof. T. C. Sharma (1966a). In his thesis titled *"Prehistoric Archaeology of Assam- A Study of the Neolithic Cultures"* he established occurrence of cord marked pottery and double shouldered Celts from stratified Neolithic site of Daojali Hading. He studied Pitt Rivers Museum collection from Assam and classified stone tools based on typology and manufacturing techniques.

A. Chipped implements: Major tool types are – various types of handaxe, choppers and scrappers, simple flakes, fluted cores, chipped celts, broad axes, tanged lance-heads
B. Edge-ground implements: Major tool types are – parallel-sided axes, large axes, short axes, pebble tools.
C. Pecked-and-ground implements: Major tool types are – Quadrangular Axes, Naga Hill Axes, Naga Hill tanged celts, Notched Axes, Shoe-last celts, Wedge-shaped celts, Rounded Adzes, Rounded chisels
D. Fully ground implements: Major tool types are – Tanged or Shouldered celts, Flat celts, Small celts, Quadrangular Axes, Large Axes with lenticular section, Quadrangular Adzes, Chisels and Wedges, Splayed Axes, Perforated Axes, Miniature Axes of jadeite, Lance-heads.

Other stone implements such as grinding stones, smoothing stones for making wooden implements, grooved hammers, notched pebbles, querns or milling troughs, muller, nut-crushers have been also reported from the various Neolithic sites of Northeast India.

2.1.1 Excavated Neolithic Sites from Northeast India

Excavated Neolithic Sites from Northeast India are Daojali Hading and Sarutaro from Assam, Parsi Parlo from Arunachal Pradesh, Law Nongthroh and Gawak Abri from Meghalaya, Napachik and Nongpok Keithelmanbi from Manipur, Ranyak Khen from Nagaland.

The excavation at Daojali Hading was conducted by T. C. Sharma and M. C. Goswami in 1961-63 and been studied by T. C. Sharma (1966a). Findings of excavation are 32 edged tools, 22 grinding stones, 4 querns, 6 mullers, 11 Quartzite pebbles and fossil wood. Quartzite, sandstone, shale and fossil wood were used as raw materials for manufacturing tools which is locally available. Presence of quern-muller indicates that available grains were grinded which indicates use of plants and grains as food items. But, in present research scenario it is difficult to conclude that whether these plant food items are domesticated or wild. The major types are 595 potsherds of cord-impressed, 19 stamped dull red and 11 brick red varieties. It is difficult to reconstruct shape of potsherds due to heavily weathered and broken into small pieces. Clay is very coarse and large Quartz particles used as tempering materials. Typologically sherds can be divided into four groups as follows:

1. Cord impressed ware
2. Incised ware
3. Stamped ware (Course dull red ware)
4. Plain Ware (fine red ware)

According to Sharma (1966b), assemblage of Daojali Hading can be closely associated with Late Bacsonian of Indo-China and Tam-Toa in Annam. Typologically, tools types, manufacturing techniques, form of small and shouldered axes of both sites Daojali Hading and Tam-Toa almost indistinguishable to each other. The grinding slabs, polishing stones for wooden and bone tools, and mullers or rubbers are other common implements of both sites. Cord-impressed and incised pottery of both sites is closely similar. The difference between both sites is incised ware of Somrong-Sen type which is present earlier and absent in later phase. Annam is located between north of Bac-Son and south of Somrong-Sen, which are two important cultural zones. Stone tools such as shouldered axes and cord-marked pottery from Daojali Hading also have similarity with stone tools and pottery from Somrong Sen. Typological similarity of stone tools and cord-marked pottery which Neolithic culture of Assam has confirms former hypothesis that closely connection with East Asiatic Neolithic complex (Worman, 1949, pp. 188-89; Dani, 1960, p. 77).

This finding opened up new opportunities for Neolithic investigation in Northeast India.

In 1973, S. N. Rao from Dept. of History, Northeast Hill University and D. K. Dutta carried out an excavation at Sarutaru situated about 25 km. southwest of Guwahati. Sarutaru is a Neolithic site. It revealed a single cultural deposit of 35 cm. in thickness and 9 specimens of ground Celts, double shouldered celts were recovered. Handmade cord – impressed pottery were also found along with tools. Ground stone celts manufactured on slate of dark grey color and sandstone of cream to buff colour. They were made in two stages: chipping and grinding (Rao 1977).

H. C. Sharma and A. A. Asharaf of Dept. of Anthropology Gauhati University (GU) (1991) explored area of Langting river valley around Langting and Maibong village. Potsherds and other materials were found from explored area. The section exhibited 4 layers and all layers belonged to single culture deposit of ceramic-Neolithic Phase.

Napachik (NPC) is located on right bank of Manipur River at Wangu village, Bishnupur district, Manipur. This site was discovered by Yumjao Singh in 1981. Neolithic pottery and stone tools have been discovered such as plain ware, cord- marked ware, ring footed ware, tripod leg ware associated with flake and pebbles tools as well as ground Celts. Potsherds are handmade and low fired. Corded wares have similarity with cord marked pottery samples recovered from site of Phunan (Dikshit & Hazarika, 2011-12, pp. 111-114).

Nongpok Keithelmanbi (KM) is located on top of a flattened hill range towards left bank of Thoubal River in Manipur. This site was discovered by O.K. Singh, assisted by S.R. Singh of Department of Archaeology, Government of Manipur in 1983. A trail trench has been dug. Three cultural sequences have been revealed. A charcoal samples have been dated to 4460±120 year BP reported from corded ware stratum. Neolithic pottery has been reported such as corded ware and curved paddle impressed ware along with other artefacts. Corded ware has similarity with Hoabinhian stratum in cultural layer. Pottery is ill fired, handmade and weathered. Design pattern is zigzag and linear on cord marked pottery. Fine clay is used to make pottery. Fine sand and Quartz particles are used as tempering material. Potsherds are often red and brown in color.

Neolithic sites in Meghalaya are most well-known among all states of Northeast India. Exploration in West Garo Hills revealed information about relics of Neolithic culture, habitation of Neolithic men and practicing of ground and polished techniques. Main Neolithic sites are

Selbalgiri 2, Gawak Abri, Sohpet Bneng, Sawmer, Sanmer, Law Nongthroh, Barapani. Among these Gawak Abri and Law Nongthroh have been excavated.

Gawak Abri is located approximately 8 km downstream from source of river Rongram, Central Garo Hills, Meghalaya. Site is located at an altitude of approximately 550 m msl and area is 3 sq. km. Artefacts were recovered from northern side of mound. Cultural material collected from site consisted of lithic tools and pottery. It can be called a single culture site on basis of typological character of tools. Lithic assemblage consisted of ground and polished celts, short axes, chipped celts, and a very high percentage of micro-size flakes, some of which were probably used. A thin patina is seen on surface of tools. Flake scars are sharp and fresh. The pottery consisted of two varieties classified on basis of colour and fabric. One was very coarse black-colour pottery and other was also of coarse variety but comparatively finer with a thin grey slip (Sharma, 2007)

Law Nongthroh (25° 72.386’ N 91° 94.90’ E) is situated on northern slope of Sohpet Bneng Hill in Ri-Bhoi district of Khasi Hills. Excavation was carried out by Marco Mitri in February, 2015. Fourteen finished stone tools in various forms have been recovered. Although, few large stone fragments and small flakes are also reported. A grinding stone made of red sandstone is also found from one of the trench in excavation. A number of 643 potsherds which have various patterns of impressions on outer body are recovered from excavation. Main patterns are cord impressed, criss-cross, zigzag, zigzag and criss-cross, horizontal and vertical. Among them, 147 potsherds are belongs to plain ware. The wild seeds such as Xanthoxyllum sp., Polygonum sp. and Ocimum have identified. Two charcoal samples which are reported from excavation have dated at Beta Analytic, USA. These dates are Cal BC 1220 -1020 (BP 3170 to 2990) and Cal BC 770 to 415 (2720 to 2365) (Mitri, Kharmawphlang, & Syiemlieh, 2015, pp. 33-41).

Ranyak Khen is situated in Kiphire district, Nagaland. Ranyak Khen is also known as cave no. 2 among other limestone caves at Mimi. Mouth of cave facing west is 8.2-9.0 m high and 7.0 m wide. Assemblage found during excavation includes Edge grinding tools of serpentine and limestone made from river pebbles, a few hammer stones, disc shaped scraper tools, bone tools and cord-marked pottery besides a human burial. Evidence of animal bones such as deer, monkeys and bats are also reported. Radio carbon dating has been determined of layer three, depth 111-126 cm and calibrated date is 4450-4350 BC.

In 1983, Shri B. C Poddar and Shri N. R. Ramesh of Geological Survey of India (GSI) find Stone Age sites in Haora and Khowai river valley, Tripura. This works included comprehensive documentation of the late quaternary deposits of North-Eastern India and accompanying Stone Age sites in Tripura. Lower level with a silicified fossil wood industry in Tripura has been dated by C14 method to 32,500 BC and upper layer with Neolithic tools has been dated to 1500 BC by C14 method (Ramesh & Rajagopalan, 1999, pp. 13-30). Neolithic Tools include chipped, grounded and polished celts, edge ground adze, hammer stones, grinding stones, pick axe, chisels etc and raw material used was silicified fossil wood.

2.1.2 Pottery

Pottery has been reported from almost all Neolithic sites of northeast India with ground and polished celts. Typologically, potsherds have been classified into several groups such as (A) Cord-impressed ware – cord-impressed coarse gritty ware, cord-impressed buff ware, cord-impressed mottled ware, cord-impressed coarse grey ware, cord-hatched coarse grey ware, cord-hatched dull red ware and cord-impressed thick brown ware; (B) Incised ware: Cross-hatched dull red ware and Herring bone pattern grey ware; Stamped Coarse ware; (C) Stamped Ware and (D) Plain fine ware. The design in form of impressed patterns includes simple cord impressions, twisted cord impressions, herringbone pattern and zigzag patterns. Due to small size, it is difficult to reconstruct the shape of potsherds. Provenance could not be determined in study area. Clay was not well levigated and it indicates that use of poor quality clay mixed with Quartz grits. All potsherds are low fired.

The cord-impressed pottery tradition in Indian context was first reported form Daojali Hading Dima Hasao, Assam (Sharma 1966a). Since, lots of explorations and excavation have been conducted in North-eastern India and resultants huge quantity of stone artefacts and pottery and other materials have been reported from various archaeological sites of Northeast India. According to Hazarika, (2006a, p. 29), there are three distinguished cultural features of Neolithic period of Northeast India viz. Stone manufacture trends (specially shoulder Celts), cord-impressed pottery and rice agriculture. Dani (1960) has reported that the connection between Southeast Asia and East Asia and various archaeological sites of Northeast India on basis of similarities in stone implements. In short, major types of Neolithic pottery that have been reported from archaeological sites of Northeast India by numerous scholars are –

- Square-gird, honey-comb (web) and beater impressed ware-Parsi-Parlo from Arunachal Pradesh by A. A. Ashraf.
- Cord impressed ware, incised ware, stamped ware (Course dull red ware), plain Ware (fine red ware) from Daojali Hading and Sarutaru, Assam classified by TC Sharma (1967).
- Cord marked ware with linear and criss-cross patterns-Nongpok Keithelmanbi and plain ware, cord marked ware, ring footed ware, tripod legs ware- Napachik from Manipur.
- Handmade grey color pottery and coarse red ware with cord-marked form Gawak Abri, Selbalgiri-2 and Pynthorlang form Meghalaya.
- 643 potsherds have been recovered from Law Nongthroh (Sohpetbneng Hill, Meghalaya), major types are-cord-impressed, criss-cross, zigzag, criss-cross, faded and plain wares (Mitri, Kharmawphlang, & Syiemlieh, 2015). Other sites from Meghalaya where Neolithic pottery are also reported – Sawmer, Barapani, Sohpet and Sanmer. Major pottery types are – cord-impressed, plain ware with chocolate color etc.
- Coarse types, grooved pattern ill fired handmade pottery – Chungliyimati and cord – impressed pottery-Ranyak Khen from Nagaland.

Potsherds selected (Appendix II) for analysis from various Neolithic sites of North East India (Appendix I) are - Daojali Hading (DJL) – Assam; Nongpok Keithelmanbi (KM) and Napachik (NPC) – Manipur; Barapani (BRP), Sophet (SPT), Sawmer (SMR), Law Nongthroh (LNGH) and Gawak Abri (GWK) – Meghalaya and Ranyak Khen (RYK) – Nagaland.

Material physiognomies of the ceramics may be subjected to many complex factors, such as technological, regional, cultural and functional ones. This chapter is designed to determine firing temperature, firing condition and investigate morphology of Neolithic pottery from Northeast India. It can provide us information on origin of clay used for making pottery by ancient potters of region and also on technical skills of potters of area. By this, we hope to get closer towards solving archaeometric, petrological and past problems emerging in study of ancient pottery of NE India. To achieve these objectives, interdisciplinary methodologies such as mineralogical and chemical techniques have been adopted.

The organized and scientific study of ancient pottery replicates technical development. Numerous spectroscopic and magnetic techniques are typically extensively used for study of archaeological pottery.

Archaeological pottery is most prolific relics that have been studied to get better understanding of past cultures. Archaeological study based on the investigation of pottery shape, color, decoration style and materials that provide plenty of information about typology and invaluable information regarding dating and provenance (Papachristodoulou, *et al.*, 2006, p. 347). The studies of mineralogical properties of ceramics play a vital role to understand earlier technologies. However, manufacturing techniques, cleaning of clays, adding of temper, shaping of objects and firing atmosphere can significantly change mineralogical composition of ceramic materials. In addition to this, life cycle of ceramics, its usage and post-depositional changes are also important and can help to change mineralogical constitution (Gasparic, Horvat, & Mirtic, 2014, p. 225).

The key objective of provenance investigation and manufacturing technologies of an artifact in archaeological science are to identify source of manufacture site and reconstruct making procedures. The role of analytical methods is mostly pertinent with qualitative and quantitative characteristics of artifacts that suitably explain, produce specific information on source of raw materials and therefore, indirectly on site of production. Provenance analyses mainly related to characterization and study of raw material of ceramic.

The technological studies or analytical methods are belongs to investigation of manufacturing techniques including usages of raw materials, clay preparation, firing technique, and pre/post firing treatment of pottery. Therefore, technological studies also deal with provenance and dating. According to Tite (1972, p. 256), physical methods of chemical examinations deal with determining chemical constitute, identifying mineral phases and investigating isotopic of certain components. Wide range of analytical method can be applied to determine and identify geographic source of raw materials used and to get information on manufacturing techniques. Mineralogical and physicochemical methods can be combining used for studies of manufacturing technologies and provenance of pottery.

Present work attempts to examine mineralogical and chemical compositions of pottery using several analytical techniques. Optically Stimulated Luminescence (OSL) has been used to determine date of two pottery (one from Daojali Hading and one from Gwak Abri). This work also investigates provenance of archaeological pottery by using thin section petrography. FTIR, Raman Spectroscopy, SEM and XRD have been applied to determine firing temperature and atmosphere. Physical characteristics of analysed pottery, such as hardness have been determined by using Mohs' scale and MHT. To identify and characterize mineralogical composition in analysed potsherds, thin section petrography, XRD and FTIR have been used. In addition, Raman spectroscopy also identifies minerals that are present in pottery. Identification of elements in analysed potsherds has carried out using EDX and LIBS. This study will find out their origin and we can replicate pottery technology of Neolithic potters.

2.2 OSL dating

2.2.1 Sample Treatment

Material: Pottery; Mineral: Quartz; Size: 90-125 μm

Protocol: Single Aliquot Regeneration (SAR) protocol (Murray & Wintle, 2000)

The age of two Neolithic potsherds has been determined using OSL dating technique. For this samples are prepared as follow:

- Samples were crushed and removed light exposed materials from surfaces under subdued red light in laboratory.
- Then we sieved samples to get 90-125 μm size fraction. After that, we remove carbonate and organic materials from samples by treating 1 N HCl and 30% H_2O_2 respectively.
- Quartz grains (density 2.65 gm/cc) were extracted using high density liquid (sodium polytungstate solution $Na_6[H_2W_{12}O_{40}]$) (Aitken, 1985).
- 40% hydrofluoric acid was used for 80 minutes to etch Quartz, which removes Feldspar contaminations from outer layer of extracted Quartz grains.
- Subsequently, we treated Quartz grains with HCl and washed in distilled water and re-sieved.
- Lastly, infrared stimulated luminescence (IRSL) technique was used to confirm purity of etched Quartz. The etched Quartz grains were pasted by silicon oil (adhesive) on center of stainless steel discs (3 mm diameter mono layer of samples in 10 mm diameter steel

discs) for determining radiation energy absorbed by sample during burial (i.e. paleo dose or equivalent dose).

2.2.2 Instrumentation

For equivalent dose (De) determination Single Aliquot Regeneration protocol was used (Murray and Wintle, 2000). OSL measurements were carried out in an automated Riso TL/DA-20 reader equipped with blue light emitting diodes. Equivalent dose (De) values were calculated using initial integral (0.8sec) of OSL (by Duller's Annalyse software). For annual dose rate estimation, quantity of uranium, thorium and potassium in pottery and surrounding sediments needs to be measured. However, in present case no surrounding sediments were available and hence only chemical concentration of U, Th and K in pottery sample were measured and for cosmic rays Grun's software is used. Concentrations of uranium, thorium and potassium in pottery were measured by ICP. Grun's software is used for dose rate (including cosmic ray contribution) and age calculation.

Equation 2.1

$$\mathbf{Age} = \frac{\textbf{equivalent dose (Gy)}}{\textbf{dose rate}\left(\frac{\textbf{Gy}}{\textbf{ka}}\right)}$$

Table 2.1 OSL dating of potsherds

Lab No.	Sample No.	Depth (cm)	U (ppm)	Th (ppm)	Potassium K (%)	Equivalent Dose (Gy)	Dose Rate (Gy/ka)	Age (ka)
LD1727	Gawak Abri	72	5.83±0.06	22.52±0.22	2.18±0.02	12.0±0.8	5.2±0.1	2.3±0.2
LD1728	Daojali Hading	105	2.41±0.02	32.52±0.3	2.23±0.02	13.9±1.5	5.1±0.1	2.7±0.3

OSL dating carried out for 2 potsherds from Daojali Hading (DJL) and Gawak Abri (GWK) and it is summarized in Table 2.1 with lab no. LD1727 (GWK) and LD1728 (DJL) are studied using Riso TL/DA-20 reader equipped with blue light emitting diodes. Age of pottery from Daojali Hading and Gawak Abri is 2.7 ka and 2.3 ka respectively.

2.3 Petrographic Analysis

2.3.1 Analysis of Thin Section Petrography

Thin section analysis has been used to investigate composition of raw materials, texture of clay paste and provenance. Further, it has been used to examine manufacturing technique and morphology of neolithic potsherds. In present study, petrographic analysis revealed information about raw materials such as occurrence of natural and artificial temper/inclusion, organic or inorganic substances used by Neolithic potters in pottery of Northeast India during manufacturing process. Thin section photomicrographs of all Neolithic potsherds are shown in figure 2.1 to 2.4.

2.3.1.1 The description of the thin section matrices of Neolithic pottery

I. DJL

A large sub rounded and fractured Quartz grains floating in a matrix composed irregularly distributed reddish and opaque materials, which forms the matrix. Mineralogically larger fragments are composed a mixture of fine Quartz and clayey materials indicating that these particles are derived from pre-existing rock fragments. Other properties of matrix are difficult to identify. Reddish color may indicate high iron concentration or staining of matrix constituent by ferruginous materials

II. GWKF

Highly angular and Quartz grain sited in a reddish ferruginous matrix. Few larger irregular grains of non-opaque materials are also found which is irregularly distributed throughout matrix. Those grains may be of Quartz.

III. BRP-01

Irregular and fibrous woody materials with cracks spaces between fibrous and crack are filled up by opaque materials. The grains show grey interference colors. Matrix is composed of ferruginous and siliceous materials.

IV.BRP-02

Matrix supported framework showing presence of angular and highly fractured Quartz grains on a reddish micaceous matrix. Elongated strick of vegetative materials are found unevenly disperse throughout into matrix. Irregular opaque bodies may represent ferruginous materials.

V. SMR-01

The specimen is composed of opaque matrix, which shows a reddish linge at places. This indicates high ferruginous materials (iron). The few angular grains of Quartz are found floating in matrix. Quartz grains are of mono-crystalline variety. Larger Quartz grains shows presence of opaque inclusions. These black inclusions may be carbon particles.

VI.SMR-02

Interlocking green supported framework comprising primarily of angular to sub angular Quartz grains show development of fracture. Quartz grains of intermediate sizes show corrosions of grains boundaries (provably due to corrosive action of inter-granular fluid movement). The intergranular spaces are filled up by reddish cementing materials.

VII. SMR-03, SMR-04, SMR-06, SMR-08 and SMR-09

Thin section composed of numerous Hornblende crystals setted in brown ferruginous matrix. The characteristic individual matrix elements are difficult to identify.

VIII. SMR-05

The matrix of thin section shows sub-hedral Feldspar distributed in fine grains ferruginous matrix. The Feldspars are highly weathered.

IX.SMR-07

Thin section matrix shows poorly aligned crystals of Muscovite (mica), Hornblende and Quartz in a fine grain brown ferruginous matrix. There are also irregular distributions of opaque mineral within fine grain matrix.

X. SPT-01

Angular Quartz particle and siliceous rock fragments floating in silty/clayey matrix. Twinkling affects is observed due to presence of slightly coarser Quartz particles within matrix.

XI.SPT-02

Thin Section showing a matrix supported mineralogical framework. Matrix is primarily constituted of fine clayey and siliceous materials together with irregular opaque deposits. A larger framework grain show a micaceous habit with two sets of cleavages aligned perpendicular to each other and is comparatively higher relief together, indicates these to be Kyanite. Concentration of reddish opaque materials along cleavages planes indicate leaching of ferruginous minerals which might have occurred during earlier times.

XII. KM-01

Thin section shows a very fine grain, reddish color matrix in which some small sub-rounded Quartz particle together with some brownish elongated flakes of unknown materials show a parallel alignment. There are also some large irregular aggregate of unknown materials observed.

XIII. KM-02 (figure 3)

Portion of rounded siltstone/mud stone particle composed of fine angular Quartz particle and irregularly shaped opaque materials are cemented together by reddish ferruginous materials.

Sub-angular and elongated particles of pre-existing rock cemented together by ferruginous materials. Pore shapes have an opaque lining and content opaque and non-opaque inclusions.

XIV. KM-03

Thin section shows a very fine, light brown matrix in which contains deep red color elongated rounded siliceous materials (Quartz), Quartz aggregate –silt stone particles etc.

XV. NPC-01, NPC-03

Thin section shows very fine brown color matrix with irregular ferruginous deposits. At some places there is removal of ferruginous materials probably due to leaching.

XVI. NPC-02

Matrix of NPC-02 shows very fine greyish to brown color materials who’s optical character cannot be identify individually. At some places there are irregular depositions of opaque materials.

XVII. NPC-04

Matrix of NPC-04 shows that there is occurrence of nummulites (foraminifera) and gastropod fossils. This probably indicates use of lime stone in preparation of clay during manufacturing pottery.

XVIII. NPC-05 (figure 4)

Specimen is primarily composed of fine micaceous materials and siliceous materials. There is also presence of irregular opaque materials and pore spaces. Pore spaces have a thin lining of opaque materials.

XIX. RYK-01 (figure 5)

Presence of particles with fibrous habit is seen may be of tree trunks/ petrified wood particles. Specimen is primarily composed of fine micaceous materials and siliceous materials. There is also presence of irregular opaque materials and pore spaces.

XX. RYK-02, RYK-03, RYK-04 and RYK-05

Thin section of these potteries show a deep brown granular matrix with presence of numerous sub-angular to sub rounded fragments of fine grain sand stone, silt stone, Quartzite and weathered Feldspar fragments. Matrix of RYK-03 contains woody materials which makes different from others potteries from Ranyak Khen.

XXI. LNGH-01

Matrix is composed of micaceous materials. The opaque may present as carbonaceous matter. There are few irregular grains of Plagioclase Feldspar also identified. They show typical polysynthetic twin. Some irregular Quartz grains are also observed.

XXII. LNGH-02

Matrix of LNGH-02 is composed of dark brownish micaceous with extensive occurrence of angular Quartz grains. Voids spaces are observed. Carbonaceous materials are also identified.

XXIII. LNGH-03

Framework of LNGH-03 is composed of less dense brownish color materials. There is extensive amount of voids spaces, some of which have been found due to removal of vegetative materials. There is also extensive occurrence of partially burnt spaces. Mud fragments and woody materials have been identified.

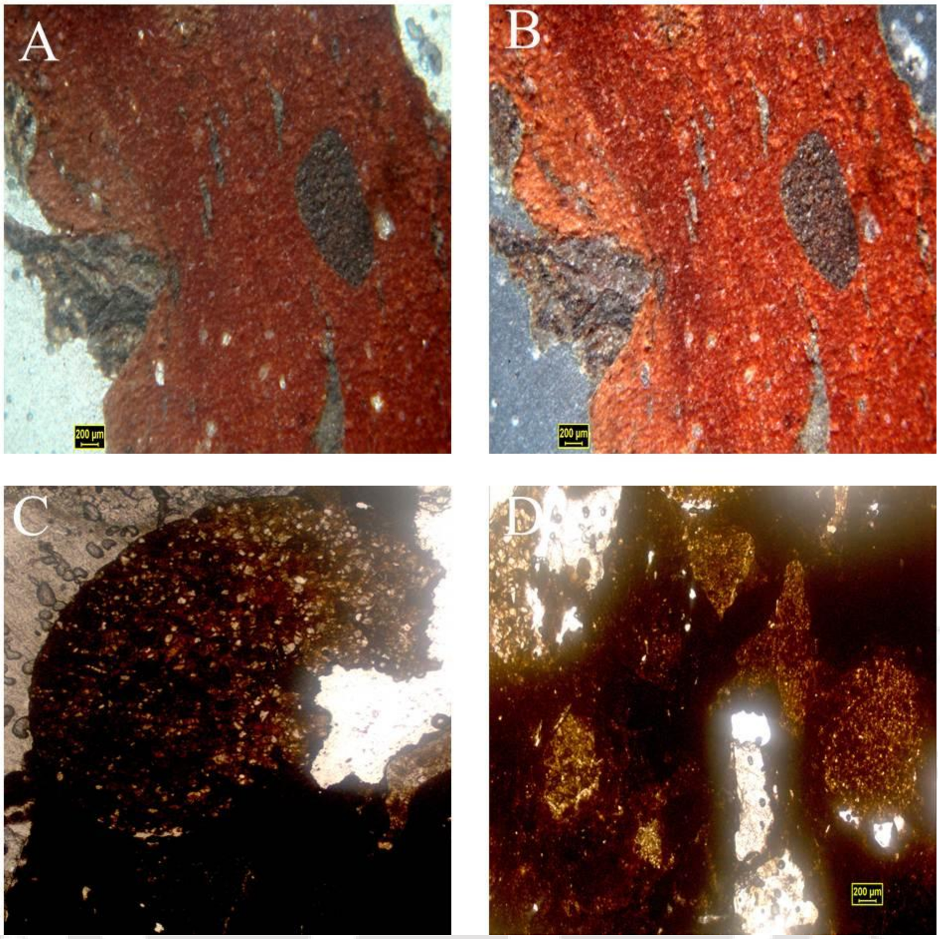

Figure 2.1 Thin Section Photomicrographs (A) KM-01 under transmitted light (B) KM-01 under reflected light (C) KM-02 rounded silt/mud stone fragment (D) KM-02 is showing opaque and non-opaque inclusions.

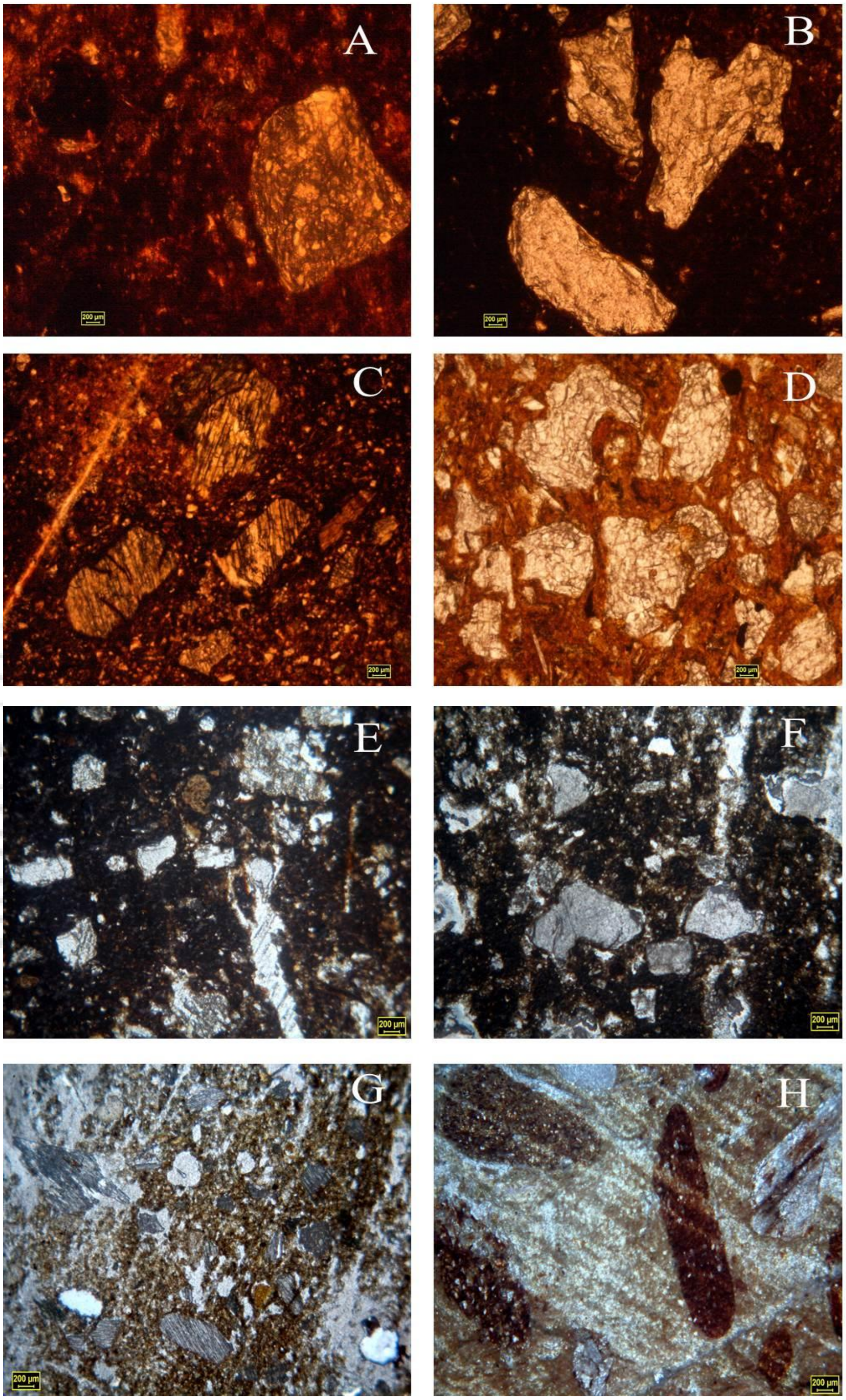

Figure 2.2 Thin Section Photomicrographs of (A) DJL (B) GWKF (C) BRP-01 (D) BRP-02 (E) LNGH-01 (F) LNGH-02 (G) LNGH-03 and (H) KM-03

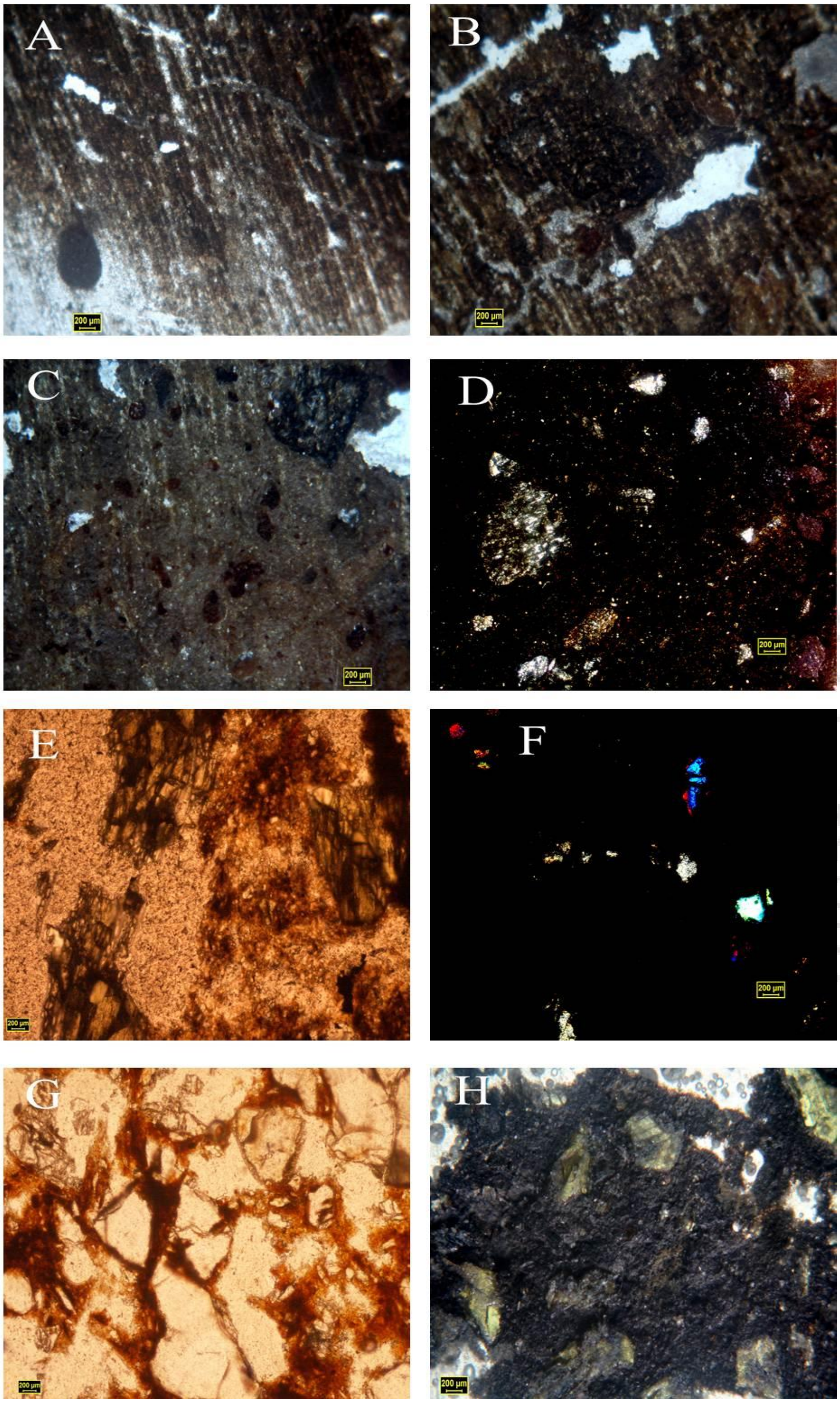

Figure 2.3 Thin Section Photomicrographs of (A) NPC-01 (B) NPC-03 (C) NPC-04 (D) SPT-01 (E) SPT-02 (F) SMR-01 (G) SMR-02 and (H) SMR-03

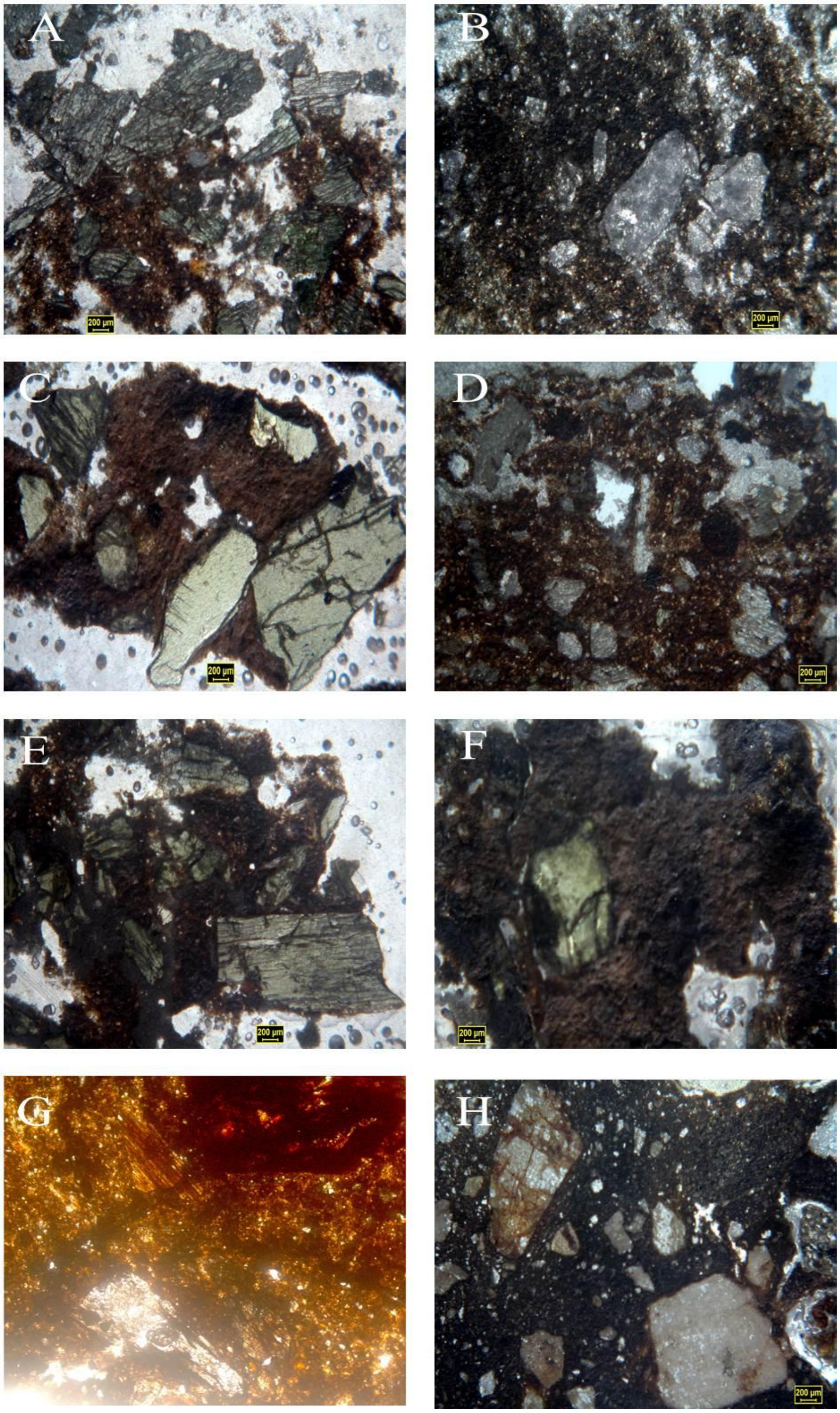

Figure 2.4 Thin Section Photomicrographs of (A) SMR-04 (B) SMR-05 (C) SMR-06 (D) SMR-07 (E) SMR-08 (F) SMR-09 (G) RYK-01 and (H) RYK-02

2.3.1.2 Classification of Neolithic pottery by Thin Section Petrography

Based on its mineralogical content and composition of matrix, thin section of Neolithic potsherds are grouped as A, B, C D, E, F, G, H, I, J and K with fabric group A having A1 and F having subgroups F1 and F2 respectively.

a. Fabric group A

Matrix of fabric group A (DJL and SMR-01) is composed of reddish color ferruginous materials. Mineralogically, larger fragments are composed of a mixture of Quartz and clayey materials indicating that these particles are derived from pre-existing rock fragments. Reddish color indicates high iron concentration. Opaque inclusions are also observed in SMR-01. These black opaque inclusions may be carbon particles.

1.1 Fabric Group A1

Fabric group A1 include KM-02 and GWKF, has reddish color ferruginous material matrix. Grains are angular, sub-angular to elongated and irregular in shape. Silt/mud stone particles which are composed of fine Quartz particle and irregular shaped opaque materials and pre-existing rock cemented together by ferruginous materials are observed. Pores have opaque lining and contain opaque and non-opaque materials.

b. Fabric group B

Thin sections of fabric group B (BRP-01, 02; NPC-05, RYK-01) is composed of fine micaceous and siliceous materials. Grains are elongated and irregular in shape. Presences of particles with fibrous habits are seen in all potsherds in this fabric group. It is observed that Quartz, micaceous and siliceous materials are present in this fabric group.

c. Fabric group C

Fabric group C (SMR-02) have interlocking green color supported framework which composed of angular to sub-angular Quartz grains show development of fracture. The inter-granular spaces are filled up by reddish cementing materials.

d. Fabric Group D

Matrix of SPT-01 and SPT-02 is constituted of fine clayey and siliceous materials together with irregular opaque deposits. Mineralogy includes Kyanite, mica and ferruginous materials.

Concentration of reddish opaque materials along with cleavages planes indicate leaching of ferruginous minerals which might have occurred during earlier times.

e. Fabric Group E

Thin sections falling in fabric group E (KM-01 and KM-03) have reddish to light brown matrix. Grains are sub-rounded to sub rounded elongate in shapes with varying sizes and do not show any specific orientations. Voids are not observed. Mineralogy includes Quartz, siliceous materials, silt stone particles and aggregate Quartz and some unknown irregular aggregate materials observed.

f. Fabric Group F

Matrix of the potsherds (SMR-03, 04, 06, 08, 09; NPC-01, 03) falling under fabric group F have fine grains, brown color ferruginous matrices. Minerals are Quartz and Hornblende identified. The characteristics individual matrix elements are difficult to identify.

i. Fabric Group F1

Matrix of fabric group F and F1 (SMR-05) are same and difference is that F1 matrix show sub-hedral Feldspar, which are highly weathered.

i. Fabric Group F2

Matrices of fabric group F, F1, and F2 (SMR-07) is same and distinctive factor being composition of minerals. Sub fabric group F2 matrix shows poorly aligned crystals of mica (Muscovite), Hornblende and irregular distributions of opaque minerals within matrix.

The main reasons of changes in these thin sections of Neolithic potsherds are attributed to difference in clay preparation and raw materials sources.

g. Fabric Group G

Thin section of NPC-02 is falling under fabric group G has greyish to brown color and composed of very fine materials. Optical character of individual grains cannot be identified. Irregular depositions of opaque materials have been observed at some places of matrix.

h. Fabric Group H

Matrix of thin section falling under fabric group H (NPC-04) shows occurrence of nummulites (foraminifera) and gastropod fossils, which indicates use of lime stone in preparation of clay during manufacturing pottery.

i. Fabric Group I

Thin sections of RYK-02 to 05 are characterized as fabric group I which has deep brown color granular matrices. Grains are sub angular to sub rounded in shapes. Non-plastic inclusion includes Quartz, sand stone, silt stone, Quartzite and weathered silt stone.

j. Fabric Group J

Thin section of LNGH-01 is characterized as fabric group J has micaceous matrix. Mineralogically, Plagioclase Feldspars and Quartz grains observed. Grains are irregular in shape. Carbonaceous substances are also present in matrix.

k. Fabric Group K

Thin section of LNGH-02 and LNGH-03 are falling under Fabric group K. Matrix is composed of dark brown micaceous materials and extensive occurrence of angular Quartz. Voids are observed in matrix. Carbonaceous matters are also present. The burnt spaces, mud fragments and woody materials are observed in LNGH-03.

Neolithic potsherds have been analysed and characterized by using thin section petrography. Characterization of Neolithic potsherds is based on microstructure and mineralogical composition of groundmass. Therefore, characterized Neolithic potsherds and their fabric groups and sub-groups have determined provenance. Thin section analysis indicates that Neolithic pottery is made of primary/residual clay. Matrices of fabric groups and sub fabric groups A, C, D and F and A1, F1, F2 are showing ferruginous habits. While, fabric groups B, J and K are showing micaceous habits. Analysis revealed that homogeneity in existence of fabric along with their secondary materials, such as organic matter and tempering materials. Colour of groundmasses varies from red, green, light brown and brown under plane polarized light. Petrographic results show Quartz is primarily present in most of samples. Plagioclase Feldspars are also observed in fabric group J and sub fabric group F1. All fabric groups have organic substances in their composition. The inclusion of organic substances is not easy to identify that it use intentionally or occurred naturally in their composition. In fabric group H, foraminifera and gastropod fossils are identified. Grains are mostly coarse in size and angular to sub-angular and sub elongate rounded in shape and poorly sorted. The angularity of grains indicates that clay used have formed in-situ and have not travelled long distances which otherwise would have resulted into complete rounding of grains. Rounded and sub-rounded shape is rare in all fabric groups. It is indicating primary clays have been used to make Neolithic pottery. In all fabric groups we found iron bearing materials which show that they use local clay for making pottery, because in region, soil has sufficient amount of ferruginous compound. The raw materials are less tempered and less sieved which might have been required as potters could attain maximum plasticity without using much temper and sieving.

2.4 Mineralogical characterization of Neolithic Pottery

Mineralogical analysis has been carried out on Neolithic pottery from Northeast India. Minerals are identified using XRD, Raman spectroscopy and FTIR in composition of analysed pottery.

Applying to present study, ***Powder XRD*** revealed information about mineralogical phases and composition of prehistoric pottery from North East India. Its patterns with identified crystalline phases of investigated potsherds are shown in Table 2.2 and figure 2.5-2.12.

The major mineral substances identified in all Neolithic pottery are Quartz, Kaolinite, Goethite and Hematite, Biotite, Hornblende. All potsherds contain above minerals in varying quantity.

Quartz is present in all samples as primary mineral. According to Papachristodoulou and co-workers (2006), Quartz is an indigenous mineral present in clay deposits and it is not easy to conform that either added intentionally as tempering materials or present initially. Due to higher rate of expansion, first cracking occur in Quartz crystal during firing of clay paste (Hoard & O'Brien, 1995; Tite, Kilikoglou, & Vekinis, 2001). Pottery workability or functionality may be affected due to Quartz mineral supposed to be used as temper, because it has less resistant to mechanical and thermal shocks.

Kaolinite is identified in all prehistoric potsherds in varying quantity. Tabulated data indicates that Kaolinite is the second most abundant mineral after Quartz in all analysed potsherds.

Feldspars mineral group such as Microcline, Orthoclase and Anorthite are present in most of potsherds. Microcline are present in GWKF, GWKC, NPC-01, 02, 04, 05, SMR-02, 03, 04, KM-03 and RYK-05. Orthoclase is present in NPC-03. In addition, Anorthite is identified in BRP-02; SPT-01; LNGH-01, 03; SMR-02, 03, 06, 07 and RYK-01, 02.

Hematite is identified in most of Neolithic pottery. For instance, Hematite is presents in BRP-01; 02 SPT-02; NPC-02, 03, 05; LNGH-02, 03; SMR-02, 03, 04, 05, 06; KM-02, 03 and RYK-01, 02, 03, 04, 05. Hematite can affect color and firing atmosphere of potsherds. Rutile is presents in BRP-01; LNGH-01, 02, 03; SMR-04, 05, 06 and RYK-01.

Hornblende is present in composition of Neolithic pottery, such as, BRP-01, 02; SPT-02; NPC-01, 02, 04, 05; SMR-01, 02; KM-02, 03 and RYK-01-05. Hornblende is a non-clay magnesium rich minerals which can be defined as an amphibole group of Calcium subgroup with $Fe_2+>Mg$ and $Al>Fe_3+$ in C position and (OH) as dominant ion in W position (Barnes, 1930). Presence of a group of non-clay minerals, such as Feldspars, Biotite, Hornblende, etc., representative of igneous material, may indicate occurrence of volcanic-ash (Grim, 1953, p. 362). Biotite is identified in mineralogical composition of potsherds, such as BRP-01; LNGH-01, 02, 03 and SMR-03, 04, 08, 09.

In addition to XRD, Raman Spectroscopy also provides information about mineralogical composition of analysed Neolithic potsherds. Anatase, Quartz, and Hematite are most common minerals identified in analysed Neolithic pottery. Powder XRD has good agreement with this. Carbonaceous materials are also identified in some samples, such as NPC-01, 03, 05; RYK-04; SPT-01, 02 and SMR-03, 04. Plagioclase is also detected in DJL; RYK-04, 05; SPT-01 SMR-03 and LNGH-01. Presence of Ilmenite in GWKF and SMR-03 (table 2.13) confirmed that composition of raw material is rich in iron and titanium (Olivares, *et al.*, 2010). Wollastonite is identified into KM-01, KM-03, SPT-02 and SMR-03.

Furthermore, FTIR analysis has been used to extract some information about mineralogical composition of Neolithic pottery. FTIR results demonstrated that Quartz is present in all analysed pottery. Hematite has been detected (excluding SPT-01, GWKF, KM-01, KM-02, SMR-06 and SMR-09) in most of potsherds. Microcline is identified in potsherds of DJL, GWKF, GWKC, KM-01, KM-02, NPC-02, SMR-06 and SMR-09.

Powder XRD, Raman spectroscopy and FTIR results reveal mineralogical composition of Neolithic potsherds from archaeological sites of Northeast India. Mineralogical analysis indicates that all Neolithic potsherds contain Quartz in their composition in varying quantity. Discussed earlier, presence of Quartz may be elucidated as indigenous minerals of natural clay or added intentionally as tempering materials are not easy to identify. Therefore, presence of Quartz in raw materials allows improving its workability and gradual vaporization of water (Medeghini, *et al.,* 2013, p. 889; Maggetti, 1982). Feldspar minerals (Anorthite, Microcline and orthoclase) are present in Neolithic potsherds as showed in table 2.2. These minerals are key constituent of original clay matrix. Clay mineral such as Kaolinite is detected in all potsherds in good amounts which indicate that firing temperature would have not been exceeded 700 °C. Also, this is confirmed by presence of Hematite and Anatase in Neolithic potsherds. Presence of Hematite also confirmed that all potsherds are fired in oxidizing atmosphere. Anatase is detected in most of Neolithic potsherds only by Raman spectroscopic analysis. The presence of non-clay minerals such as Hornblende, Feldspars, Biotite which is representative of igneous materials confirmed that volcanic origin of raw materials or metamorphic rock present in area. Rutile is identified only by Powder XRD in BRP-01; LNGH-01, 02, 03; SMR-04, 05, 06 and RYK-01. The presence of both Anatase and Rutile suggest that firing temperature range should be within Anatase-Rutile transformation phase which take place between 600-900 °C. Amorphous carbons and organic substances in Neolithic potsherds have been detected by Raman spectroscopy and FTIR analysis.

Table 2.2 Identified Minerals and phase obtained by XRD of neolithic pottery from Northeast India, ++++ = very abundant, ++ =abundant, + = present, - = absent

Minerals Identified by XRD analysis of samples from Neolithic period											
Si. No.	**Sample name**	**Quartz**	**Kaolinite**	**Hematite**	**Goethite**	**Hornblende**	**Microcline**	**Anorthite**	**Biotite**	**Rutile**	**Orthoclase**
1	DJL	++++++	++	-	+	-	-	-	-	-	-
2	GWKF	++++	++	-	+	-	+	-	-	-	-
3	GWKC	++++++++	+	-	+	-	+	-	-	-	-
4	BRP-01	+++++++++	++	+	+	+	-	-	-	+	-
5	BRP-02	++++++++++++	+++	+	+	+	-	+	+	-	-
6	SPT-01	++++++	+	-	+	-	-	+	-	-	-
7	SPT-02	+++++	++	++	+	+	-	-	-	-	-
8	NPC-01	++++	++	-	+	-	+	-	-	-	-
9	NPC-02	+++	+	++	+	+	+	-	-	-	-
10	NPC-03	+++	+++	+	+	+	-	-	-	-	+
11	NPC-04	+++	++	-	+	+	+	-	-	-	-
12	NPC-05	+++	+	+	+	+	+	-	-	-	-
13	LNGH-01	++++	++	+	-	-	-	+	+	+	-
14	LNGH-02	+++++	++	+	+	-	-	-	+	+	-
15	LNGH-03	++++	+++	-	+	-	-	+	+	+	-
16	SMR-01	++++++++	+	-	+	+	-	-	-	-	-
17	SMR-02	+++++++	+	+	-	+	+	+	-	-	-
18	SMR-03	++++	++++	+	+	-	+	+	+	-	-
19	SMR-04	++++++	+++++	+	+	-	+	-	+	+	-
20	SMR-05	+++++	+++	+	+	-	-	-	-	+	-
21	SMR-06	++++++++	+++++	+	+	-	-	++	-	+	-
22	SMR-07	++++++++	++++	-	+	-	-	+	-	-	-
23	SMR-08	++++++	+++++	+	+	-	-	-	+	-	-
24	SMR-09	++++++	++++	+	+	-	-	-	+	-	-
25	KM-01	+++++++	+++	+	+	-	-	-	-	-	-
26	KM-02	+++++++++	+	+	+	+	-	-	-	-	-
27	KM-03	+++++++++	+++	-	+	+	+	-	-	-	-
28	RYK-01	+++++++++	+++++	+	+	+	-	+	-	+	-
29	RYK-02	++++++++	++++	+	+	+	-	+	-	-	-
30	RYK-03	++++++++	++++	++	+	+	-	-	-	-	-
31	RYK-04	++++++++	+++	+	+	+	-	-	-	-	-
32	RYK-05	+++++++++	++++	+	+	+	+	-	-	-	-

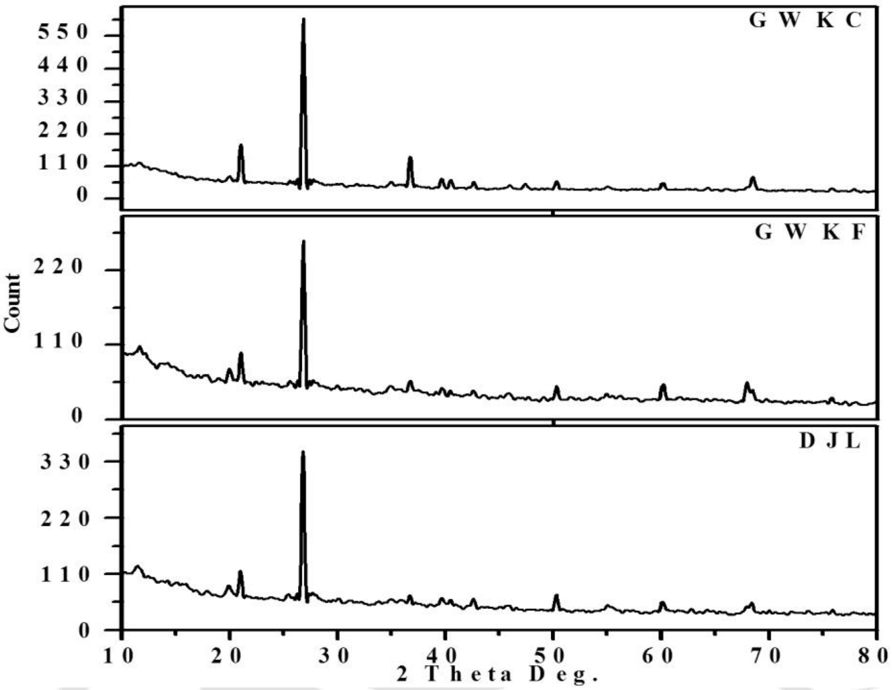

Figure 2.5 XRD Patterns of DJL, GWKC and GWKF

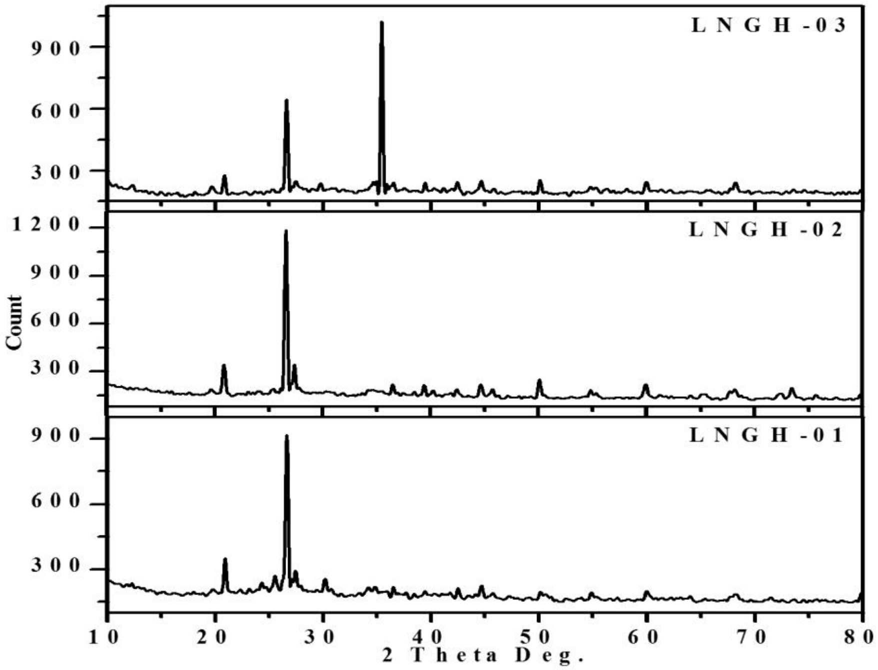

Figure 2.6 XRD Patterns of LNGH-01, LNGH-02 and LNGH-03

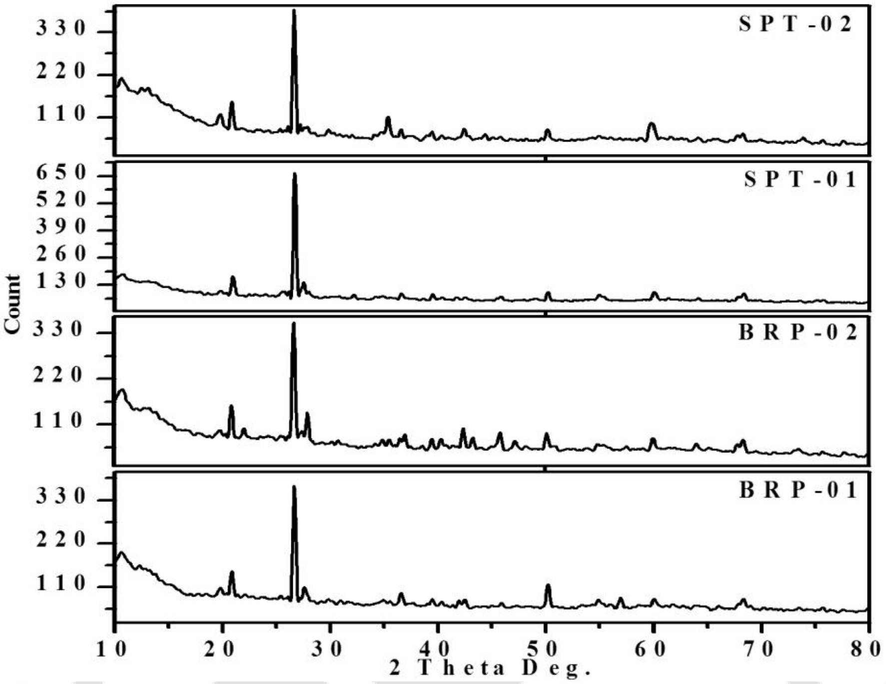

Figure 2.7 XRD Patterns of BRP-01, BRP-02, SPT-01 and SPT-02

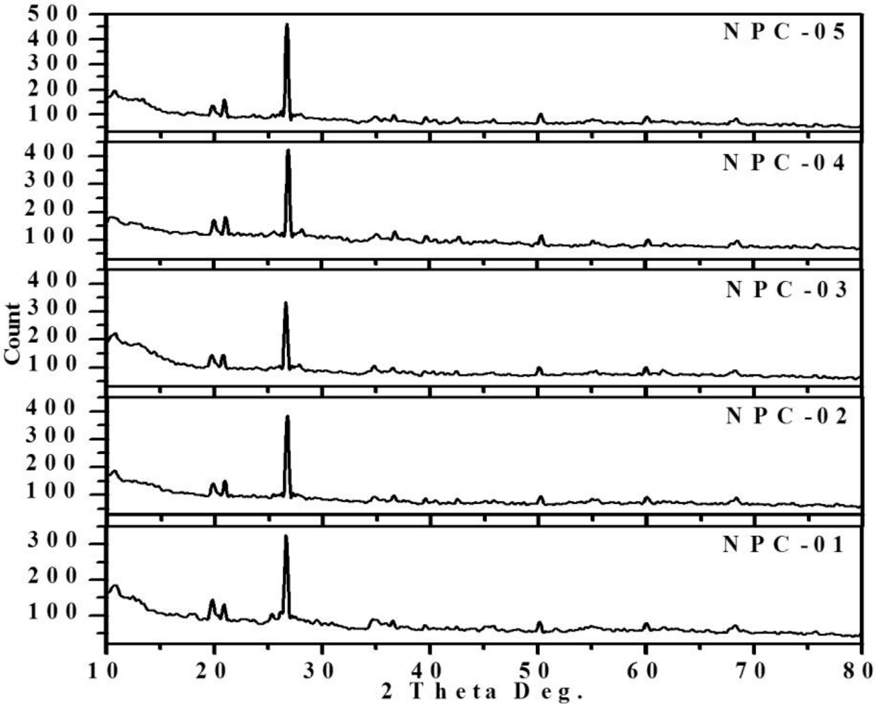

Figure 2.8 XRD Patterns of NPC-01, NPC-02, NPC-03, NPC-04 and NPC-05

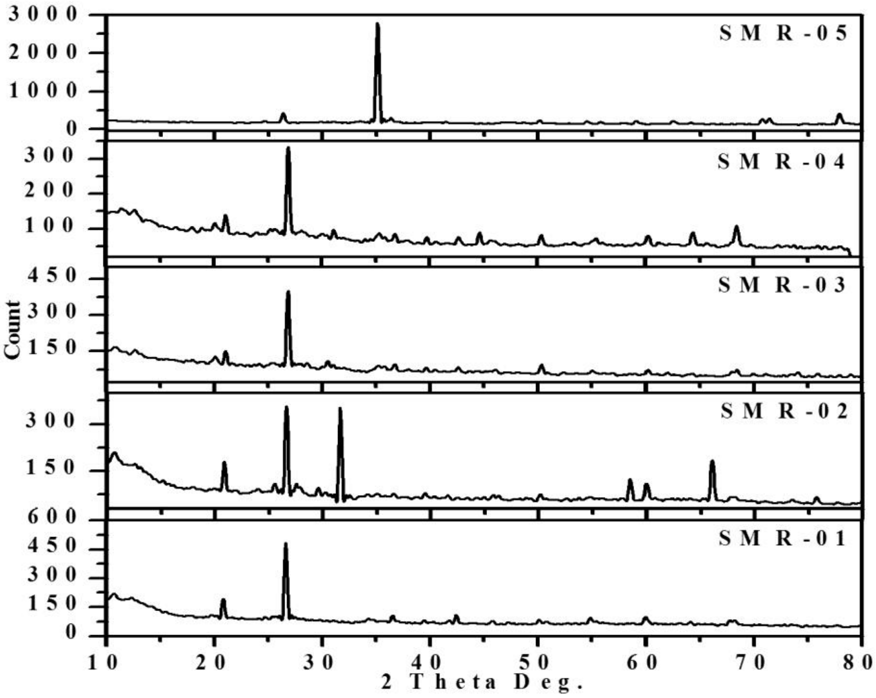

Figure 2.9 XRD Patterns of SMR-01, SMR-02, SMR-03, SMR-04 and SMR-05

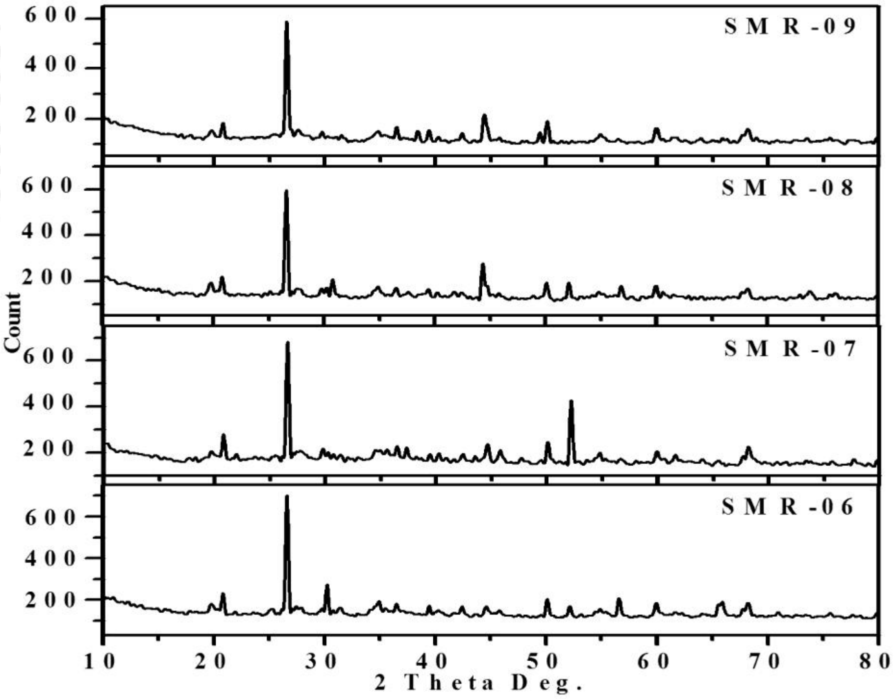

Figure 2.10 XRD Patterns of SMR-06, SMR-07, SMR-08, AND SMR-09

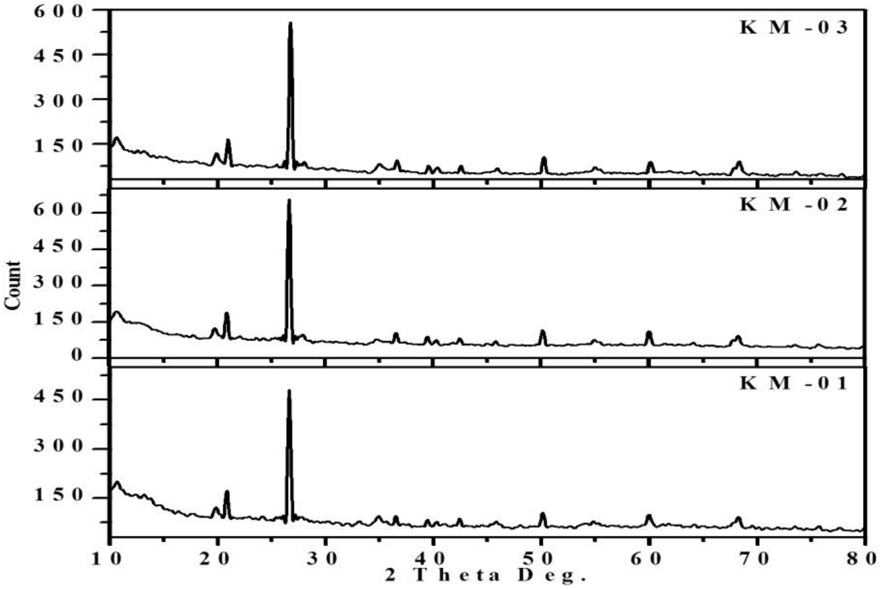

Figure 2.11 XRD Patterns of KM-01, KM-02 and KM-03

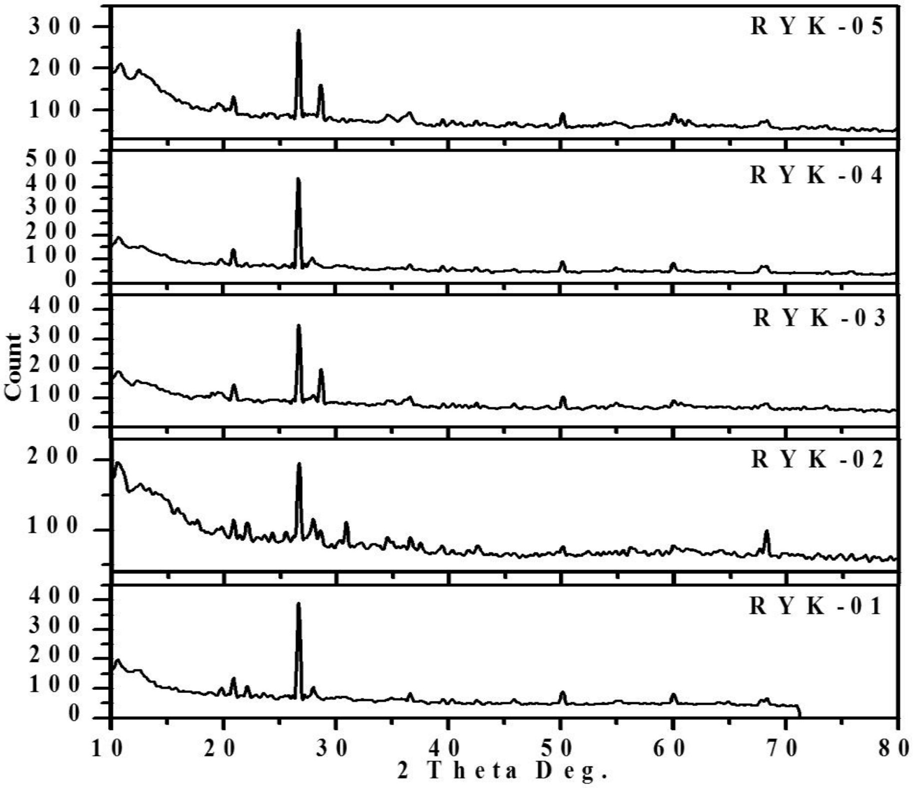

Figure 2.12 XRD Patterns of RYK-01, RYK-02, RYK-03, RYK-04 and RYK-05

2.5 Infrared analysis of Neolithic Pottery

In present study, FTIR is used to determine minimum and maximum firing temperature range and also firing atmosphere (redox condition) of analysed Neolithic potsherds. FTIR spectra, peak positions and their tentative assignments of analysed Neolithic potsherds are summarized in table 2.3 to 2.9 and figure 2.13-16.

The IR spectrum of RYK-03 and RYK-04 show medium bands around 3778 cm^{-1} due to O-H stretching of interlayer water.

The very weak IR spectrum around 3652 cm^{-1} is due to O-H stretching of inner hydroxyl group that is present in KM-03, RYK-03 and RYK-04 (Velraj *et al.*, 2009).

Infrared spectra of KM-03, LNGH-01, 02, 03; NCP-01, 02, 03; SMR-03, 09 and RYK-05, shows very weak band around 3620 cm^{-1} is due to Inner O-H group of adsorbed water.

The medium to strong spectra around 3426 cm^{-1} and a weak to medium spectra around 1630 cm^{-1} (observed in all samples) is due to O-H stretching and O-H bending of absorbed water molecule in samples (Venkatachalapathy *et al.* 2002; Manoharan *et al.*, 2007; Ramasamy & Kamalakannan, 1987; Wolf, 1965a; 1969b).

The weak spectrum at 2950-2220 cm^{-1} is probably due to aliphatic C–H stretching bands as observed in organic materials. Due to higher abundance of silicon based materials, it is difficult to identify C–H stretching bands in samples accurately (Claret, *et al.*, 2003). C–H stretching bands are detected in SPT-01, LNGH-01, 03, KM-01, 03, NPC-02, 04, SMR-01-09 and RYK-02-05.

Infrared bands around 1800 cm^{-1}, 1400 cm^{-1} and 1300 are due to carbonate overtone/ combinations detected in BRP-01, 02; SPT-01; DJL; KM-01, 03; LNGH-01, 02, 03; NPC-01, 02, 03, 04; SMR-03, 0405, 06, 07, 08, 09 and RYK-02, 03, 04, 05.

Maniyatis and Tite (1975) have stated that band around 1034 cm^{-1} is due to red clay origin and band around 1080 cm^{-1} is due to white clay origin. Spectrum around 1079-1100 cm^{-1} with very strong intensity is due to white clay origin of Kaolinite which is present in SPT-01, KM-01, LNGH-03, NPC-03, RYK-02 and RYK-05, that it indicates that white clay origin of Kaolinite was used in making above pottery.

Rest of Neolithic specimen recorded IR spectrum around 1040-1015 cm^{-1} with very strong intensity is due to presence of red clay origin Kaolinite. It indicates that red clay origin of Kaolinite used in making pottery.

LNGH-01 and RYK-04 shows very weak peaks around 920 cm^{-1} is due to Al-OH of octahedral sheet.

The weak bands appear at 778 cm^{-1} and 694 cm^{-1} is due to presence of Quartz as observed in all samples.

The presence of very weak bands around 539 cm^{-1} and 475 cm^{-1} are attributing to presence of Hematite in all samples. Formation of Magnetite and Hematite minerals depends on firing temperature prevalent at time of manufacture. Hematite peak around 535-560 cm^{-1} in all samples indicates that pottery are fired in oxidizing condition (Velraj *et al.* 2009). So, it is concluded that Neolithic men were well aware of technique of firing pottery in oxidizing atmosphere.

The presence of absorption bands in sample around 481 cm^{-1} and 465 cm^{-1} are due to presence of Microcline. Microcline ($KAlSi_3O_8$) is a K-Feldspar. Microcline is present in DJL, SPT-01, GWKF, GWKC, KM-01, KM-02, NPC-02, NPC-04, SMR-06 and SMR-09. Venkatachalapathy and co-workers (2002) have reported that Infrared band of Microcline is free from any temperature effects and stable up to 800 °C (Venkatachalapathy, *et al.*, p. 770, 2002; Akyuz *et al.*, 2008, p. 278).

The FTIR investigation of Neolithic pottery infers firing atmosphere and firing temperature. Determination of firing atmosphere and temperature will provide technological aspects of Neolithic people and extract some information about the kiln. The FTIR analysis of all potsherds indicates firing temperature range should be within 500-700 °C. Presence of Hematite in most of potsherds is indicating towards oxidizing firing atmosphere of Neolithic pottery. This indicates that open firing process (open firing kiln) was used to fire pottery in Neolithic period. C–H stretching spectrum reveals presence of organic matters in composition of pottery. Generally, natural clay contains varying quantity of organic materials in their composition. However, potters may use organic materials externally as additive to improve plasticity during clay preparation. Furthermore, pottery can absorb organic substances during burial period (Damjanovic, *et al.*, 2011, p. 826). The FTIR result has good agreement with thin section petrography and Powder X-Ray diffraction analysis.

Table 2.3 The IR spectra and probable Assignments of DJL, BRP-01, BRP-02, SPT-01, SPT-02 and GWKF

Peak position (cm^{-1})	**As received State**	**Tentative Vibrational Assignments**
DJL		
3426	Medium	O-H stretching of absorbed water molecule
1630	Very Weak	H-O-H bending of water
1034	Very Strong	Si-O-Si (Kaolinite)
797, 694	Very Weak	Si-O of Quartz
559	Very Weak	Fe_2O_3
482	Very Weak	Si-O-Si bending (Microcline)
BRP-01		
3441	Medium	O-H stretching of absorbed water molecule
1639	Very Weak	H-O-H bending of water
1035	Very Strong	Si-O-Si (Kaolinite)
797	Very Weak	Si-O of Quartz
540	Very Weak	Fe_2O_3
BRP-02		
3445	Medium	O-H stretching of absorbed water molecule
1638	Very Weak	H-O-H bending of water
1037	Very strong	Si-O-Si (Kaolinite)
778, 692	Very Weak	Si-O of Quartz
537	Very Weak	or Fe_2O_3
SPT-01		
3419	Medium	O-H stretching of absorbed water molecule
2924, 2853	Very Weak	C–H stretching
1625	Very Weak	H-O-H bending of water
1087	Very Strong	Si-O-Si (Kaolinite)
796, 692, 669	Very Weak	Si-O of Quartz
517	Very Weak	Si-O-M^{IV}
466	Very Weak	Si-O-Si bending (Microcline)
SPT-02		
3419	Medium	O-H stretching of absorbed water molecule
1637	Very Weak	H-O-H bending of water
1037	Very Strong	Si-O-Si (Kaolinite)
797, 778, 693, 668	Very Weak	Si-O of Quartz
541	Very Weak	Fe_2O_3
GWKF		
3448	Medium	O-H stretching of absorbed water molecule
1631	Very Weak	H-O-H bending of water
1043	Very Strong	Si-O-Si (Kaolinite)
797, 778, 693	Very Weak	Si-O of Quartz
479	Very Weak	Si-O-Si bending (Microcline)

Table 2.4 The IR spectra and probable Assignments of GWKC, LNGH-01, LNGH-02, LNGH-03 and KM-01

GWKC		
Peak position (cm^{-1})	**As received State**	**Tentative Vibrational Assignments**
3430	Medium	O-H stretching of absorbed water molecule
1628	Weak	H-O-H bending of water
1043	Very Strong	Si-O-Si (Kaolinite)
797, 777	Weak	Si-O of Quartz
693	Very Weak	Fe_2O_3
481	Medium	Si-O-Si bending (Microcline)
LNGH-01		
3620	Medium	Inner O-H group of adsorbed water
2925	Very Weak	C–H stretching
1635	Very Strong	H-O-H bending of water
1384	Very Weak	Carbonate overtone/combinations
1031	Very Strong	Si-O-Si (Kaolinite)
920	Shoulder Very Weak	Al-OH
777, 694	Very Weak	Si-O of Quartz
546	Very Weak	Fe-O of Hematite
LNGH-02		
3566	Very Strong	Inner O-H group of adsorbed water
1635	Very Strong	H-O-H bending of water
1384	Very Weak	Carbonate overtone/combinations
777, 694	Very Weak	Si-O of Quartz
560	Very Weak	Fe-O of Hematite
LNGH-03		
3595	Very Strong	Inner O-H group of adsorbed water
2921, 2852, 2356	Very Weak	C–H stretching
1635	Very Strong	H-O-H bending of water
1392	Very Weak	Carbonate overtone/combinations
1090	Very Strong	Si-O-Si (Kaolinite)
797, 693	Very Weak	Si-O of Quartz
559	Very Weak	Fe-O of Hematite
KM-01		
3485	Medium	O-H stretching absorbed water molecule
2921, 2346	Very Weak	C–H stretching
1639	Very Weak	H-O-H bending of water
1384	Very Weak	Carbonate overtone/combinations
1083	Very Strong	Si-O-Si (Kaolinite)
1035	Very Weak	Si-O-Si (Kaolinite)
797, 778	Weak	Si-O of Quartz
694	Very Weak	Si-O of Quartz
482	Very Weak	Microcline

Table 2.5 The IR spectra and probable Assignments of KM-02, KM-03, NPC-01, NPC-02, NPC-03

Peak position (cm^{-1})	As received State	Tentative Vibrational Assignments
KM-02		
3443	Medium	O-H stretching of absorbed water molecule
1639	Very Weak	H-O-H bending of water
1036	Very Strong	Si-O-Si (Kaolinite)
797, 778, 694, 668	Very Weak	Si-O of Quartz
480	Very Weak	Si-O-Si bending (Microcline)
KM-03		
3647	Weak	O-H stretching of inner hydroxyl group
3375	Very Weak	Inner O-H group of adsorbed water
2921, 2859	Very Weak	C–H stretching
1883	Very Weak	Carbonate overtone/combinations
1688	Very Weak	H-O-H bending of water
1037	Very Strong	Si-O-Si (Kaolinite)
798, 778	Weak	Si-O of Quartz
694	Very Weak	Si-O of Quartz
559	Very Weak	Fe-O of Hematite
NPC-01		
3639	Medium	Inner O-H group of adsorbed water
2070	Very Weak	C–H stretching
1636	Medium	H-O-H bending of water
1384	Very Weak	Carbonate overtone/combinations
1041	Very Strong	Si-O-Si (Kaolinite)
798, 778, 694	Very Weak	Si-O of Quartz
560, 474	Very Weak	Fe-O of Hematite
NPC-02		
3552	Medium	Inner O-H group of adsorbed water
2924, 2279	Very Weak	C–H stretching
1636	Medium	H-O-H bending of water
1384	Very Weak	Carbonate overtone/combinations
1037	Very Strong	Si-O-Si (Kaolinite)
798, 778,694	Very Weak	Si-O of Quartz
560	Very Weak	Fe-O of Hematite
480	Very Weak	Microcline
NPC-03		
3631	Medium	Inner O-H group of adsorbed water
3186	Very Weak	Inner O-H group of adsorbed water
1880	Very Weak	Carbonate overtone/combinations
1639	Medium	H-O-H bending of water
1100	Medium	Si-O-Si (Kaolinite)
798, 778	Very Weak	Si-O of Quartz
694	Very Weak	Si-O of Quartz
563	Very Weak	Fe-O of Hematite
476	Very Strong	Fe-O of Hematite

Table 2.6 The IR spectra and probable Assignments of NPC-04, NPC-05, SMR-01, SMR-02 and SMR-03

NPC-04		
Peak position (cm^{-1})	**As received State**	**Tentative Vibrational Assignments**
3442	Medium	O-H stretching absorbed water molecule
2924, 2849	Very Weak	C–H stretching
1635	Weak	H-O-H bending of water
1384	Very Weak	Carbonate overtone/combinations
1033 VS	Very Strong	Si-O-Si (Kaolinite)
798, 778	Very Weak	Si-O of Quartz
477	Very Weak	Microcline
NPC-05		
3440	Medium	O-H stretching of absorbed water molecule
1636	Very weak	H-O-H bending of water
1100	Very strong	Si-O-Si (Kaolinite)
798, 778, 693	very weak	Si-O of Quartz
548	Very weak	Fe_2O_3
SMR-01		
3444	Very Strong	O-H stretching of absorbed water molecule
2921, 2850	Sh. VW	C–H stretching
1638	Medium	H-O-H bending of water
1026	Very Strong	Si-O-Si (Kaolinite)
783	Very Weak	Si-O of Quartz
539, 475	Very Weak	Fe-O of Hematite
SMR-02		
3444	Medium	O-H stretching of absorbed water molecule
2910	Sh. VW	C–H stretching
1640	Weak	H-O-H bending of water
1027	Very Strong	Si-O-Si (Kaolinite)
796	Medium	Si-O of Quartz
691	Very Weak	Si-O of Quartz
527, 472	Very Weak	Fe-O of Hematite
SMR-03		
3626	Very Weak	Inner O-H group of adsorbed
3436	Medium	O-H stretching of absorbed water molecule
2926, 2850	Sh. VW	C–H stretching
1877	Very Weak	Carbonate overtone/combinations
1635	Very Weak	H-O-H bending of water
1035	Very Strong	Si-O-Si (Kaolinite)
920	Sh. VW	Al-OH
798, 776	Medium	Si-O of Quartz
694, 673	Very Weak	Si-O of Quartz
633	Very Weak	Al-O co-ordination vibration
523, 472	Very Weak	Fe-O of Hematite

Table 2.7 The IR spectra and probable Assignments of SMR-04, SMR-05 SMR-06 and SMR-07

	SMR-04	
Peak position (cm^{-1})	**As received State**	**Tentative Vibrational Assignments**
3436	Very Strong	O-H stretching of absorbed water molecule
2923, 2850	Sh. VW	C–H stretching
1877	Very Weak	Carbonate overtone/combinations
1637	Very Weak	H-O-H bending of water
1035	Very Strong	Si-O-Si (Kaolinite)
798, 779, 694	Very Weak	Si-O of Quartz
634	Very Weak	Al-O co-ordination vibration
515	Very Weak	Si-O-M
471	Very Weak	Fe_2O_3
	SMR-05	
3448	Very Strong	O-H stretching of absorbed water molecule
2923, 2850	Shoulder Very Weak	C–H stretching
2356	Very Weak	C–H stretching
1885	Very Weak	Carbonate overtone/combinations
1631	Medium	H-O-H bending of water
1392	Very Weak	Carbonate overtone/combinations
1035	Very Strong	Si-O-Si (Kaolinite)
797	Medium	Si-O of Quartz
693	Very Weak	Si-O of Quartz
647	Very Weak	Al-O co-ordination vibration
535	Very Weak	Fe-O of Hematite
472	Medium	Fe-O of Hematite
	SMR-06	
3436	Medium	O-H stretching of absorbed water molecule
2923, 2850	Shoulder Very Weak	C–H stretching
1877	Very Weak	Carbonate overtone/combinations
1636	Very Weak	H-O-H bending of water
1036	Very Strong	Si-O-Si (Kaolinite)
798, 778, 694	Very Weak	Si-O of Quartz
515	Very Weak	Si-O-M
482	Strong	Si-O-Si bending (Microcline)
	SMR-07	
3437	Medium	O-H stretching of absorbed water molecule
2923, 2850	Shoulder Very Weak	C–H stretching
1882	Very Weak	Carbonate overtone/combinations
1637	Very Weak	H-O-H bending of water
1035	Very Strong	Si-O-Si (Kaolinite)
798, 778, 694	Very Weak	Si-O of Quartz
529	Very Weak	Fe-O of Hematite
471	Medium	Fe-O of Hematite

Table 2.8 The IR spectra and probable Assignments of SMR-08, SMR-09, RYK-01, RYK-02, and RYK-03

SMR-08		
Peak position (cm^{-1})	**As received State**	**Tentative Vibrational Assignments**
3437	Medium	O-H stretching of absorbed water molecule
2923, 2850	Shoulder Very Weak	C–H stretching
1882	Very Weak	Carbonate overtone/combinations
1634	Very Weak	H-O-H bending of water
1035	Very Strong	Si-O-Si (Kaolinite)
798, 776, 694	Very Weak	Si-O of Quartz
526	Very Weak	Fe-O of Hematite
472	Medium	Si-O-Si bending (Microcline)
SMR-09		
3639	Medium	Inner O-H group of adsorbed water
3527	Medium	Inner O-H group of adsorbed water
2922	Very Weak	C–H stretching
1639	Medium	H-O-H bending of water
1384	Very Weak	Carbonate overtone/combinations
1044	Very Strong	Si-O-Si (Kaolinite)
798, 778	Very Weak	Si-O of Quartz
481	Very Weak	Si-O-Si bending (Microcline)
Ranyak Khen (RYK-01)		
3418	Medium	O-H stretching of absorbed water molecule
1647	Very weak	H-O-H bending of water
1020	Very strong	Si-O-Si (Kaolinite)
668	Very weak	Si-O of Quartz
534, 471	Very weak	Fe_2O_3
RYK-02		
3482	Medium	O-H stretching of absorbed water molecule
2924, 2850, 2605	Very weak	C–H stretching
1886	Very weak	Carbonate overtone/combinations
1635	Medium	H-O-H bending of water
1384	Very weak	Carbonate overtone/combinations
1081	Very strong	Si-O-Si (Kaolinite)
798, 778, 694	Very weak	Si-O of Quartz
471	Very weak	Fe_2O_3
RYK-03		
3777	Medium	O-H stretching of inter layer water
3660	Medium	O-H stretching on inner hydroxyl group
3484	Medium	O-H stretching of absorbed water molecule
2924, 2853, 2000	Very weak	C–H stretching
1639	Medium	H-O-H bending of water
1417, 1384	Very weak	Carbonate overtone/combinations
1018	Very strong	Si-O-Si (Kaolinite)
798, 778, 669	Very weak	Si-O of Quartz
535	Very weak	Fe_2O_3

Table 2.9 The IR spectra and probable Assignments of RYK-04 and RYK-05

RYK-04		
Peak position (cm^{-1})	**As received State**	**Tentative Vibrational Assignments**
3778	Medium	O-H stretching of inter layer water
3660	Medium	O-H stretching of inner hydroxyl group
3552	Very strong	Inner O-H group of adsorbed water
2925, 2854, 2713, 2254	Very weak	C–H stretching
1802	Very weak	Carbonate overtone/combinations
1644	Medium	H-O-H bending of water
1421, 1384	Very weak	Carbonate overtone/combinations
978	Very strong	Al-OH
798, 778	Very weak	Si-O of Quartz
670	Strong	Si-O of Quartz
543	Very weak	Fe-O of Hematite
RYK-05		
3553	Very strong	Inner O-H group of adsorbed water
2924, 2853, 2638	Very weak	C–H stretching
1645	Very weak	H-O-H bending of water
1384	Very weak	Carbonate overtone/combinations
1097	Very strong	Si-O-Si (Kaolinite)
796, 778	Very weak	Si-O of Quartz
670	Medium	Si-O of Quartz
534	Very weak	Fe-O of Hematite

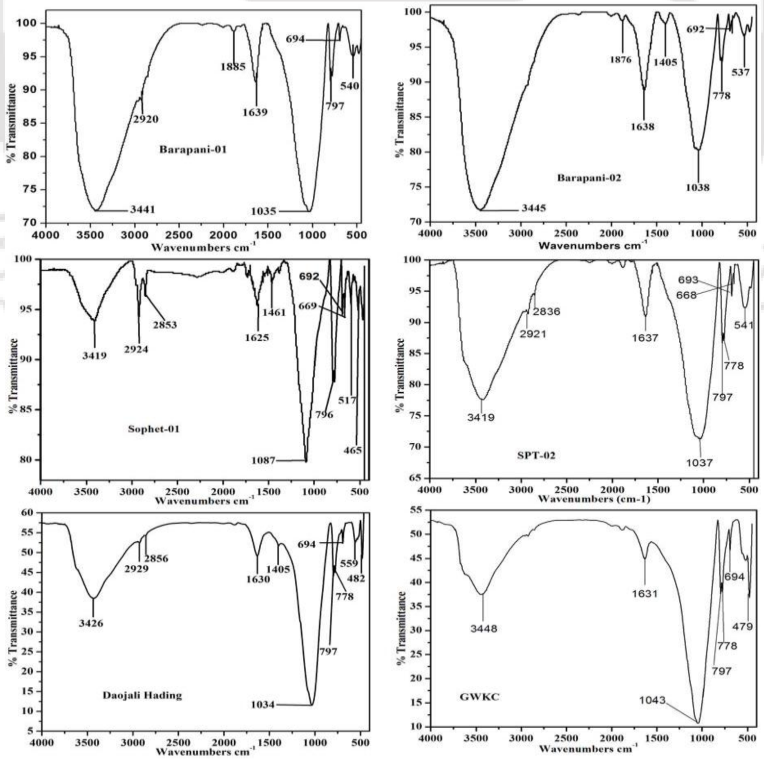

Figure 2.13 FTIR Spectra of BRP-01, BRP-02, SPT-01, SPT-02, DJL and GWKC

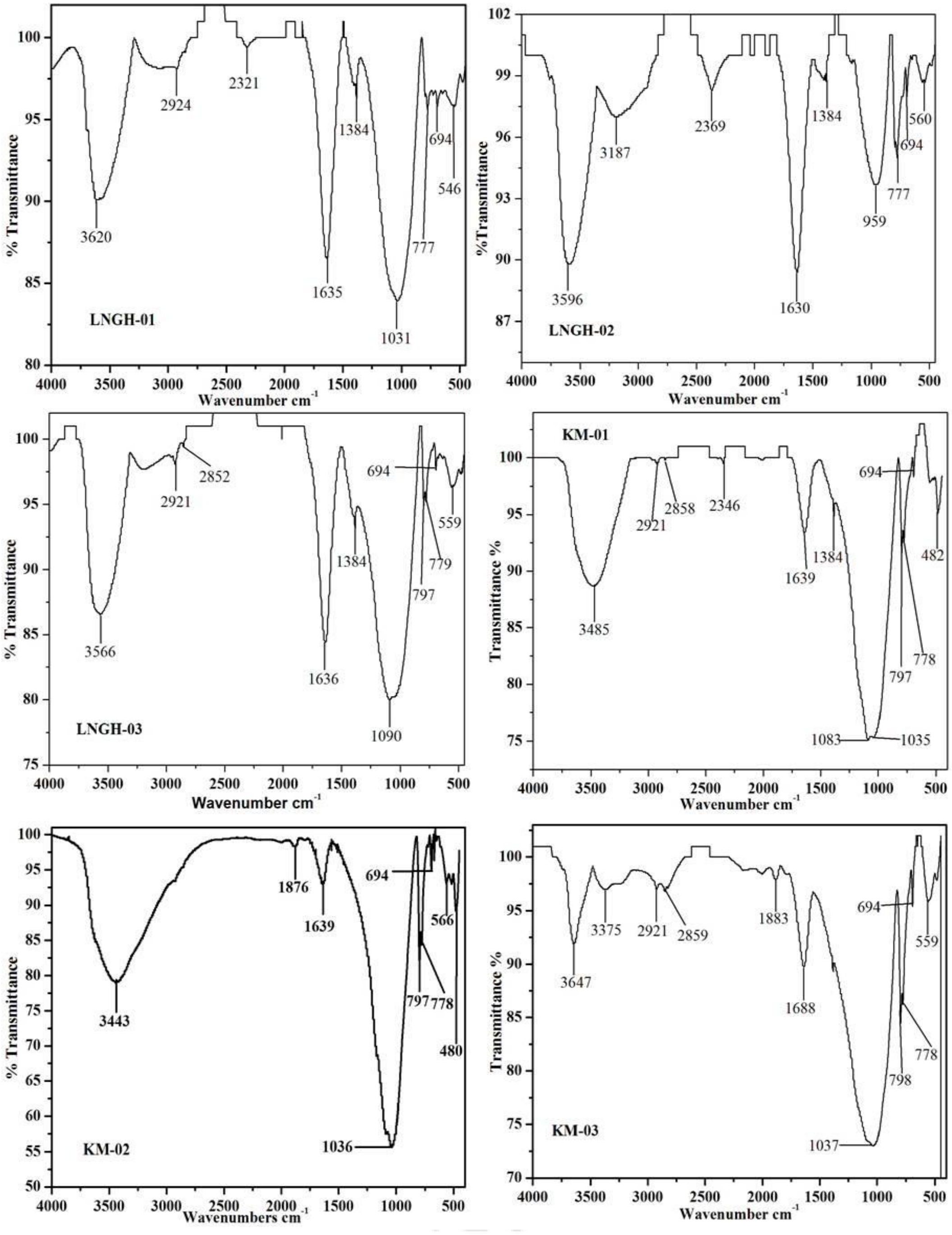

Figure 2.14 FTIR Spectra of LNGH-01, LNGH-02, LNGH-03, KM-01, KM-02 and KM-03

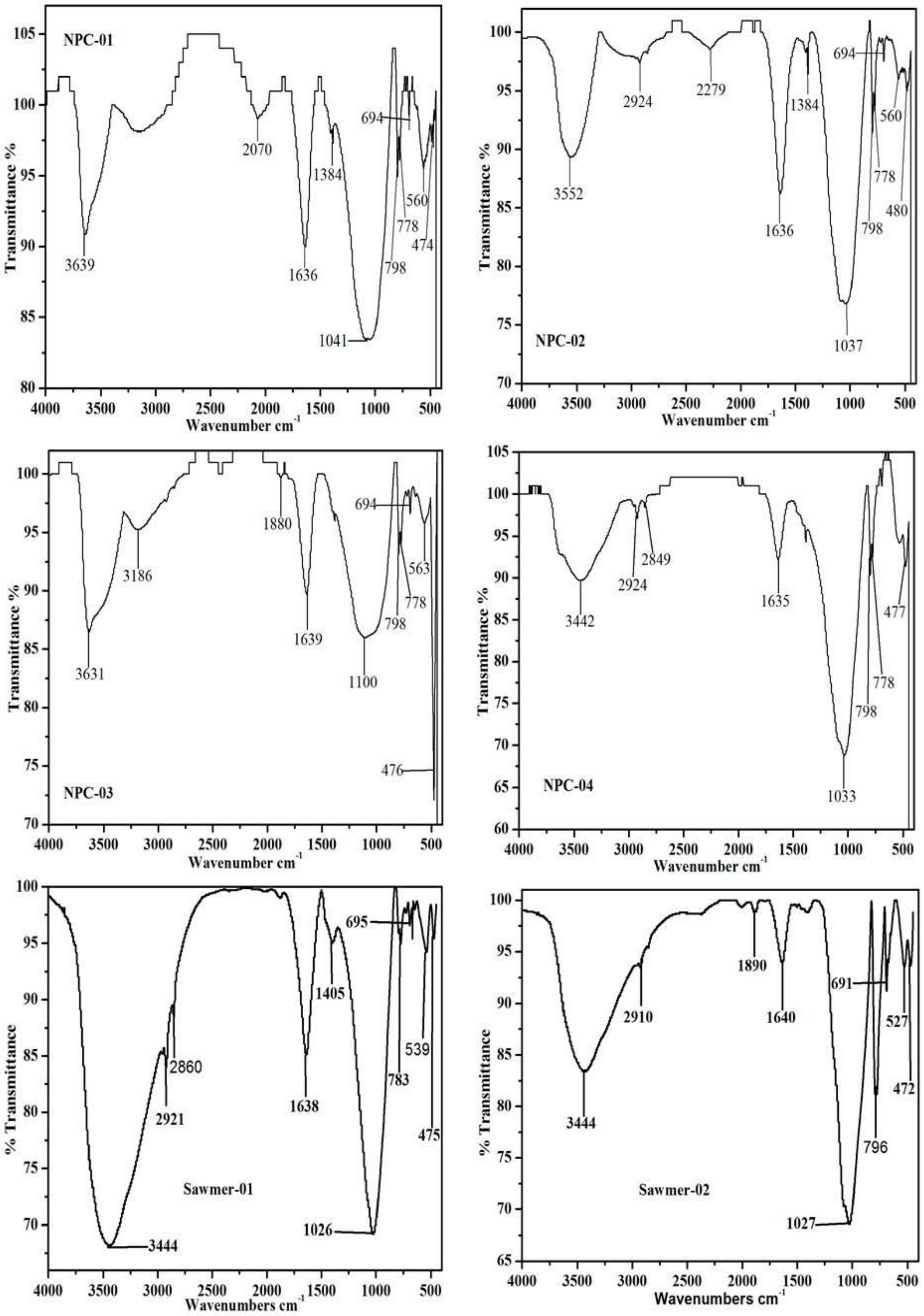

Figure 2.15 FTIR spectra of NPC-01, NPC-02, NPC-03, NPC-04, SMR-01 and SMR-02

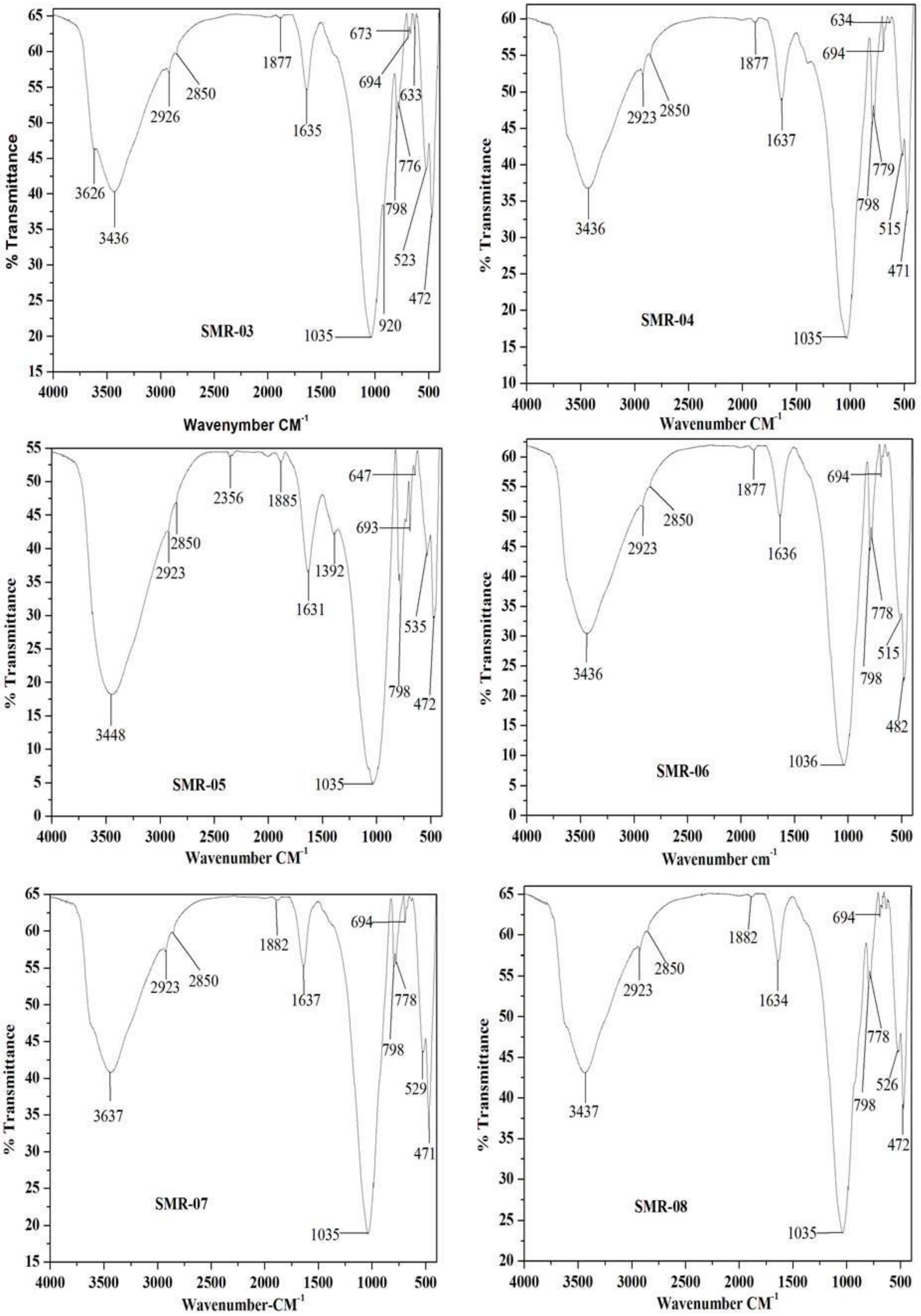

Figure 2.16 FTIR spectra of SMR-03, SMR-04, SMR-05, SMR-06, SMR-07 and SMR-08

2.6 Characteristic Study of Neolithic pottery of Northeast India using micro-Raman Spectroscopy

In present study, micro-Raman spectroscopy is used to determine firing temperature range and firing atmosphere of analysed potsherds achieved by the Neolithic potters of northeast India during manufacturing process. Raman spectrum and tentative assignment and Raman spectra of Neolithic potsherds from Northeast India are given in table 2.10 to 2.12 and figure 2.17-2.19.

The weak to medium distinctive Raman peaks due to symmetric bending vibration (Si-O-Si) of Quartz at 129, 130, 263, 462, 464, 466,467 and 470 cm^{-1} are identified and present in most of Neolithic potsherds (table 2.10-2.12). Asymmetric bending mode of silica tetrahedral is identified due to peaks at 194 and 360 cm^{-1} in samples - GWKC, GWKF, SPT-02 (table 2.10-2.12) of Raman spectrum. Quartz is identified in GWKC, GWKF, BRP-01,02, KM-02, NPC-04, 05, SPT-01, 02, SMR-01, 02, 03 and LNGH-01-03. It is well-known fact that Quartz is normally present in ancient ceramics, and this indicates that raw materials are rich in silica (Olivares, *et al.*, p. 1545, 2010; Liou *et al.*, p. 1063, 2011). On basis of XRD, thin section, EDX and FTIR results, Quartz is most abundant mineral present in composition of analysed potsherds.

Raman spectrum of Ilmenite (Fe_2TiO_3) showed weak Raman band at 228 and 360 cm^{-1}. Presence of Ilmenite in GWKF, SPT-02, and SMR-03 (table 2.10-2.12) confirmed that raw material is rich in iron and titanium (Olivares, *et al.*, p. 1545, 2010; Liou, *et al.*, p. 1066, 2011).

The characteristic Raman peaks of Hematite at 226, 296, 410, 612, 1320 cm^{-1} (table 2.10-12) are detected in analysed potsherds such as GWKC, GWKF, SPT-02, KM-01, NPC-02, NPC-05, RYK-04, 05, SMR-01, 03, 04, LNGH-01 and LNGH-02. Magnetite is identified due to presence of Raman band at 666 cm^{-1} in SMR-04 (table 2.10-2.12). Olivares, *et al.* (2010) stated that analysis of nature of oxidation state of iron can be useful to determine firing condition of clay in which it is fired. Refering to this, presence of black iron oxides (Magnetite) indicates that firing condition would be reductive. While, presence of reddish iron oxides (Hematite) indicates that firing environment would be oxidative. Presences of Hematite in all analysed potsherds are also confirmed by XRD and FTIR. EDX data has also indicated that iron oxide is present in all analysed potsherds. According to this, presence of Hematite indicates that all potsherds might have been fired under oxidative environment (Olivares, *et al.*, 2010; Liou *et al*., 2011; Striova, *et al.*, p. 1142, 2006).

The weak, medium and strong distinctive Raman peaks at 144, 153, 195, 202, 205, 395, 512, 636 cm^{-1} due to modes of Anatase identified in (table 2.10-2.12) Neolithic pottery – DJL, KM-01, 03, NPC-01, 02, 03, 05, SPT-02, SMR-01, 02, 03, 04, 06, LNGH-01 and LNGH-02. Olivares, *et al.* (2010) and Striova, *et al.*(2006) stated that Anatase is a common constituent of soil minerals and is usually extant as additional component in Kaolin. It is reported by various scholars that Raman Spectra of clay minerals are frequently masked in occurrence of Anatase. Referring to this, presence of Anatase in analysed samples may be part of raw materials or clay composition.

Raman Spectra of 1285 to 1600 cm^{-1} is detected due to carbon black materials present in Neolithic pottery – KM-02, NPC-01, 02, 03, 05, SPT-01, 02, SMR-01, 05 and RYK-04 (table 2.10-2.12) (Smith & Clark, 2004, p. 1146). According to Liou *et al*. (p. 1064, 2011), carbon black materials may presents naturally in clay source, or added intentionally as organic materials, for example, plant ash, straw, fiber etc. by potter during manufacturing pottery, or it may be deposited during using or burial on surface of ceramics.

Raman Spectroscopy has been used to get important information about minimum firing temperature and firing atmosphere in which Neolithic pottery were manufactured. Technologically, micro-Raman results demonstrate that most of analysed pottery fired in between 500-700 °C. This is confirmed by presence of only Anatase in most of analysed potsherds, because Anatase begins to transform into Rutile at 750-900 °C. The presence of Anatase and absence of Rutile indicates that most of Neolithic potsherds might have been fired below Anatase-Rutile transition phase. Presence of Hematite refers to oxidative atmosphere under which all potsherds might have been produced.

Mineralogically, micro-Raman analysis revealed that all analysed potsherds have Quartz, Anatase, Hematite and Feldspar minerals as key constituent in clay composition which is also confirmed by thin section, Powder XRD and FTIR analyses. Besides these minerals, other identified constituents in analysed potsherds are carbon black materials, Ilmenite and Wollastonite.

Table 2.10 Peak positions and tentative assignment of Neolithic potsherds

DJL		
Raman Shift (cm^{-1})	**As received State**	**Tentative Vibrational Assignments**
149	Weak	Modes of Anatase
510	Weak	Plagioclase
783	Weak	Si-O Quartz
GWKC		
129, 209, 265	Very Weak	Due the symmetric bending vibration (Si-O-Si) Quartz
356	Very Weak	Due to the asymmetric bending modes of the silica tetrahedra
470	Very Strong	Due the symmetric bending vibration (Si-O-Si) Quartz
GWKF		
134, 217	Very Weak	Due the symmetric bending vibration (Si-O-Si) Quartz
360	Very Weak	Ilmenite
470	Very Strong	Due the symmetric bending vibration (Si-O-Si) Quartz
919	Very Weak	Si-O Quartz
BRP-01		
467	Weak	Due the symmetric bending vibration (Si-O-Si) Quartz
BRP-02		
134, 213	Very Weak	Due the symmetric bending vibration (Si-O-Si) Quartz
470	Weak	Due the symmetric bending vibration (Si-O-Si) Quartz
KM-01		
156	Very Strong	Modes of Anatase
398, 635	Very Weak	Modes of Anatase
288	Very Weak	Hematite
KM-02		
466	Very Weak	Due the symmetric bending vibration (Si-O-Si) Quartz
1610	Very Weak	Carbon Black Material
KM-03		
145	Very Strong	Modes of Anatase
398, 643	Very Weak	Modes of Anatase
NPC-01		
146, 640	Very Weak	Modes of Anatase
640	Very Weak	Modes of Anatase
1604	Strong	Carbon Black Material
NPC-02		
156	Very Strong	Modes of Anatase
205	Very Weak	Si-O of Quartz
1326	Very Weak	Carbon Black Material
NPC-03		
142	Very Weak	Modes of Anatase
1610	Very Weak	Carbon Black Material

Table 2.11 Peak positions and tentative assignment of Neolithic pottery

NPC-04		
Raman Shift (cm^{-1})	**As received State**	**Tentative Vibrational Assignments**
124, 466	Very Weak	Due the symmetric bending vibration (Si-O-Si) Quartz
NPC-05		
153	Very Weak	Modes of Anatase
1621	Very Weak	Carbon Black Material
RYK-04		
591	Weak	Quartz
676	Weak	Hematite
1600	Weak	Carbon Black Material
RYK-05		
288	Very Weak	Hematite
413	Very Weak	Hematite
477	Very Weak	Due the symmetric bending vibration (Si-O-Si) Quartz
511	Strong	Plagioclase
SPT-01		
182	Very Weak	Quartz (Si-O-Si)
265	Very Weak	Due the symmetric bending vibration (Si-O-Si) Quartz
450	Very Weak	Due the symmetric bending vibration (Si-O-Si) Quartz
515	Very Weak	Plagioclase
1613	Strong	Carbon Black Material
SPT-02		
141, 194	Very Weak	Modes of Anatase
232	Very Weak	Hematite
326	Strong	Hematite
390	Strong	Ilmenite
507	Very Weak	Plagioclase
670	Very Strong	Hematite
828	Very Weak	Si-O Quartz
1013	Very Strong	Gypsum
1043	Very Weak	Wollastonite
1610	Very Weak	Carbon Black Material
SMR-01		
271	Very Strong	Hematite
285	Very Strong	Due the symmetric bending vibration (Si-O-Si) Quartz
394	Very Weak	Modes of Anatase
538	Very Weak	Calcium Oxalate (hydrated)
1613	Very Weak	Carbon Black Material
SMR-02		
153	Very Weak	Modes of Anatase
790	Weak	Si-O of Quartz
964	Very Weak	Due to the Phosphate

Table 2.12 Peak positions and tentative assignment of Neolithic pottery

SMR-03		
Raman Shift (cm^{-1})	**As received State**	**Tentative Vibrational Assignments**
141	Very Strong	Modes of Anatase
186	Very Weak	Quartz (Si-O-Si)
232, 254, 534	Very Weak	Hematite
326	Strong	Hematite
360	Very Weak	Ilmenite
394	Strong	Modes of Anatase
511	Very Weak	Plagioclase
670	Very Strong	Hematite
824, 858	Very Weak	Si-O of Quartz
1013	Very Strong	Gypsum
1043	Very Weak	Wollastonite
SMR-04		
137, 164	Very Weak	Modes of Anatase
326	Strong	Hematite
394	Strong	Modes of Anatase
666	Strong	Magnetite
1006	Strong	Gypsum
SMR-05		
1610	Very Weak	Carbon Black Material
SMR-06		
154	Very Weak	Modes of Anatase
514	Very Weak	Plagioclase
626	Very Weak	MnO_2
LNGH-01		
156	Very Strong	Modes of Anatase
285	Very Weak	Hematite
454, 473	Very Weak	Due the symmetric bending vibration (Si-O-Si) Quartz
515	Strong	Plagioclase
809	Very Weak	Si-O of Quartz
LNGH-02		
149	Strong	Modes of Anatase
235	Very Weak	Hematite
779	Very Strong	Si-O of Quartz
960	Very Weak	Due the Phosphate
LNGH-03		
119, 458	Very Weak	Due the symmetric bending vibration (Si-O-Si) Quartz

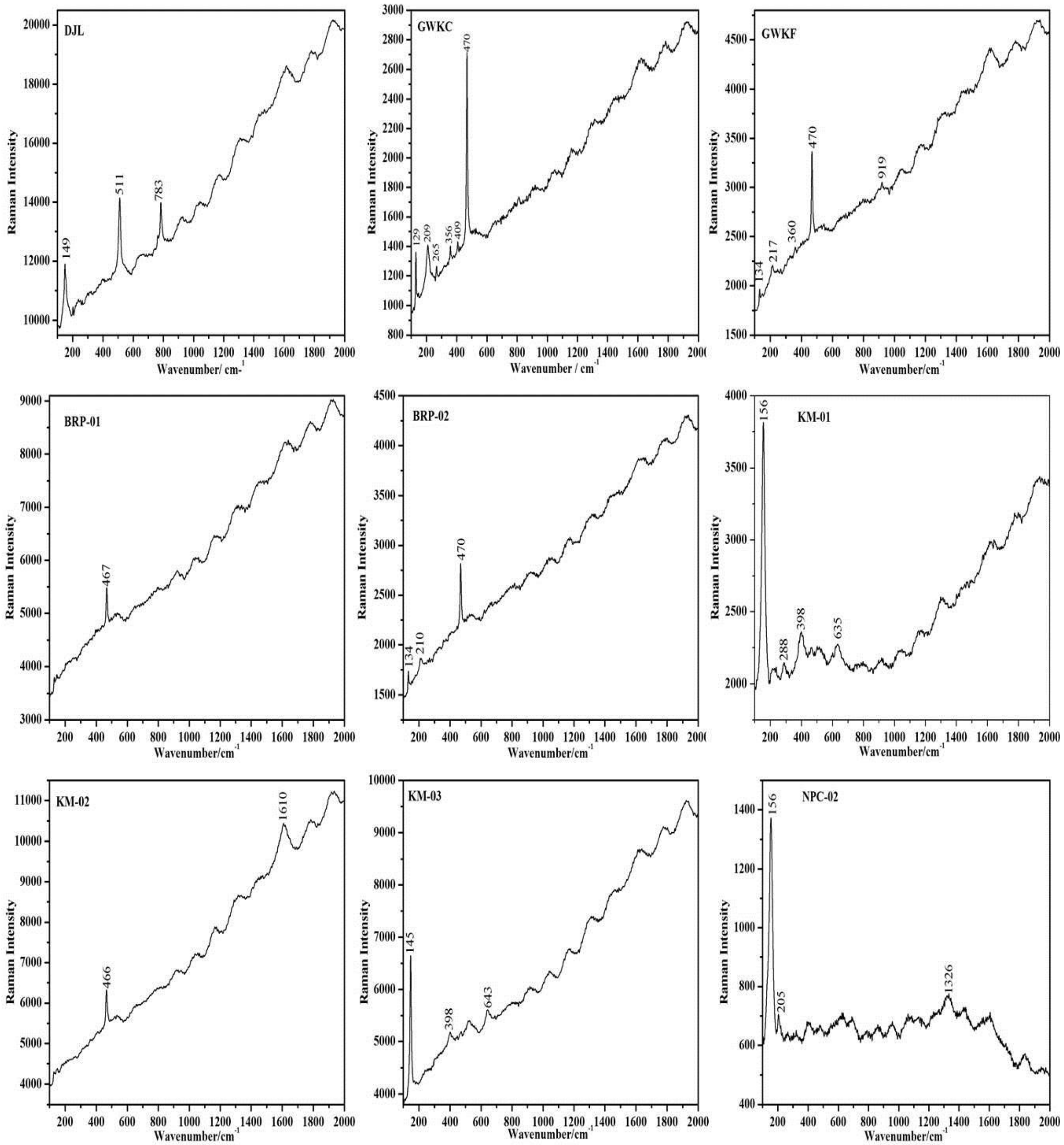

Figure 2.17 Raman Spectra of DJL, GWKC, GWKF, BRP-01, BRP-02, KM-01, KM-02, KM-03 and NPC-02

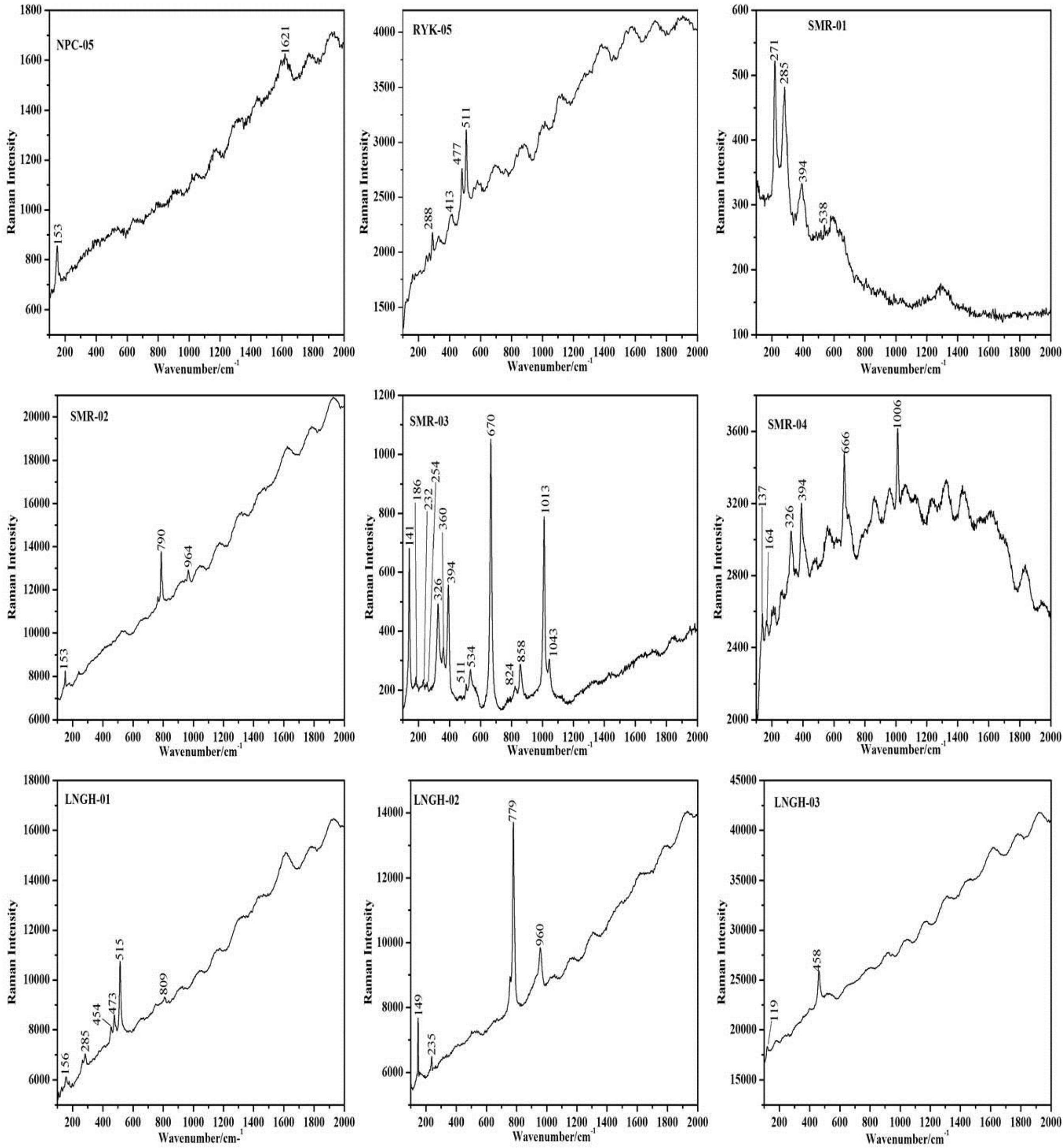

Figure 2.18 Raman Spectra of NPC-05, RYK-05, SMR-01, SMR-02, SMR-03 SMR-04, LNGH-01, LNGH-02 and LNGH-03

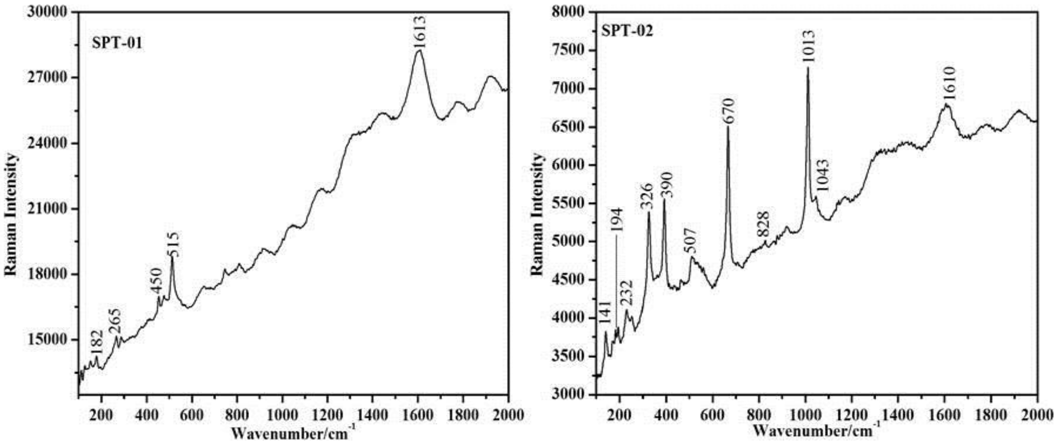

Figure 2.19 Raman Spectra of SPT-01 and SPT-02

2.7 Elemental analysis of Neolithic pottery by means of EDX and LIBS

2.7.1 Energy-dispersive X-ray analysis

In present study, chemical composition of Neolithic pottery has been investigated using EDX and LIBS. SEM-EDX analysis help to identify major elements such as Si, Al, or Mg and also their distribution can be determined by X-ray scan of a given area. X-ray spectra collected from grain inclusions can be used to infer presence of most of abundant minerals in paste (Ali *et. al.* 2011). Elemental composition of Neolithic potsherds is given in Table 2.14 and figure 2.20-2.22.

EDX analysis (table 2.14) indicates SiO_2 and Al_2O_3 occur as main constituent of Neolithic pottery. SiO_2 detected in range of 37.12-61.95 wt. %. From EDX analysis, it is evident that KM-02 has highest amount of 61.95 wt. percentage of SiO_2 in composition. RYK-01 has lowest amount of 37.12 wt. percentage of SiO_2. Most of Neolithic potsherds showed higher quantity of SiO_2 and lower content of CaO in their composition confirmed non-calcareous clay with simple mineral deposits (Damjanovic, *et al.*, 2011). The presence of Al_2O_3 is varies between 12.36-36.84 wt. percentage in composition of all analysed Neolithic pottery. SMR-01 contains highest amount of 36.84 wt. % and RYK-03 has lowest quantity of 12.36 wt. % in their composition. The presence of higher quantity of SiO_2 and Al_2O_3 reveal occurrence of Quartz and alumina-silicate rich minerals such as clay minerals and Feldspars (Vecstaudza, *et al.*, 2013).

According to tabulated data of EDX (table 2.14), amount of Fe_2O_3 varies between 7.21 and 20.00 wt. % for Neolithic pottery. From the EDX analysis, it is evident that NPC-04 has highest amount 20.00 wt. % of iron oxide in their composition. BRP-02 has lowest amount of

7.21 wt. % of iron oxide in their composition. Some of analysed potsherds such as DJL (10.56), NPC-03 (13.30 %), NPC-05 (8.04 %), and RYK-01 (12.45 %) KM-01 (11.44 %) and GWKC (13.18 %) is showing comparatively good amount of iron oxide in their composition. As reported in literature (Ali *et al*., 2011, Mangone *et al*., 2008) that higher amount of iron oxide in typical clay batch is not possible to occur naturally. There are several factors that can affect higher amount of iron oxide in the clay batch. For example, firstly, additional iron or iron rich materials must be required for enhancement of clay. Secondly, various traditional methods require choosing specific clay by potters. Third is mixing the iron rich constituents with clay composition. Redox atmosphere during firing and presence of iron oxide in raw material can affect color of ceramic body (Ali, *et. al*. 2011, Nodari, *et al*., 2007).

The occurrence of Na_2O substances is noticed in most of analysed Neolithic potsherds ranging from 0.03 to 2.09 wt. % respectively. Na_2O is not present in GWKF, SMR-01, SPT-02 and LNGH-01 respectively. Tabulated data show that presence of CaO in Neolithic pottery is in low amount, ranging from 0.04 to 12.21 weight %. Only, RYK-01 (12.21 wt. %) is showing higher percentage of CaO in their composition, which indicates that calcareous materials might have been used by ancient potters. While, some potsherds such as BRP-02, NPC-01, NPC-05, KM-01, KM-02, SMR-01, and LNGH-01 are not showing CaO content in their composition.

The amount of MgO content is approximately similar in all potsherds and it is in a reasonable range from 1.00–16.12 wt. %. RYK-03 has highest content with 16.12 wt. % and GWKF has lowest amount with 1.00 wt. % in their composition.

The presence of K_2O materials in all potsherds varies from 1.23 to 4.79 wt. %. Ali, *et al*. (2011) stated that presence of K_2O indicate that K-Feldspars might have been added deliberately or employed naturally potassium rich micaceous clays, such as Muscovite, Phlogopite or Sericite, as key minerals source. TiO_2 substances ranging from 0.44 to 2.46 wt. % is present in all potsherds.

In addition to EDX, LIBS has been used for elemental or more precisely chemical analysis of Neolithic potsherds from NE India.

Table 2.13 EDX results of Neolithic pottery

Sample Name	**Oxide (Weight %)**										
	Na_2O	**MgO**	**SiO_2**	**P_2O_5**	**Al_2O_3**	**K_2O**	**CaO**	**SO_3**	**TiO_2**	**Fe_2O_3**	**Cl**
DJL	0.36	2.03	52.51	1.74	25.67	4.17	0.72	0.59	1.48	10.56	0.18
BRP-01	0.50	2.48	47.18	0	27.92	1.53	1.01	0	1.88	17.51	0
BRP-02	0.84	1.94	49.08	1.79	34.90	2.81	0	0	1.11	7.21	0.32
NPC-01	0.64	2.04	46.44	1.22	33.92	2.62	0	0	1.19	11.93	0
NPC-03	0.63	1.92	51.52	1.27	26.21	2.73	0.93	0	1.49	13.30	0
NPC-04	1.04	1.86	43.19	4.03	23.12	3.11	1.16	0	2.49	20.00	0
NPC-05	0.34	1.65	51.75	3.77	28.88	4.06	0	0	1.49	8.04	0
RYK-01	2.09	4.90	37.32	8.01	17.85	2.84	12.21	0.46	1.46	12.45	0.42
RYK-03	0.78	16.12	45.36	0.59	12.36	1.23	3.83	0	0.56	18.91	0
KM-01	0.79	1.62	48.64	1.35	29.55	3.93	0	0.48	2.19	11.44	0
KM-02	0.46	1.62	61.95	0	24.46	2.79	0	0	1.25	7.48	0
KM-03	1.12	2.54	55.72	0	27.59	3.02	0.82	0	1.76	7.43	0
GWKF	0	1.00	56.86	0	23.92	2.35	0.04	0.01	0.44	15.39	0
GWKC	0.50	1.30	47.05	0.54	25.74	4.79	1.24	2.12	2.56	13.18	0.96
SMR-01	0	2.23	45.41	0.68	36.04	2.71	0	0.61	1.40	10.74	0.18
SMR-03	0.05	4.47	51.25	0	28.49	1.56	1.47	0	1.13	11.59	0
SMR-09	0.29	3.70	50.46	0.26	27.25	1.93	2.57	0	1.21	12.34	0
SPT-01	0	1.71	44.16	0.01	33.88	3.11	0.05	0.01	1.09	15.98	0
SPT-02	0.03	3.45	50.62	0.02	24.40	1.52	3.04	0.01	1.23	15.71	0
LNGH-01	0	1.13	40.59	0.01	36.84	2.50	0	0.01	0.94	17.98	0

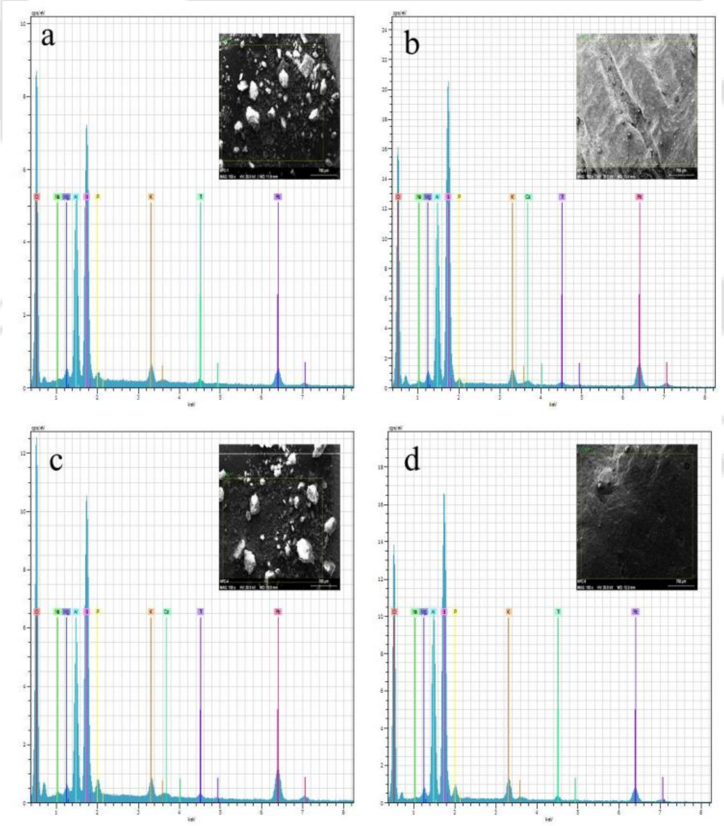

Figure 2.20 EDX spectrums of (a) NPC-01 (b) NPC-03 (c) NPC-04 and (D) NPC-5. Image indicates the spot of EDX analysis

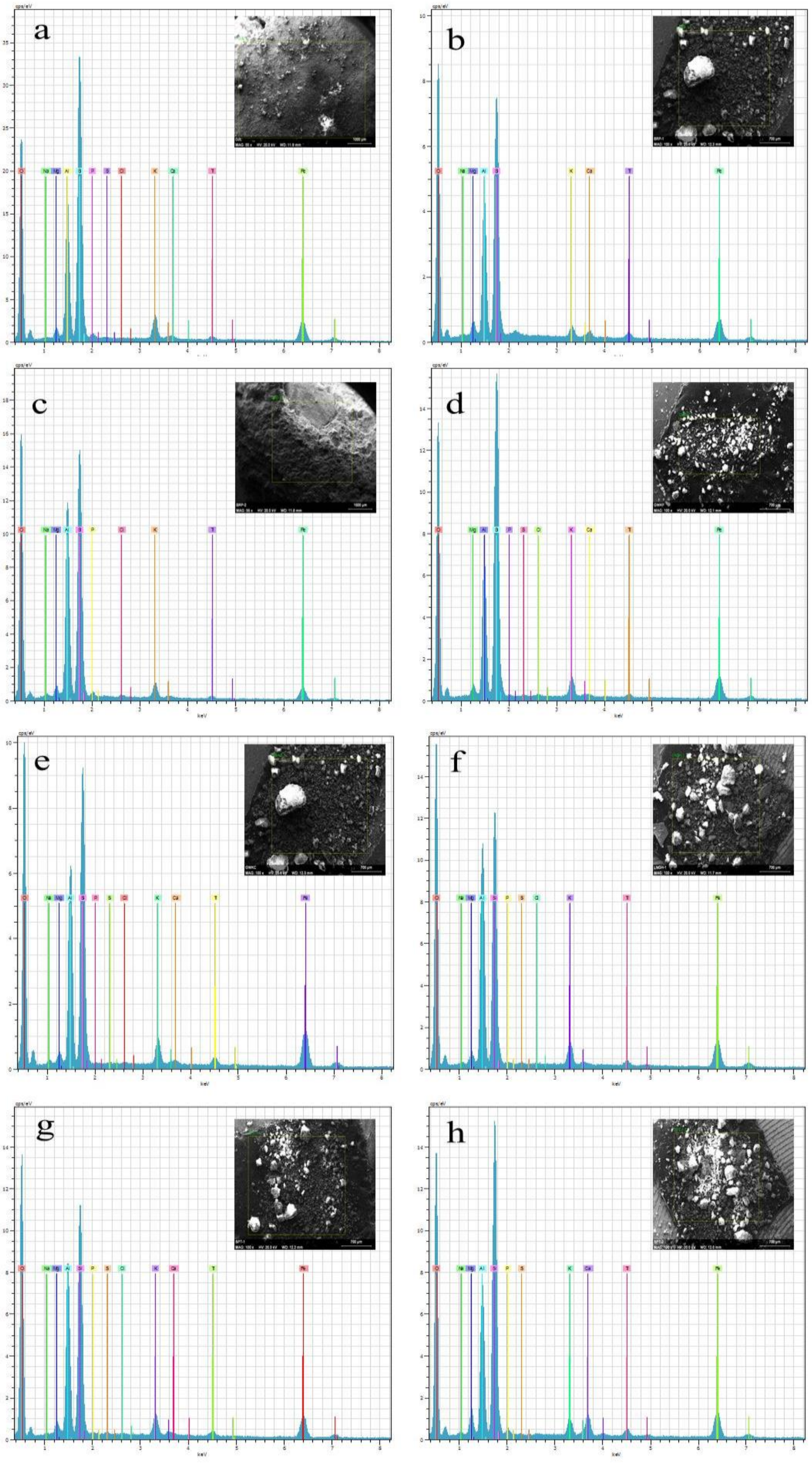

Figure 2.21 EDX spectrum of (a) DJL (b) BRP-01 (c) BRP-02 (D) GWKF (e) GWKC (f) LNGH-01 (g) SPT-01 and (h) SPT-02. Image indicates spot of EDX analysis

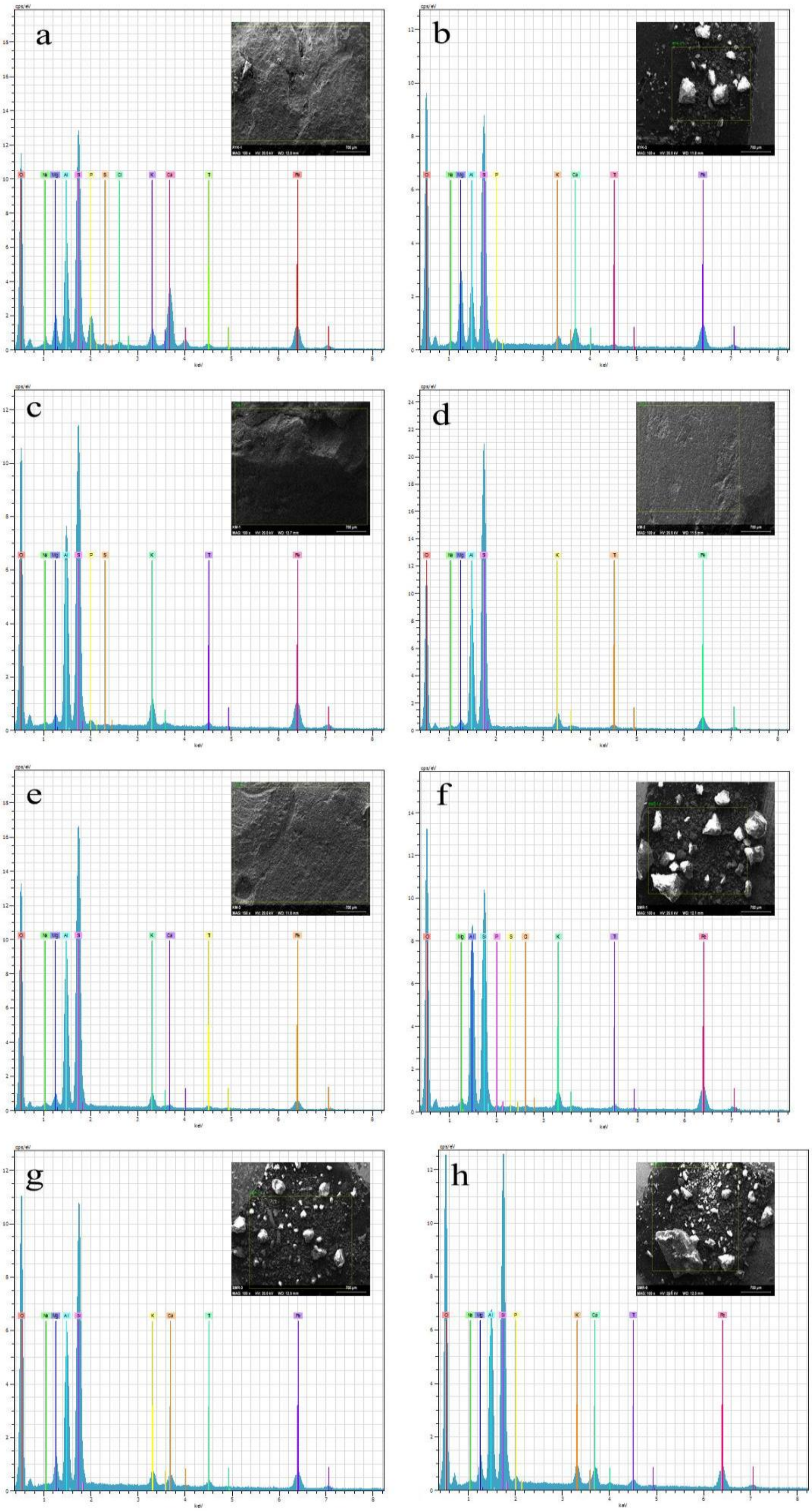

Figure 2.22 EDX spectrum of (a) RYK-01 (b) RYK-03 (c) KM-01 (D) KM-02 (e) KM-03 (f) SMR-01 (g) SMR-03 and (h) SMR-09. Image indicates spot of EDX analysis

2.7.2 Laser Induced Breakdown Spectroscopy (LIBS)

LIBS have been used for elemental analysis of Neolithic pottery from Northeast India. LIBS result (table 2.15 and figure 2.23-2.24) indicates that Fe, Ca, Si, Ti, Mg and Na present in clay composition of all analysed potsherds. In addition, potassium presents only in BRP-01 and KM-01.

LIBS spectra of 350.886, 351.2, 373.19, 374.5, are 384.375 nm are detected in all potsherds due to presence of iron-rich minerals such as Hematite (Fe2O3), Magnetite (Fe3O4) and Goethite (FeOOH) (Melessanaki, 2002). The emission lines at 393.869, 395.372, 431.66, 431.896, 444.1237, 655.41 and 705.84 nm are assigned to Ca which is present in all potsherds. Si has been detected due to emission lines at 406.486, 412.883, 455.109, 559.1295 and 616.348 nm in all potsherds. The characteristics spectrum at 430.297, 500.31, 644.957 and 671.96 nm is assign to Titanium. Magnesium (Mg) has been identified due to presence of LIBS spectrum at 517.7255 and 518.35 nm in all potsherds. LIBS spectrum lines at 587.62 and 589.16 nm are due to sodium (Na) in all pottery. The present of Na in potsherds might be adsorbed during burial. Due to low amount of sodium present in potsherds, it is difficult to identify that presence of Na either during manufacturing or adsorbed under earth in their composition.

On basis of EDX and LIBS analysis, chemical compositions of analysed Neolithic potsherds indicate that SiO_2, Al_2O_3, Fe_2O_3 and K_2O are present as main constituent in their composition. High content of SiO_2 and Al_2O_3 is attributed to occurrence of Quartz and aluminium-silicate minerals. The presence of Quartz and clay mineral such as Kaolinite and Feldspar mineral groups are also confirmed by Petrographic, XRD, FTIR and Raman spectroscopic analysis. The presence of clay minerals is also confirmed by occurrence of K_2O in good amount. Low content of CaO and MgO is attributed to carbonates and silicates constituents. It must remember that archaeological pottery is buried for long period. So, pottery must come to contact of some contamination and weathering. Other constituents such as Na_2O, P_2O_5, SO_3, TiO_2 and Cl are present in very low quantity. Therefore, it is not easy to detect presence of these elements in composition of analysed potsherds is due to contamination during burial environment or occur naturally.

Table 2.14 Elemental composition of Neolithic pottery identified using LIBS

BRP-02	
Wavelength (nm)	**Element**
351.2, 374.5, 384	Fe
395.372, 431.66, 444.1237, 655.41, 705.84	Ca
412.6, 567.1, 625.56, 634.91	Si
500.93	Ti
518.35	Mg
587.62	Na
776.769, 819.47	K
KM-01	
351.208, 373.19, 384.375	Fe
395.372, 655.06	Ca
447.422, 552.989, 635.45	Si
500.939	Ti
517.701	Mg
589.16	Na
765.773, 776.759	K
NPC-03	
350.886, 373.078, 384.2739	Fe
395.29, 431.896	Ca
406.486, 412.883, 455.109, 559.1295, 616.348	Si
500.91, 671.96	Ti
517.7255	Mg
589.338	Na
SMR-03	
350.866, 373.078, 384.273	Fe
393.869, 422.478, 444.691	Ca
559.129, 616.348	Si
430.297, 500.31, 644.957	Ti
517.725	Mg
598.338	Na

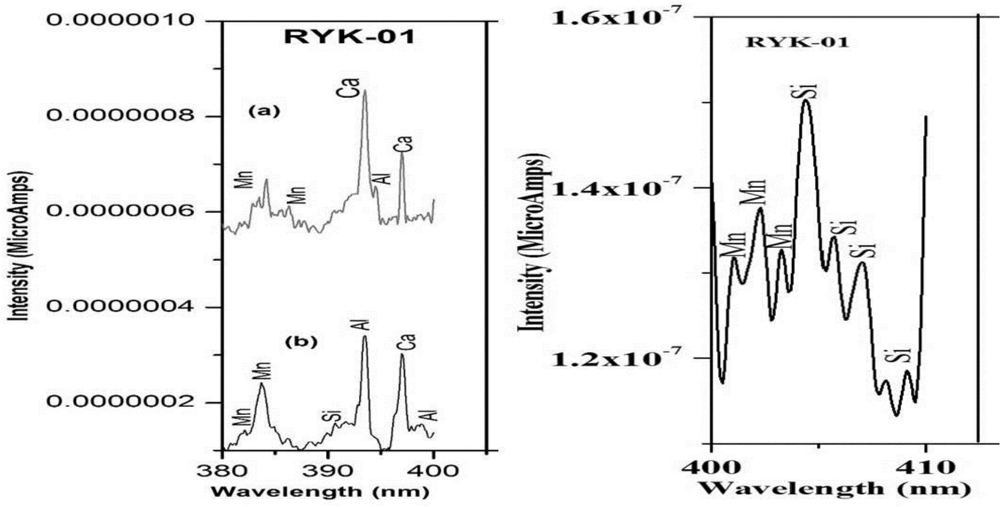

Figure 2.23 Characteristics peaks of BRP-02

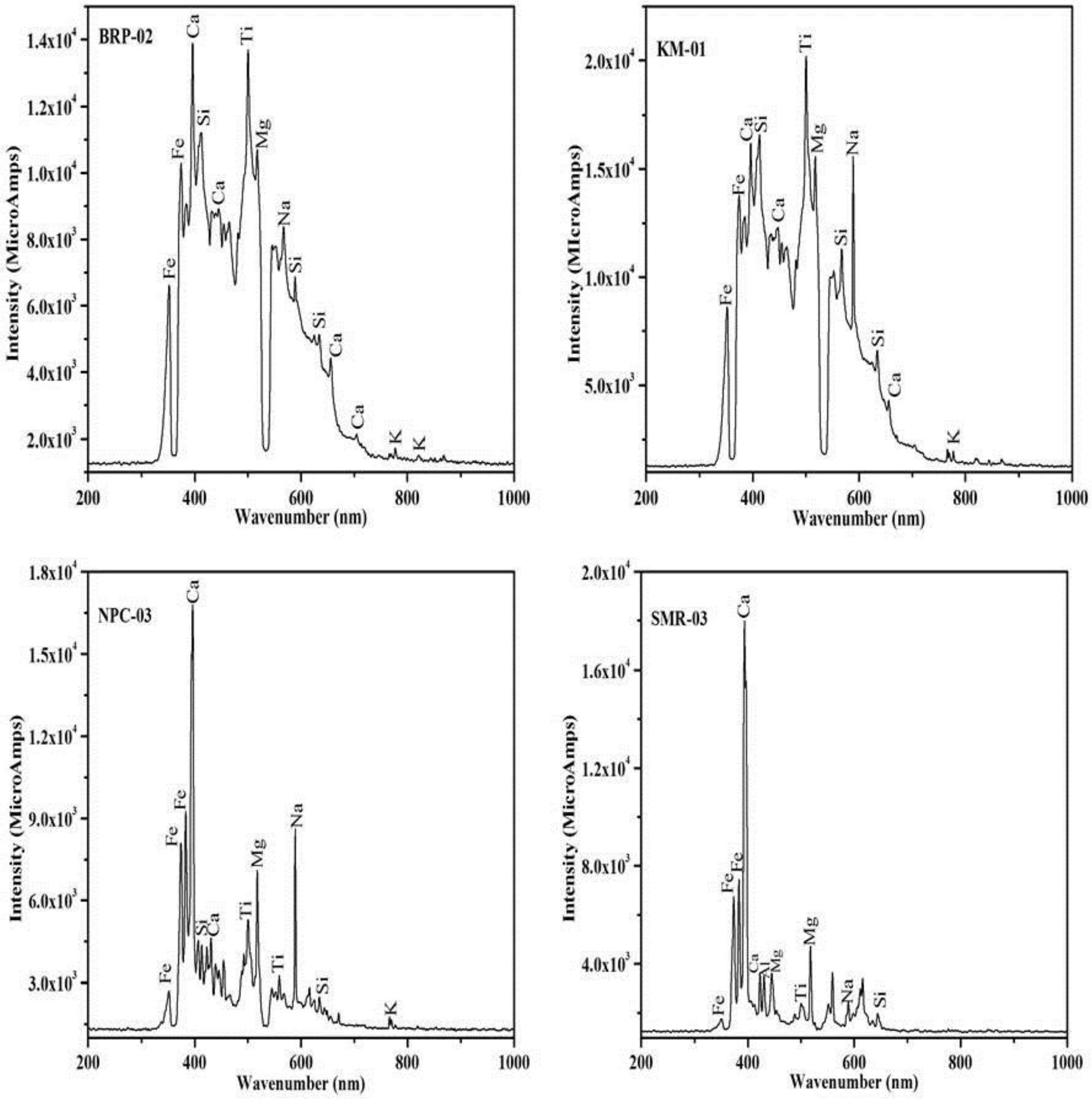

Figure 2.24 Characteristics peaks of BRP-02, KM-01, NPC-03 and SMR-03

2.8 Micro-indentation Hardness Testing (MHT)

Micro Hardness Test or Microindentation Hardness and Mohs' scale is used to measure hardness of Neolithic potsherds. All Neolithic potsherds showed hardness of 2 to 3 unit in Mohs' scale.

The tabulated (table 2.16) data gives MHT hardness value of various analysed Neolithic pottery. Tabulated data is evident that most of Neolithic potsherds showed very low hardness value which varies from 46.33 HV to 15.02 HV. Micro hardness test demonstrated that KM-01 has hardest surface with 46.33 HV and NPC-03 has softest surface with 15.02 HV. Most of potsherds have low hardness value which varies from 15.02 to 26 HV. Only, BRP-02

(42.57), KM-01 (46.33), RYK-01 (42.32) and RYK-02 (39.72) showed harder surface than other analysed Neolithic potsherds.

The Microindentation Hardness value (figure 2.25) of these potsherds indicates that technique of manufacturing, quality and preparation of clay. The result demonstrated that BRP-02, KM-01, RYK-01 and RYK-02 technologically better, because clay is well prepared and comparatively fired on higher temperature which can produce such hardness value.

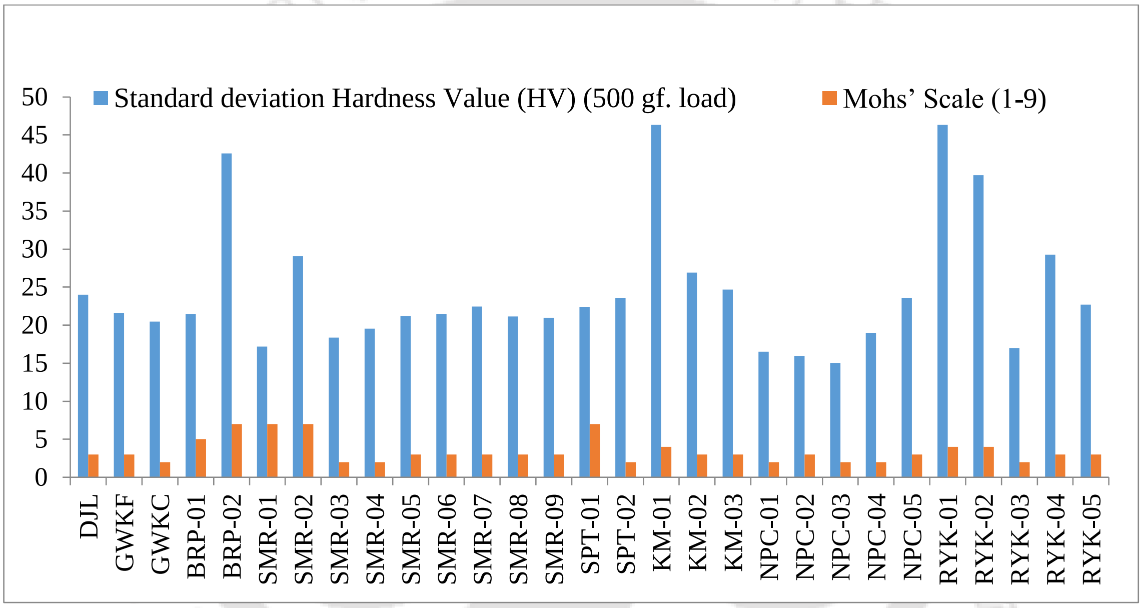

Figure 2.25 Variations of Micro Hardnes

Table 2.15 Micro Hardness and Mohs' Scale Hardness of Neolithic pottery

Si. No.	Sites Name	Standard deviation Hardness Value (HV) (500 gf. load)	Mohs' Scale (1-9)
1	DJL	23.99	3
2	GWKF	21.62	3
3	GWKC	20.45	2
4	BRP-01	21.45	2
5	BRP-02	42.57	3
6	SMR-01	17.2	2
7	SMR-02	29.05	2
8	SMR-03	18.38	2
9	SMR-04	19.53	2
10	SMR-05	21.18	3
11	SMR-06	21.47	3
12	SMR-07	22.43	3
13	SMR-08	21.15	3
14	SMR-09	20.98	3
15	SPT-01	22.42	3
16	SPT-02	23.55	3
17	KM-01	46.33	3
18	KM-02	26.92	3
19	KM-03	24.67	3
20	NPC-01	16.5	2
21	NPC-02	15.98	3
22	NPC-03	15.02	2
23	NPC-04	18.98	2
24	NPC-05	23.57	3
25	RYK-01	46.32	4
26	RYK-02	39.72	4
27	RYK-03	16.97	2
28	RYK-04	29.25	3
29	RYK-05	22.7	3

2.9 Discussion

2.9.1 Reconstructing the firing technology of Neolithic pottery

Estimation of firing range and temperature of pottery is based on fact that once clay is fired at a fixed temperature and then cooled down; property of clay will not be altered unless clay is subsequently refired at a temperature exceeding initial firing temperature (Ravisankar, *et al.* 2010; Singh & Sharma, 2016, p. 560).

Reconstruction of manufacturing technology of Neolithic pottery has been obtained from analysis of petrographic microscope, FTIR, SEM-EDX, XRD and Raman Spectroscopy.

Temperature and atmosphere are two key features of firing process for reconstructing firing technology of archaeological pottery using analytical and petrographic methods. Changes in clay properties and inclusions such as rocks or minerals during ceramic production can be detected in thin section analysis depending on their transformation in optical and physical properties during firing. For examples, Hornblende shows alteration in body color in thin section from green to brown at 750 °C due to oxidation of iron within mineral (Quinn, 2013, pp. 190-191). At 1100 °C and above Feldspars begin to melt and Na rich varieties disintegrating and before K-rich start to form.

There are several key factors that may affect redox reaction that takes place in properties of pottery. For example, maximum firing temperature, soaking time and presence of mixture of gases play significant role to determine reactions that involves in clay properties during firing. The color of pottery body is affected by free oxygen and air supplied during firing process. Other reasons to affect color of pottery body are presence of iron and organic rich substances which are present in most natural clay source, firing temperature and condition, rate and timing of firing. At 300 °C organic materials such as plant and humus present in natural clay sources is begin to carbonized. In oxidizing condition carbon mix with oxygen begin to form CO_2 and removed from clay. Though, if carbonized organic materials remain in clay property, form black carbon inclusion subjected fired in reducing environment. Presence of iron-rich materials even less than 5% in clay is enough to affect color of pottery body depending on oxidation atmosphere and its abundance. In oxidizing condition iron is converted to 'ferric' mineral (Hematite-Fe_2O_3) and it give to pottery body red, brown and orange in color at 600 °C temperature. While, in reducing atmosphere iron is exists as 'ferrous' mineral (Magnetite- Fe_3O_4) and it give to ceramic body black and grey in color due to presence of carbonized organic substances (Quinn, 2013, pp. 198-200). The lighter red, orange or brown and dark black or grey color core of ceramics is revealing that short period of firing due to lacking of oxygen penetrated in body to eliminate carbon.

The thin section result indicates that all characterised fabric groups of Neolithic potsherds shows lighter red to reddish and brown color matrices. This indicates that these potsherds might have been fired above or around 600 °C under oxidizing environment. The presence of organic matter and brown colour matrices of fabric group G and H indicates that pottery was fired in short duration under oxidizing atmosphere above or around 600 °C. However, organic materials could be removed if pottery was fired for longer-duration in oxidizing condition.

The study of chemical composition of archaeological pottery can provide information about source of raw materials, type of clay, determining firing temperature and atmosphere. Percentage of CaO (calcium oxide) gives information about type of clay whether it is calcareous or non-calcareous. According to Maniatis and Tite (1981), the presence of CaO content above 6 wt. % can be classified as calcareous clay and below 6 wt. % known as non-calcareous clay. Referring to this, EDX analysis revealed that all analysed Neolithic potsherds are made of non-calcareous clay while RYK-01 is made of calcareous clay due to the higher percentage CaO (table 2.14). If occurrence of K_2O, Fe_2O_3, CaO, MgO and TiO_2 in clay composition is greater than 9 wt. percentage, then it indicates that clay is low refractory clay (Velraj 2010). Considering this, EDX analysis (table 2.14) indicates that low refractory clay have been used to manufacture all Neolithic potsherds. According to Velraj *et al.* (2010), occurrence of iron oxide such as Hematite and Magnetite provide information about redox firing atmosphere of archaeological pottery (Benedetto *et al.*, 2002; Velraj *et al.* 2010). Referring to this, identification of Fe_2O_3 (Hematite) using EDX analysis indicates that all analysed Neolithic potsherds might have been fired under oxidizing condition during manufacturing.

Table 2.16 SEM-EDX analysis estimating the firing temperature and condition NV – No vitrification; C – calcareous clay; NC – Non calcareous clay

Si. No.	Sample Name	Clay Type	Stage of Vitrification	Firing Temperature (°C)	Firing Atmosphere
01	DJL	NC	NV	<800	Oxidizing
02	BRP-01	NC	NV	<800	Oxidizing
03	BRP-02	NC	NV	<800	Oxidizing
04	NPC-01	NC	NV	<800	Oxidizing
05	NPC-03	NC	NV	<800	Oxidizing
06	NPC-04	NC	NV	<800	Oxidizing
07	NPC-05	NC	NV	<800	Oxidizing
08	RYK-01	C	NV	<800	Oxidizing
09	RYK-03	NC	NV	< 800	Oxidizing
10	KM-01	NC	NV	<800	Oxidizing
11	KM-02	NC	NV	< 800	Oxidizing
12	KM-03	NC	NV	< 800	Oxidizing
13	GWKF	NC	NV	<800	Oxidizing
14	GWKC	NC	NV	<800	Oxidizing
15	SMR-01	NC	NV	<800	Oxidizing
16	SMR-03	NC	NV	<800	Oxidizing
17	SMR-09	NC	NV	<800	Oxidizing
18	SPT-01	NC	NV	<800	Oxidizing
19	SPT-02	NC	NV	<800	Oxidizing
20	LNGH-01	NC	NV	<800	Oxidizing

SEM photomicrographs (figure 2.26-2.28; table 2.17) of all analyzed potsherds revealed no vitrification (NV) occurred during firing. According to Tite and Maniyatis (1975a), low refractory clay with no vitrification stage and fired in oxidizing atmosphere indicate that firing temperature will not exceed 800 °C during manufacturing pottery. Considering this, these potsherds are fired < 800 °C under oxidizing condition. The FTIR, Powder XRD and thin section petrography analysis also showed similar results.

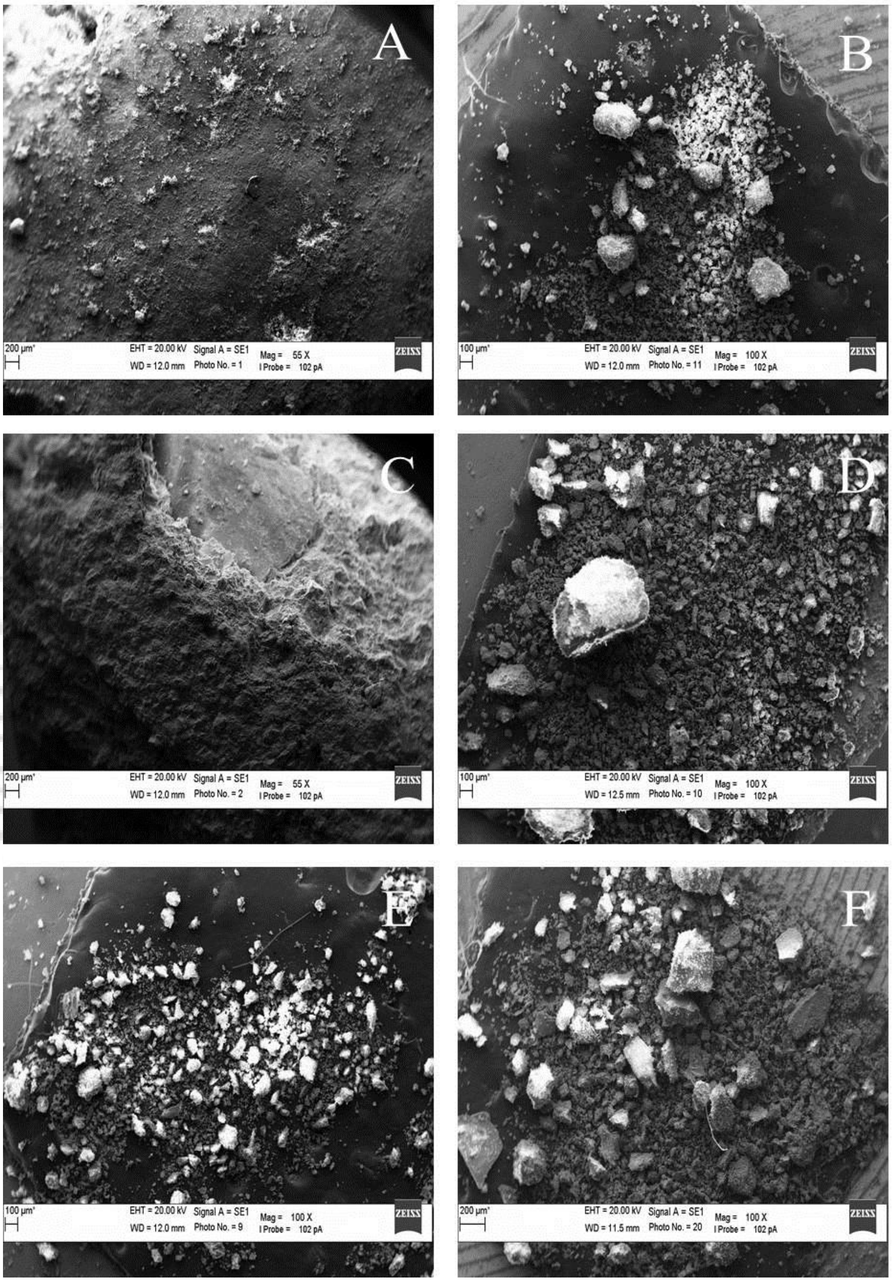

Figure 2.26 SEM images of (A) DJL (B) BRP-01 (C) BRP-02 (D) GWKC (E) GWKF and (F) LNGH-01

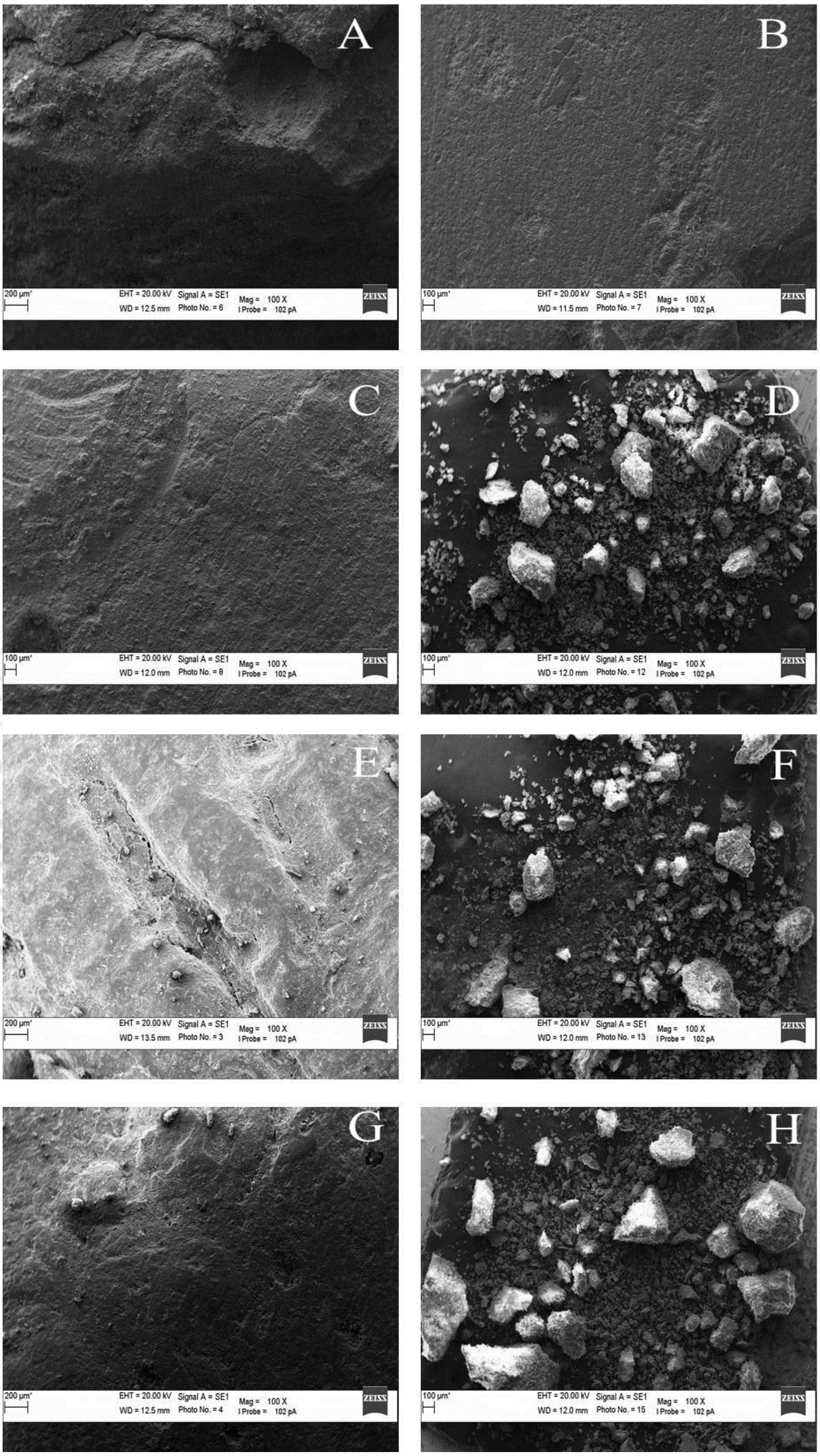

Figure 2.27 SEM images of (A) KM-01 (B) KM-02 (C) KM-03 (D) NPC-01 (E) NPC-03 (F) NPC-04 (G) NPC-05 and (H) SMR-01

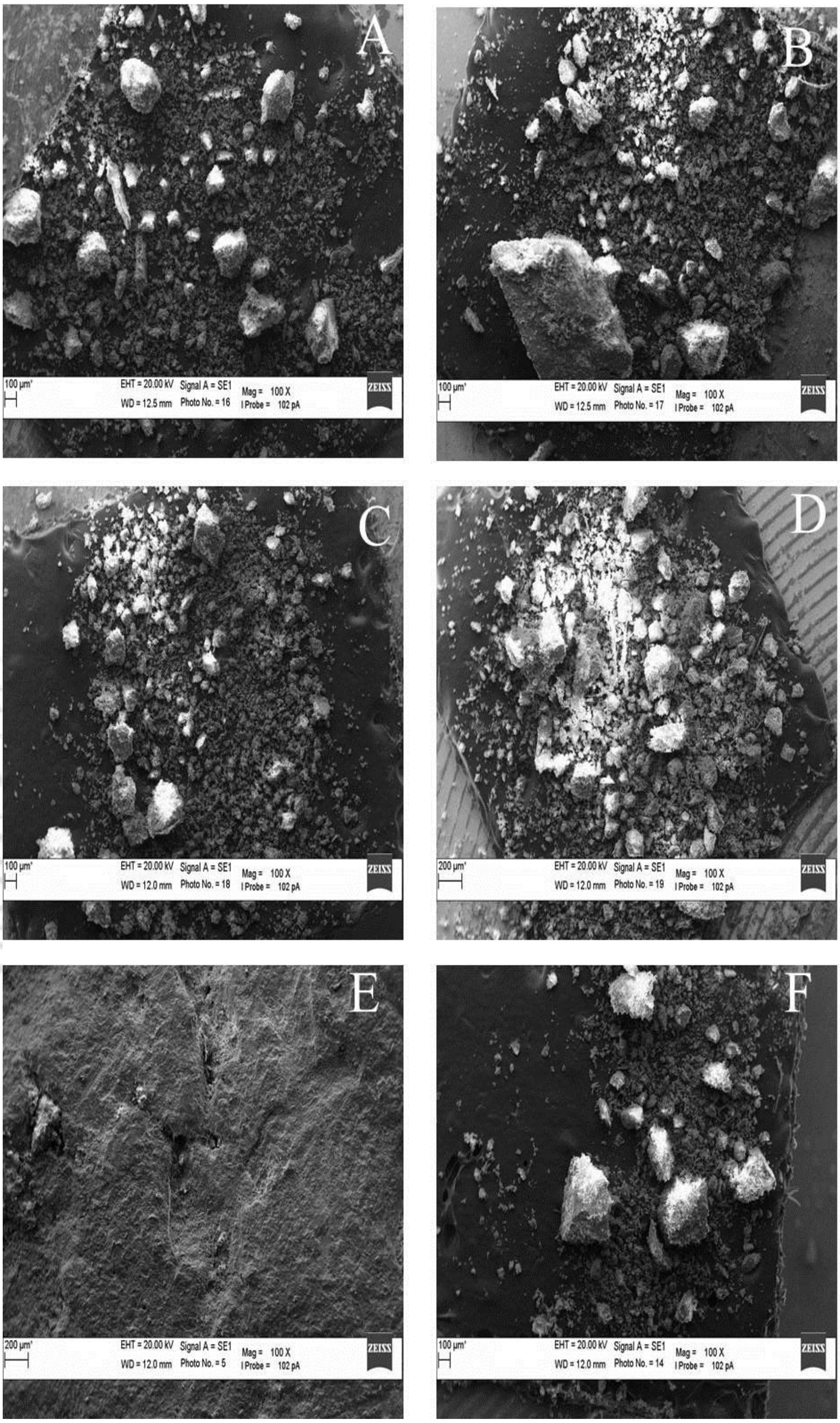

Figure 2.28 SEM images of (A) SMR-03 (B) SMR-09 (C) SPT-01 (D) SPT-02 (E) RYK-01 and (F) RYK-03

The IR band around 3700-3800 cm^{-1} is assigned to crystalline hydroxyl (OH-stretching of interlayer water), and takes complete destruction of clay structure when subjected to fire above 450-500 °C, but only weak peaks around 3600-3640 cm^{-1} is persist above this temperature (Venkatachalapathy *et al.* pp. 771-774, 2002; Ravisankar *et al.*, 2010 pp. 1016-1017; Singh & Sharma, 2016). Referring to this, only RYK-03 and RYK-04 is showing infrared bands around 3700-3800 cm^{-1} which indicates that both samples might have been fired around 500 °C. IR spectra of analysed Neolithic potsherds, such as KM-03; LNGH-01, 02, 03; NPC-01, 02, 03; RYK-03, 04, 05; SMR-03 and 09 from Northeast India demonstrate bands around at 3352-3620 cm^{-1} which assigned to disorder Kaolinite and might have been fired above 500 °C (Manoharan *et al*., 2007).

The very weak bands around 920 cm^{-1} is due to Al-OH octahedral sheet which is present in LNGH-02 and RYK-04. This band is sensitive to firing temperature. Manoharan *et al.* (2007) and Venkatachalapathy *et al*. (2002) reported that during firing this band is exist at steps of 300 °C in decreasing order and disappear at 500 °C. According to this, presence of band around 920 cm^{-1} in LNGH-02 and RYK-04 indicates that firing temperature might have been around or above 500 °C. IR spectra of DJL; BRP-01, 02; SPT-01, 02; GWKC, GWKF; KM-01, 02, NPC-04, 05; RYK-01, 02, SMR-01, 02, 04, 05, 06 07 and 08 do not demonstrate peaks around 3700 cm^{-1} (inner hydroxyl) and 920 cm^{-1} (Al-OH octahedral sheet) in their composition. This indicates that these Neolithic potsherds are probably fired above 500 °C (Ravishankar *et al*., 2010; Venkatachalapathy *et al*., 2002; Miller, 1961).

Venkatachalapthy and co-workers (2002) reported that in clay, there are three characteristic bands around 1100 cm^{-1} and 1080 cm^{-1} and 915 cm^{-1}, which are regarded as sensitive to temperature effect when clay minerals are fired at 650 °C complete destruction takes place. The very strong bands around 1040 cm^{-1} is present in most of potsherds, such as DJL; GWKF, GWKC; BRP-01, 02, SPT-02; KM-02,03; LNGH-01; NPC-01,02, 04; RYK-01, 03, 04 and SMR-01-09 respectively indicates that firing temperature should be around 650 °C. The infrared bands of around 1080 cm^{-1} is due to presence of Wollastonite in some Neolithic potsherds such as SPT-01; KM-01; LNGH-03; NPC-03, 05 and RYK-02, 05 (Palanivel & Kumar, 2011). Barilaro *et al.* (2008) stated that Wollastonite formed during firing temperature at 900 ºC from chemical reaction between Quartz and carbonates (Palanivel & Kumar, 2011).

The composition of clay helps to estimate firing temperature of ancient pottery. But, it is not easy to replicate actual firing duration and firing condition achieved by ancient ceramics during manufacturing and has possibilities to small errors to do (Ravishankar *et al.* 2010). The presence of Hematite is due to IR band around 561 cm^{-1}, 535 cm^{-1} and 475 cm^{-1} in all Neolithic potsherds. This suggests that all pottery are fired under oxidizing atmosphere.

According to Venkatachalapathy *et al.* (2002) and Akyuz *et al.* (2008), Infrared band around 481 cm^{-1} and 465 cm^{-1} is assigned to Microcline that is free from any temperature effects and stable up to 500-800 °C. Presence of Microcline also confirmed by Powder XRD, indicates that firing temperature of these analysed potsherds, would not been exceeded above 800 °C.

In addition to FTIR, Raman spectroscopy provides comprehensive information regarding mineralogical constituents and technological process of firing of Neolithic pottery from Northeast India. It is permitted to detect minerals, which is formed during firing of pottery. Moreover, it is also allowed to approximation of maximum temperature and oxidoreduction states of firing condition with reference to Hematite-Magnetite transformation phase during firing process (Maggetti, 1982; Maritan, *et al.*, 2005, 2006; Medeghini *et al.*, p. 890, 2013).

Anatase is identified by Raman spectroscopy in most of samples. According to Liou *et al.* (2011) Medeghini *et al.* (2013) and Olivares, *et al.*, (2010), Anatase is most common form of titanium dioxide and ancillary with many clay minerals. It stays stable at lower temperature in between 600-900 °C, beyond this temperature Anatase transforms into Rutile (Bouzid, *et. al.* 2013). This process is called Anatase-Rutile transformation phase. Presence of Anatase and absence of Rutile in most of analysed potsherds indicates a firing temperature below Anatase-Rutile transition phase, which occurs at 750-950 °C, although, this is reliant on various involving iron concentrations. This indicates that firing temperature might have been below Anatase-Rutile transition phase (Olivares, *et al.*, 2010; Smith & Clark, p. 1145, 2004; Gennary & Pasquevich, 1999). It is indicating that analysed pottery might have been fired below 750-950 °C. Apparently, it is also confirmed by Powder XRD and FTIR analysis.

Hematite (Fe_2O_3) has been identified using Raman spectroscopy in most of analyzed potsherds. According to Medeghini *et al.* (2013) and Hradil *et al.* (2003), Hematite may be present naturally in mineralogical composition of raw materials or formed during firing oxidizing environment. Magnetite present in SPT-02 and SMR-04 associated with Hematite indicates that incomplete transition process in oxidizing atmosphere during firing which takes place at 600-900 °C (Lomfrumento *et al.* 2004).

Above discussion revealed that all analyzed neolithic potsherds might have been fired in oxidizing environment at temperature of below or around 800 °C.

2.10 Conclusion

The combination of Petrography, Powder XRD, FTIR, OSL, micro-Raman Spectroscopy, SEM-EDX, LIBS, MHT and Scratch hardness (Mohs' Scale) have been applied to mineralogical and physiochemical investigation of neolithic pottery of Northeast India.

The OSL dates of two samples are 2.9 ka and 2.7 ka of potsherds from Daojali Hading and Gawak Abri respectively. The past studies are also showed similar results (Sharma, 1967; Sharma, 2002 and 2007; IAR Report-1963-64).

Thin section petrography coupled with polarizing microscope has revealed information regarding wide variety of natural and artificial clay minerals, inorganic and organic, materials which occurred naturally or inclusions as temper in neolithic potsherds from Northeast India. Mineralogically, Quartz, Feldspars, silt stone, clayey and siliceous materials, ferruginous materials and organic substances have been identified. These materials first of all reflects properties of local geology of place of manufacture of pottery, but also potters' habits, which largely determined use of particular kinds of temper and addition of artificial components, in an effort to improves properties of clay and to impart particular properties to pottery. Use of organic substances, Quartz grit and sand as tempering materials are also confirmed by past studies on neolithic pottery (Sharma, 1967).

Micro Raman analysis revealed that Quartz and Anatase are present in almost all potsherds. Other minerals such as Plagioclase, Hematite, Wollastonite, organic materials, Apatite and Gypsum have been identified. The presence of Anatase, carbonate and Hematite indicate the firing range between 500-700 °C and oxidizing atmosphere. Raman spectroscopy results have good agreement with other experiments such as FT-IR, XRD, thin section and SEM-EDX.

SEM analysis is revealed that no vitrification occurred in all Neolithic potsherds which indicate that all samples might have been fired below 800 °C. In addition, EDX data showed SiO_2, Al_2O_3 and Fe_2O_3 are occurred as primary constituent in all samples. EDX analyses are also revealed that in divergent concentrations of Ca, Na, Mg, Ti, Cl, K, S, and P occurred in all Neolithic pottery. The high content of silica and alumina rich constituent indicates high quantity of Kaolinite is used to manufacture analysed Neolithic potsherds. Powder XRD results also revealed good amount of Kaolinite in composition of analysed pottery. The presence of Feldspars mineral group is confirmed by thin section and Powder XRD analysis. Report of Geological survey of India (GIS) supports above findings (Kesari, 2011). In studied region, deposition of both minerals Kaolinite and weathered Feldspars are well known. The deposition of Kaolinite and weathered Feldspars indicate that analysed pottery manufactured locally. It is also confirmed by high content of Magnetite, Hematite and goethite in samples (Kesari, 2011).

It is also concluded that Neolithic potters of studied region used open firing techniques. As discussed earlier all methods of open firing differ from approximately 400-700 °C. The firing temperature of Neolithic pottery has been determined using various analytical methods. Combining results of applied various techniques revealed that all potsherds were fired in range of 500-700 °C. Moreover, open firing techniques acquire an oxidizing atmosphere. It means that firing is completed in such a fashion, where oxygen is supplied throughout firing duration resulting burning of gases and accomplishment of combustion. Sometimes, it is also affects color of ceramic body. All Neolithic potsherds were fired under oxidation atmosphere, which confirmed by all applied archaeometric techniques.

The micro hardness test results of potsherds of BRP-02, KM-01, RYK-01 and RYK-02 have comparatively harder surfaces. The typological studies of this pottery are also indicating towards this fact. Except these potsherds, all potsherds showed very low hardness.

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Chapter 3
Mineralogical and Physicochemical analysis of Historical Pottery of Assam

3.1 Introduction

Assam is situated in north-eastern part of India, lying between 26.2006° N, 92.9376° E covering an area of 78523 sq. km. Assam includes Barak and Brahmaputra valleys with numerous mountains and plain region and major part of the state are covered by forest. The Brahmaputra River valley is more fertile for agriculture of the state. Geographically, Assam is contact root zone with China and Myanmar.

Dhavalikar (1973) stated that archaeological investigation in Assam was started with systematic excavation in the Guwahati city (Ambari). The archaeological site of Ambari, Assam (Lat. 26° 11′ 8″ N; Long. 91° 45′ 21″ E) was discovered in 1969 AD. The excavation was carried out by M. C. Goswami and T. C. Sharma of Department of Anthropology, Gauhati University in collaboration with Z. D. Ansari and M. K. Dhavalikar of Deccan College Post-Graduate and Research Institute, Pune (Ansari & Dhavalikar, 1971). Later, excavation was carried out by T. C. Sharma in 1987. The excavations began soon after. Recently, Ambari was excavated by Archaeological Survey of India (ASI) in 2008–09. The pottery samples are used from 2008–09 excavation. The wheel-made and handmade pottery have been found at the site. Numerous pottery are manufactured by wheel and beater. Texture is fine to coarse and whole assemblage is well fired, but some potsherds are poorly fired. The major decorative designs on pottery are loops, rosettes, diamonds and leaves. The main pottery shapes such as different sizes of bowls, vase, jars, grey ware, kaolin ware, carinated handi, dish, lota, lamp, lamp-on-stand, inkpot and few smoking pipes have been reported from Ambari (Tiwari & Dimri, 2015, pp. 12-17).

Gauhati (now Guwahati) mentioned in various ancient literature was earlier known as Pragjyotishpura. Later, the region referred to as Kamprup, and name continues even today. The epics refer to city as Pragjyotishpura. According to some scholars, evidence of habitation traced back to the later Vedic period. Paul Pelliot stated that Pragjyotishpura was regular trade route with China through upper Burma (Myanmar) and Yunan, at least from second the century BC. It is confirmed by evidence of an ink-pot shaped lid of Kushan period that is found in the excavation at Ambari (Dhavalikar, 1973).

On the basis of excavation materials from the Ambari, the occupation can be divided into two different cultural periods. Kaolin pottery characterizes period I approximately dated to 7th to 12th century AD. On the basis of the typology of sculptures, the upper levels can be dated to 8th–9th century AD. According to Dhavalikar (1973), assemblages of Ambari can be dated between 7th–12th centuries AD. During this period, Pragjyotishpura witnessed its golden age under Bhaskarbarman (595–650 AD.). Period II is dated to 13th to 17th century AD belongs to the Muslim period.

Significant findings are — exquisitely carved sculptures of Lord Vishnu, Lord Surya, Mahishasurmardini, Ganga, Yamuna, Nataraja, Sivalingas, etc. Besides terracotta figurines, a Kushana inkpot lead, a seal and architectural pieces carved in granite, gneiss and basalt, etc. are recovered. Brick walls, covered drains, ring wells were found at the habitation site. Rouletted ware and Kaolin Pottery and red ware found in the period 1, and Chinese Celadon and green glazed Mughal Pottery found in Period II. The Kaolin pottery found in enormous quantity in Period I of Ambari has not been found elsewhere in India. Therefore, it has been named as the 'Ambari Ware'. Principal shapes are globular jars with flaring mouth and short neck, flat based goblets, sharp-rimmed cups, carinated bowls with rusticated bases, dish-on stand and horn-shaped vessels. This horn-shaped vessel resembling a cornucopia interpreted as a decorative vase similar to the Vrishabhasringa, a symbol of prosperity referred in the epics Ramayana and Mahabharata. This shape has not reported elsewhere in India. It is sometimes depicted in Gandhara art. The red ware reported in small amounts and the fabric is coarse and devoid of any surface treatment. Torch with wide flaring mouth and ribbed body is the unique vessel has been reported in this ware group (Dhavalikar, 1973).

The pottery samples belong to period I of Ambari which was reopened in 2008–2009 by Archaeological Survey of India, Guwahati Circle. The details of all analysed potsherds are given in Appendix III.

Another archaeological site, Bamuni Hill (Lat. 26°36'59"N Long. 92°48'54"E) is situated in Tezpur, Sonitpur district, Assam. The remains of Panchayatan group of temples have been discovered, situated at the right bank of Brahmaputra River. The remains of the 3 Panchayatan temple group is dated 10th–12th century AD. The central remains of temple which is located on the top of the hill has been dedicated to Lord Vishnu and four subsidiary shrines are located on each of corner of hill. On the north-eastern side, another temple has been discovered. The major pottery types are bowls, vase, jars, grey ware, kaolin ware and diya. The color of potsherds mainly is red, grey and buff. The fabric is medium to coarse.

Sri Sri Suryapahar is situated in Goalpara district Assam, India. D. Bhengra, assisted by T. J. Baidya, S. K. Choudhary, B. Sinha and T. K. Srivastava of Guwahati Circle, Archaeological Survey of India, conducted a small scale horizontal excavation at Sri Sri Suryapahar with a view to co-relate the rock-cut sculptures with the votive stupas in area. The excavated site Sri Sri Suryapahar has been dated to two different periods- period I and II. The major pottery types reported from period I are (dated 6th to 7th century AD) button-knobbed lid, knobbed lid, incurved bowls, sprinkler, basin, chilam, diya and miniature vases. The miniature vases are dull red in color and coarse to medium in fabric. From the period 2 (dated 8th to 9th century AD) main pottery types are jar, chilam, basin, knobbed-lid and diya (Indian Archaeology 1992-93: A Review, 1997, pp. 4-5).

Studies on ancient pottery of Assam were mainly done by using ethnoarchaeological paradigms. These studies were able to provide important insights of manufacturing techniques used in ancient pottery of the region. For example, Goswami and Sharma (1963) described art of making the Neolithic pottery of Daojali Hading. Sharma (1967) explained a stratified cultural deposit from Daojali Hading (North Cachar Hill). Roy, (1977) studied ceramics from Daojali Hading and Ambari. He used Hira and Kumar traditional potter groups as ethnoarchaeological implements. Medhi (1990) pointed out that cord-marked pottery reported from Daojali Hading and Sarutaru has close similarity with findings in Southeast Asia. Besides this, some observations have been made by individual excavators of Sri Sri Surya Pahar-Goalpara; Ambari-Guwahati; Charaideo-Sibsagar etc (Singh P. , 2015a, pp. 117-119).

None of these scholars had used scientific techniques for analysing the pottery. No attempt was made to place pottery types in a broader chronological sequence. Stratigraphical sequencing was not done. As a result, ceramic tradition of Assam is still a missing chapter in history and archaeology books of India and the world. The present study attempts to fill these gaps in pottery studies of Assam.

A selection of 49 potsherds from Ambari (AMB) Kamrup, Shri Shri Suryapahar (SPR) Goalpara district, Bamuni Hill (BMH) - Tezpur, Sonitpur district, Assam dated from 7-12th century AD and Kangla fort (KGF) - Imphal Manipur (Appendix I) are studied by applying scientific techniques in present work.

The primary objectives of the present chapter are identifying chemical properties, reconstruction of manufacturing techniques, determine provenance of the pottery and to understand the technical skills of ancient potters of Assam. To achieve these objectives, numerous scientific techniques have been used for analysis. They are thin section petrography, Powder XRD, Micro-Raman Spectroscopy, FTIR, SEM-EDX, LIBS, MHT, and Mohs, scale. The TG-DSC analytic method focuses on the thermal behavior of the pottery, and it is used for the characterizing the clay minerals (Singh & Sharma, 2016).

3.2 Petrographic Analysis

3.2.1 Analysis of Thin Section Petrography

The thin section analysis has been used to investigate the composition of raw materials, texture of clay paste and provenance. In present study, petrographic analysis revealed information about raw materials such as occurrence of natural and artificial temper/inclusion, organic or inorganic substances used by historical potters. The thin section photomicrographs of all historical potsherds are shown in figure 3.1 to 3.10.

3.2.1.1 The description of the thin section matrices of Historical pottery

i. AMB-01

The thin section shows brownish color matrix supported framework with few angular grains of Quartz floating in it. The Quartz shows poor sorting. The larger Quartz are fractured and filled up with completely opaque to deep brown ferruginous habit (It might be organic material or carbonaceous materials). The larger Quartz fragments are of mono-crystalline undolose variety. The fine grains matrix shows numerous minute angular Quartz fragments. There is also one plagioclase feldspar grain, which is identified by its typical poly synthetic twin and deposition of irregular spaces of black color opaque material materials (It might be organic/carbonaceous materials).

ii. AMB-02

The sample under polarize light shows a yellowish grey color and contains numerous minute angular fragments of Quartz in its matrix. A faint foliation/Schistosity is observed which becomes distinct. Dew to presence of alternate yellowish and greenish irregular sands, towards one end of the slide there is presence of large size rounded partially Quartz and rock fragments.

iii. AMB-03

The matrix is brownish in color and is composed of highly angular fragments of mono-crystalline undolose Quartz and few grains of partially altered plagioclase feldspar. The larger grains comprise about 20% of total constitution of sample. Irregular opaque particles are distributed unevenly throughout matrix (presence of comparatively high amount of feldspar in this sample may indicates that people at that time realized the importance of using clay in making pottery).

iv. AMB-04, AMB-08, AMB-12, AMB-15, AMB-16, AMB-19 and AMB-20

Thin section shows a red color fine ferruginous matrix and contains numerous angular and sub-angular fragments of Quartz. There are wide variations in size of Quartz fragments. The matrix of AMB-15 shows lesser amount of coarse angular Quartz.

v. AMB-05

This sample is composed of a fine siliceous matrix with good sorting of fine components. There are few angular grains of mono-crystalline undolose Quartz. Opaque grains of irregular shapes are distributed throughout the mineralogical framework. Few pores /voids spaces are also found. They have an opaque inner lining.

vi. AMB-06

This slide shows reddish color and is composed of a fine siliceous matrix together with appreciable amount of sub angular to rounded coarse siliceous Quartz particles. The larger grains are fractured and contain deposition of ferruginous materials within the fractures. There are numerous rounded to irregular deposit are seen with a prominent rim surrounding them.

vii. AMB-07

The matrix shows very fine yellowish to red color with numerous minute angular Quartz particles. There is little highly angular and fractured large sized Quartz are also seen. There are few large irregular to rounded particles with reddish tinge. These might be of organic or vegetative origin.

viii. AMB-09

The matrix is composed of fine grains siliceous with few large irregular grains of plagioclase and Microcline feldspar and mono-crystalline undolose Quartz. At few places there is development of porosity due to weathering of larger feldspar grains.

ix. AMB-10

The matrix is composed of greenish grey of crypto-crystalline and fine siliceous materials together with elongated stricks of black opaque material. Presence of numerous large spaces indicates porous nature of matrix. The pore spaces are fine dark color inner lining. The percentage of porosity is approximately 10% in matrix.

x. AMB-11

The matrix is composed of dark brown clayey material together with silty coarser angular Quartz grains. The larger fragments showing an angular habit and is composed of Quartz and plagioclase feldspar.

xi. AMB-13

The matrix shows fine grains yellowish clayey material and minute grains of Quartz distributed throughout matrix. Opaque materials are also found and show irregular grains. There is one grain which shows orange color and weakly pleockroic irregular pore spaces are found throughout the mineralogical framework.

xii. AMB-14

The matrix shows reddish brown clayey matrix which contains numerous fine Quartz particles. There is irregular concentration of reddish (ferruginous) deposits. Opaque substances of various dimensions are distributed throughout the matrix. Pore spaces of different dimension are seen. They comprise about 10% of mineralogical framework.

xiii. AMB-17

The matrix of AMB-17 show fine grains, reddish color matrix which contain angular to sub-angular coarse Quartz grains. There are also irregular deposits of reddish ferruginous materials of varying in size and shapes.

xiv. AMB-18

The matrix shows grey color fine siliceous together with coarse, angular to sub-angular mono crystalline Quartz grains. Minor amount of ferruginous materials is also present.

xv. AMB-21 and AMB-22

The Thin section of AMB-21 shows fine groundmass of reddish ferruginous materials and sub-angular monocrystalline Quartz fragments. There is also presence of mica flakes within fine groundmass. The coarser fragments are angular monocrystalline Quartz grains with fractured margin. Some silt stone particles is also observed.

Only difference between AMB-21 and AMB-22 is that AMB-22 shows fine alternate dark and light layering are observed in the groundmass.

xvi. AMB-23

The matrix show very fine brown coloured muddy materials which are containing sub-rounded reddish coloured patches. There are also irregular shaped voids which have a black coloured internal lining. There are numerous minute of Quartz specks. They do not show any color under reflected light which indicates that these particles may be of carbonaceous materials.

xvii. AMB-24 and AMB-26

The matrices of AMB-24 and AMB-26 showed pale red to yellow coloured groundmass with thin irregular ferruginous streaks. The groundmass is composed fine siliceous materials with minor amounts of coarse elongate monocrystalline Quartz grains. Few mica flakes and minor amount of opaque carbonaceous materials are observed.

xviii. AMB-25

The matrix shows irregular fractured Quartz grains which are filled with ferruginous materials. The pale red to yellow groundmass is composed of fine siliceous materials. There are some voids which have a ferruginous lining. Feldspar is also observed which show weathering along its cleavage planes.

xix. AMB-27 and AMB-33

The matrix of AMB-33 shows fine red to deep brown coloured with numerous angular to sub-angular, rounded to sub-rounded and elongated Quartz grains. The sizes of Quartz grains are much coarser than AMB-33. The difference between AMB-33 and AMB-27 is that matrix of AMB-27 is composed of finer materials with a much coarser Quartz grains and weathered plagioclase Feldspars.

xx. AMB-28 and AMB-29

The Groundmass show black color carbonaceous materials together with monocrystalline Quartz grains. There are few voids spaces and groundmass show almost homogenous distributions.

AMB-29 is same as AMB-28, but difference is that voids are comparatively larger in dimension.

xxi. AMB-30

The matrix is composed of pale red yellow coloured siliceous materials with monocrystalline Quartz grains and monocrystalline rock fragments. There are some angular voids which have ferruginous lining.

xxii. AMB-31

The matrix is composed of fine grains siliceous with ferruginous materials. Angular fragments of twined plagioclase feldspar are also seen together with angular grains of monocrystalline undulose/undulatroy Quartz.

xxiii. AMB-32

Fine red to deep brown coloured matrix composed of numerous sub-angular to sub-rounded siliceous and opaque materials. The larger grains are of monocrystalline Quartz and weathered Feldspars.

xxiv. SPR-01

Red coloured matrix comprising of fine granular matrix which becomes isotropic under crossed nicols. The fine grain matrix also contains numerous black coloured opaque speck of varying dimensions. They do not show any reflectance under reflected light, this is indicating their organic composition. The coarser detrital fragments are composed of angular to sub rounded monocrystalline Quartz grains. Few feldspar (weathered) grains are also found. They are identified by their cloudy appearance and cleavages under polarized light. One prismatic coloured of Hornblende could also be identified; it is green in color and pleochroic. At some locations, there is presence of materials showing reduction in volume due to loss of firing.

xxv. SPR-02

Fine reddish siliceous matrix with coarser Quartz grains distributed throughout matrix.

xxvi. SPR-03

Fine reddish coloured matrix with angular monocrystalline Quartz feldspar. Minor amounts of irregular opaque bodies are also present.

xxvii. SPR-04 and SPR-05

Fine reddish matrices with siliceous materials larger angular grains of monocrystalline Quartz grains are observed. Sedimentary rock fragments distributed throughout groundmass. Minor organic materials are also observed.

xxviii. SPR-06

The matrix is composed of deep reddish to yellow coloured which containing high amount of coarser siliceous particles. The coarser fragments in the groundmass are of angular monocrystalline Quartz.

xxix. SPR-07

Fine groundmass contains lesser amount of siliceous materials. There is ill developed mineral orientation within fine grain groundmass. The coarser grains are monocrystalline Quartz and opaque reddish coloured bodies.

xxx. SPR-08

The fine yellowish matrix composed of coarser grains of monocrystalline Quartz and deep brown and black deposits of ferruginous and organic materials are also observed.

xxxi. KGF-01 and KGF-02

Matrix composed of brown to black coloured carbonaceous materials. There are also numerous highly fractured monocrystalline Quartz grains distributed throughout framework.

xxxii. KGF-03

Same as KGF-01 and KGF-02, but difference is that matrix composed of pale yellow to grey coloured very fine grained clayey which contain numerous sub-angular to sub-rounded particles of silt stone and mudstone. The intragranular particles are connected together by red ferruginous materials.

xxxiii. BMH-01

The matrix is composed of reddish coloured ferruginous materials together with sub-angular Quartz grains. Some ferruginous concentrations are observed.

xxxiv. BMH-02 and BMH-03

The matrix is composed of reddish coloured siliceous materials with large elongate monocrystalline Quartz grains which is floating throughout matrix.

xxxv. BMH-04 and BMH-05

Same as previous (BMH-02, 03), but red coloured rounded patches are found distributed throughout matrix. The matrix show siliceous and minor amount of carbonaceous materials with fibrous materials.

3.2.1.2 Classification of Historical pottery by Thin Section Petrography

Based on its mineralogical content and composition of matrix, the thin section of historical pottery samples are grouped as A, B, C D, E, F, G, H, I, J, K, L, M, N and O with fabric groups A, B, C, E, F and O having subgroups A1, B1, C1, E1, F1 and O1 respectively.

a. Fabric group A

The thin section falling in fabric group A (AMB-01) have brownish color framework. The grains are poorly sorted. Monocrystalline Quartz and plagioclase feldspar are present. The fractured grains of Quartz are filled with opaque to deep ferruginous materials which might be organic/carbonaceous materials? There is deposition of irregular spaces of black color opaque material which might be organic/carbonaceous materials.

1.1 Fabric Group A1

The thin section falling in fabric subgroup A1 (AMB-03, 11) and Fabric Group A have same matrix. The grains are angular and irregular in shape. Mineralogically, Quartz and plagioclase Feldspars are identified. Irregular opaque minerals are also present. Comparatively, quantity of Feldspars is higher in fabric group A1 than fabric group A.

b. Fabric group B

The matrix of fabric group B (AMB-02) has yellowish grey to greenish color framework. The grains are angular, rounded and irregular in shapes. Mineralogy includes Quartz, sands and rock fragments. The grains are poorly sorted and do not show specific orientations. Voids are not seen.

1.1. Fabric Group B1

The thin section of AMB-07 falling in category of fabric group B1 has yellowish to red color very fine matrix. The grains are angular, rounded and irregular in shape. The minerals are Quartz and few large irregular to rounded reddish tinge which might be organic/vegetative origin.

c. Fabric group C

The thin section falling under fabric group C (AMB-04, 08, 12, 15, 16, 19, 20; SPR-02-05) have red color ferruginous matrix. The grains are angular to sub angular in shapes. Only Quartz is identified in Fabric group C.

1.1.Fabric Group C1

The thin section falling in category of fabric group C1 (AMB-21, 22) have reddish color ferruginous groundmass. The grains are sub-angular in shape. Minerals include monocrystalline Quartz, mica flakes and silt stone.

d. Fabric Group D

The thin sections falling in category of fabric group D (AMB-24, 25, 26, 30) have pale to red color fine siliceous ferruginous groundmass. The grains are elongate and irregular in shape. The minerals are monocrystalline Quartz, feldspar and minor amount of opaque carbonaceous materials.

e. Fabric Group E

The thin sections falling in fabric group E (AMB-05, 06, 13, 17; SPR-06, 07) have fine siliceous matrices. The matrices show good sorting. The minerals include monocrystalline Quartz and some fractured larger grains which contain ferruginous materials within it.

1.1. Fabric Group E1

The thin section of AMB-18 falling in category of fabric group E1 has fine grains siliceous matrix. The grains are angular to sub-angular in shapes. Some coarser monocrystalline Quartz grains are identified and some minor amount of ferruginous materials also present.

f. Fabric Group F

The thin sections falling under fabric group F (AMB-09, AMB-31; BMH-01) have fine grains siliceous matrices. The grains are angular to sub angular and irregular in shapes. Mineralogy includes Quartz, plagioclase and Microcline Feldspars groups. Some coarser grains are also presents.

1.1. Fabric Group F1

The matrices of fabric group E and fabric group F1 (AMB-32) are same. They differentiated by their composition of minerals. The fabric group F1 has monocrystalline Quartz and plagioclase Feldspars in their compositions.

g. Fabric Group G

The thin section of AMB-10 is falling under fabric group G has greenish grey matrix. The grains are elongate in shapes. The fine siliceous and black opaque materials are identified. The numerous large spaces indicate the porous nature of matrix. The percentage of porosity is approximately 10 % in the matrix.

h. Fabric Group H

The thin section of AMB-14 falling in category of fabric group H has reddish brown clayey matrix which contains numerous Quartz. The grains are irregular in shapes. Only Quartz is identified. The matrix is composed of irregular concentration of reddish (ferruginous) deposits. Opaque substances observed. The percentage of porosity is approximately 10% of mineralogical framework.

i. Fabric Group I

The thin section of AMB-23 is falling in category of fabric group I has brown color matrix. The grains are sub-rounded in shape. Some irregular voids are presents. The matrix show muddy materials which have reddish color patches on it. Quartz, muddy materials and carbonaceous materials are identified.

j. Fabric Group J

The thin sections of AMB-27 and AMB-33 falling in category of fabric group J have red to deep brown coloured matrix. The majority of grains are angular to sub-angular, rounded to sub-rounded and elongated in shape. The non-plastic inclusions include Quartz, weathered plagioclase Feldspars.

k. Fabric Group K

The thin sections of AMB-28 and AMB-29 falling in category of fabric group K have black color carbonaceous matrix. The voids are seen and varying in sizes and shapes. Monocrystalline Quartz is only identified.

l. Fabric Group L

The thin section of SPR-01 and SPR-08 falling in category of fabric group L composed of red color fine granular grains which become isotropic in crossed nicols. The fabric group L has numerous black coloured opaque specks in varying dimensions which do not show any reflectance under light, it is indicating their organic origin. The grains are angular and sub rounded in shape. Minerals include monocrystalline Quartz, feldspar and Hornblende.

m. Fabric group M

The thin sections of historical pottery samples KGF-01 and KGF-02 falling under fabric group M have brown to black coloured carbonaceous matrices. The grains of monocrystalline Quartz are highly fractured.

n. Fabric group N

The fabric group N (KGF-03) is composed of pale yellow to grey coloured very fine grain clayey materials, which contain numerous angular to sub-rounded particles of silt stone and mudstone. The intragranular particles are connected together by red color ferruginous materials.

o. Fabric group O

The samples BMH-02 and BMH-03 falling under fabric group O have reddish coloured siliceous matrix. The grains are elongated in shape. Only, Quartz is identified.

1.1. Fabric group O1

The matrix of fabric group O and fabric group O1 (BMH-04, BMH-05) are same. They differentiated by the presence of red coloured patches which are distributed throughout matrix. The minor amounts of carbonaceous and fibrous materials are identified.

The thin section results demonstrated that Quartz is present as key mineral composition in all fabric groups. Monocrystalline Quartz grains are observed in the fabric groups of A, D, J, K, L and subgroups C1, C2, D1, and E1. The presence of monocrystalline grains indicates the igneous source of raw materials. The feldspar minerals group such as plagioclase, Microcline and Orthoclase etc. are observed in fabric groups A, D, F, J, K and subgroups A1 and F1. Carbonaceous substances are also present which either may be added intentionally as inclusions or might have occurred naturally. But sometimes it is difficult to distinguish. Moreover, fabric groups D, E and F contain fine siliceous materials in their composition. They are differentiated by grain, size and proportion of their raw materials and minerals. They present clear evidence of clay mixing. The few sub-rounded to rounded sand grains appearing as non-plastic inclusions within fine matrices of fabric groups are sand tempers added by potter. Thin section analysis revealed that historical pottery is made of both type of clay – primary/residual and secondary/sedimentary. Most of fabric groups are composed of ferruginous, micaceous siliceous and sometimes clayey materials. The grains are present in almost all forms such as angular to sub angular, rounded to sub-rounded and elongate in most of fabric groups which indicates that historical potters used secondary clay as temper. Generally, raw materials are well sieved and well levigated. Historical pottery samples might have been fired under both oxidizing and reducing atmosphere, which is confirmed by red, brown, greyish and black color matrices.

XRD, FTIR and Raman Spectroscopy analysis also showed similar results. The presence of organic matter in fabric group and sub groups A to O1 are confirmed. It is quite common that straw or fragments of plants added to the clay in order to increase the porosity of archaeological pottery. This is confirmed by the presence of voids left behind after combustion of organic matter during firing. Since these organic fragments are enclosed by clay, they are often carbonized and not oxidized completely, particularly when firing was performed under reducing conditions. In any case, remaining voids retain shape of original plant material. Hence studies of the void structure permits an identification of parts of a plant used, like leaves, stalks, seeds or roots. Voids have been observed in all shapes in historical potteries fabric groups such as A, G, H and J etc. The voids are more abundant in historical pottery than Neolithic pottery.

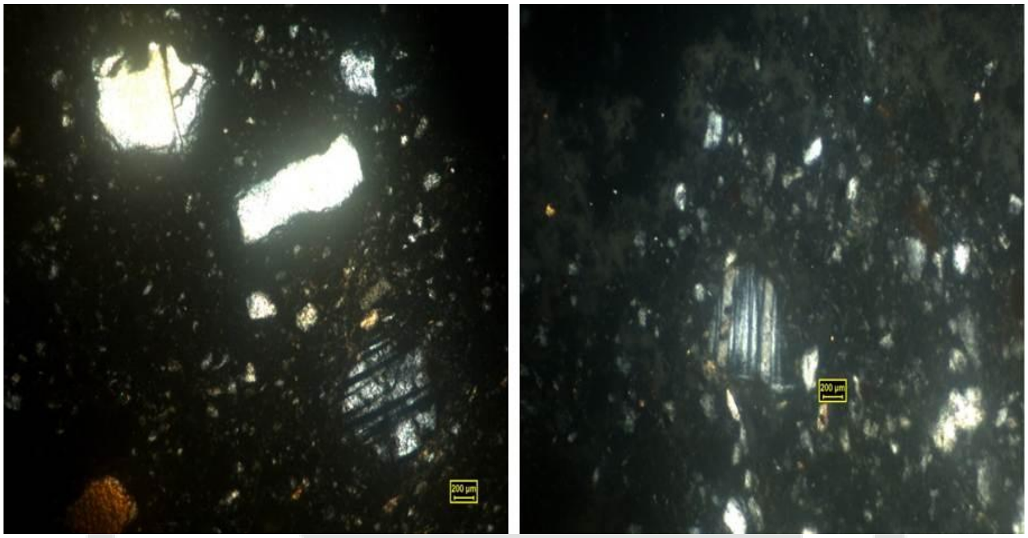

Figure 3.1 Thin section photomicrographs of AMB-01 shows brownish color matrix with few angular grains of Quartz floating in it and twined plagioclase.

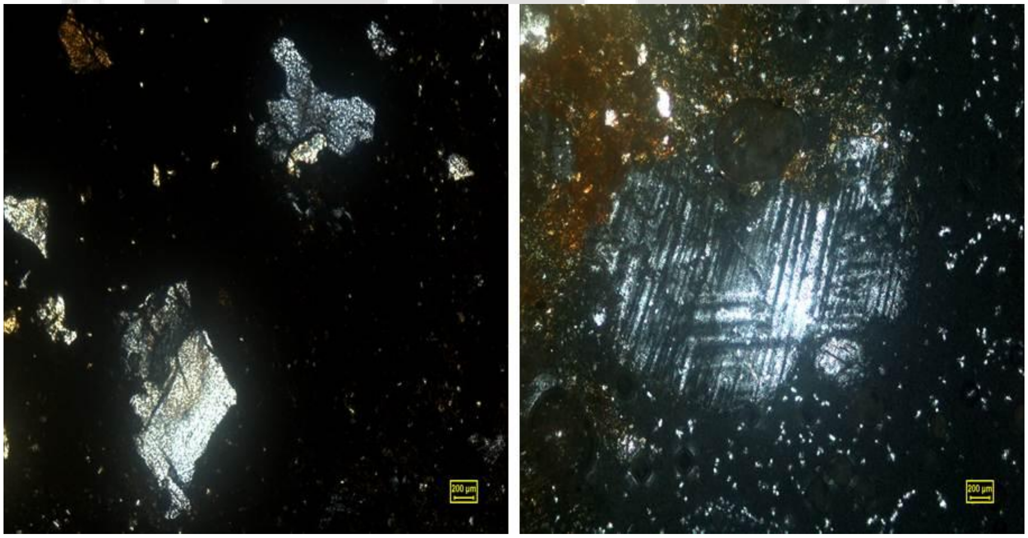

Figure 3.2 Thin section photomicrographs of AMB-03 shows highly angular fragments of mono-crystalline undolose Quartz and few grains of partially altered plagioclase feldspar

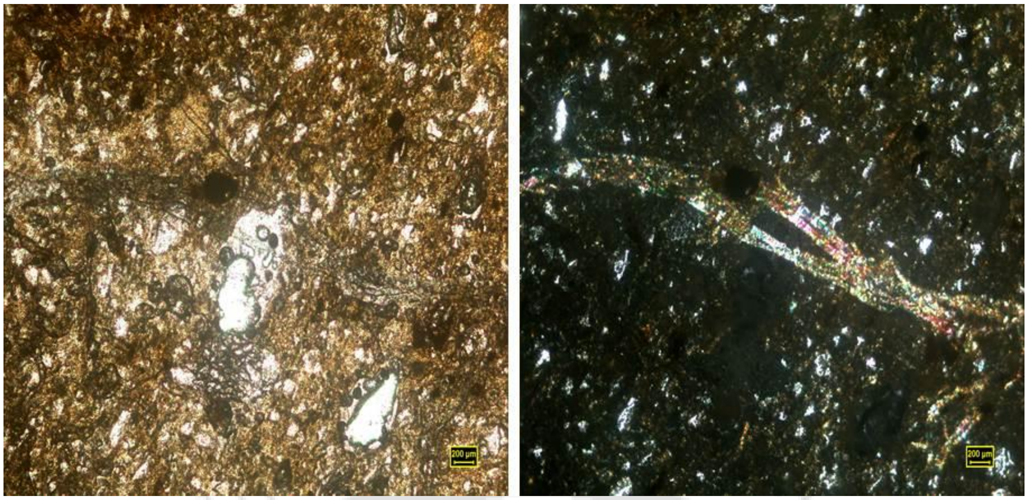

Figure 3.3 Thin section photomicrographs of AMB-05 is showing fibrous habit

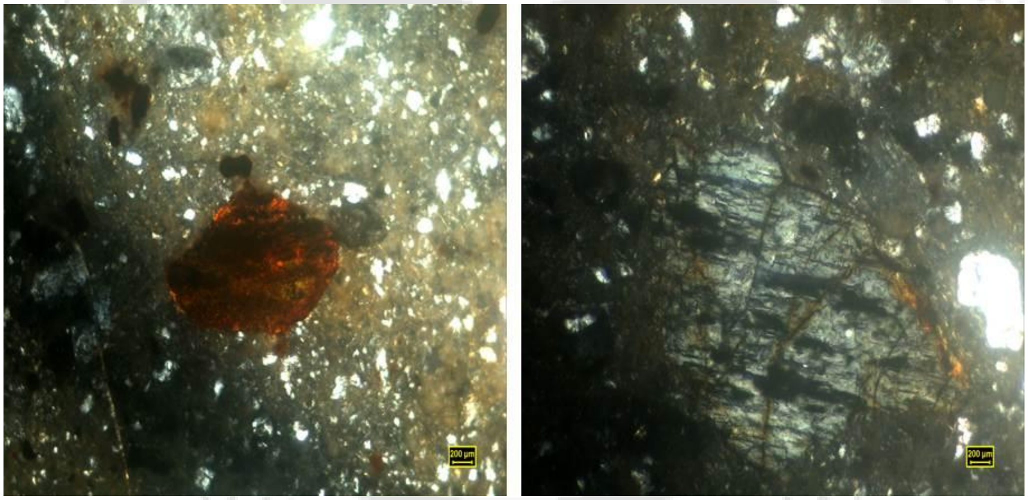

Figure 3.4 Thin section photomicrographs of AMB-10: reddish micaceous mineral and twinned feldspar with ferruginous stain along with twin plane

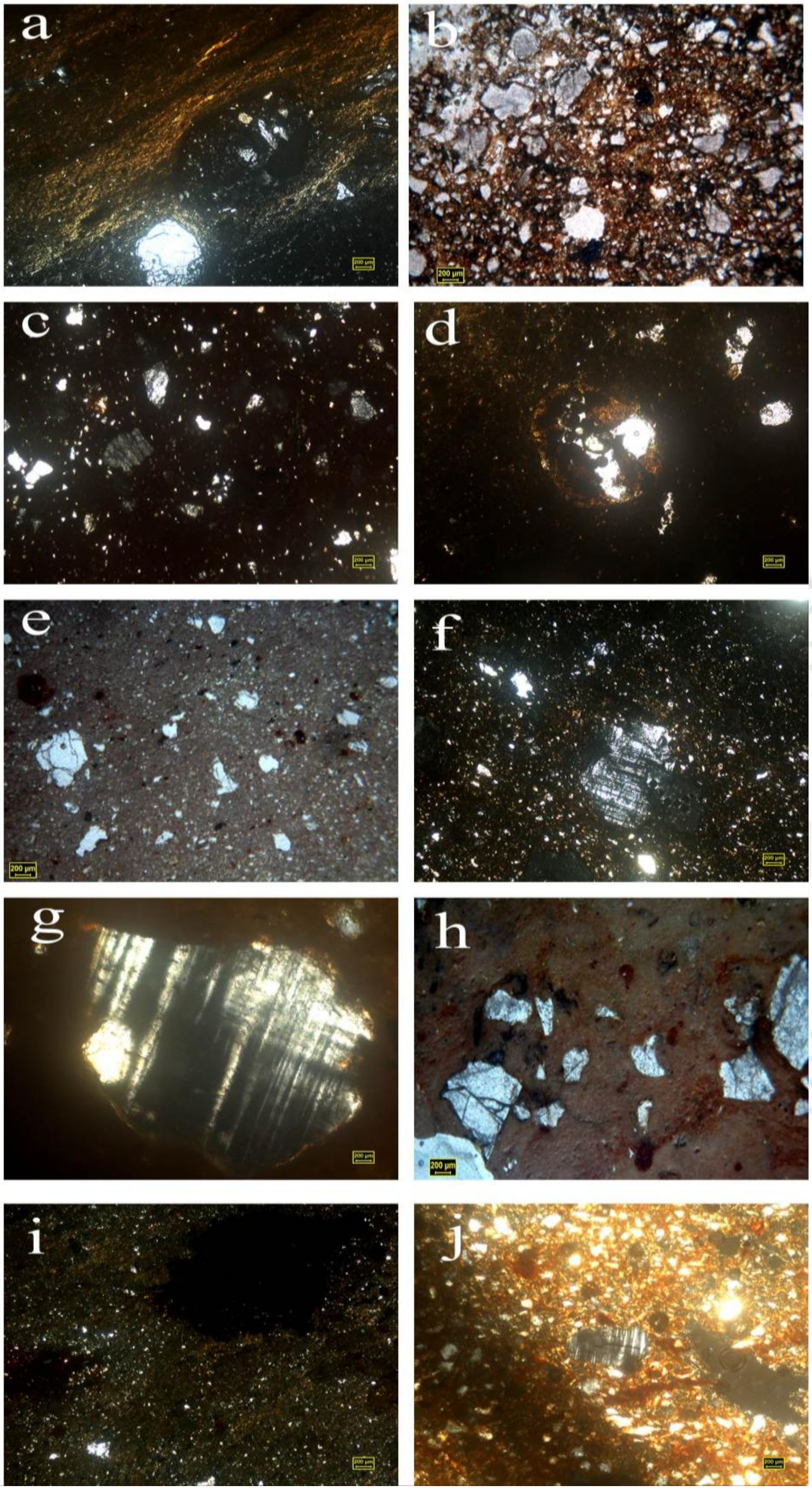

Figure 3.5 Thin section photomicrographs of (a) AMB-02 (b) AMB-04 (c) AMB-06 (d) AMB-07 (e) AMB-08 (f) AMB-09 (g) AMB-11 (h) AMB-12 (i) AMB-13 and (j) AMB-14

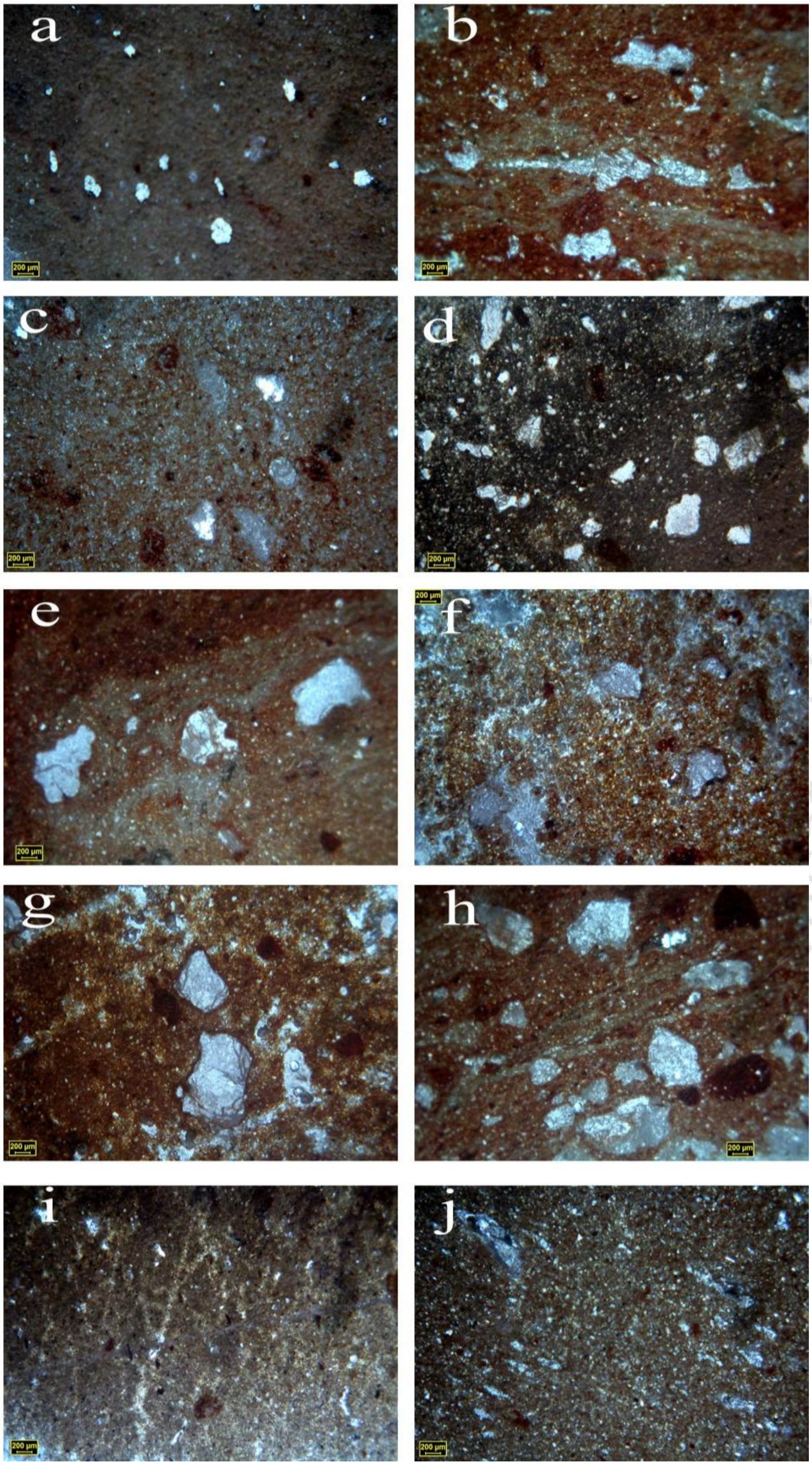

Figure 3.6 Thin section photomicrographs of (a) AMB-15 (b) AMB-16 (c) AMB-17 (d) AMB-18 (e) AMB-19 (f) AMB-20 (g) AMB-21 (h) AMB-22 (i) AMB-23 and (j) AMB-24

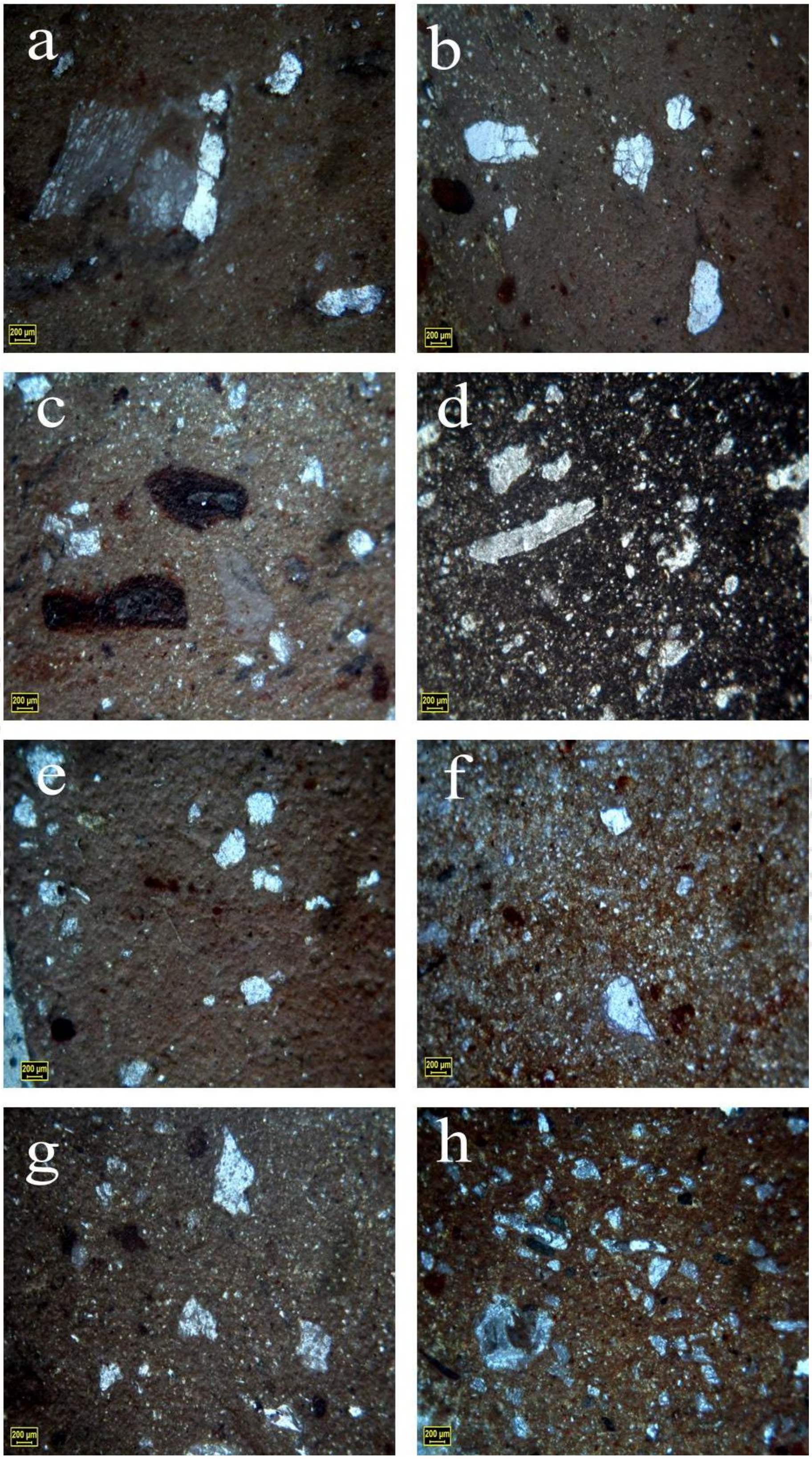

Figure 3.7 Thin section photomicrographs of (a) AMB-25 (b) AMB-26 (c) AMB-27 (d) AMB-29 (e) AMB-30 (f) AMB-31 (g) AMB-32 (h) AMB-33

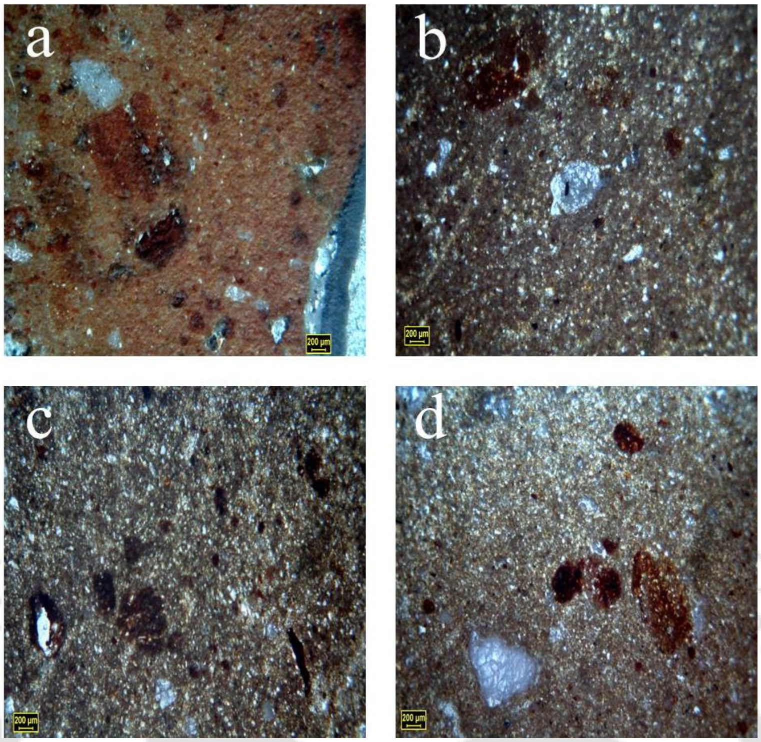

Figure 3.8 Thin section photomicrographs of (a) BMH-01 (b) BMH-03 (c) BMH-04 (d) BMH-05

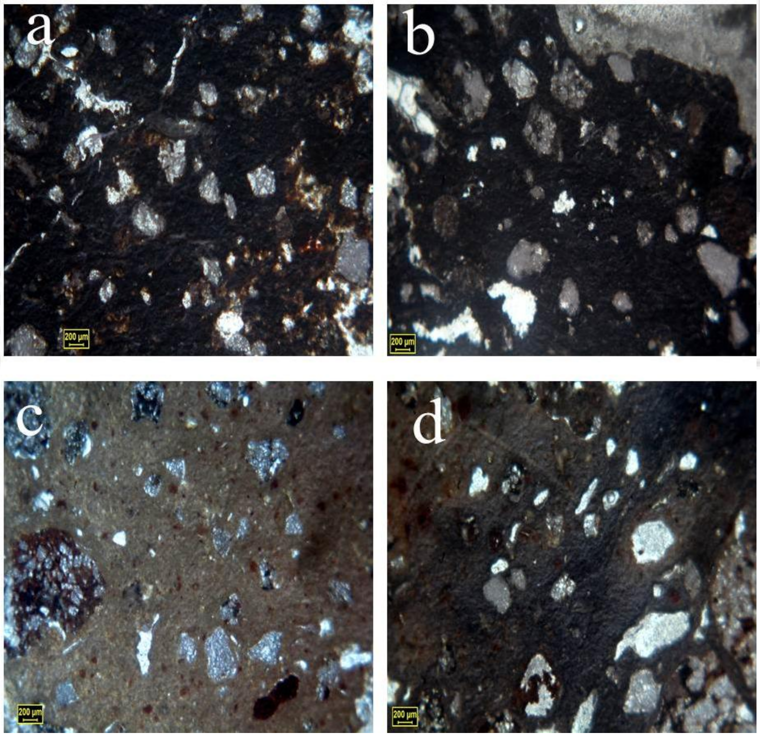

Figure 3.9 Thin section photomicrographs of (a) KGF-01 (b) KGF-02 (c) KGF-03 reddish matrix (d) KGF-03 blackish matrix

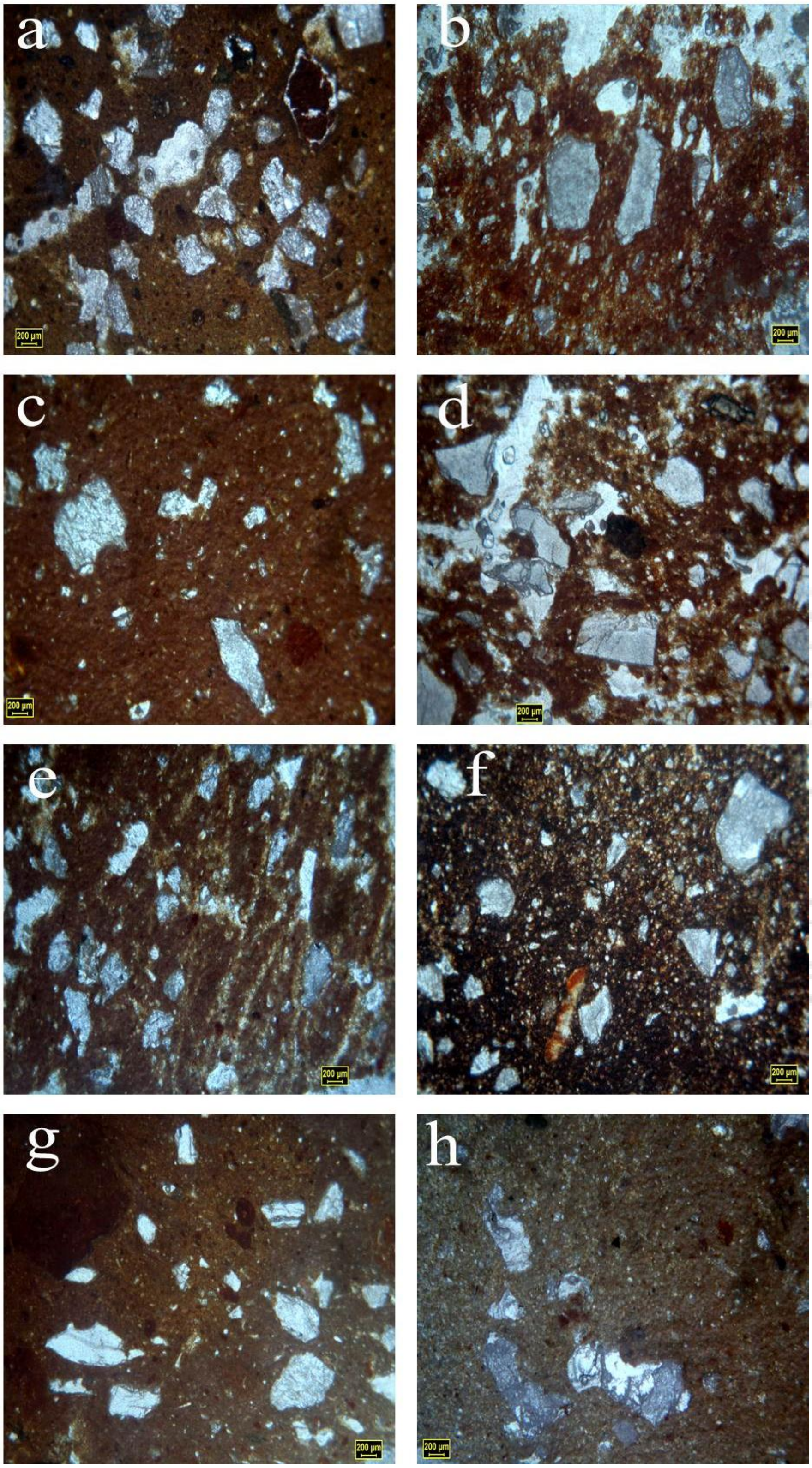

Figure 3.10 Thin section photomicrographs of (a) SPR-01 (b) SPR-02 (c) SPR-03 (d) SPR-04 (e) SPR-05 (f) SPR-06 (g) SPR-07 (h) SPR-08

3.3 Mineralogical characterization of Historical Pottery

Mineralogical analysis has been carried out on historical pottery from Assam and Manipur. The minerals are identified using Powder XRD, Raman spectroscopy and FTIR.

Powder XRD revealed the information about mineralogical phases and composition of historical pottery from Assam and Manipur. Its patterns with identified crystalline phases of investigated potsherds are shown in Tables 3.1-3.2 and figures 3.11-3.21.

The major mineral substances identified in all historical pottery are Quartz, Kaolinite, Goethite, Hematite, Biotite, Hornblende, Orthoclase, K-Feldspars (Microcline/Orthoclase). Other minerals such as Anorthite, Muscovite, Montmorillonite, Calcite and Anatase also have been identified in the composition of historical potsherds. Quartz and Kaolinite are present as major minerals in all analysed historical potsherds. However, both minerals are present in varying quantity in composition of all these potsherds. The tabulated data (table 3.1-3.2) indicate that Kaolinite is second most abundant mineral after Quartz present in composition of all analysed potsherds.

Feldspars mineral group such as Microcline, Orthoclase and Anorthite are present in most of analysed potsherds. Microcline is present in AMB-21, 22, 24 and 32. Orthoclase is present in AMB-08, 09, 20, 23, 32; SPR-01, 02, 03, 05; BMH-01, 03, 04; KGF-01 and 02. In addition, Anorthite is identified in AMB-03, 22 33; SPR-05 and 07. K-Feldspars is identified in most of analysed historical potsherds (excluding AMB-01, 02-06, 12-15, 18, 22, 24; SPR-03, 08; BMH-02 and KGF-02).

Hematite is identified in most of historical pottery. For instance, Hematite is presents in AMB-01-33; SPR-01-08; BMH-01-05 and KGF-01-03. Presence of Hematite indicates that all potsherds are fired in oxidizing atmosphere.

Hornblende is present in composition of historical pottery, such as, AMB-13, 23, 27, 28, 30, 31, 32; SPR-01 and SPR-05. The presence of a group of non-clay minerals, such as Feldspars, Biotite, Hornblende, etc., representative of igneous material, may indication for occurrence of volcanic-ash (Grim, 1953, p. 362).

Biotite is identified in mineralogical composition of historical potsherds of AMB-15, 18, 21, 22, 24, 32; SPR-01, 02, 05, 06 08; BMH-01, 02, 04, 05; KGF-01, 02 and 03. Goethite is present in almost all historical potsherds (excluding AMB-21, 22 and 30). Montmorillonite is present in pottery samples of AMB-07, 08, 09, 11-14, 18, 19, 25; BMH-01, 02, 03 and 05. Muscovite is detected in potsherds such as, AMB-05, 10, 20 and 23. Calcite is present only in AMB-07, 16 and 25.

Quartz is a major mineral which makes clay self-tempered. Tite *et al.* (2001) stated that Quartz-tempered pottery is less resistant than Calcite-tempered pottery to mechanical and thermal stresses during use. Calcite gets decomposed in between firing temperature 700–900 °C. The presence of Calcite in AMB-07, 16 and 25 indicate that it must have been fired in between 700-900 °C. The presence of Microcline in AMB-21, 22, 24 and 32 indicate firing temperature would have not been exceeding above 800 °C. Muscovite indicates that AMB-05, 10, 20 and 23 is fired around 950 °C. Hematite is one of most concentrated coloring materials and its minor presents can make pottery reddish (Singh & Sharma, 2016).

In addition to XRD, Raman Spectroscopy results provided significant information about mineralogical composition and firing process of analysed historical potsherds. Micro-Raman analysis revealed that all analysed potsherds have Quartz and Anatase (table 3.3 and 3.11) as key constituent in clay composition. In addition, other experiments such as thin section and FTIR analyses also confirmed that Quartz is key constituent in all analysed historical pottery. Carbonaceous materials are also observed. Hematite and other minerals are not detected in the most of potsherds due to the high florescence at analysing point. Anatase is observed only by m-Raman in the most of analysed historical potsherds. Besides these minerals, other identified constituents in analysed pottery samples are Ilmenite, plagioclase and Wollastonite. Technologically, the micro-Raman results reveal that most of analysed potsherds fired below 800-950 °C. This is confirmed by presence of only Anatase in most of analysed pottery samples, because Anatase-rutile process occurs at 800-950 °C. The sifting of Raman peaks from 144 cm^{-1} to 149, 151, 156 and 164 cm^{-1} and presence of 398, 632 and 637 cm^{-1} are also confirmed this.

In addition to powder XRD and Raman spectroscopy, FTIR analysis has also provided some information about mineralogical composition of historical potsherds. FTIR results demonstrated that Quartz is present in all analysed potsherds. Hematite has been detected (excluding AMB-09, 17, 26, 31-33) in most of potsherds. Magnetite is detected in AMB-17, 23 and SPR-01. Microcline is identified in pottery samples of AMB-01, 03, 17, 21, 26, 27, 30, SPR-01, 03, 04, 05 and 08.

Mineralogical investigation of historical potsherds has done using XRD, Raman spectroscopy and FTIR. Combining analytical method results revealed that historical potsherds contain Quartz and Kaolinite in their composition in varying quantity. Abundance of Quartz indicates that raw materials might have been procured from the piedmont area (Ravisankar 2010). Discussed earlier, presence of Quartz may be elucidated as indigenous minerals of natural clay or added intentionally as tempering materials are not easy to identify. Therefore, presence of Quartz in raw materials is allowed to improve its workability and to allowed water vaporize gradually (Medeghini, *et al.*, 2013, p. 889; Maggetti, 1982). Feldspar minerals (Microcline and Orthoclase) are identified in composition of all historical pottery samples. These minerals are key constituent of the original clay matrix. The presence of Hematite also confirmed that all potsherds are fired in oxidizing atmosphere. Anatase is detected only by Raman spectroscopy in all analysed historical potsherds. The presence of non-clay minerals such as Hornblende, Feldspars, Biotite which is representative of igneous materials confirmed that volcanic origin of raw materials or metamorphic rock present in the area. Clay minerals such Montmorillonite is present in AMB-07, 08, 09, 11-14, 18, 19, 25; BMH-01, 02, 03 and 05 and Muscovite detected in AMB-05, 10, 20 and 23. Calcite is present only in AMB-07, 16 and 25. Amorphous carbons and organic substances in historical pottery samples have been detected by Raman spectroscopy and FTIR analysis.

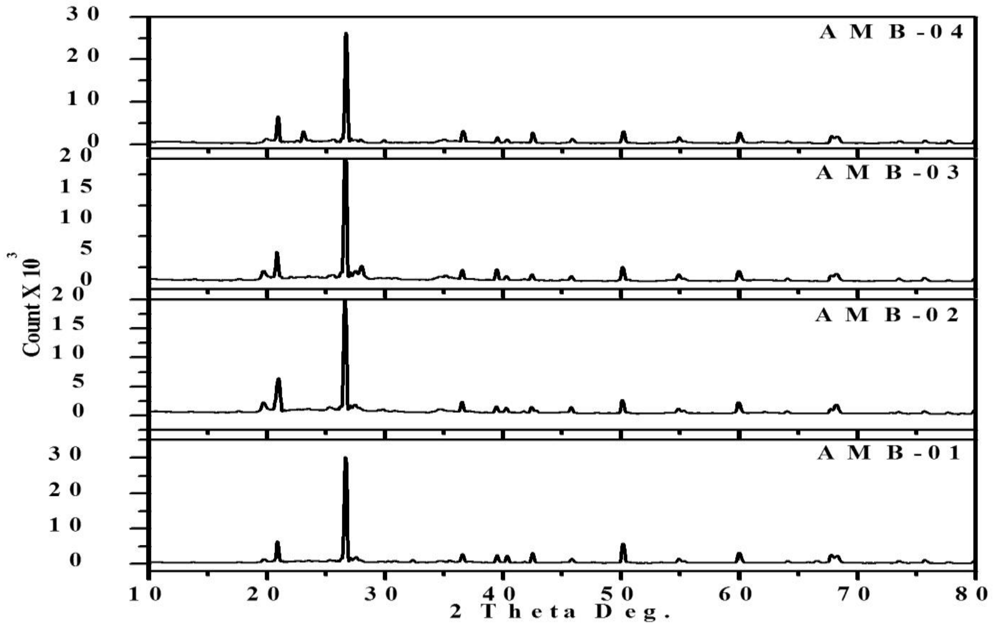

Figure 3.11 Powder XRD Patterns of AMB-01, AMB-02, AMB-03 and AMB-04

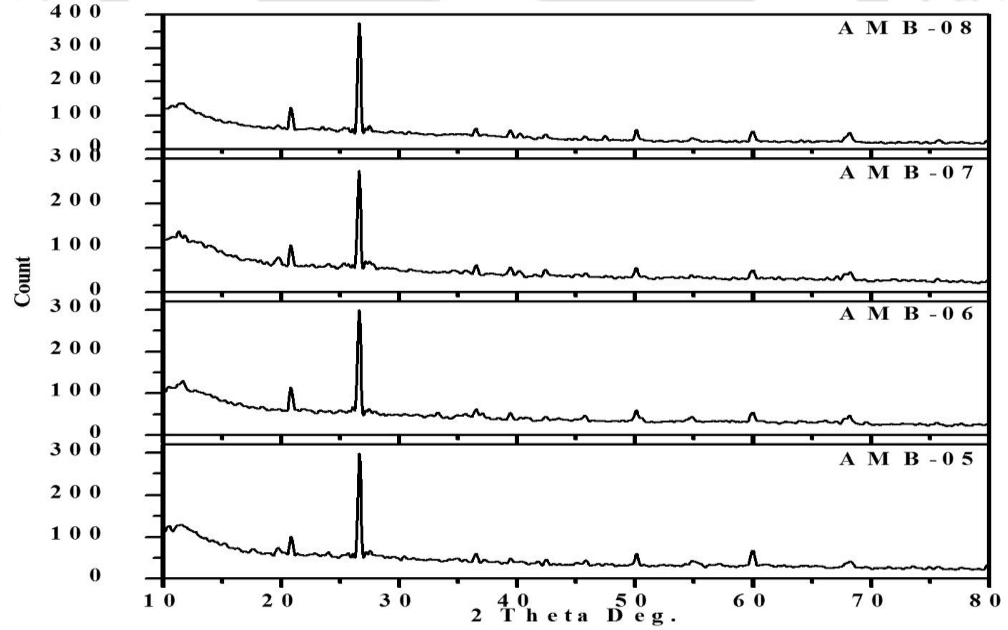

Figure 3.12 Powder XRD Patterns of AMB-05, AMB-06, AMB-07 and AMB-08

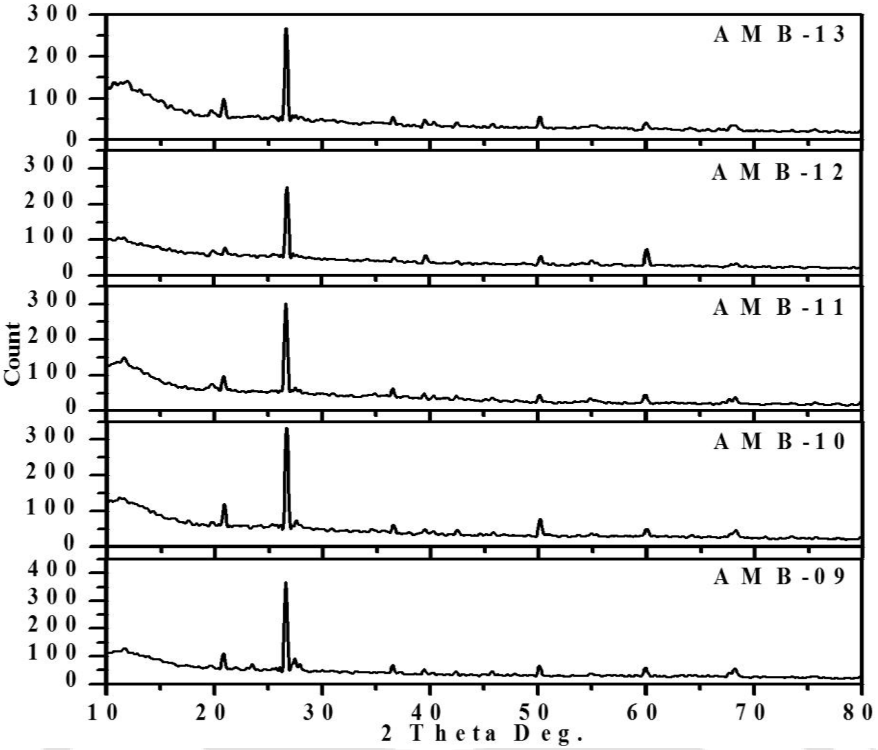

Figure 3.13 Powder XRD Patterns of AMB-09. AMB-10, AMB-11, AMB-12 and AMB-13

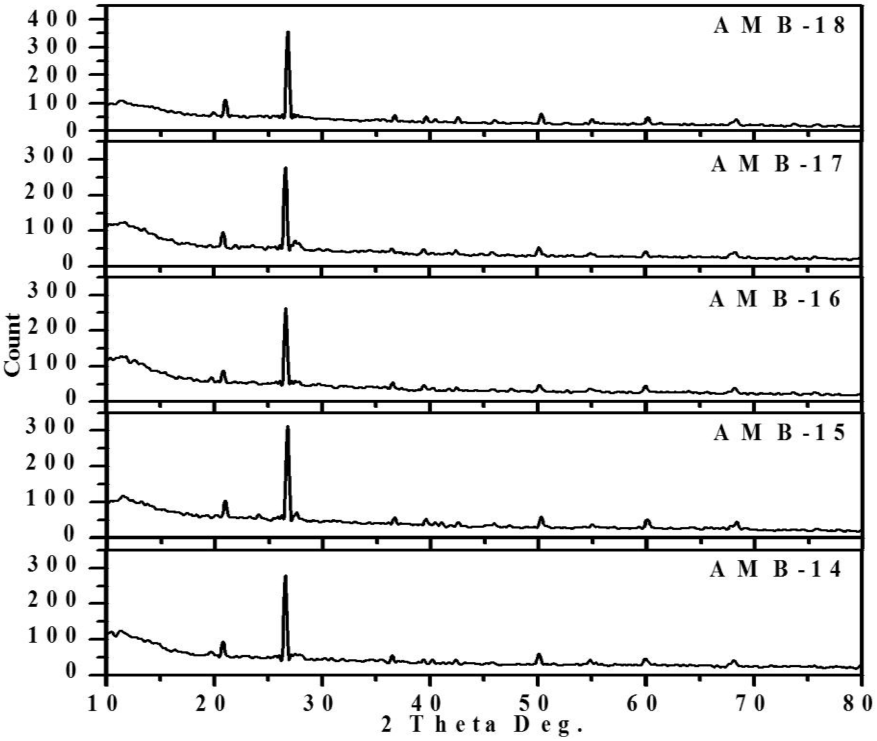

Figure 3.14 Powder XRD Patterns of AMB-14. AMB-15, AMB-16, AMB-17, and AMB-18

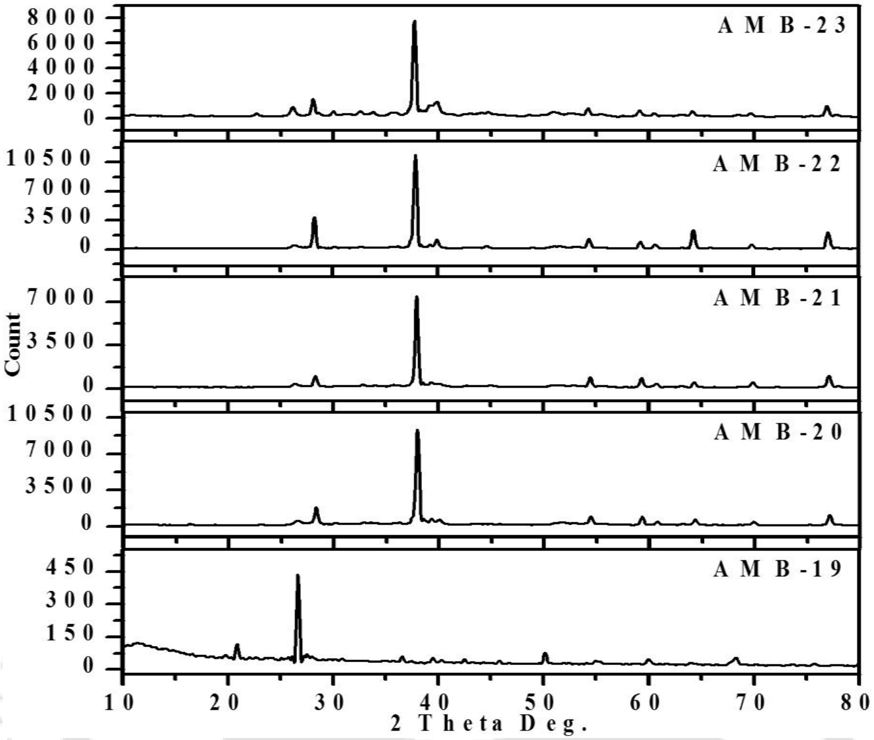

Figure 3.15 Powder XRD Patterns of AMB-19. AMB-20, AMB-21, AMB-22, and AMB-24

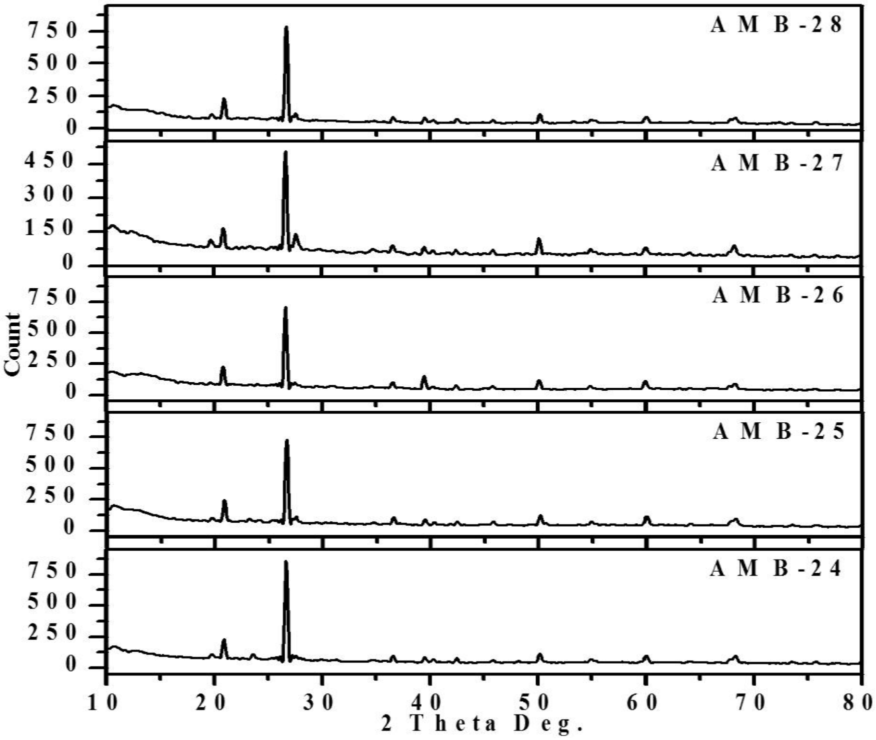

Figure 3.16 Powder XRD Patterns of AMB-24. AMB-25, AMB-26, AMB-27, and AMB-28

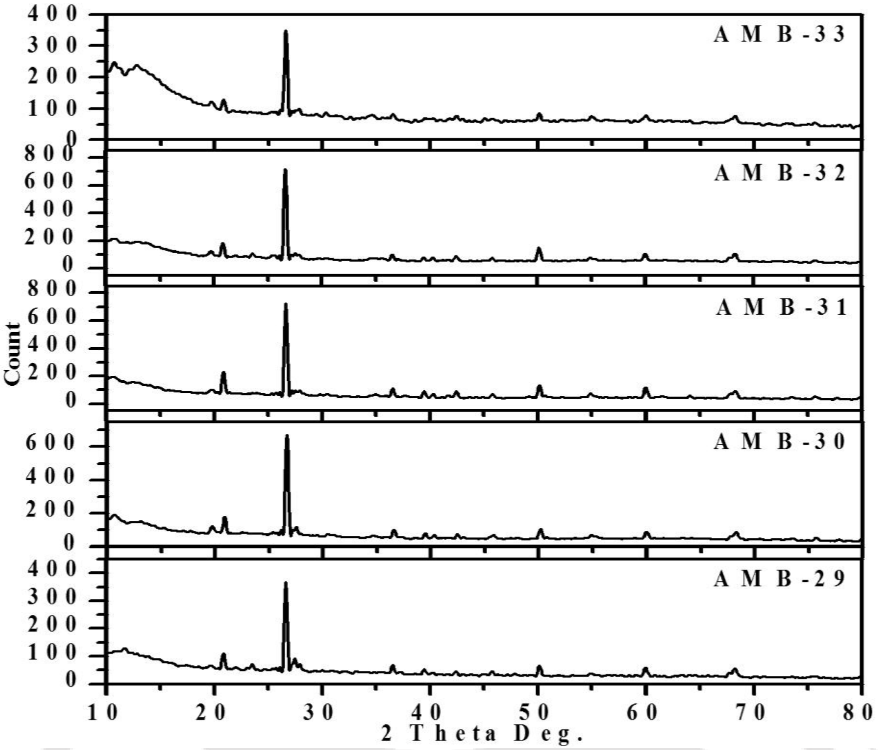

Figure 3.17 Powder XRD Patterns of AMB-29. AMB-30, AMB-31, AMB-32, and AMB-33

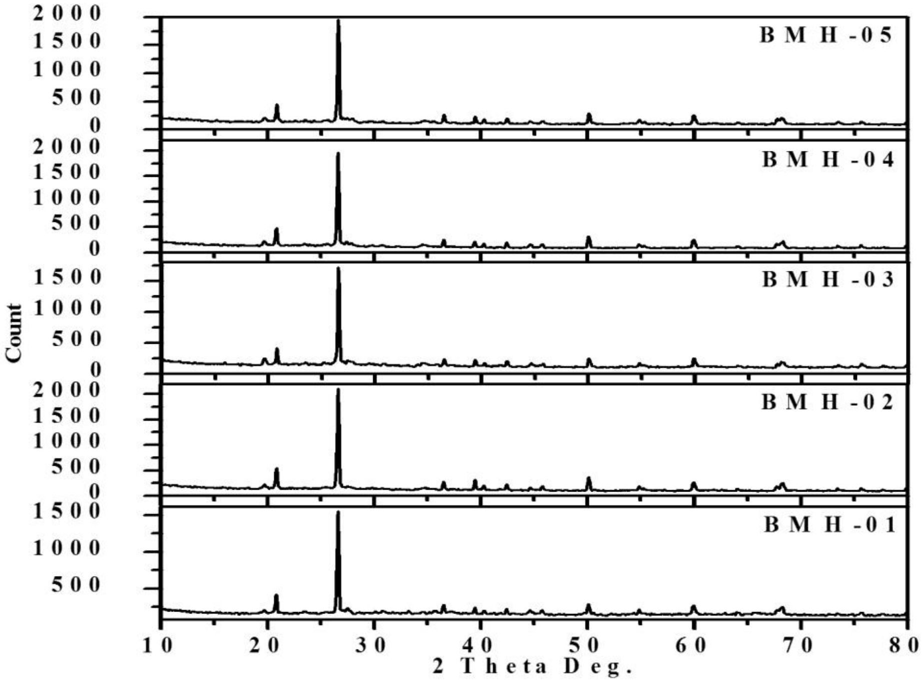

Figure 3.18 Powder XRD patterns of BMH-01, BMH-02, BMH-03, BMH-04 and BMH-05

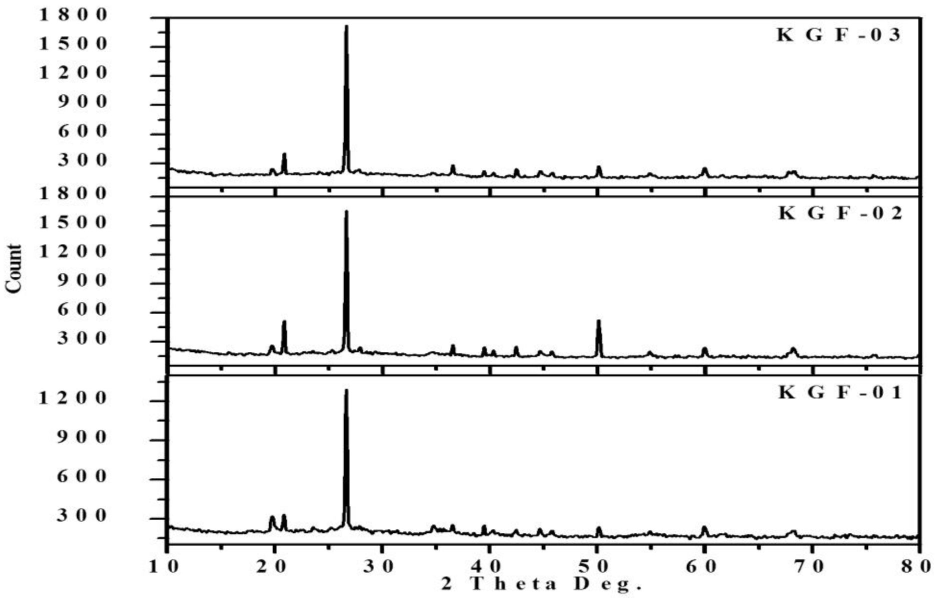

Figure 3.19 Powder XRD patterns of KGF-01, KGF-02 and KGF-03

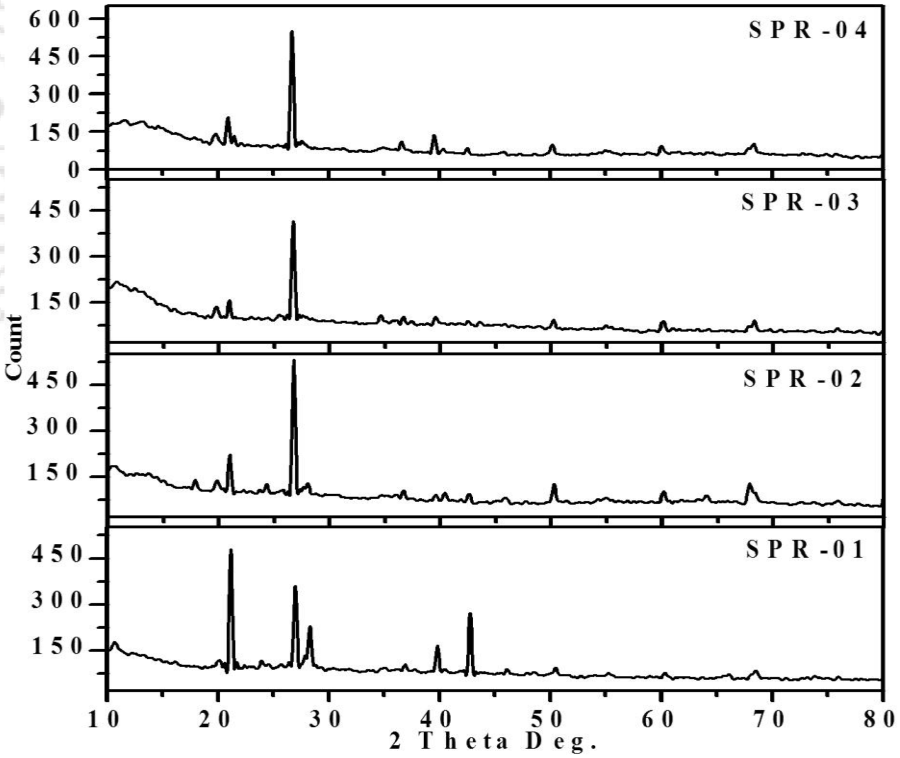

Figure 3.20 Powder XRD patterns of SPR-01, SPR-02, SPR-03 and SPR-04

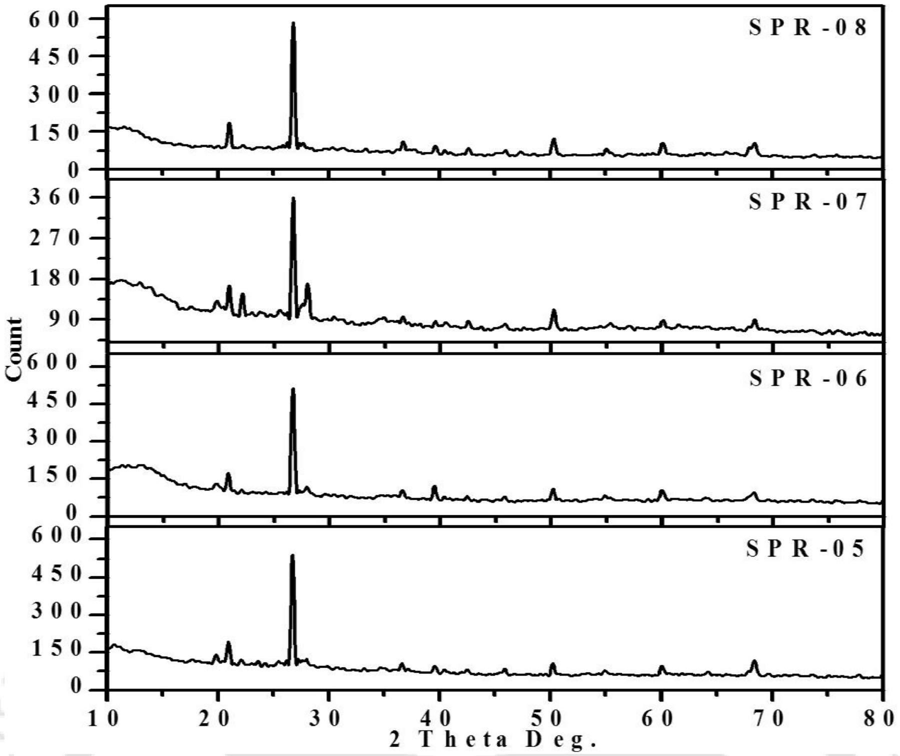

Figure 3.21 Powder XRD patterns of SPR-05, SPR-06, SPR-07 and SPR-08

Table 3.1 Identified Minerals and phase obtained by Powder XRD of Historical period (Sri Sri Suryapahar, Bamuni Hill, Kangla Fort), ++++ = very abundant, ++ =abundant; + = present, - = absent

Si. No.	**Sample name**	**Quartz**	**Kaolinite**	**Hematite**	**Goethite**	**Microcline/ Orthoclase**	**Anorthite**	**Montmorillonite**	**Hornblende**	**Orthoclase**	**Biotite**
1	SPR-01	+++++++	++	+++	+	+	-	-	+	+	+
2	SPR-02	++++++++	+++	++	+	+	-	-	-	-	+
3	SPR-03	++++++	+	++	-	-	-	-	-	+	-
4	SPR-04	++++	++++	++	+	+	-	-	-	-	-
5	SPR-05	++++++++	++	++	+	+	+	-	+	+	+
6	SPR-06	++++++	+++	++	+	+	-	-	-	-	-
7	SPR-07	++++++	++	++	+	+	+	-	-	-	-
8	SPR-08	++++++++	+++	+	+	-	-	-	-	-	+
9	BMH-01	++++++++	+	+	+	+	-	+	-	+	+
10	BMH-02	+++++++	++	+++	+	-	-	+	-	-	+
11	BMH-03	+++++++	+	++++	+	+	-	+	-	+	+
12	BMH-04	+++++++++	++	+++	+	+	-	-	-	+	+
13	BMH-05	+++++++	++	++	+	+	-	+	-	-	-
14	KGF-01	+++++++++	+++	+	+	-	-	-	-	+	+
15	KGF-02	++++++++	+++	++	+	+	-	-	-	+	+
16	KGF-03	++++++++	++++	+	+	+	-	-	-	-	+

Table 3.2 Identified Minerals and phase obtained by Powder XRD of Historical period (Ambari), ++++ = very abundant, ++ =abundant, + = present, - = absent

Si. No.	**Sample name**	**Quartz**	**Kaolinite**	**Hematite**	**Goethite**	**Microcline/ Orthoclase**	**Anorthite**	**Microcline**	**Muscovite**	**Montmorillonite**	**Calcite**	**Hornblende**	**Orthoclase**	**Biotite**
1	AMB-01	+++++++	+	++	+	-	-	-	-	-	-	-	-	-
2	AMB-02	+++++++	+++++	++	+	-	-	-	-	-	-	-	-	-
3	AMB-03	++++++++	++++	++	+	-	+	-	-	-	-	-	-	-
4	AMB-04	+++++++	+	++	+	-	-	-	-	-	-	-	-	-
5	AMB-05	++++++++	+++++	++	+	-	-	-	+	-	-	-	-	-
6	AMB-06	+++++++	++++	+	+	-	-	-	-	-	-	-	-	-
7	AMB-07	++++++++	++++	+	+	+	-	-	-	+	+	-	-	-
8	AMB-08	+++++++++	+++	+	+	+	-	-	-	+	-	-	+	-
9	AMB-09	++++++	+++	+	+	+	-	-	-	+	-	-	+	-
10	AMB-10	+++++++	++	+	+	+	-	-	+	-	-	-	-	-
11	AMB-11	+++++++	++++	+	+	+	-	-	-	+	-	-	-	-
12	AMB-12	+++	++	+	+	-	-	-	-	+	-	-	-	-
13	AMB-13	+++++++	+	+	+	-	-	-	-	+	-	+	-	-
14	AMB-14	++++++	+++	+	+	-	-	-	-	+	-	-	-	-
15	AMB-15	+++++++	+	+	+	-	-	-	-	-	-	-	-	+
16	AMB-16	+++++++	+++++	++	+	+	-	-	-	-	+	-	-	-
17	AMB-17	+++++	+	++	+	+	-	-	-	-	-	-	-	-
18	AMB-18	++++++	++++	++	+	-	-	-	-	+		-	-	+
19	AMB-19	++++++	++++	+	+	+	-	-	-	+	-	-	-	-
20	AMB-20	+++++++	++	++	+	+	-	-	+	-	-	-	+	-
21	AMB-21	+++++	+++	+++	-	+	-	+	-	-	-	-	-	+
22	AMB-22	+++++++	++	+++	-	-	+	-	-	-	-	-	-	+
23	AMB-23	+++++++	+++++	++	+	+	-	-	+	-	-	+	+	-
24	AMB-24	++++++	+	++	+	-	-	+	-	-	-	-	-	+
25	AMB-25	++++++++	++	++	+	+	-	-	-	+	+	-	-	-
26	AMB-26	++++++++	++	++	+	+	-	-	-	-	-	-	-	-
27	AMB-27	++++++++	++	+	+	+	-	-	-	-	-	+	-	-
28	AMB-28	++++++	++	+++	+	+	-	-	-	-	-	+	-	-
29	AMB-29	++++++++	++	+	+	+	-	-	-	-	-	-	-	-
30	AMB-30	++++++++++	++	++	+	+	-	-	-	-	-	+	-	-
31	AMB-31	++++++++	++	+++	+	+	-	-	-	-	-	+	-	-
32	AMB-32	+++++++	++	++	+	+	-	+	-	-	-	+	+	+
33	AMB-33	++++++	++	++	+	+	+	-	-	-	-	-	-	-

3.4 Infrared analysis of Historical Pottery

FTIR has been used to reconstruct the firing techniques and manufacturing processes of historical potsherds. The FTIR spectra and their peak positions of various bands with varied intensity of pottery samples of historical period are observed. These spectra along with their tentative assignments are summarized in Table 3.3-3.12 and figures 3.22-3.30.

The IR spectrum of AMB-04 shows very weak peak at 3703 cm^{-1} is due O-H stretching of interlayer water. AMB-04, AMB-11, SPR-05 and SPR-06 are show very weak infrared bands around 3660-3616 cm^{-1} are due to Inner O-H group of adsorbed water (Velraj *et. al.*, 2009).

The medium to strong infrared spectra around 3480-3418 cm^{-1} and weak to medium spectra around 1630 cm^{-1} are observed in all historical potsherds due to O-H stretching and O-H bending of absorbed water molecule (Venkatachalapathy *et al.,* 2002; Manoharan *et al.*, 2007.

The shoulder very weak IR spectra around 2950-2220 cm^{-1} are probably due to aliphatic C–H stretching bands as observed in organic materials. These bands are observed in AMB-01, 02, 04, 06, 09, 15, 17, 18, 20, 22, 24, 25, 28, 30, 31, 32, 33; SPR-04, 07, 08; BMH-02, -05; KGF-02 and KGF-03 (Claret, *et al.,* 2003).

The infrared bands around 1890 cm^{-1}-1814 cm^{-1} are due to carbonate overtone/combinations detected in analysed specimens, such as AMB-01, 03, 08, 10, 17, 18, 19, 20, 21, 22, 23, 24, 25, 28, 28; SPR-02 and SPR-08.

The spectra around 1140-1080 cm^{-1} with very strong intensity is due to white clay origin of Kaolinite which is present in the AMB-01, 06, 08, 09, 10, 12, 15, 17, 23, 25, 26, 28, 29 and SPR-01 and SPR-05.

Rest of analysed historical pottery specimens recorded the IR spectra around 1050-1015 cm^{-1} with very strong intensity is due to red clay origin of Kaolinite. This is indicating that red clay origin of Kaolinite used in making pottery.

The very weak bands appear at 798-778 cm^{-1} and 694-664 cm^{-1} are due to presence of Quartz detected in all analysed historical potsherds.

The presence of very weak bands around 565-530 cm^{-1} and 477-471 cm^{-1} are attributing to the presence of Hematite in most of analysed potsherds (excluding AMB-08, 09, 15, 17, 20, 23, 26, 31, 32, 33 and SPR-01). AMB-17, 23 and SPR-01 showed infrared bands of 574 cm^{-1}, 581 cm^{-1} and 578 cm^{-1} respectively. These peaks are assigned to Fe_3O_4 or FeO of Magnetite.

The presence of absorption bands in the sample around 481 cm^{-1}, and 465 cm^{-1} are due to Microcline and identified in AMB-01, 03, 17, 21, 26, 27, 30; SPR-01, 03, 05 and 08.

On the basis of above results, FTIR analysis indicates the firing temperature range of AMB-01, 06, 08, 09, 10, 12, 15, 17, 23, 25, 26, 28, 29; SPR-01 and 05 should be within 800-900 °C. Rest of historical potsherds would have been fired approximately 650-850 °C. The presence of Hematite in most of potsherds is indicating towards oxidizing firing atmosphere of historical potsherds. Presence of magnetite in AMB-17, 23, SPR-01 confirmed that these pottery fired around 800-900 °C under reducing firing atmosphere. C–H stretching spectra reveal presence of organic matters in the composition of pottery. Generally, natural clay contains varying quantity of organic materials in their composition. However, potters may use organic materials externally as additive to improve plasticity during clay preparation. Furthermore, pottery can absorb organic substances during burial period (Damjanovic, *et al.*, 2011, p. 826). The FTIR result has good agreement with thin section petrography and Powder XRD analysis.

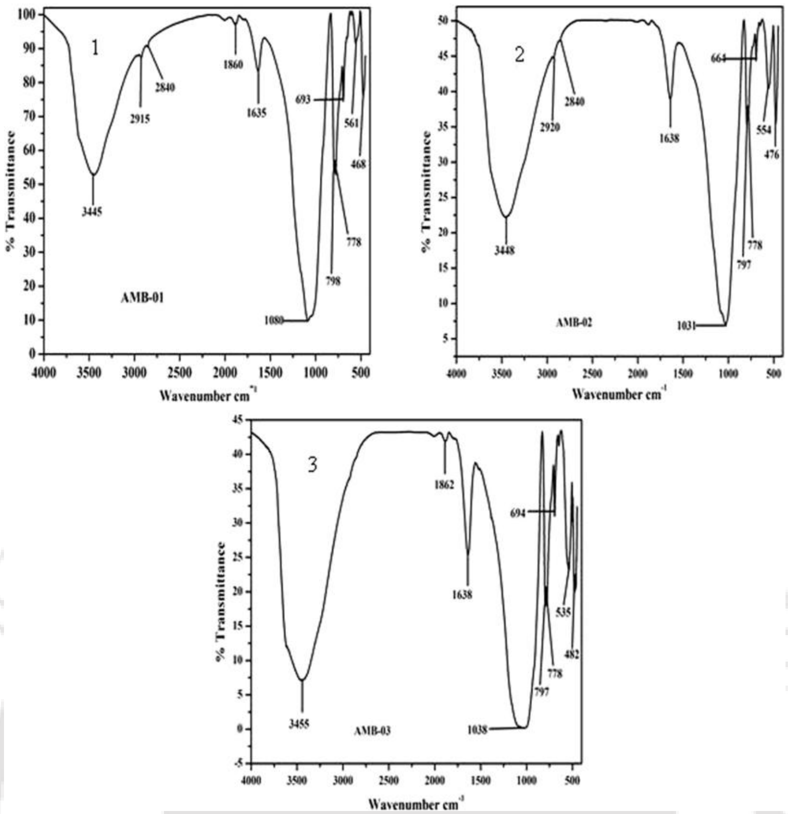

Figure 3.22 FT-IR graphs of Ambari pottery: (1) AMB-01 (2) AMB-02 (3) AMB-03

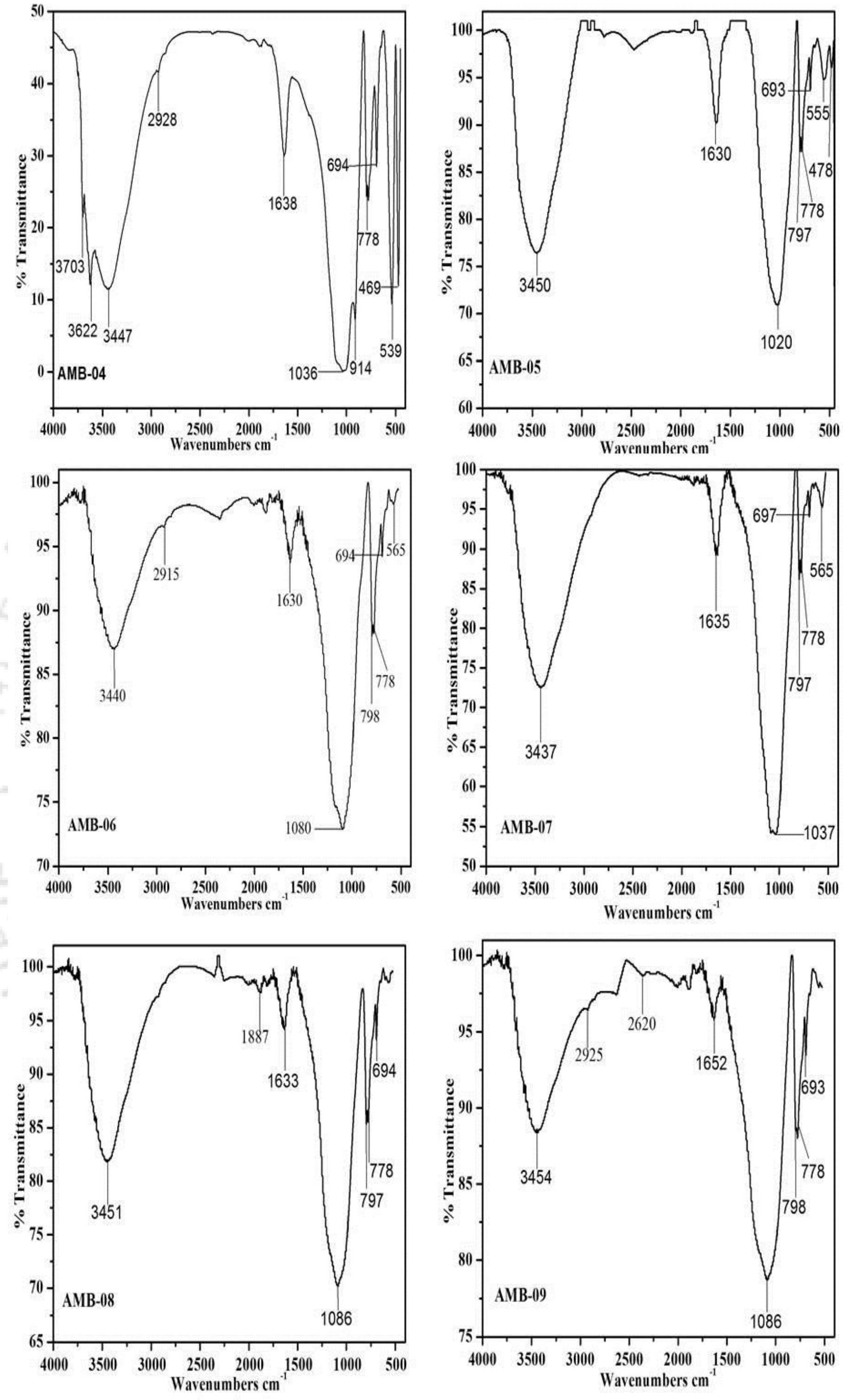

Figure 3.23 FT-IR graphs of AMB-04, AMB-05, AMB-06, AMB-07, AMB-08 and AMB-09

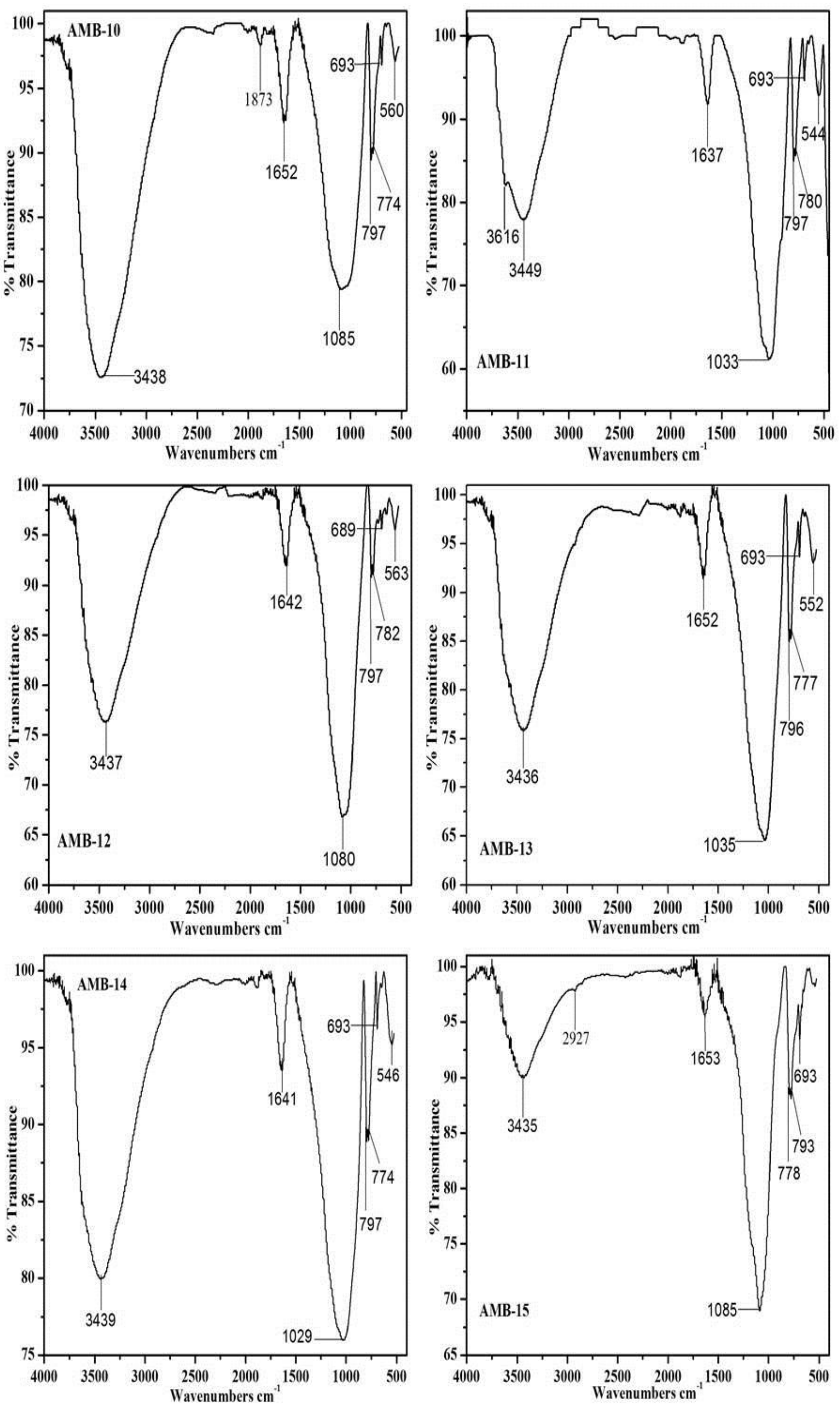

Figure 3.24 FT-IR graphs of AMB-10 AMB-11, AMB-12, AMB-13, AMB-14 and AMB-15

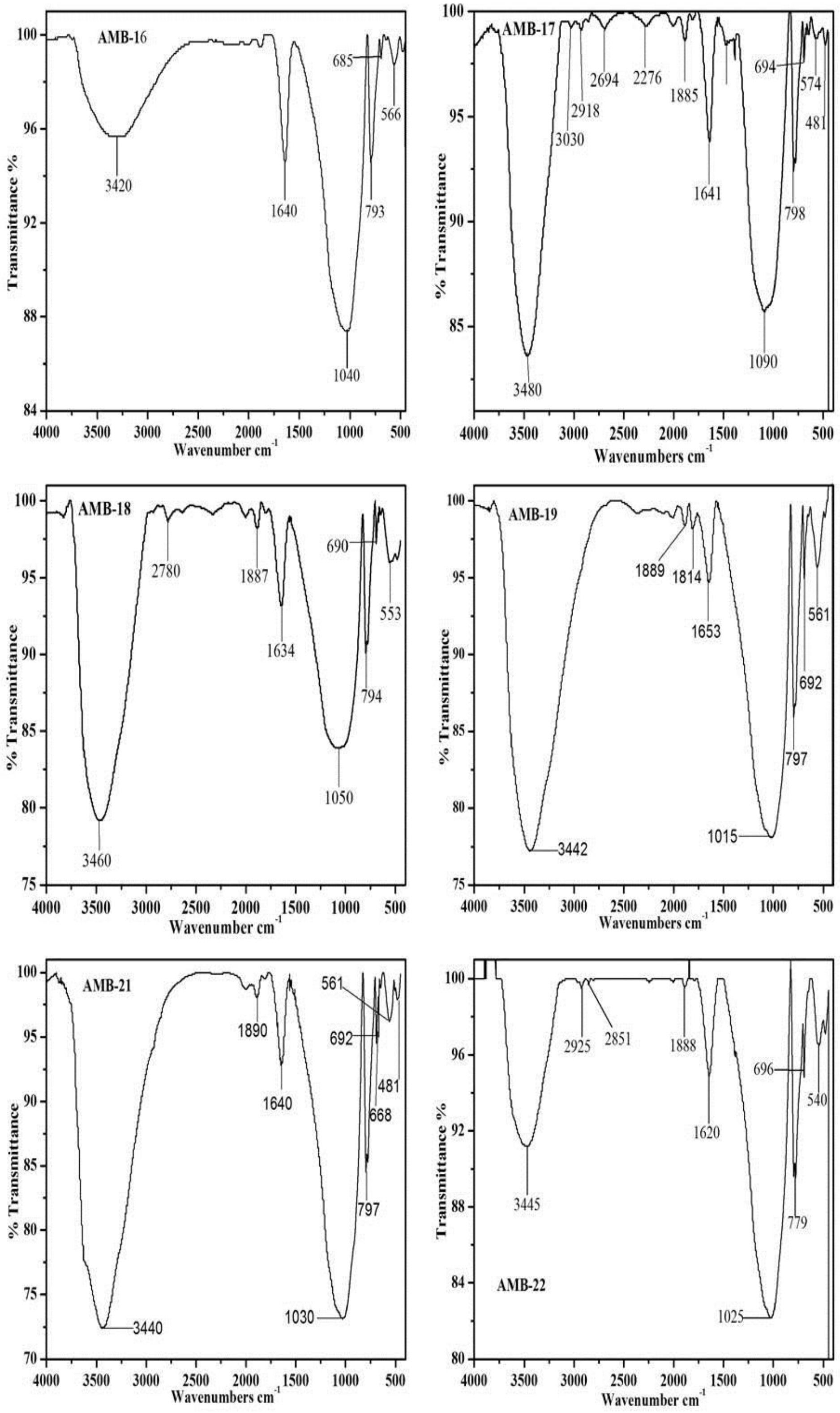

Figure 3.25 FT-IR graphs of AMB-16, AMB-17, AMB-18, AMB-19, AMB-21 and AMB-22

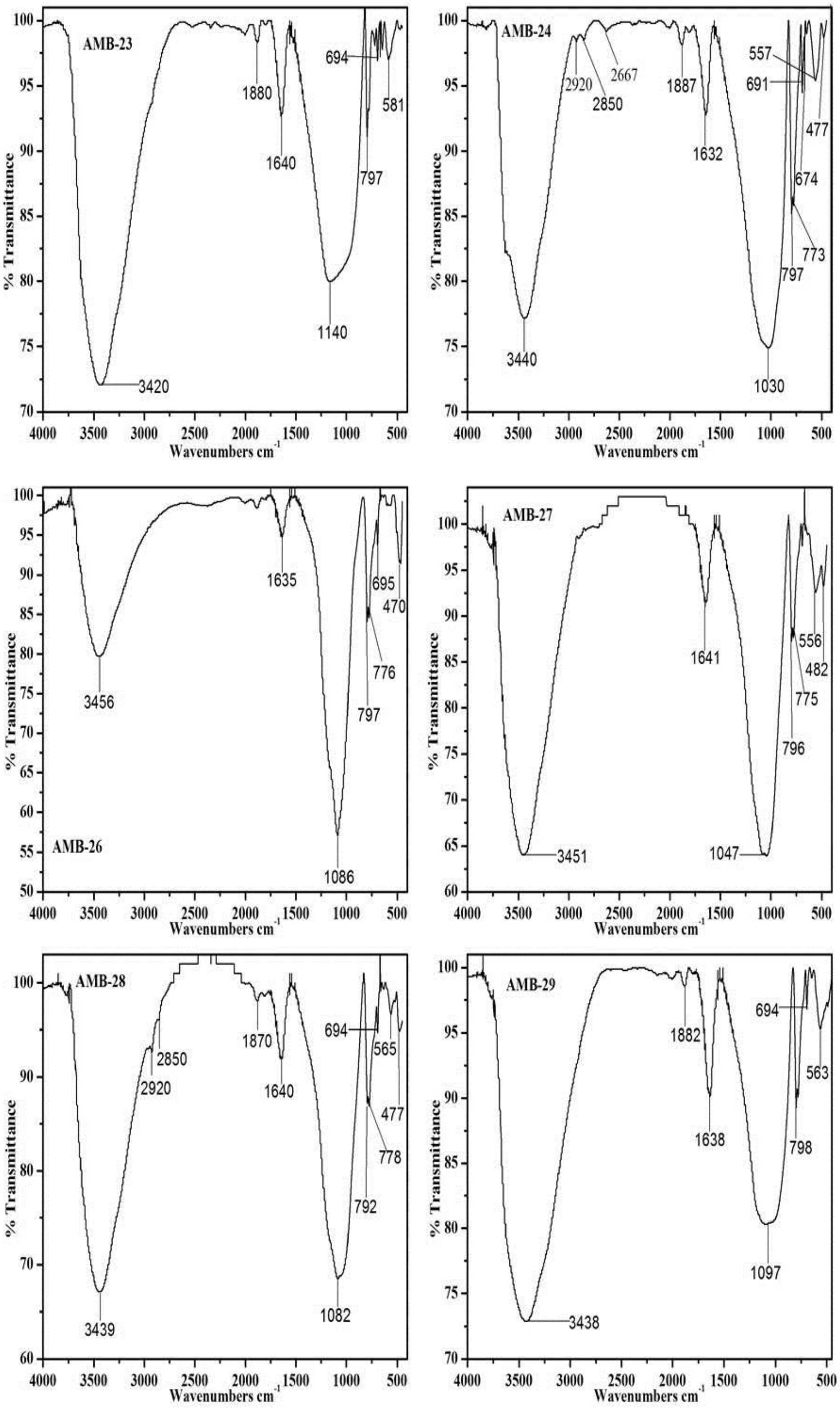

Figure 3.26 FT-IR graphs of AMB-23 AMB-24, AMB-26, AMB-27, AMB-28 and AMB-29

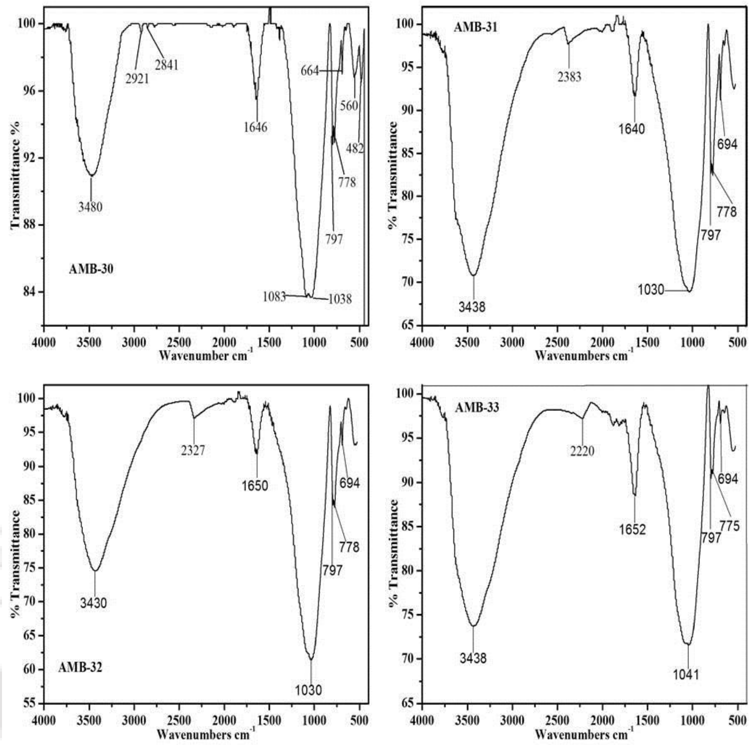

Figure 3.27 FT-IR graphs of AMB-30 AMB-31, AMB-32 and AMB-33

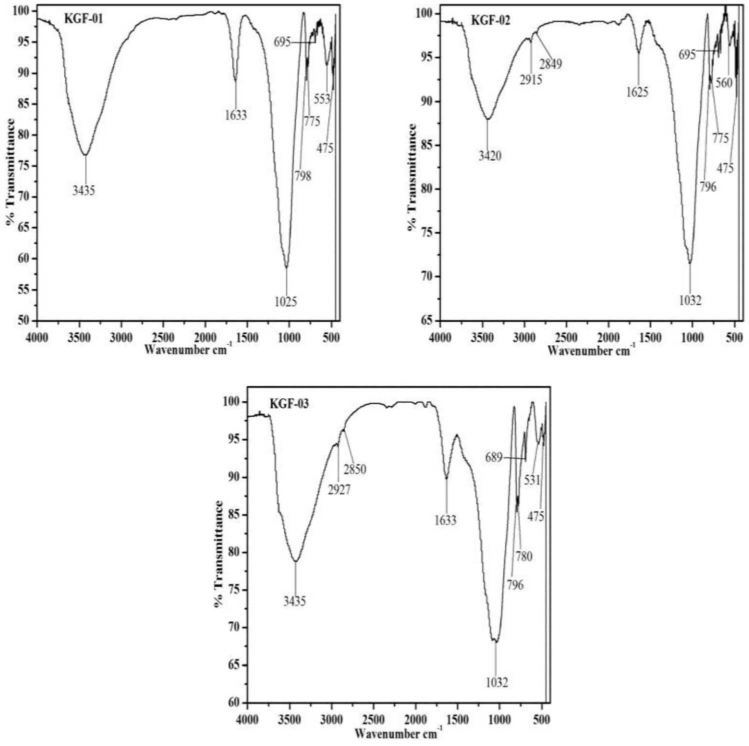

Figure 3.28 FT-IR graphs of KGF-01, KGF-02 and KGF-0

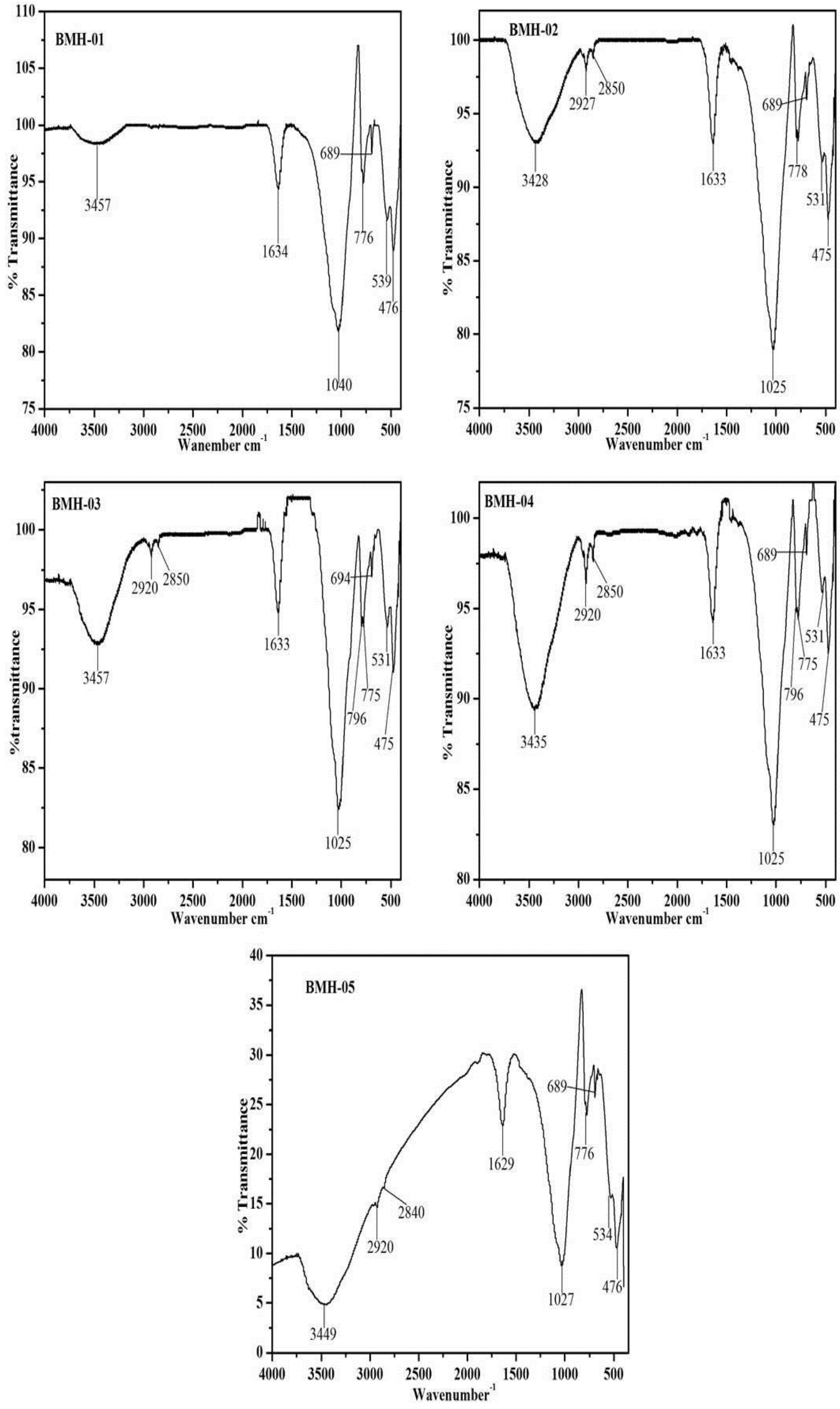

Figure 3.29 FT-IR graphs of BMH-01, BMH-02, BMH-03, BMH-04 and BMH-05

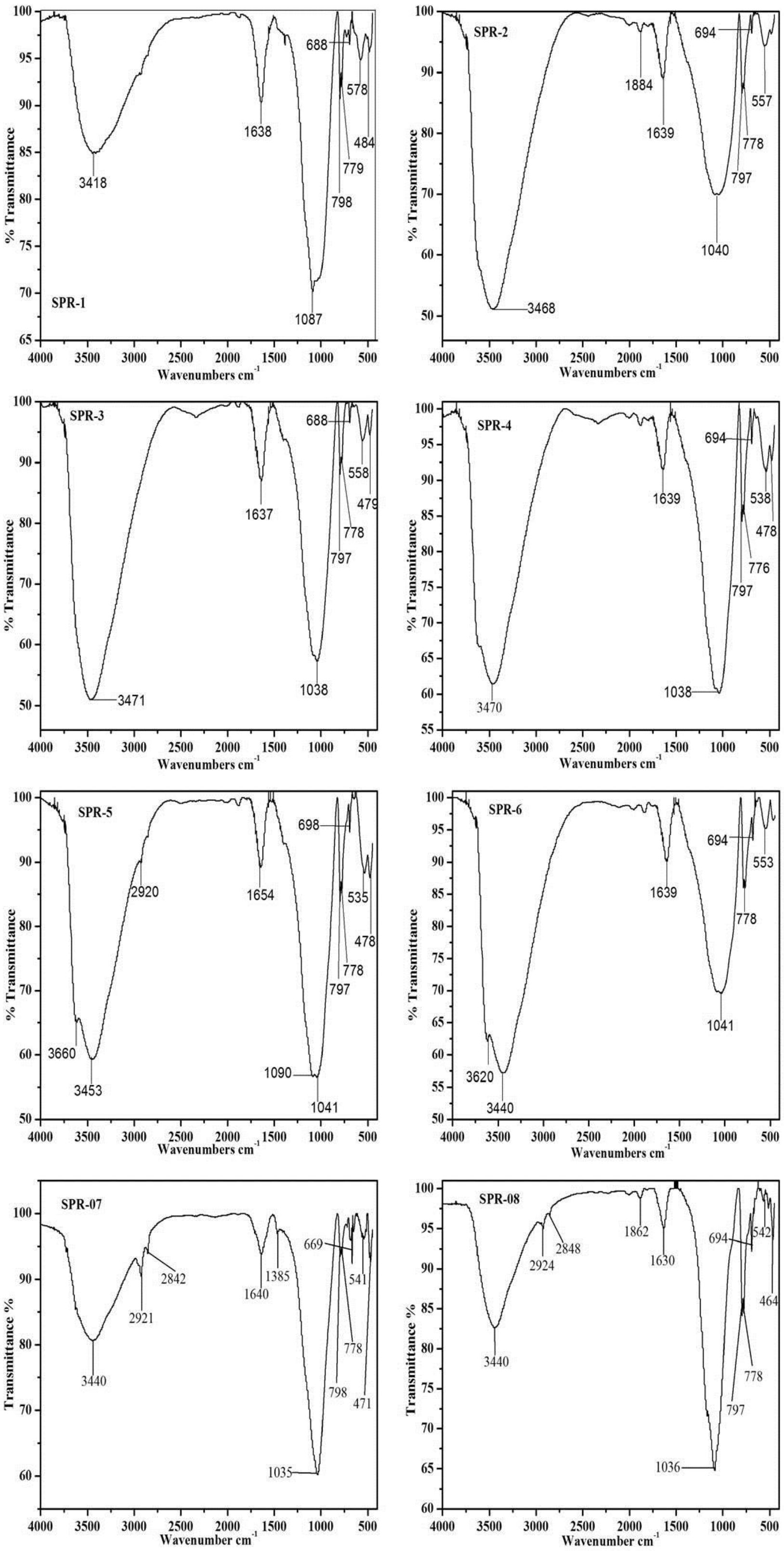

Figure 3.30 FT-IR graphs of SPR-01, SPR-02, SPR-03, SPR-04, SPR-05, SPR-06, Spry-07 and SPR-08

Table 3.3 The IR spectra and probable assignments of AMB-01, AMB-02, AMB-03, AMB-04 and AMB-05 were recorded in ARS (As Received State)

Peak position (cm^{-1})	**As received State**	**Tentative Vibrational Assignments**
AMB-01		
3445	Medium	O-H stretching of absorbed water molecule
2915, 2840	Sh. Very Weak	C–H stretching
1860	Very Weak	Carbonate overtone/combinations
1635	Very Weak	H-O-H bending of water
1080	Very Strong	Si-O-Si (Kaolinite)
798, 778	Medium	Si-O of Quartz
693	Very Weak	Si-O of Quartz
561	Very Weak	Fe_2O_3
468	Very Weak	Microcline
AMB-02		
3448	Medium	O-H stretching of absorbed water molecule
2920, 2840	Sh. Very Weak	C–H stretching
1638	Very Weak	H-O-H bending of water
1031	Very Strong	Si-O-Si (Kaolinite)
797, 778, 664	Very Weak	Si-O of Quartz
554, 476	Very Weak	Fe_2O_3
AMB-03		
3455	Very Strong	O-H stretching of absorbed water molecule
1862	Very Weak	Carbonate overtone/combinations
1638	Very Weak	H-O-H bending of water
1038	Very Strong	Si-O-Si (Kaolinite)
798, 778	Medium	Si-O of Quartz
694	Very Weak	Si-O of Quartz
535	Very Weak	Fe_2O_3
482	Medium	Microcline
AMB-04		
3703	Very Weak	O-H stretching of inter layer water
3622	Very Weak	Inner O-H group of absorbed water
3447	Strong	O-H stretching of absorbed water molecule
2928	Very Weak	C–H stretching
1638	Very Weak	H-O-H bending of water
1036	Very Strong	Si-O-Si (Kaolinite)
914	Very Weak	Al-OH
778, 694	Very Weak	Si-O of Quartz
539	Weak	Fe_2O_3
469	Very Strong	Microcline
AMB-05		
3450	Strong	O-H stretching of absorbed water molecule
1630	Weak	H-O-H bending of water
1020	Very Strong	Si-O-Si (Kaolinite)
797, 778	Weak	Si-O of Quartz
693	Very Weak	Si-O of Quartz
555, 478	Very Weak	Fe_2O_3

Table 3.4 The IR spectra and probable assignments of AMB-06, AMB-07, AMB-08, AMB-09, AMB-10 and AMB-11 were recorded in ARS (As Received State)

AMB-06		
Peak position (cm^{-1})	**As received State**	**Tentative Vibrational Assignments**
3440	Strong	O-H stretching of absorbed water molecule
2915	Very Weak	C–H stretching
1630	Very Weak	H-O-H bending of water
1080	Very Strong	Si-O-Si (Kaolinite)
798	Very Weak	Si-O of Quartz
778	Weak	Si-O of Quartz
694	Very Weak	Si-O of Quartz
565	Very Weak	Fe_2O_3
AMB-07		
3437	Medium	O-H stretching of absorbed water molecule
1635	Weak	H-O-H bending of water
1037	Very Strong	Si-O-Si (Kaolinite)
797, 778	Very Weak	Si-O of Quartz
697	Very Weak	Si-O of Quartz
565	Very Weak	Fe_2O_3
AMB-08		
3451	Medium	O-H stretching of absorbed water molecule
1887	Very Weak	Carbonate overtone/combinations
1633	Very Weak	H-O-H bending of water
1086	Very Strong	Si-O-Si (Kaolinite)
797, 778	Very Weak	Si-O of Quartz
694	Very Weak	Si-O of Quartz
567	Very Weak	Fe_3O_4 or FeO of Magnetite
AMB-09		
3454	Medium	O-H stretching of absorbed water molecule
2925, 2620	Very Weak	C–H Stretching
1652	Very Weak	H-O-H bending of water
1086	Very Strong	Si-O-Si (Kaolinite)
798, 778	Very Weak	Si-O of Quartz
693	Very Weak	Si-O of Quartz
AMB-10		
3438	Very Strong	O-H stretching of absorbed water molecule
1873	Very Weak	Carbonate overtone/combinations
1652	Very Weak	H-O-H bending of water
1085	Very Strong	Si-O-Si (Kaolinite)
797, 774	Very Weak	Si-O of Quartz
693	Very Weak	Si-O of Quartz
560	Very Weak	Fe_2O_3
AMB-11		
3616	Very Weak	Inner O-H group of absorbed water
3449	Medium	O-H stretching of absorbed water molecule
1637	Very Weak	H-O-H bending of water
1033	Very Strong	Si-O-Si (Kaolinite)
797, 780	Very Weak	Si-O of Quartz
693	Very Weak	Si-O of Quartz
544	Very Weak	Fe_2O_3

Table 3.5 The IR spectra and probable assignments of AMB-12, AMB-13, AMB-14, AMB-15, AMB-16 and AMB-17 were recorded in ARS (As Received State)

AMB-12		
Peak position (cm^{-1})	**As received State**	**Tentative Vibrational Assignments**
3437	Strong	O-H stretching of absorbed water molecule
1642	Very Weak	H-O-H bending of water
1080	Very Strong	Si-O-Si (Kaolinite)
797, 782, 689	Very Weak	Si-O of Quartz
563	Very Weak	Fe_2O_3
AMB-13		
3436	Strong	O-H stretching of absorbed water molecule
1652	Very Weak	H-O-H bending of water
1035	Very Strong	Si-O-Si (Kaolinite)
796, 777, 693	Very Weak	Si-O of Quartz
552	Very Weak	Fe_2O_3
AMB-14		
3439	Strong	O-H stretching of absorbed water molecule
1641	Very Weak	H-O-H bending of water
1029	Very Strong	Si-O-Si (Kaolinite)
797, 774	Weak	Si-O of Quartz
693	Very Weak	Si-O of Quartz
546	Very Weak	Fe_2O_3
AMB-15		
3435	Strong	O-H stretching of absorbed water molecule
2927	Very Weak	C–H Stretching
1653	Very weak	H-O-H bending of water
1085	Very Strong	Si-O-Si (Kaolinite)
793, 793, 693	Very Weak	Si-O of Quartz
AMB-16		
3420	Medium	O-H stretching of absorbed water molecule
1640	Medium	H-O-H bending of water
1040	Very Strong	Si-O-Si (Kaolinite)
793	Medium	Si-O of Quartz
685	Very Weak	Si-O of Quartz
566	Very Weak	Fe_2O_3
AMB-17		
3480	Very Strong	O-H stretching of absorbed water molecule
3030, 2918, 2694, 2276,	Very Weak	C–H Stretching
1885	Very Weak	Carbonate overtone/combinations
1641	Medium	H-O-H bending of water
1090	Very Strong	Si-O-Si (Kaolinite)
798	Medium	Si-O of Quartz
694	Very Weak	Si-O of Quartz
574	Very Weak	Fe_3O_4
481	Very Weak	Microcline

Table 3.6 The IR spectra and probable assignments of AMB-18, AMB-19, AMB-22, AMB-21, and AMB-22 were recorded in ARS (As Received State)

AMB-18		
Peak position (cm^{-1})	**As received State**	**Tentative Vibrational Assignments**
3460	Very Strong	O-H stretching of absorbed water molecule
2780	Very Weak	C–H Stretching
1887	Very Weak	Carbonate overtone/combinations
1634	Weak	H-O-H bending of water
1050	Very Strong	Si-O-Si (Kaolinite)
794	Medium	Si-O of Quartz
690	Very Weak	Si-O of Quartz
553	Very Weak	Fe_2O_3
AMB-19		
3442	Very Strong	O-H stretching of absorbed water molecule
1889, 1814	Very Weak	Carbonate overtone/combinations
1653	Very Weak	H-O-H bending of water
1015	Very Strong	Si-O-Si (Kaolinite)
797	Strong	Si-O of Quartz
692	Very Weak	Si-O of Quartz
561	Very Weak	Fe_2O_3
AMB-20		
3428	Very Strong	O-H stretching of absorbed water molecule
2767	Very Weak	C–H Stretching
1890	Very Weak	Carbonate overtone/combinations
1647	Medium	H-O-H bending of water
1050	Strong	Si-O-Si (Kaolinite)
778	Very Weak	Si-O of Quartz
AMB-21		
3440	Strong	O-H stretching of absorbed water molecule
1890	Very Weak	Carbonate overtone/combinations
1640	Very Weak	H-O-H bending of water
1030	Very Strong	Si-O-Si (Kaolinite)
797	Very Weak	Si-O of Quartz
692, 668	Very Weak	Si-O of Quartz
561	Very Weak	Fe_2O_3
481	Very Weak	Microcline
AMB-22		
3445	Medium	O-H stretching of absorbed water molecule
2925, 2851	Very Weak	C–H Stretching
1888	Very Weak	Carbonate overtone/combinations
1620	Weak	H-O-H bending of water
1025	Very Strong	Si-O-Si (Kaolinite)
779	Strong	Si-O of Quartz
696	Very Weak	Si-O of Quartz
540	Very Weak	Fe_2O_3

Table 3.7 The IR spectra and probable assignments of AMB-23, AMB-24, AMB-25, AMB-26, AMB-27 and 28 were recorded in ARS (As Received State)

AMB-23		
Peak position (cm^{-1})	**As received State**	**Tentative Vibrational Assignments**
3420	Very Strong	O-H stretching of absorbed water molecule
1880	Very Weak	Carbonate overtone/combinations
1640	Very Weak	H-O-H bending of water
1140	Very Strong	Si-O-Si (Kaolinite)
797, 694	Very Weak	Si-O of Quartz
581	Very Weak	Fe_3O_4
AMB-24		
3440	Very Strong	O-H stretching of absorbed water molecule
2920, 2850, 2667	Very Weak	C–H stretching
1887	Very Weak	Carbonate overtone/combinations
1632	Weak	H-O-H bending of water
1030	Very Strong	Si-O-Si (Kaolinite)
797, 773	Strong	Si-O of Quartz
691	Very Weak	Si-O of Quartz
557, 477	Very Weak	Fe_2O_3
AMB-25		
3439	Very Strong	O-H stretching of absorbed water molecule
2920, 2850	Sh. Very Weak	C–H stretching
1890	Very Weak	Carbonate overtone/combinations
1638	1090	H-O-H bending of water
1090	Strong	Si-O-Si (Kaolinite)
798, 778, 694	Very Weak	Si-O of Quartz
540, 475	Very Weak	Fe_2O_3
AMB-26		
3456	Strong	O-H stretching of absorbed water molecule
1635	Very weak	H-O-H bending of water
1086	Very Strong	Si-O-Si (Kaolinite)
797, 776, 695	Very Weak	Si-O of Quartz
470	Very Weak	Microcline
AMB-27		
3451	Very Strong	O-H stretching of absorbed water molecule
1641	Very Weak	H-O-H bending of water
1047	Very Strong	Si-O-Si (Kaolinite)
796, 775	Very Weak	Si-O of Quartz
556	Very Weak	Fe_2O_3
482	Very Weak	Microcline
AMB-28		
3439	Strong	O-H stretching of absorbed water molecule
2920, 2850	Sh. Very Weak	C-H stretching
1870	Very Weak	Carbonate overtone/combinations
1640	Very Weak	H-O-H bending of water
1082	Very Strong	Si-O-Si (Kaolinite)
792, 778	Weak	Si-O of Quartz
694	Very Weak	Si-O of Quartz
565, 477	Very Weak	Fe_2O_3

Table 3.8 The IR spectra and probable assignments of AMB-29, AMB-30, AMB-31 AMB-32 and AMB-33 were recorded in ARS (As Received State)

AMB-29		
Peak position (cm^{-1})	**As received State**	**Tentative Vibrational Assignments**
3438	Very Strong	O-H stretching of absorbed water molecule
1882	Very Weak	Carbonate overtone/combinations
1638	Weak	H-O-H bending of water
1097	Very Strong	Si-O-Si (Kaolinite)
798	Weak	Si-O of Quartz
694	Very Weak	Si-O of Quartz
563	Very Weak	Fe_2O_3
AMB-30		
3483	Very Strong	O-H stretching of absorbed water molecule
2910, 2808	Very Weak	C-H stretching
1636	Weak	H-O-H bending of water
1083	Very Strong	Si-O-Si (Kaolinite)
1038	Very Strong	Si-O-Si (Kaolinite)
797, 778	Weak	Si-O of Quartz
664	Very Weak	Si-O of Quartz
560	Very Weak	Fe_2O_3
482	Very Weak	Microcline
AMB-31		
3438	Strong	O-H stretching of absorbed water molecule
2383	Very Weak	C-H stretching
1640	Very Weak	H-O-H bending of water
1030	Very Strong	Si-O-Si (Kaolinite)
797, 778	Weak	Si-O of Quartz
694	Very Weak	Si-O of Quartz
AMB-32		
3430	Strong	O-H stretching of absorbed water molecule
2327	Very Weak	C-H stretching
1650	Very Weak	H-O-H bending of water
1030	Very Strong	Si-O-Si (Kaolinite)
797, 778	Weak	Si-O of Quartz
694	Very Weak	Si-O of Quartz
AMB-33		
3438	Very Strong	O-H stretching of absorbed water molecule
2220	Very Weak	C-H stretching
1652	Weak	H-O-H bending of water
1041	Very Strong	Si-O-Si (Kaolinite)
797, 775	Weak	Si-O of Quartz
694	Very Weak	Si-O of Quartz

Table 3.9 The IR spectra and probable assignments of SPR-01, SPR-02, SPR-03, SPR-04 and SPR-05 were recorded in ARS (As Received State)

SPR-01		
Peak position (cm^{-1})	**As received State**	**Tentative Vibrational Assignments**
3418	Medium	O-H stretching of absorbed water molecule
1638	Weak	H-O-H bending of water
1087	Very Strong	Si-O-Si (Kaolinite)
798, 779	Weak	Si-O of Quartz
688	Very Weak	Si-O of Quartz
578	Very Weak	Fe-O of Fe_3O_4
484	Very Weak	Microcline
SPR-02		
3468	Very Strong	O-H stretching of absorbed water molecule
1884	Very Weak	Carbonate overtone/combinations
1639	Very Weak	H-O-H bending of water
1040	Very Strong	Si-O-Si (Kaolinite)
797, 778, 694	Very Weak	Si-O of Quartz
557	Very Weak	Fe_2O_3
SPR-03		
3471	Very Strong	O-H stretching of absorbed water molecule
1638	Weak	H-O-H bending of water
1038	Very Strong	Si-O-Si (Kaolinite)
797, 778	Weak	Si-O of Quartz
688	Very Weak	Si-O of Quartz
558	Very Weak	Fe_2O_3
479	Medium	Microcline
SPR-04		
3435	Medium	O-H stretching of absorbed water molecule
2920, 2850	Very Weak	C–H Stretching
1633	Weak	H-O-H bending of water
1025	Very Strong	Si-O-Si (Kaolinite)
796, 775	Weak	Si-O of Quartz
689	Very Weak	Si-O of Quartz
531	Very Weak	Fe_2O_3
475	Weak	Fe_2O_3
SPR-05		
3660	Very Weak	Inplane degenerated vibration of water molecule
3453	Very Strong	O-H stretching of absorbed water molecule
1654	Weak	H-O-H bending of water
1090	Very Weak	Si-O-Si (Kaolinite)
1041	Very Strong	Si-O-Si (Kaolinite)
797, 778	Weak	Si-O of Quartz
698	Very Weak	Si-O of Quartz
535	Very Weak	Fe_2O_3
478	Very Weak	Microcline

Table 3.10 The IR spectra and probable assignments of SPR-05, SPR-06, SPR-07 SPR-08, KGF-01, KGF-02 and KGF-03 were recorded in ARS (As Received State)

Peak position (cm^{-1})	**As received State**	**Tentative Vibrational Assignments**
SPR-06		
3620	Very Weak	Inplane degenerated vibration of water molecule
3440	Very Strong	O-H stretching of absorbed water molecule
1639	Very Weak	H-O-H bending of water
1041	Very Strong	Si-O-Si (Kaolinite)
778, 694	Very Weak	Si-O of Quartz
553	Very Weak	Fe_2O_3
SPR-07		
3440	Medium	O-H stretching of absorbed water molecule
2921, 2842	Sh. Very Weak	C–H Stretching
1640	Very Weak	H-O-H bending of water
1385	Very Weak	Carbonate overtone/combinations
1035	Very Strong	Si-O-Si (Kaolinite)
798, 778, 669	Very Weak	Si-O of Quartz
541, 471	Very Weak	Fe_2O_3
SPR-08		
3440	Medium	O-H stretching of absorbed water molecule
2924, 2848	Sh. Very Weak	C–H Stretching
1862	Very Weak	Carbonate overtone/combinations
1630	Very Weak	H-O-H bending of water
1036	Very Strong	Si-O-Si (Kaolinite)
797, 778	Medium	Si-O of Quartz
694	Very Weak	Si-O of Quartz
542	Very Weak	Fe_2O_3
464	Very Weak	Microcline
KGF-01		
3435	Medium	O-H stretching of absorbed water molecule
1633	Very Weak	H-O-H bending of water
1025	Very Strong	Si-O-Si (Kaolinite)
798, 775, 695	Very Weak	Si-O of Quartz
553, 475	Very Weak	Fe_2O_3
KGF-02		
3440	Medium	O-H stretching of absorbed water molecule
2915, 2849	Sh. Very Weak	C–H Stretching
1625	Very Weak	H-O-H bending of water
1032	Very Strong	Si-O-Si (Kaolinite)
796, 775, 695	Very Weak	Si-O of Quartz
560, 475	Very Weak	Fe_2O_3
KGF-03		
3435	Strong	O-H stretching of absorbed water molecule
2927, 2850	Sh. Very Weak	C–H Stretching
1633	Very Weak	H-O-H bending of water
1032	Very Strong	Si-O-Si (Kaolinite)
796, 780, 695	Very Weak	Si-O of Quartz
531, 475	Very Weak	Fe_2O_3

Table 3.11 The IR spectra and probable assignments of BMH-01, BMH-02, BMH-03, BMH-04 and BMH-05 were recorded in ARS (As Received State)

BMH-01		
Peak position (cm^{-1})	**As received State**	**Tentative Vibrational Assignments**
3457	Very Weak	O-H stretching of absorbed water molecule
1634	Weak	H-O-H bending of water
1040	Very Strong	Si-O-Si (Kaolinite)
776, 689	Very Weak	Si-O of Quartz
539, 476	Very Weak	Fe-O of Fe_2O_3
BMH-02		
3428	Medium	O-H stretching of absorbed water molecule
2927, 2850	Very Weak	C–H Stretching
1633	Medium	H-O-H bending of water
1025	Very Strong	Si-O-Si (Kaolinite)
778, 689	Very Weak	Si-O of Quartz
531, 475	Very Weak	Fe_2O_3
BMH-03		
3457	Medium	O-H stretching of absorbed water molecule
2920, 2850	Very Weak	C–H Stretching
1638	Medium	H-O-H bending of water
1025	Very Strong	Si-O-Si (Kaolinite)
796, 775	Medium	Si-O of Quartz
694	Very Weak	Si-O of Quartz
531, 475	Very Weak	Fe_2O_3
BMH-04		
3435	Medium	O-H stretching of absorbed water molecule
2920, 2850	Very Weak	C–H Stretching
1633	Weak	H-O-H bending of water
1025	Very Strong	Si-O-Si (Kaolinite)
796, 775	Weak	Si-O of Quartz
689	Very Weak	Si-O of Quartz
531	Very Weak	Fe_2O_3
475	Weak	Fe_2O_3
BMH-05		
3453	Very Strong	O-H stretching of absorbed water molecule
2920, 2840	Very Weak	C–H Stretching
1629	Very Weak	H-O-H bending of water
1027	Very Strong	Si-O-Si (Kaolinite)
776, 689	Very Weak	Si-O of Quartz
534, 476	Very Weak	Fe_2O_3

3.5 Thermo-gravimetric analysis

A selection of three pottery from Ambari, Assam has been used for the TG-DSC analysis and objective is to investigate firing techniques of these potsherds. The TG–DSC curves are respectively shown in Fig. 3.31 (I, II, III) for the samples AMB-01, AMB-02, and AMB-03. DSC curves of all pottery show different endothermic and exothermic peaks at various temperatures. Sharp endothermic/exothermic peaks in the range of 450–650 °C in AMB-01, AMB-02 and AMB-03 is due to the combustion of the organic constituent and it is indicating that all pottery have organic material in it. So observations clearly suggest the presence of organic materials which have been incorporated during the preparation of clay. It is evident that the peak at 450–650 °C is due to the dehydroxylation of Kaolinite which indicates that this characteristic endothermic peak survived around firing range between 450 and 650 °C (Chakchouk *et al.*, 2009). As in Fig. 3.31 (I, II and III) the DSC curves of the samples AMB-01 and AMB-02 shows peaks at 445 °C and 491 °C in AMB-01 and around 482 °C in AMB-02 is due to dehydroxylation of Kaolinite. While in AMB-03 exothermic peak around 575 °C is observed. It is indicating that the pottery samples might have been fired at 650–800 °C (Leach *et al.*, 2008).

According to Drebushchak *et al.* (2005) there are three steps of the weight loss of the pottery in thermo-gravimetric analysis:

1. The dehydration (room temperature to 100 °C)
2. Decomposition of hydroxyls (400–500 °C)
3. Decomposition of carbonates, mainly Calcite (700–800 °C).

From thermo-gravimetric curves, it is noted that the mass loss of the ancient pottery by increasing the temperature from room to 900 °C is not gradual but the variation is changed in different temperature regions as shown in table 3.12. The TG curves presented the gradual weight loss in all three samples due to the dehydration of water molecules in the temperature range 30–300 °C. Further, the curve exemplifies increases in weight loss from 400 to 650 °C in all three samples representing the decomposition of hydroxyls. These weight losses are due to the presence of organic molecule in all three potsherds. The oxidation of the organic compound begins at 250 °C temperature when carbon in the clay oxidizes as CO and CO2. Above 600–700 °C organic compound totally eliminate. Besides water and organics, weight loss occurs due to the presence of Calcite, dolomite, marcasite, pyrite or gypsum which is decomposed in the temperature range of 700–900 °C.

All analysed sherds showed less mass loss at dehydration than at dehydroxylation. The sherds AMB-01, AMB-02, and AMB-03 (Table 2.12) showed weight loss due to water 1.69%, 5.34% and 4.75% respectively at 200 °C. AMB-01, AMB-02 and AMB-03 showed weight loss due to the organic materials 4.91%, 8.71% and 7.00% respectively at 450–650 °C. While none of the samples showed, weight loss is due to decomposition of Calcite. It occurs between 700 and 800 °C indicating Calcite was absent in all the samples (Chakchouk *et al.*, 2009). The TG-DSC analysis in conjunction with FTIR and XRD provide information for the estimation of original firing temperature of the ancient pottery. The TG analysis of Ambari pottery indicates that the firing temperature must be between 650 and 800 °C, which are in complete agreement with the FTIR and XRD results (Singh & Sharma, 2016).

Table 3.12 Weight loss from thermo-gravimetric curves of the samples

Sample	Weight loss at			Total weight loss
	Dehydration (room temperature-200° C)	Decomposition of hydroxyls (400-650° C)	Decomposition of Calcite (700-800° C)	
AMB-01	1.68 %	4.91 %	0	5.04 %
AMB-02	5.33%	8.71 %	0	10.17 %
AMB-03	4.75%	7.00%	0	8.21%

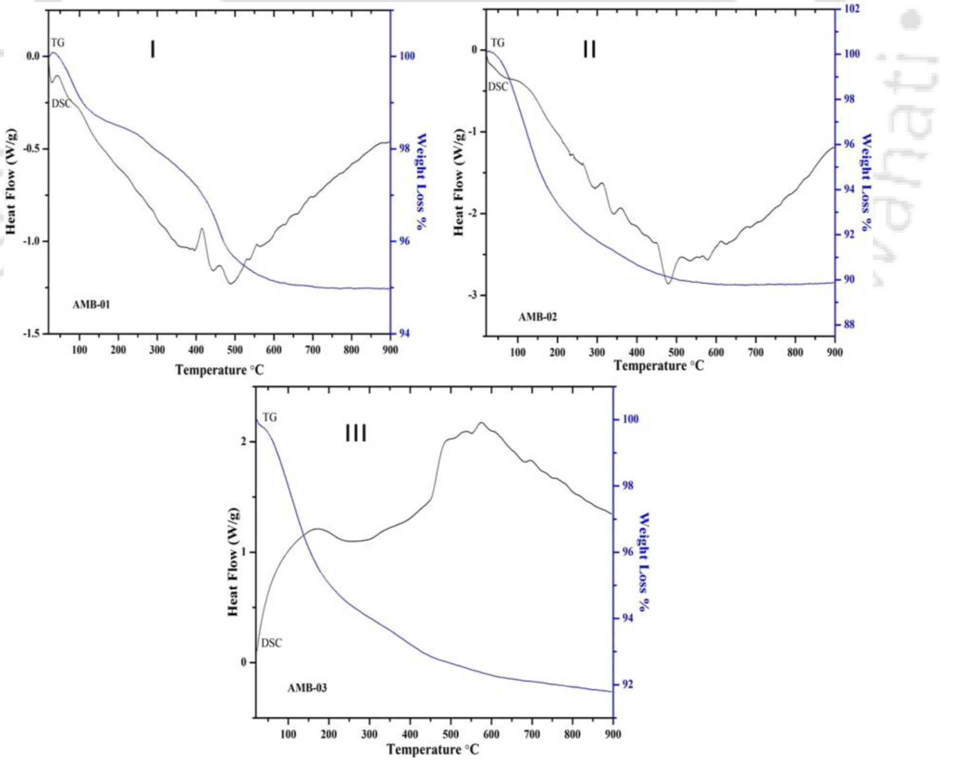

Figure 3.31 TG-DSC graph of pottery samples from Ambari: (I) AMB-01 (II) AMB-02 (III) AMB-03

3.6 Characteristic Study of Historical pottery of by Micro-Raman Spectroscopy

Micro-Raman spectroscopy has been used to identify mineralogical composition and to determine firing temperature range and atmosphere of analysed historical potsherds. Sometimes, due to high fluorescence of potsherds, explainable Raman spectra were not recorded. The Raman spectrum and tentative assignment of historical potsherds are given in tables 3.13-3.15 and figures 3.32-3.36.

The weak to medium distinctive Raman peaks due to symmetric bending vibration (Si-O-Si) of Quartz at 123-130, 263, 462, 464, 466,467 and 470 cm^{-1} are identified in most of historical potsherds. For instance, Raman peaks around 123-131 are observed in AMB-05, 09, 14, 17, 24, 25, 30, 31; SPR-02, 04; BMH-01 and 03 (tables 3.13-3.15). The Raman spectra around 203 to 210 cm^{-1} are observed in analysed historical potsherds AMB-05, 09, 11, 14, 15, 21, 23, 24, 25, 26, 27, 30, 31, SPR-02, 04, 06, BMH-01 and KGF-03. The Raman bands around 460 to 470 cm^{-1} are identified in analysed historical potsherds AMB-01, 05, 06, 09, 11, 14, 17, 24, 25, 30, 31, 33, SPR-02, 04, 06, 08, BMH-01 and 03 (tables 3.13-3.15). Raman Peak at 355 cm^{-1} is recorded in potsherds AMB-30 due to asymmetric bending modes of silica tetrahedral. Raman spectrum at 1155 cm^{-1} is detected in AMB-30 due to Si-O asymmetric stretching mode. On the basis of XRD, thin section and FTIR results, Quartz is most abundant mineral present in composition of analysed historical potsherds (Olivares, *et al.*, p. 1545, 2010; Liou *et al.*, p. 1063, 2011).

Raman spectrum of Ilmenite (Fe_2TiO_3) showed weak band at 228 and 360 cm^{-1}. The presence of ilmenite in SPR-02, 04, 06 and BMH-01 (tables 3.13-3.15) confirmed that raw material was rich in iron and titanium (Olivares, *et al.*, p. 1545, 2010; Liou, *et al.*, p. 1066, 2011). Raman peaks at 228, 285, 410, 612, 1320 cm^{-1} are assigned to Fe_2O_3 (Hematite). Hematite is observed in analysed historical potsherds AMB-05, SPR-02, 08 and BMH-01 (tables 3.13-3.15).

The weak, medium and strong distinctive Raman peaks at 144, 153, 195, 202, 395, 512 and 636 cm^{-1} are assigned to modes of Anatase. The presence of Anatase is observed in analysed potsherds (tables 3.13-3.15) – AMB-01, 03, 05, 08, 12, 14, 15, 21-29, 32, 32, SPR-08 and KGF-01-03. Olivares, *et al.* (2010) and Striova, *et al.*(2006) stated that Anatase is a common constituent of soil minerals and is usually extant as additional component in Kaolin. It is reported by various scholars that Raman Spectra of clay minerals are frequently masked in occurrence of

Anatase. Referring to this, presence of Anatase in analysed samples may be part of raw materials or clay composition.

Raman Spectra of 1280 to 1610 cm^{-1} and 1754 cm^{-1} is detected due to carbon black materials in composition of historical potsherds. Carbon black materials are detected in AMB-01, 05, 06, 09, 14, 15, 28, 32, KGF-02 and 03 (tables 3.13-3.15) (Smith & Clark, p. 1146, 2004). According to Liou *et al*. (p. 1064, 2011), carbon black materials may presents naturally in clay source, or added intentionally as organic materials, for example, plant ash, straw and fiber etc. added by potter during manufacturing, or it may be deposited during use or burial on surface of ceramics. Plagioclase is detected due to Raman spectra at 511, and 519 cm^{-1} in AMB-21, AMB-26, and SPR-08.

Raman Spectroscopy results have provided significant information about mineralogical composition and firing process of analysed historical pottery. Technologically, micro-Raman results reveal that most of analysed potsherds fired below 800-950 °C. This is confirmed by presence of only Anatase in most of analysed potsherds, because Anatase-rutile transition phase occurs at 800-950 °C. The Presence of Hematite in AMB-05, SPR-02, 08 and BMH-01 refers to oxidative atmosphere under which these potsherds would have been produced.

Table 3.13 Raman spectra and tentative assignments of AMB-01, AMB-03, AMB-06, AMB-09, AMB-10, AMB-11, AMB-12, AMB-12, AMB-14 and AMB-15

AMB-01		
Raman Shift (cm^{-1})	**As received State**	**Tentative Vibrational Assignments**
145	Very Weak	Modes of Anatase
470	Very Weak	Due the symmetric bending vibration (Si-O-Si) Quartz
1285, 1368, 1398	Very Weak	Carbon black material
AMB-03		
145	Very Weak	Modes of Anatase
AMB-05		
123,210, 264, 462	Very Weak	Due the symmetric bending vibration (Si-O-Si) Quartz
164,	Very Weak	Modes of Anatase
694,	Very Weak	Si-O of Quartz
865,	Very Weak	Fe_2O_3
1325, 1439	Very Weak	Carbon black material
AMB-06		
462	Very Weak	Due the symmetric bending vibration (Si-O-Si) Quartz
1375, 1405	Very Weak	Carbon black material
AMB-08		
144	Very Weak	Modes of Anatase
AMB-09		
123, 206, 462,	Very Weak	Due the symmetric bending vibration (Si-O-Si) Quartz
1280, 1633	Very Weak	Carbon black material
AMB-10		
1329	Very Weak	Carbon black material
AMB-11		
210, 469	Very Weak	Due the symmetric bending vibration (Si-O-Si) Quartz
AMB-12		
147	Very Weak	Modes of Anatase
240	Very Weak	Rutile
764, 786	Very Weak	Si-O of Quartz
967	Very Weak	Wollastonite
AMB-14		
130, 203, 263, 462,	Very Weak	Due the symmetric bending vibration (Si-O-Si) Quartz
147, 397, 515	Very Weak	Modes of Anatase
1462	Very Weak	Carbon black material
AMB-15		
144,	Very Weak	Modes of Anatase
205	Very Weak	Due the symmetric bending vibration (Si-O-Si) Quartz
727	Very Weak	Si-O of Quartz
1754	Very Weak	Carbon black material

Table 3.14 Raman spectra and tentative assignments of AMB-17, AMB-21, AMB-22, AMB-23, AMB-24, AMB-25, AMB-26, AMB-27, AMB-28, AMB-29, AMB-30, AMB-31, AMB-32 and AMB-33

AMB-17		
Raman Shift (cm^{-1})	**As received State**	**Tentative Vibrational Assignments**
128, 466	Very Weak	Due the symmetric bending vibration (Si-O-Si) Quartz
AMB-21		
153, 398, 632	Very Weak	Modes of Anatase
205	Very Weak	Due the symmetric bending vibration (Si-O-Si) Quartz
511	Very Weak	Plagioclase
AMB-22		
153	Very Weak	Modes of Anatase
201	Very Weak	Due the symmetric bending vibration (Si-O-Si) Quartz
AMB-23		
147, 395, 631	Very Weak	Modes of Anatase
205	Very Weak	Due the symmetric bending vibration (Si-O-Si) Quartz
AMB-24		
131, 208, 470	Very Weak	Due the symmetric bending vibration (Si-O-Si) Quartz
AMB-25		
131, 208, 266, 466	Very Weak	Due the symmetric bending vibration (Si-O-Si) Quartz
150	Very Weak	Modes of Anatase
AMB-26		
153, 398, 632	Very Weak	Modes of Anatase
205	Very Weak	Due the symmetric bending vibration (Si-O-Si) Quartz
511	Very Weak	Plagioclase
AMB-27		
153	Very Weak	Modes of Anatase
201	Very Weak	Due the symmetric bending vibration (Si-O-Si) Quartz
AMB-28		
149	Very Weak	Modes of Anatase
1379, 1602	Very Weak	Carbon black material
AMB-29		
149	Very Weak	Modes of Anatase
1379, 1605	Very Weak	Carbon black material
AMB-30		
127, 206, 263, 466,	Very Weak	Due the symmetric bending vibration (Si-O-Si) Quartz
355	Very Weak	Due to the asymmetric bending modes of the silica tetrahedra
1155	Very Weak	Si-O asymmetric stretching mode
AMB-31		
123, 206, 466	Very Weak	Due the symmetric bending vibration (Si-O-Si) Quartz
AMB-32		
149	Very Weak	Modes of Anatase
1599	Very Weak	Carbon black material
AMB-33		
141,	Very Weak	Modes of Anatase
466	Very Weak	Due the symmetric bending vibration (Si-O-Si) Quartz

Table 3.15 Raman spectra and tentative assignments of SPR-02, SPR-03, SPR-04, SPR-06, SPR-08, BMH-01, BMH-03, KGF-01, KGF-02 and KGF-03

Raman Shift (cm^{-1})	**As received State**	**Tentative Vibrational Assignments**
	SPR-02	
130, 228, 470	Very Weak	Due the symmetric bending vibration (Si-O-Si) Quartz
228		Ilmenite
413		Hematite
	SPR-03	
1598	Very Weak	Carbon black material
	SPR-04	
130, 205, 265, 467	Very Weak	Due the symmetric bending vibration (Si-O-Si) Quartz
360	Very Weak	Ilmenite
	SPR-06	
130, 205, 265, 466	Very Weak	Due the symmetric bending vibration (Si-O-Si) Quartz
360	Very Weak	Ilmenite
	SPR-08	
149, 643	Very Weak	Modes of Anatase
402		Hematite
467		Due the symmetric bending vibration (Si-O-Si) Quartz
519		Plagioclase
	BMH-01	
126, 202, 465	Very Weak	Due the symmetric bending vibration (Si-O-Si) Quartz
285	Very Weak	Hematite
360	Very Weak	Ilmenite
	BMH-03	
130, 470	Very Weak	Due the symmetric bending vibration (Si-O-Si) Quartz
	KGF-01	
152	Very Weak	Modes of Anatase
	KGF-02	
149	Very Weak	Modes of Anatase
1375, 1605	Very Weak	Carbon black material
	KGF-03	
153	Very Weak	Modes of Anatase
205	Very Weak	Due the symmetric bending vibration (Si-O-Si) Quartz
1379, 1605	Very Weak	Carbon black material

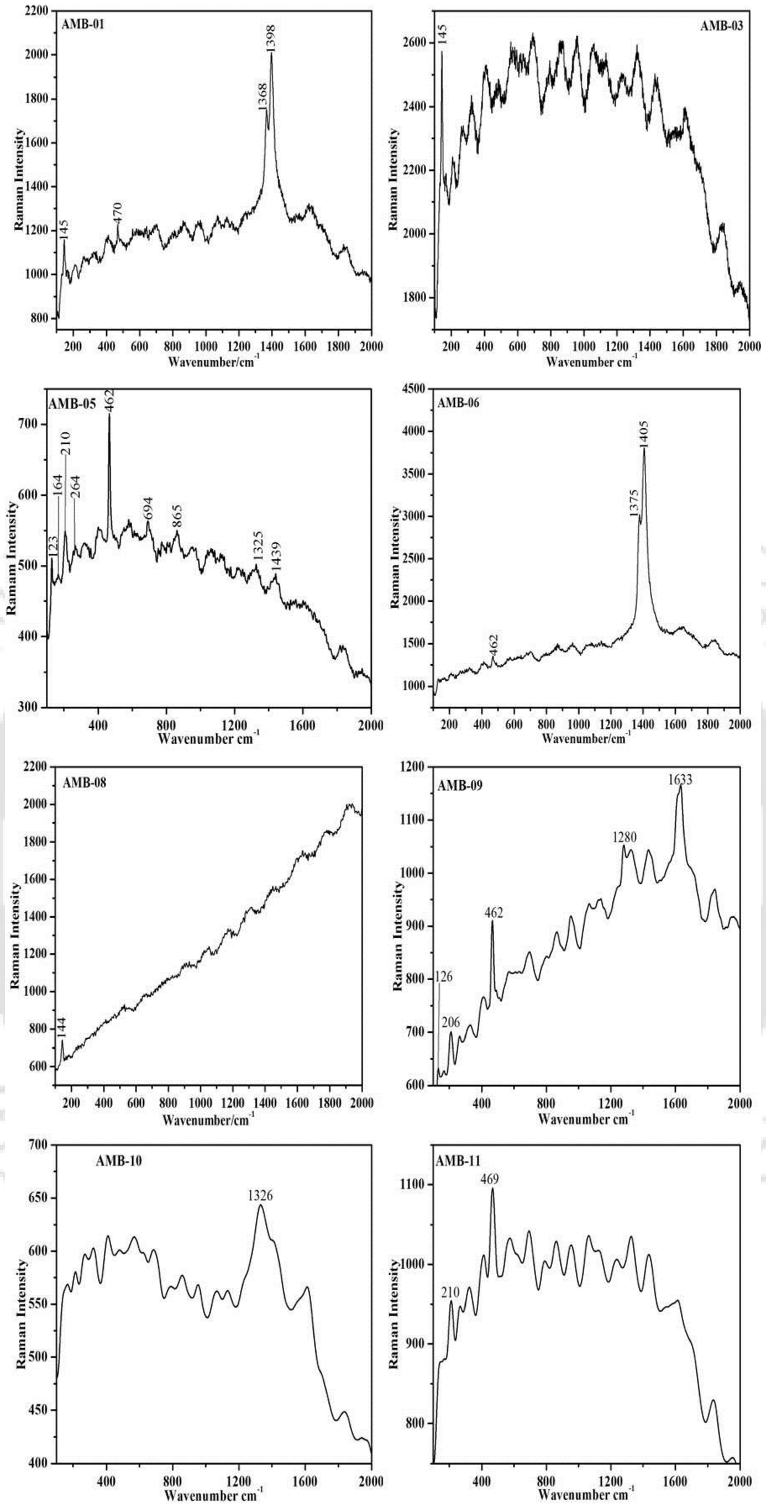

Figure 3.32 Raman Spectra of AMB-01, AMB-03, AMB-05, AMB-06, AMB-08, AMB-09, AMB-10 and AMB-11

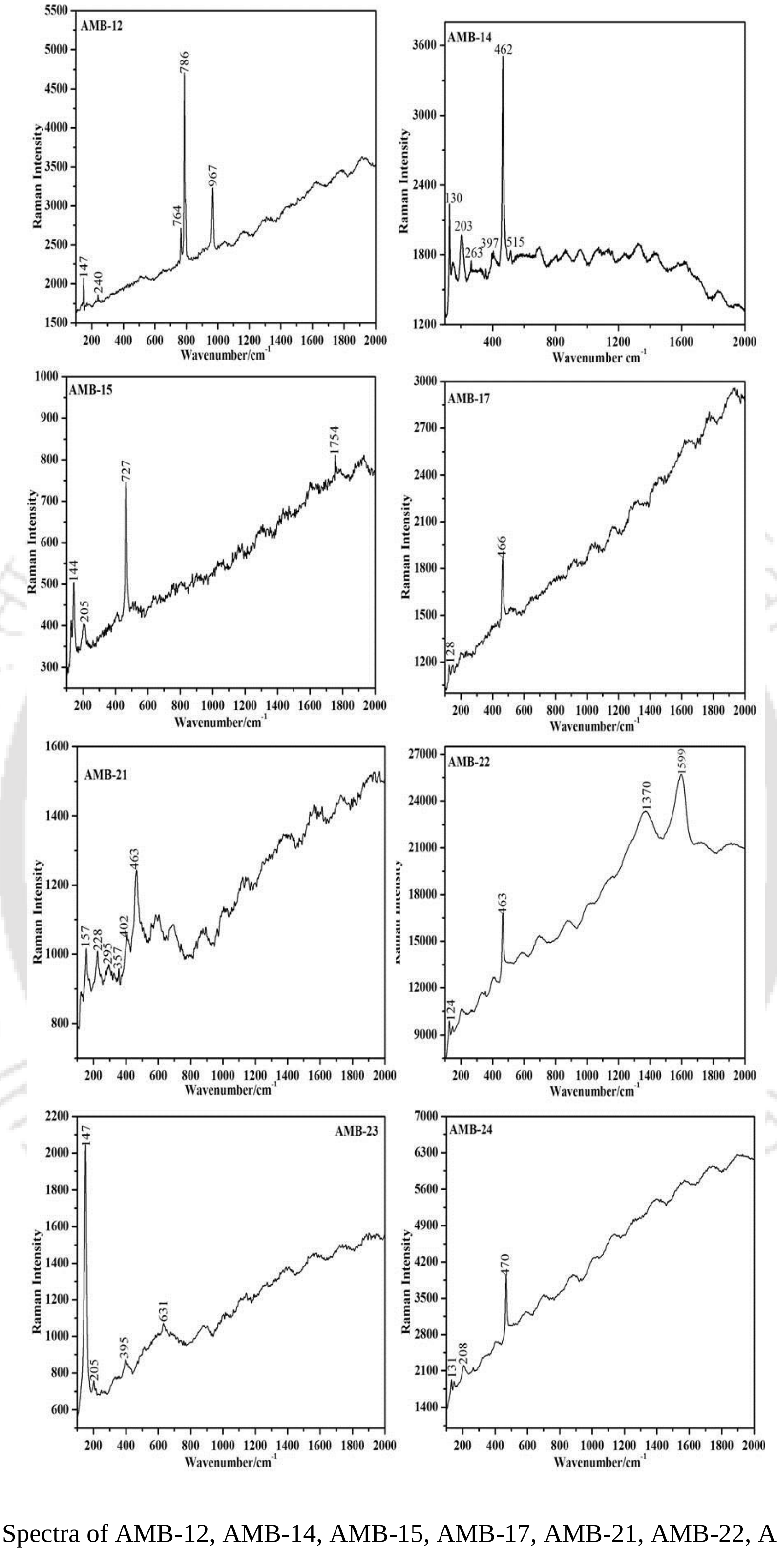

Figure 3.33 Raman Spectra of AMB-12, AMB-14, AMB-15, AMB-17, AMB-21, AMB-22, AMB-23 and AMB-24

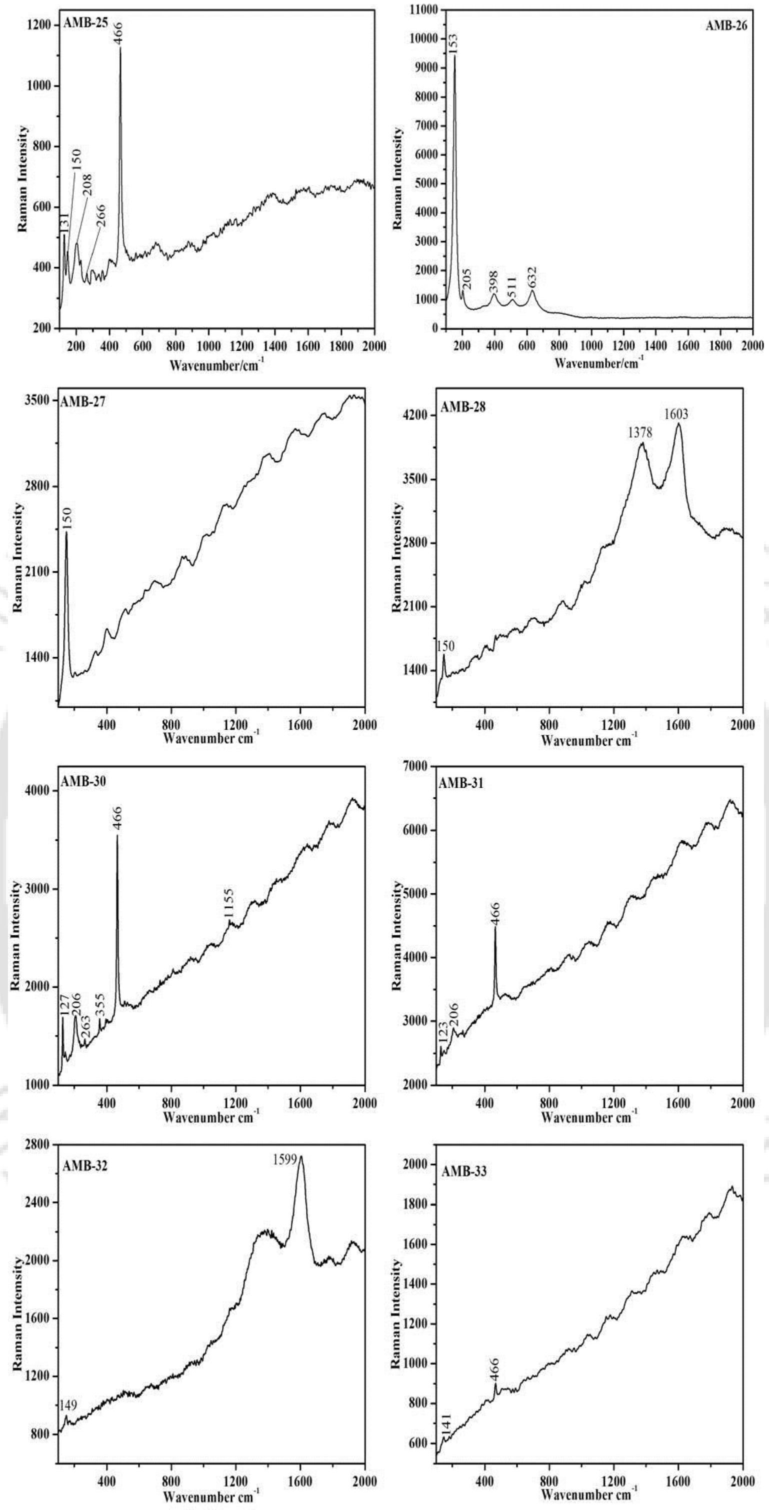

Figure 3.34 Raman Spectra of AMB-25, AMB-26, AMB-27, AMB-28, AMB-30, AMB-31, AMB-32 and AMB-33

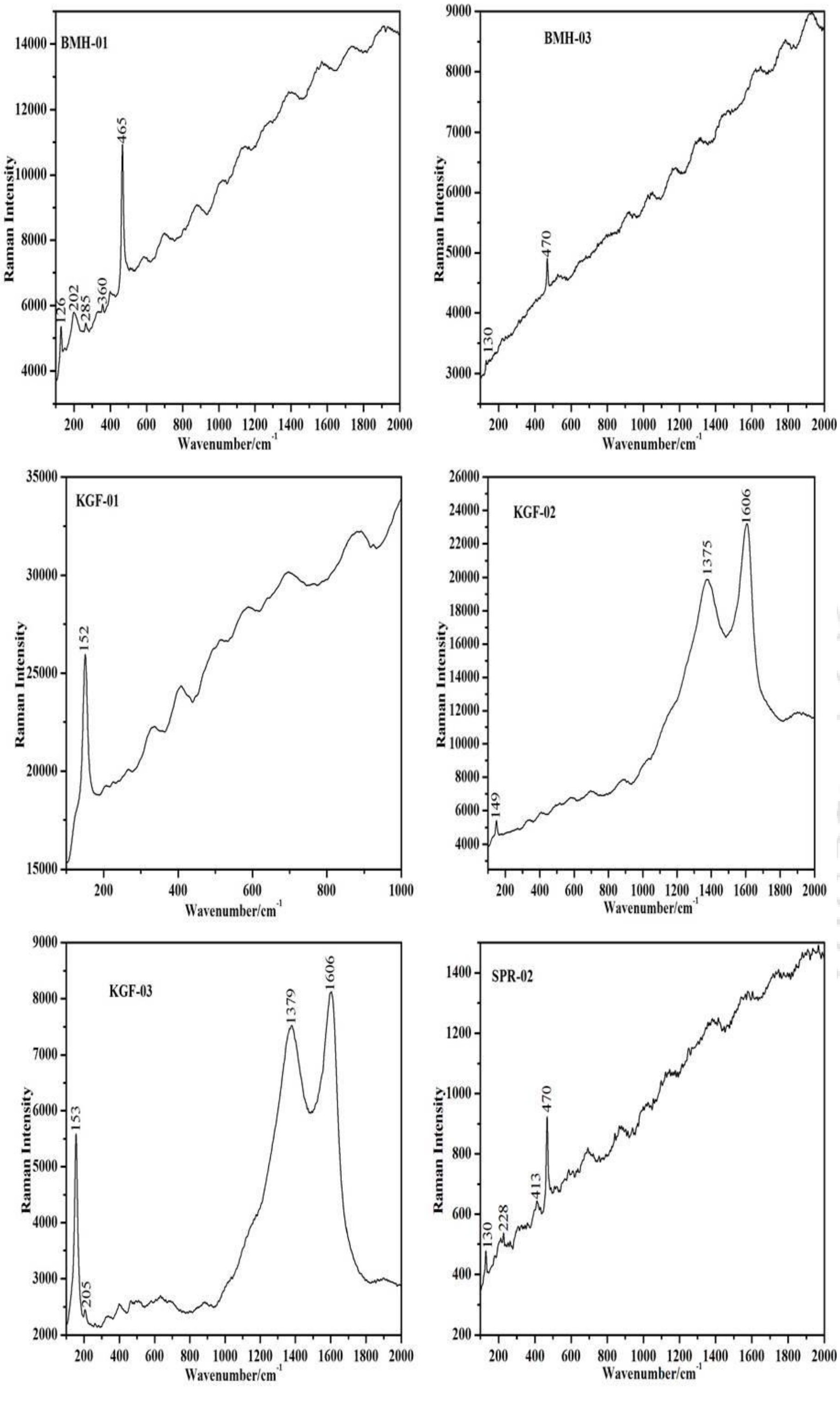

Figure 3.35 Raman Spectra of BMH-01, BMH-03, KGF-01, KGF-02, KGF-03 and SPR-02

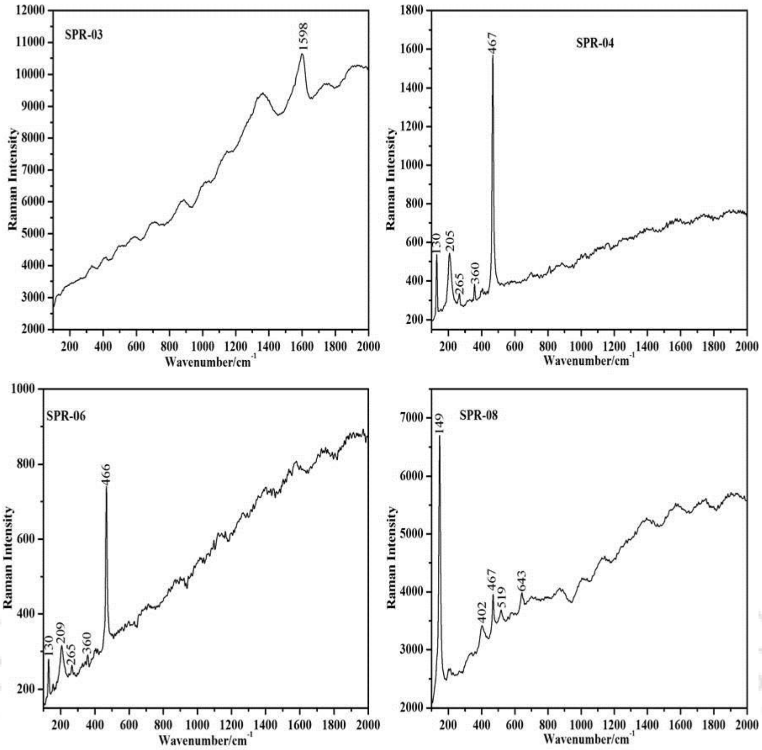

Figure 3.36 Raman Spectra of SPR-03, SPR-04, SPR-06 and SPR-08

3.7 Elemental analysis of Historical pottery by means of EDX and LIBS

3.7.1 Energy-dispersive X-ray analysis

In present study, chemical composition of historical pottery has been investigated using SEM-EDX. EDX results have been summarized in table 3.16 and figures 3.37-3.38. In addition, LIBS has been used on selected historical potsherds.

Elemental composition of potsherds by EDX analysis (table 3.16) indicates SiO_2 and Al_2O_3 as main constituent. SiO_2 detected in range of 37.85-62.85 wt. %. From EDX analysis, it is evident that AMB-13 has highest amount 62.85 wt. percentage of SiO_2 in composition. SPR-01 has lowest amount 37.85 wt. percentage of SiO_2. The presence of silica in such percentages in composition of all analysed potsherds indicates that raw materials were rich in Quartz mineral.

The presence of Al_2O_3 is varies between 23.10-43.96 wt. percentage in composition of all analysed historical potsherds. AMB-15 contains highest amount of 43.96 wt. % and AMB-21 has lowest quantity of 23.10 wt. % in their composition. The presence of higher quantity of SiO_2 and Al_2O_3 reveal occurrence of alumina-silicate rich minerals such as clay minerals and Feldspars (Vecstaudza, *et al.*, 2013).

According to tabulated data of EDX (table 3.16), amount of Fe_2O_3 varies in between 0.71 and 20.54 wt. percentage for historical pottery. From EDX analysis, it is evident that AMB-17 has highest amount 20.00 wt. percentage of iron oxide in their composition. AMB-15 has lowest amount 0.71 wt. percentage of iron oxide in their composition. AMB-17, AMB-21, SPR-01, KGF-03, BMH-01 and BMH-02 is showing comparatively higher amount of iron oxide of 20.54 wt. %, 10.25 wt. %, 16.35 wt. %, 12.84 wt. %, 16.88 wt. %, 10.76 wt. %, 11.43 weight percentages respectively in their composition. As discussed earlier, occurrence of iron oxide in significant quantity in clay is affected by factors such as additional use of iron rich materials. The traditional potters (Hira and Kumar potter communities) of studied area (Assam) are using ochre (Geru) as colouring materials to colour pottery.

The occurrence of Na_2O substances in most of analysed historical potsherds is ranging from 0.38 to 2.35 wt. % respectively. Na_2O is not present in AMB-15. P_2O_5 is present in historical potsherds varying from 0.47 to 3.69 wt. percentages. AMB-02, AMB-15, AMB-17 and BMH-01 are not showing P_2O_5 in their composition. SO_3 is detected only in AMB-02 and SPR-07 with amount of 0.83 and 0.51 wt. percentages respectively. Cl is found only in AMB-02 with amount of 0.67 wt. percentage. The presence of Na_2O, P_2O_5, SO_3 and Cl in very low amount may be adsorbed by all analysed historical potsherds during burial.

Tabulated data (table 3.16) show that presence of CaO in historical pottery is in very low amount, ranging from 0.33 to 1.97 weight %. While, AMB-15 and BMH-01 are not showing CaO content their composition.

The amount of MgO content is approximately similar in all potsherds and it is in a reasonable range from 0.23–3.75 wt. %. KGF-01 has highest content with 3.75 wt. % and SPR-07 has lowest amount with 1.00 wt. % in their composition.

The presence of K_2O materials in all potsherds is ranging from 0.86 to 3.83 wt. %. Ali *et al*. (2011) stated that presence of K_2O indicate that K-Feldspars might have been added deliberately or employed naturally potassium rich micaceous clays. TiO_2 substances ranging from 0.18 to 3.06 wt. % is present in all potsherds.

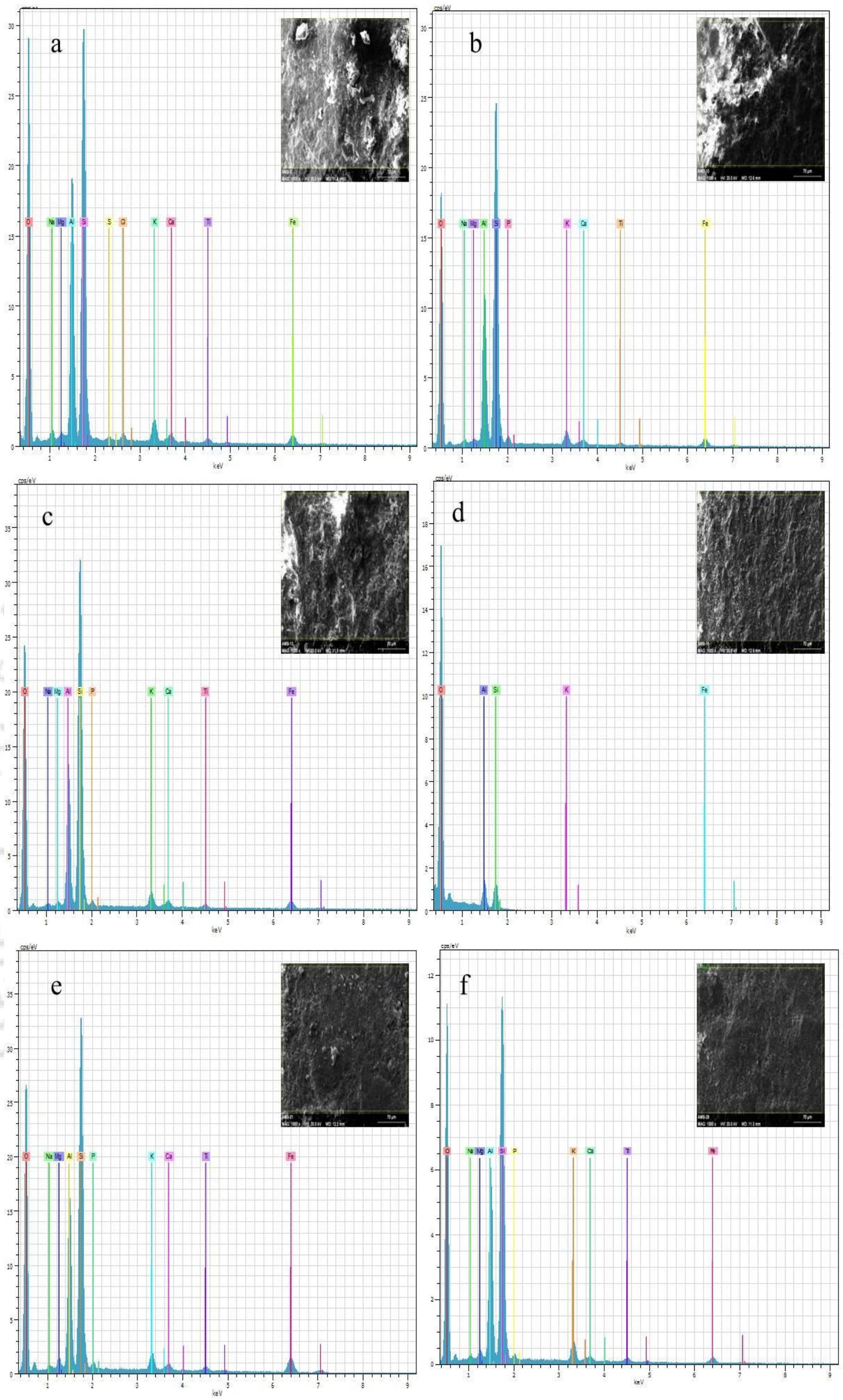

Figure 3.37 EDX spectrum of (a) AMB-02 (b) AMB-10 (c) AMB-13 (D) AMB-15 (e) AMB-21 and (f) AMB-29. Image indicates the spot of EDX analysis

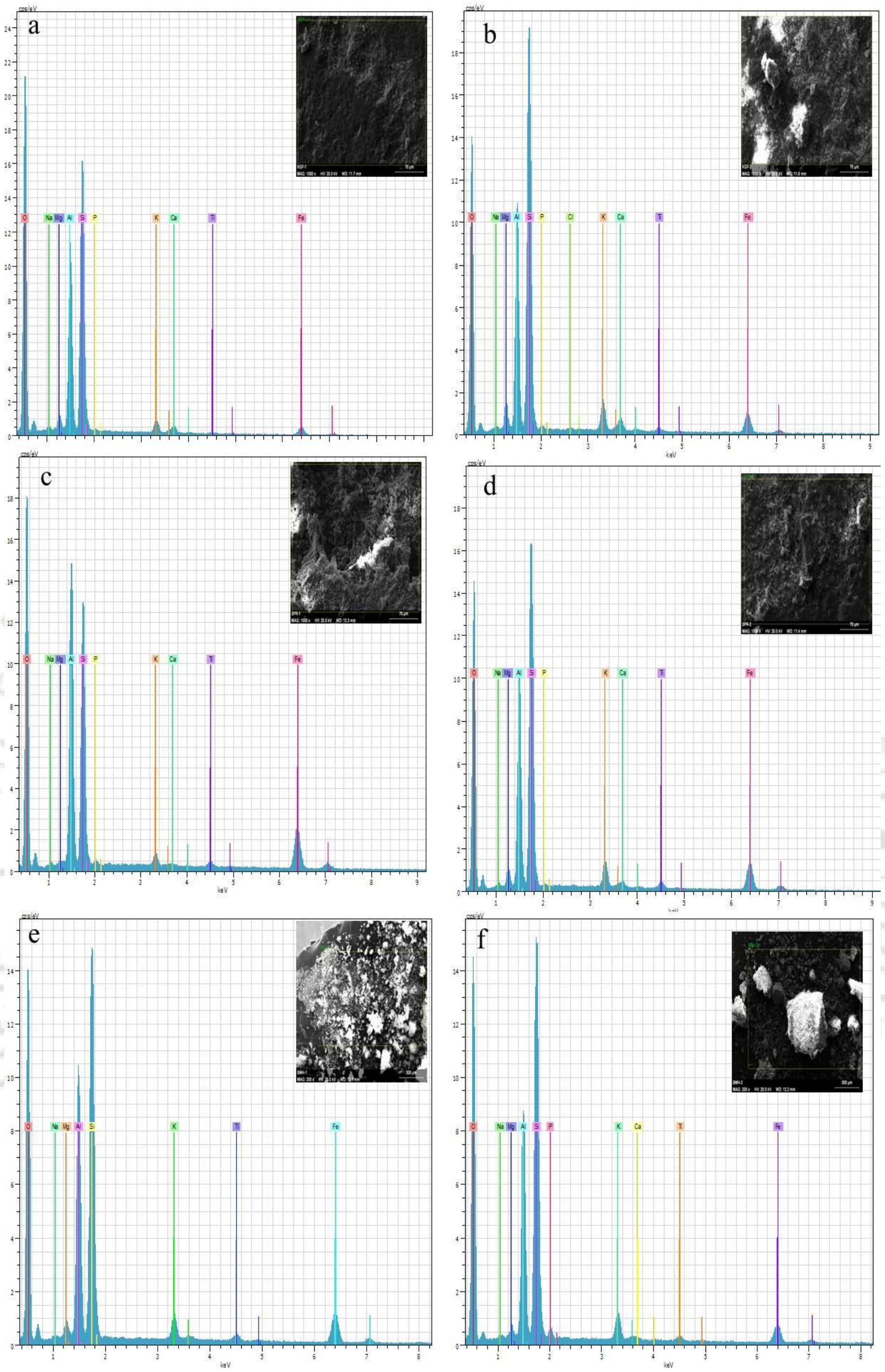

Figure 3.38 EDX spectrum of (a) KGF-01 (b) KGF-02 (c) SPR-01 (D) SPR-02 (e) BMH-01 and (f) BMH-02. Image indicates spot of EDX analysis

3.7.2 Laser Induced Breakdown Spectroscopy

In addition to EDX, LIBS has been used for chemical analysis of five selected historical potsherds. These samples are AMB-01, AMB-10, AMB-31, SPR-03 and KGF-03. LIBS result (table 3.17 and figure 3.39) indicates Fe, Ca, Si, Ti, Mg and Na are present in clay composition of all analysed potsherds.

Table 3.16 EDX results of historical pottery

Si No.	**Sample Name**	**Oxide (Weight %)**											
		Na_2O	**MgO**	**SiO_2**	**P_2O_5**	**Al_2O_3**	**K_2O**	**CaO**	**SO_3**	**TiO_2**	**Fe_2O_3**	**Cl**	**O**
01	AMB-02	1.99	1.00	53.98	0	31.67	2.96	1.32	0.83	1.27	4.31	0.67	17.61
02	AMB-10	0.93	0.61	61.30	2.28	25.02	2.42	1.13	0	1.02	5.30	0	13.32
03	AMB-11	0.87	0.60	60.57	1.93	26.58	2.27	0.87	0	1.13	5.19	0	13.71
04	AMB-13	0.66	0.66	62.75	1.92	23.70	2.80	1.51	0	1.37	4.63	0	13.66
05	AMB-15	0	0	54.47	0	43.96	0.86	0	0	0	0.71	0	30.21
06	AMB-17	0.90	0.94	44.80	0	28.21	2.31	0.97	0	1.32	20.54	0	18.30
07	AMB-21	0.53	0.88	55.95	2.91	23.10	3.61	1.53	0	1.23	10.25	0	11.31
08	AMB-23	2.35	3.17	54.88	1.24	24.81	3.68	1.56	0	1.53	6.78	0	11.70
09	AMB-25	1.55	1.81	53.48	1.28	32.97	2.79	0.81	0	1.45	3.86	0	25.15
10	AMB-27	1.00	0.97	53.33	3.69	27.95	3.10	1.67	0	1.47	6.81	0	8.82
11	AMB-28	1.26	0.87	58.32	1.40	29.47	2.89	1.10	0	1.36	2.96	0	24.01
12	AMB-29	1.08	1.44	57.45	1.66	28.52	3.34	1.01	0	1.64	3.85	0	18.62
13	AMB-31	0.64	1.46	55.04	1.77	25.87	2.80	1.33	0	1.68	9.41	0	14.39
14	AMB-33	0.66	1.75	55.57	1.15	27.07	3.18	1.54	0	1.69	7.39	0	3.69
15	SPR-01	0.52	0.33	37.85	0.92	40.74	1.58	0.33	0	1.38	16.35	0	43.98
16	SPR-02	0.66	1.98	48.58	0.47	28.79	3.56	1.11	0	2.02	12.84	0	11.72
17	SPR-07	2.19	0.23	50.74	0.65	34.26	1.57	0.52	0.51	1.31	8.01	0	11.11
18	KGF-01	0.41	3.75	53.63	1.51	34.40	1.70	1.08	0	0.18	3.36	0	22.34
19	KGF-02	0.78	3.28	52.63	1.16	27.42	3.68	1.97	0	1.14	7.63	0	1.96
20	KGF-03	0.96	2.33	47.17	0.84	26.59	2.96	1.01	0	1.26	16.88	0	2.64
21	BMH-01	0.38	2.84	48.57	0	33.29	2.67	0	0	1.49	10.76	0	10.35
22	BMH-02	0.96	1.69	48.82	2.77	26.39	3.83	1.04	0	3.06	11.43	0	11.12

The LIBS spectra around 352.465, 374.677, 777.51 nm in AMB-01; 352.2, 552.72, 616.35, 655.975, 767.214, 776.0 nm in AMB-10; 353, 374.67, 552.732, 777.0 nm in AMB-31; 350.89, 374.67, 552.732, 655.975 nm in SPR-03 and 350.86, 374.1, 559.12 in KGF-03 nm are detected respectively due to presence of iron-rich minerals such as Hematite (Fe_2O_3), Magnetite (Fe_3O_4) and Goethite (FeOOH) (Melessanaki, 2002). The emission lines at 395.5, 396.890, 822.934, 867.736 and 869.07 are assigned to Ca which is presents in AMB-01, AMB-10, AMB-31, SPR-03 and KGF-03. Silica (Si) has been detected due to emission lines at 406.486, 412.883, 454.109, 559.1295, 616.348, 617.947 and 633.762 nm in all potsherds. LIBS spectrum of 430.297 nm is due to Al detected in SPR-03 and KGF-01. The characteristics spectrum at 500.31 nm is assign to Titanium. Magnesium (Mg) has been identified spectrum at 444.301 and 517.7255 nm in AMB-01, AMB-10, AMB-31 and KGF-03. LIBS spectrum lines at 587.62 and 589.16 nm are due to sodium (Na) in all potsherds. The present of Na in potsherds might be adsorbed during burial. Due to low amount of sodium present in pottery samples, it is difficult to identify that presence of Na either during manufacturing or adsorbed under earth in their composition.

On the basis of elemental analysis (EDX and LIBS analysis), chemical compositions of analysed historical pottery samples indicate that SiO_2, Al_2O_3, Fe_2O_3 and K_2O are present as main constituent in their composition. High content of SiO_2 and Al_2O_3 is attributed to occurrence of Quartz and aluminium-silicate minerals. The presence of Quartz and clay mineral such as Kaolinite and feldspar mineral groups are also confirmed by Petrographic, XRD, FTIR and Raman spectroscopic analysis. The presence of clay minerals is also confirmed by occurrence of K_2O in good amount. Low content of CaO and MgO is attributed to carbonates and silicates constituents. It must remember that archaeological pottery is buried for long period. So, pottery must come to contact of some contamination and weathering. Other constituents such as Na_2O, P_2O_5, SO_3, TiO_2 and Cl are present in very low quantity. It is not easy to detect presence of these elements in composition of analysed pottery is due to contamination during burial environment or occur naturally.

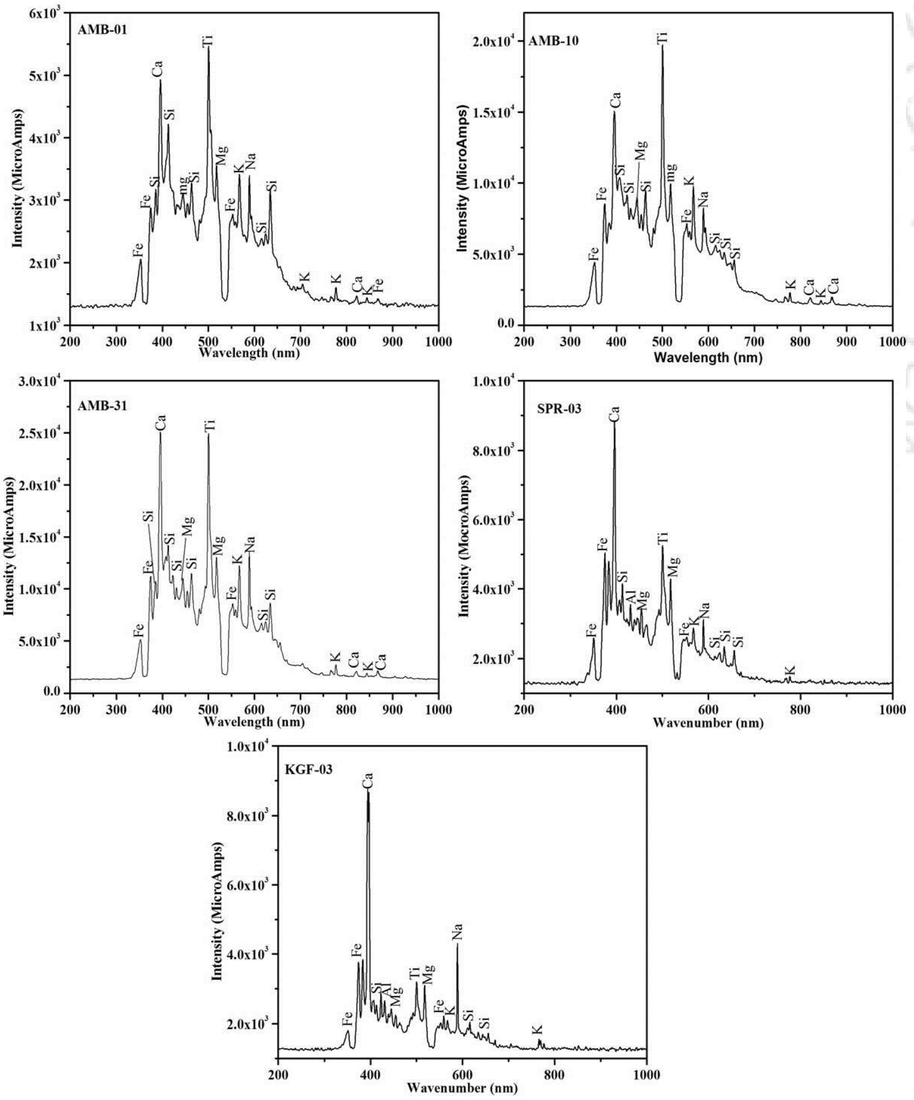

Figure 3.39 Characteristics spectra of AMB-01, AMB-10, AMB-31, SPR-03 and KGF-03

Table 3.17 Elemental composition of AMB-01, AMB-10, AMB-31, SPR-03 and KGF-03 identify using LIBS

AMB-01	
Wavelength (nm)	**Element**
352.465, 374.677, 777.51	Fe
396.890, 822.934, 869.07	Ca
385.872, 413.881, 634.362	Si
500.510	Ti
517.725	Mg
589..338	Na
705.196, 845.046	K
AMB-10	
352.2, 552.72, 616.35, 655.975, 767.214, 776.0	Fe
395.5, 822.834, 867.736	Ca
423.90, 463.50, 634,	Si
500.5	Ti
517.725	Mg
589.34	Na
567.5, 845.447	K
AMB-31	
353, 374.67, 552.732, 777.0	Fe
396, 822.834, 867.436	Ca
412.883, 463.5, 634.5	Si
500.5	Ti
444.5, 517.725	Mg
589	Na
567.5, 843.447	K
SPR-03	
350.89, 374.67, 552.732, 655.975	Fe
395.290,	Ca
384.273, 412.88, 454.109, 625.766, 634.109,	Si
430.297	Al
500.12	Ti
589.338	Na
567.212, 778.409	K
KGF-03	
350.86, 374.1, 559.12,	Fe
395.29,	Ca
384.71, 412, 42, 454.10, 559.12, 617.94, 633.76	Si
501.90	Ti
444.69, 517.725	Mg
589.338	Na
567.948, 768.81	K

3.8 Micro-indentation Hardness Testing (MHT)

Microindentation hardness or Micro Hardness tester (MHT) and Mohs' scale has been used to measure hardness of historical potsherds. MHT and Mohs' scale measurements value has been summarized in table 3.18 and figure 3.40.

The tabulated data (table 3.18) indicate that most of analysed historical potsherds showed comparatively higher micro hardness values than analysed Neolithic potsherds. The tabulated data is evident that most of historical pottery samples showed very hard, medium and very low hardness value. Hardness value is varying from 93.65 HV to 20.85 HV. AMB-02, AMB-14 and AMB-15 show hardest surface with hardness values 85.67, 89.10 and 93.3.65 respectively. Further, Microindentation test demonstrated that AMB-05, AMB-10, AMB-11, AMB-18, AMB-19 have harder surface with hardness values 62.05, 66.70, 66.95, 61.22, 65.35 and 64.00 HV respectively. Although, AMB-01, AMB-03, AMB-06, AMB-07, AMB-09, AMB-13, AMB-22, AMB-30, SPR-01, SPR-07, SPR-08, BMH-01 and BMH-04 shows medium hardness values – 59.4, 42.45, 59.9, 42.35, 54.59, 43.62, 53.52, 42.63, 53.40, 43.42, 43.32, 57.78, 41.21 and 44.12 HV respectively. Rest of potsherds have very low hardness values such as AMB-12 (26.65), AMB-17 (29.18), AMB-21 (32.6), AMB-23 (33.4), AMB-24 (37.13), AMB-29 (34.3), AMB-31 (25.3), SPR-02 (20.85), SPR-03 (33.93), SPR-04 (29.57), SPR-05 (26.12), SPR-06 (30.7), BMH-02 (35.5), BMH-03 (31.1), BMH-05 (28.9), KGF-01 (29.38), KGF-02 (28.7) and KGF-03 (33.8) HV.

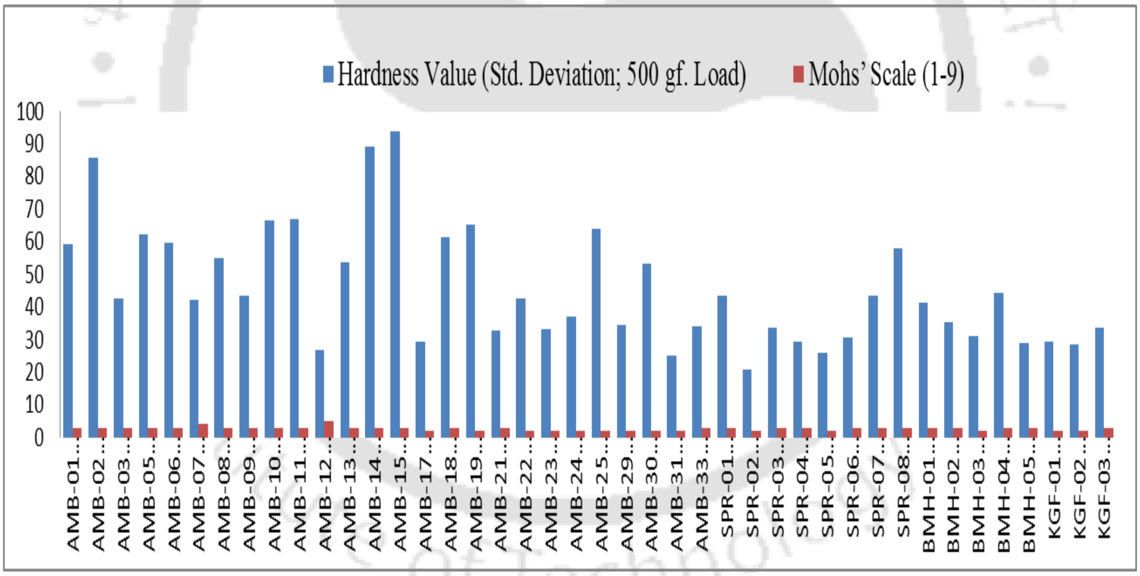

Figure 3.40 variations of micro hardness and Mohs' scale

Table 3.18 Micro Hardness and Mohs' scale hardness of Historical pottery

Si. No.	Samples Name	Hardness Value (Std. Deviation; 500 gf. Load)	Mohs' Scale (1-9)
1	AMB-01	59.4	3
2	AMB-02	85.67	3
3	AMB-03	42.43	3
4	AMB-05	62.05	3
5	AMB-06	59.9	3
6	AMB-07	42.35	4
7	AMB-08	54.9	3
8	AMB-09	43.62	3
9	AMB-10	66.7	3
10	AMB-11	66.95	3
11	AMB-12	26.65	5
12	AMB-13	53.52	3
13	AMB-14	89.1	3
14	AMB-15	93.65	3
15	AMB-17	29.18	2
16	AMB-18	61.22	3
17	AMB-19	65.35	2
18	AMB-21	32.6	3
19	AMB-22	42.63	2
20	AMB-23	33.4	2
21	AMB-24	37.13	2
22	AMB-25	64	2
23	AMB-29	34.4	2
24	AMB-30	53.4	2
25	AMB-31	25.3	2
26	AMB-33	33.93	3
27	SPR-01	43.42	3
28	SPR-02	20.85	2
29	SPR-03	33.7	3
30	SPR-04	29.57	3
31	SPR-05	26.12	2
32	SPR-06	30.7	3
33	SPR-07	43.32	3
34	SPR-08	57.78	3
35	BMH-01	41.21	3
36	BMH-02	35.5	3
37	BMH-03	31.1	2
38	BMH-04	44.12	3
39	BMH-05	28.9	3
40	KGF-01	29.38	2
41	KGF-02	28.7	2
42	KGF-03	33.8	3

3.9 Discussion

3.9.1 Provenance Analysis

Petrographic investigation of archaeological pottery may provide information about both provenance and technology. Study of provenance determination and technological reconstruction of archaeological pottery are interrelated.

The Powder XRD analysis of historical pottery is revealed that Quartz, Kaolinite and Hematite present in varying quantity in clay composition. Abundance of Quartz indicates that raw materials might have been procured from piedmont area (Ravisankar 2010). Discussed earlier, presence of Quartz may be elucidated as indigenous minerals of natural clay or added intentionally as tempering materials are not easy to identify. Interestingly, thin section analysis revealed that inclusion of non-plastic materials such as Quartz is very abundant in all characterized fabric groups. This is also confirmed by Powder XRD, FTIR and Raman spectroscopy. It is also indicate that Quartz would have been preferred as tempering inclusions over other materials such as Calcite. Powder XRD analysis confirmed that presence of K-feldspar (Orthoclase/Microcline) might be part of parent clay composition.

Thin section petrographic analysis has revealed that fifteen distinguished fabric groups with sub-fabric groups A1, B1, C1, E1, F1 and O1. This is indicating that historical potters of these places (Ambari, Suryapahar, Bamuni Hills and Kangla Fort) used different fabric for pottery production. Thin section investigation revealed shapes of grains in fabric groups. For examples, angular to sub-angular, irregular shapes are observed in fabric groups A, F, H and subgroups A1. Fabric group B and subgroup B1 shows angular, rounded and and irregular shapes. The grains of fabric groups C, E and subgroup C1 and E1 are showing angular to sub-angular. The fabric groups D and O reveal elongated grains shape. The grains of fabric group J are angular to sub-angular, rounded and elongate in shape. The fabric groups L and N are angular and sub-rounded grain shape. In addition, presence of grains in varying shapes provides information about types of clay and inclusions. The presence of angular to sub-angular and elongate shapes in various fabric groups indicates that primary/residual clay would have been used to produce these potsherds. The rounded shapes might have been used as tempering materials such as sand. Mineralogically, Quartz, feldspar minerals groups, siliceous and muddy materials are observed in all fabric groups. The matrix of fabric group B showed poor sorting because size of grains is not uniform. While fabric group E showed good sorting. The raw materials are less tempered and less sieved which might have been required as potters could attain maximum plasticity without using much temper and sieving.

3.9.2 Reconstructing firing technology of Historical pottery

Reconstruction of manufacturing technology of historical pottery has been obtained using petrographic analysis, FTIR, SEM-EDX, XRD, TGA-DSC and Raman Spectroscopy.

The thin section analysis of characterized fabric groups of historical potsherds – A to D, G, H, I, J, L O and subgroups A1, B1, C1, C2, revealed deep, lighter red to reddish, yellow, green grey and brown color matrices. As discussed earlier, in oxidizing atmosphere iron is converted to ferric mineral such as Hematite and it give to pottery body red, brown, and orange in color at 600 °C temperatures. While, in reducing atmosphere iron is exists as 'ferrous' mineral (magnetite- Fe_3O_4) and it give to ceramic body black and grey in color due to presence of carbonized organic substances. Lighter red, orange or brown and dark black or grey color core of ceramics is revealing that short period of firing due to lacking of oxygen penetrated body to eliminate carbon. Considering this, above mentioned fabric groups and sub groups might have been fired under oxidizing atmosphere above 600 °C. In addition, fabric groups B, F and M might have been fired for short duration. The presence of organic matter and brown color matrices also indicate that pottery would have been fired for short duration under oxidizing atmosphere above 600 °C. However, organic materials could be removed if pottery was fired for longer-duration in oxidizing condition. FTIR, XRD and Raman Spectroscopy also showed similar results.

EDX analysis revealed that quantity CaO is below 6 wt. %. According to Maniatis and Tite (1981), presence of CaO content above 6 wt. % can classified as calcareous clay and below 6 wt. % known as non-calcareous clay. Referring to this, EDX analysis revealed that non-calcareous clay has been used to produce all analyzed historical potsherds during production.

If the occurrence of K_2O, Fe_2O_3, CaO, MgO and TiO_2 in clay composition is greater than 9 wt. percentage, then it indicate that clay is low refractory clay (Velraj *et al.*, 2010). Considering this, tabulated data confirmed (table 3.16) that low refractory clay has been used for production of analyzed pottery. EDX analysis also revealed comparatively higher content of iron oxide (Hematite) which indicates all analyzed historical potsherds might have been fired under oxidizing condition. Table 3.19 showed distinguished characteristics of various analyzed pottery samples.

SEM photomicrographs of AMB-06 (fig. 3.41D), AMB-11 (fig. 3.42C), AMB-15 (fig. 3.42E), AMB-17 (fig. 3.42F), AMB-23 (fig. 3.42H), AMB-29 (fig. 3.43D), AMB-31 (fig. 3.43E), AMB-33 (fig. 3.43F), KGF-01 (fig. 3.44A), KGF-02 (fig. 3.44B), KGF-03 (fig. 3.44C) BMH-01 (fig. 3.43G) and BMH-02 (fig. 3.43H) is revealed that there is no vitrification (NV) occurred during firing. According to Maniyatis and Tite (1975), low refractory clay with no vitrification stage and fired in oxidizing atmosphere indicate that firing temperature will not exceed 800 °C. Considering this, these potsherds are fired < 800 °C under oxidizing condition.

SEM photomicrographs of AMB-02 (fig. 3.42A), AMB-05 (fig. 3.41C) and AMB-08 (fig. 3.41F) are showed extensive vitrification (EV) during firing. According to Maniyatis and Tite (1975), non-calcareous and low refractory clay with extensive vitrification (EV) stage and fired in oxidizing atmosphere indicate that firing temperature range in between 850-950 °C (Velraj *et al*., 2010). This indicates that AMB-02, AMB-05 and AMB-08 are fired in between 850-950 °C under oxidizing atmosphere.

SEM photomicrographs of AMB-01 (fig. 3.41A), AMB-03 (fig. 3.41B), AMB-07 (fig. 3.41E), AMB-10 (fig. 3.42B), AMB-13 (fig. 3.42D), AMB-21 (fig. 3.42G), AMB-25 (fig. 3.42A), AMB-27 (fig. 3.42B), AMB-28 (fig. 3.42C), SPR-01 (fig. 3.44D), SPR-02 (fig. 3.44E and SPR-07 (fig. 3.44F) are showed initial stage of vitrification (IV). Initial stage of vitrification of clay occurs in firing range of 800-850 °C under oxidizing condition. This is indicating that these potsherds are fired under oxidizing atmosphere in range of 800-850 °C (Maniatis and Tite, 1981; 1975a; Velraj *et al*., 2010).

The FTIR investigation of historical pottery infers about firing atmosphere and firing temperature. According to Ravishankar and co-workers (2010), compositions of clay help to estimate firing temperature of ancient pottery. But, it is not easy to replicate actual firing duration and firing condition achieved by ancient ceramics during manufacturing and has possibilities of small errors. IR band around 3700 cm^{-1} is assigned to crystalline hydroxyl (OH-stretching of interlayer water), and it reflects destruction of clay structure when subjected to fire above 500–600 °C. While, IR absorption band around 915 cm^{-1} is due to Al-OH vibrations in octahedral sheet structure which completely collapses near 550 °C.

Table 3.19 SEM analysis estimating the firing temperature and condition IV- initial vitrification, EV – Extensive vitrification, NV, No vitrification; NC – Non calcareous clay

Si. No.	Sample Name	Clay Type	Stage of Vitrification	Firing Temperature (°C)	Firing Atmosphere
01	AMB-01	NC	IV	800-850	Oxidizing
02	AMB-02	NC	EV	850-950	Oxidizing
03	AMB-03	NC	IV	800-850	Oxidizing
04	AMB-05	NC	EV	850-950	Oxidizing
05	AMB-06	NC	NV	< 800	Oxidizing
06	AMB-07	NC	IV	800-850	Oxidizing
07	AMB-08	NC	EV	850-950	Oxidizing
08	AMB-10	NC	IV	800-850	Oxidizing
09	AMB-11	NC	NV	< 800	Oxidizing
10	AMB-13	NC	IV	800-850	Oxidizing
11	AMB-15	NC	NV	< 800	Oxidizing
12	AMB-17	NC	NV	< 800	Oxidizing
13	AMB-21	NC	IV	800-850	Oxidizing
14	AMB-23	NC	IV	800-850	Oxidizing
15	AMB-25	NC	NV	< 800	Oxidizing
16	AMB-27	NC	IV	800-850	Oxidizing
17	AMB-28	NC	IV	800-850	Oxidizing
18	AMB-29	NC	NV	< 800	Oxidizing
19	AMB-31	NC	NV	< 800	Oxidizing
20	AMB-33	NC	NV	< 800	Oxidizing
21	SPR-01	NC	IV	800-850	Oxidizing
22	SPR-2	NC	IV	800-850	Oxidizing
23	SPR-07	NC	IV	800-850	Oxidizing
24	BMH-01	NC	NV	< 800	Oxidizing
25	BMH-02	NC	NV	< 800	Oxidizing
26	KGF-01	NC	NV	< 800	Oxidizing
27	KGF-02	NC	NV	< 800	Oxidizing
28	KGF-03	NC	NV	< 800	Oxidizing

Presence of these two infrared bands indicates that AMB-04 would have been fired around 600 °C. While, these two bands are totally absent in rest of analyzed potsherds. It indicates that all potsherds might have been fired above 500 °C. Infrared bands at 3622 cm^{-1}, 3616 cm^{-1}, 3660 cm^{-1} and 3620 cm^{-1} are indicating that disordered Kaolinite present in potsherds AMB-04, AMB-11, SPR-05 and SPR-06 respectively. These bands persist above firing temperature of 400-600 °C, which indicate that these samples would have been fired above 600 °C (Prost *et al.*, 1989; Ravisankar, *et al.*, 2011; Venkatachalapathy *et al.* pp. 771-774, 2002; Singh & Sharma, 2016).

The infrared bands at around 1080 cm^{-1} are due to present of wollastonite in some historical potsherds (AMB-01, 06, 08, 09, 10, 12, 15, 17, 23, 25, 26, 28, 29; SPR-01 and 05) (Palanivel & Kumar, 2011). Barilaro *et al.* stated that wollastonite formed during firing temperature at 900 ºC from chemical reaction between Quartz and carbonates (Palanivel & Kumar, 2011). Excluding above mentioned potsherds, infrared bands around 1040 cm^{-1} with very strong peaks recorded in rest of samples. The destruction of octahedral sheet structure takes place at 700 °C. It indicates that analyzed historical potsherds might have been fired in between 600-850 °C.

All historical potsherds showed absorption bands around 561 cm^{-1}, 535 cm^{-1} and 475 cm^{-1} due to Hematite. It indicates all pottery are fired in oxidizing atmosphere. Presence of Hematite reveals existence of iron oxide which also confirms firing temperature as above 600 ºC. The infrared bands at 574 cm^{-1}, 581 cm^{-1} and 578 cm^{-1} detected in AMB-17, 23, SPR-01 respectively are attributed to magnetite. Transformation of Hematite to magnetite occur around 800 to 900 ºC. Presence of only Hematite in most of historical potsherds indicates that firing temperature should be in range of 650-800 ºC under oxidizing condition. While, presence of magnetite confirmed that AMB-17, 23, SPR-01 would have been fired in between 800-900 ºC under reducing atmosphere.

Microcline is present in analyzed potsherds AMB-01, 03, 17, 21, 26, 27, 30; SPR-01, 03, 05 and 08. The presence of Microcline is also confirmed by XRD, indicate that firing temperature of these analyzed pottery would have been approximately 800 °C.

On the basis of above discussion, FTIR analysis indicates firing temperature range of AMB-01, 06, 08, 09, 10, 12, 15, 17, 23, 25, 26, 28, 29; SPR-01 and 05 should be within 800-900 °C. Rest of historical pottery samples would have fired approximately 800-900 °C. The presence of Hematite is indicating towards oxidizing firing atmosphere of historical pottery. Presence of magnetite in AMB-17, 23, SPR-01 confirmed that these pottery fired around 800-900 °C under reducing firing atmosphere. C–H stretching spectra reveal presence of organic matters in composition of pottery. Generally, natural clay contains varying quantity of organic materials in their composition. However, potters may use organic materials externally as additive to improve plasticity during clay preparation. Furthermore, pottery can adsorb organic substances during burial period (Damjanovic, *et al.*, 2011, p. 826). The FTIR result has good agreement with thin section petrography, SEM and Powder XRD.

In addition to FTIR, Raman Spectroscopy has been used to investigate the mineralogical composition and inferring minimum firing temperature and firing atmosphere of historical pottery. Raman bands around 1130 cm^{-1} due to Si-O asymmetric stretching mode instead of around 1000 cm^{-1} indicate high-temperature firing of AMB-30. Presence of Ilmenite in pottery samples of SPR-02, 04, 06 and BMH-01 indicate that raw material are rich in iron and titanium.

Due to high fluorescence at analyzing point of potsherds, Hematite (Fe_2O_3) has been identified by Raman spectroscopy only in AMB-05, SPR-02, 08 and BMH-01. While, Powder XRD analysis reveals that all historical potsherds have Hematite in their composition in varying quantity. EDX analysis also indicates higher content of iron oxide. It is difficult to identify that presence of Hematite in samples whether occur naturally or added intentionally by potters. Presence of Hematite also indicates that these potsherds would have been fired in between range of 700-900 °C, because above this temperature Hematite-magnetite transition phase occurs. Approximately, at 650-700 °C Hematite begins to decompose gradually and above 900 °C transform into magnetite (Olivares, *et al.*, 2010, pp. 1546-47).

Anatase is detected in pottery samples AMB-01, 03, 05, 08, 12, 14, 15, 21-29, 32, 33, SPR-08 and KGF-01-03. As discussed earlier, Anatase is stable in between 600-900 °C (Liou *et al*. 2011; Medighini *et al*; Olivares, *et al.*, 2010). Above this temperature Anatase-rutile transformation phase takes place (Bouzid, *et al.*, 2013). Sometimes, Anatase transforms directly or via brookite to rutile during firing. Only Anatase has detected in above mentioned analyzed potsherds indicates that firing temperature might have been below Anatase-rutile transition phase (Olivares, *et al.*, 2010; Smith & Clark, p. 1145, 2004). Generally, Anatase is present as associate constituent in Kaolin and naturally grows as one of three polymorphs of titanium dioxide (Olivares, *et al.*, 2010; Gennari & Pasquevich, 1999). Referring to this, Anatase may be present in raw material (used clay) of studied historical potsherds. Apparently, by SEM, XRD and FTIR analyses are also confirmed that historical pottery might have been fired in between 750-950 °C.

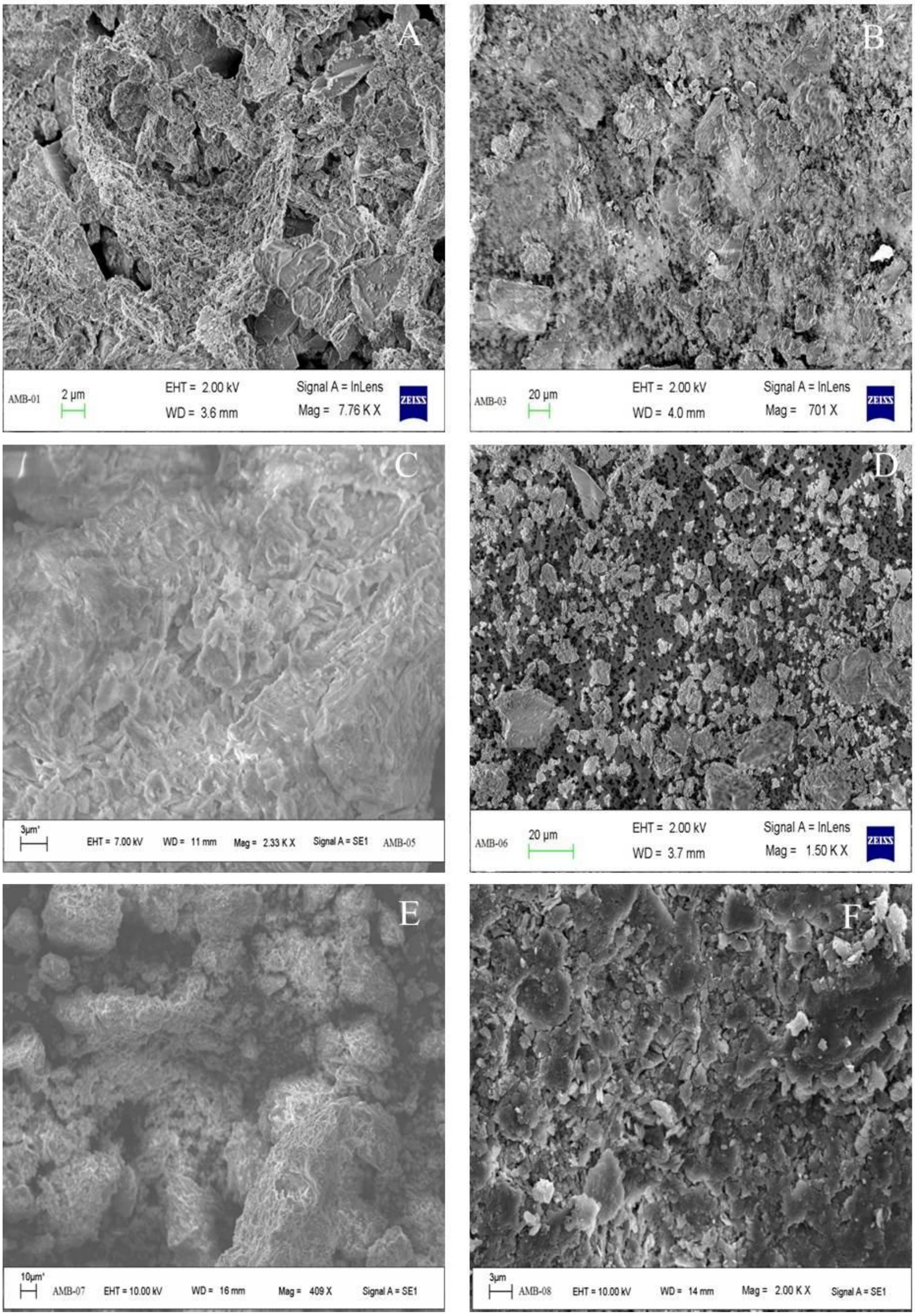

Figure 3.41 SEM photomicrographs of (A) AMB-01 (B) AMB-03 (C) AMB-05 (D) AMB-06 (E) AMB-07 and (F) AMB-08

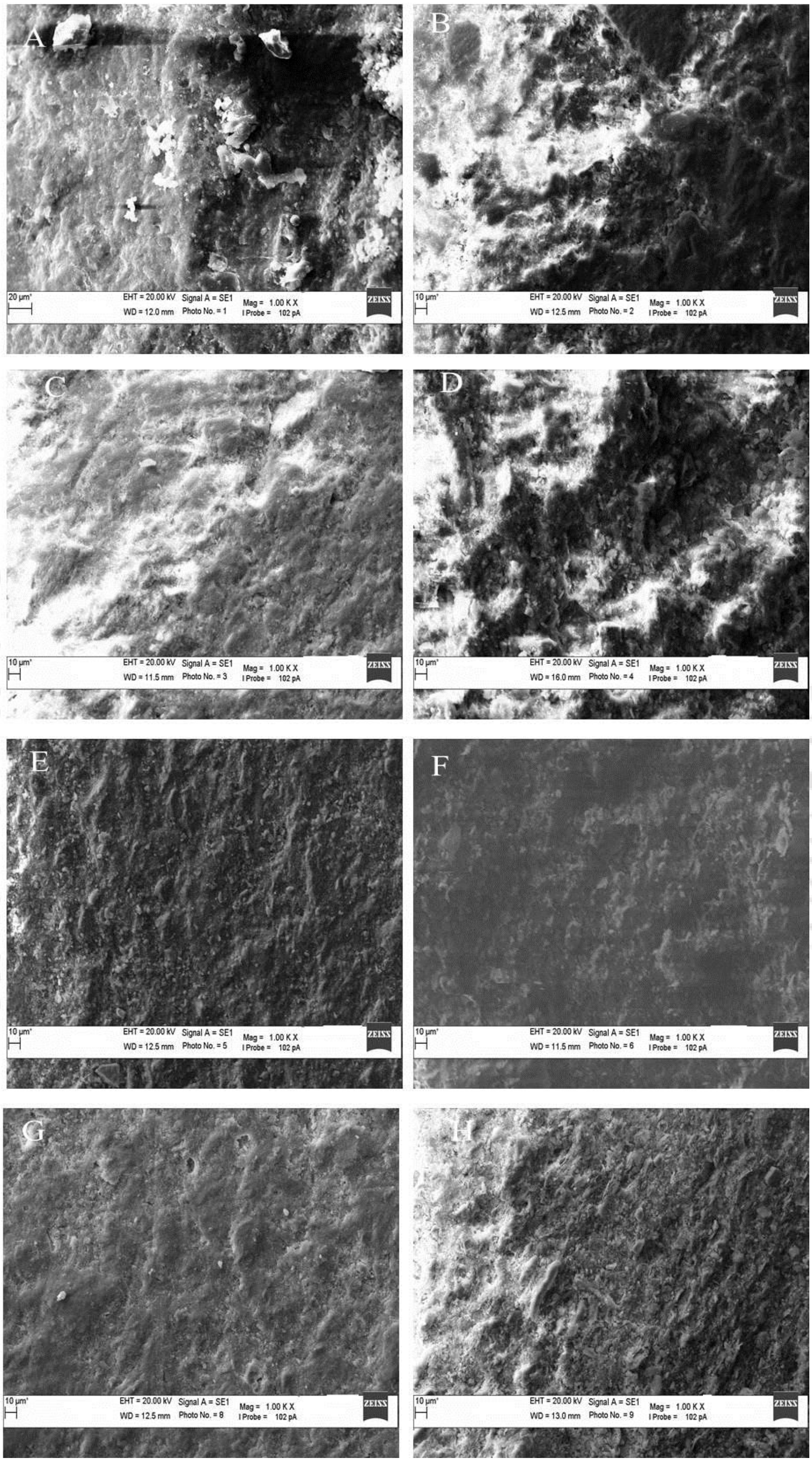

Figure 3.42 SEM photomicrographs of (A) AMB-02 (B) AMB-10 (C) AMB-11 (D) AMB-13 (E) AMB-15 (F) AMB-17 (G) AMB-21 and (H) AMB-23

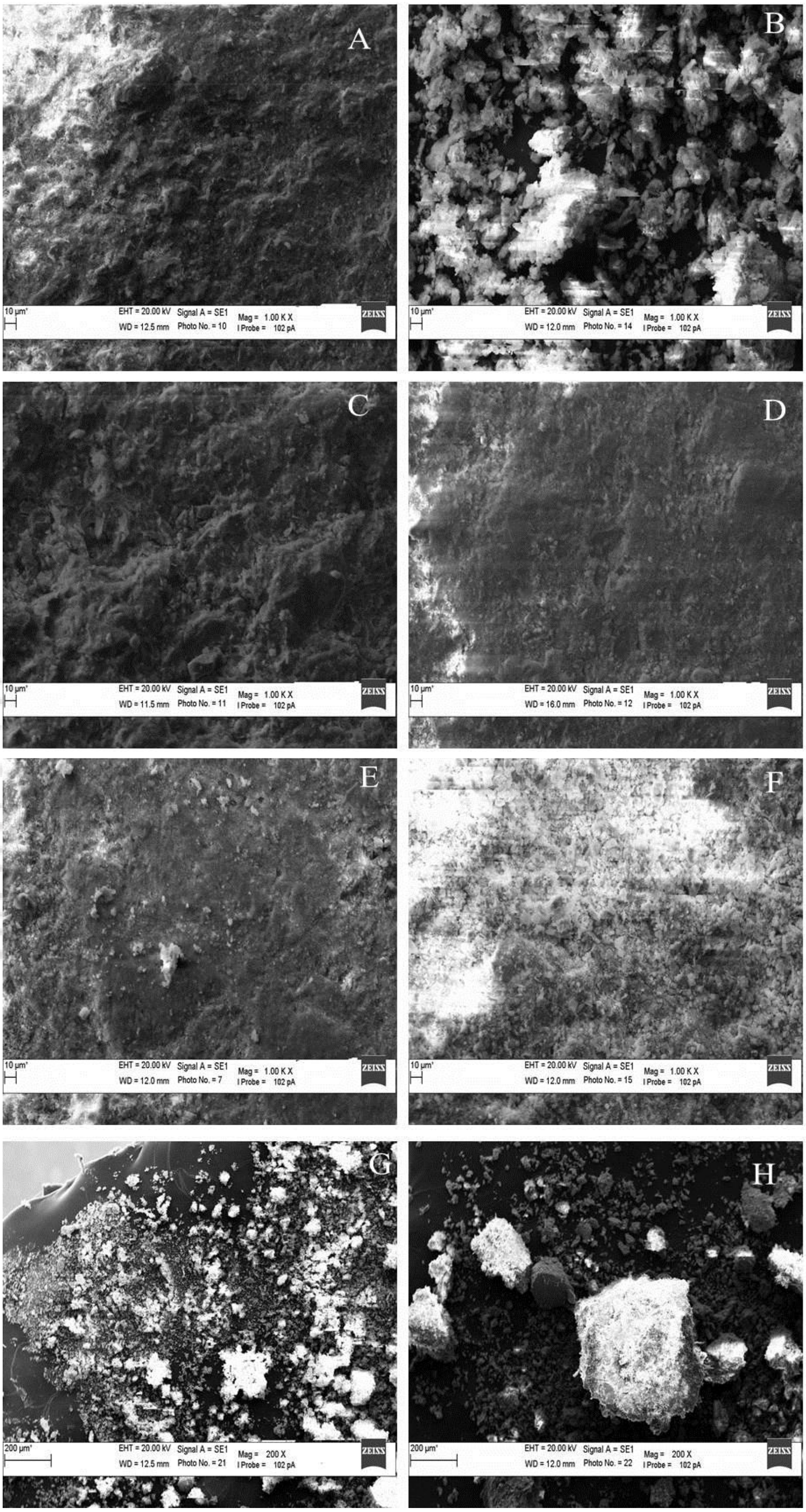

Figure 3.43 SEM photomicrographs of (A) AMB-25 (B) AMB-27 (C) AMB-28 (D) AMB-29 (E) AMB-31 (F) AMB-33 (G) BMH-01 and (H) BMH-02

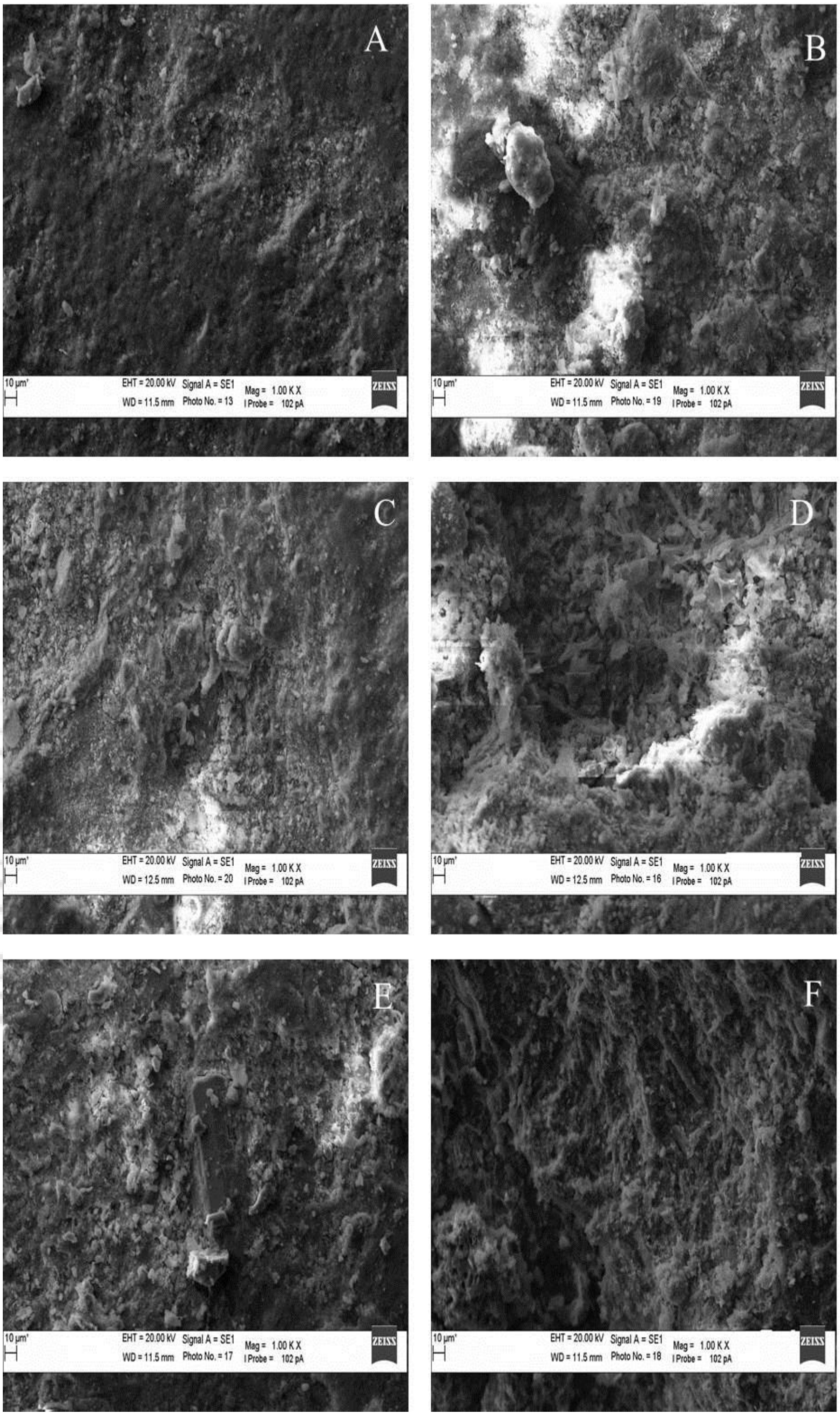

Figure 3.44 SEM photomicrographs of (A) KGF-01 (B) KGF-02 (C) KGF-03 (D) SPR-01 (E) SPR-02 and (F) SPR-0

3.10 Conclusion

The present work was carried out to get information about manufacturing techniques and provenance of historical potsherds reported in excavation at Ambari, Sri Sri Suryapahar, Bamuni Hills and Kangla Fort. The combination of Petrography, Powder XRD, FTIR, TG–DSC, micro-Raman Spectroscopy, SEM-EDX, LIBS, MHT and Scratch hardness (Mohs' Scale) have been applied to investigate mineralogical and physiochemical properties of historical pottery. Using these analytical techniques concluding remarks are as following:

- Thin section petrography analysis revealed that both type of clay (primary and residual clay) might have been used to produce analyzed potsherds. On the basis of mineralogical content, all analyzed potsherds have been characterized into fifteen distinguished fabric groups and sub-groups. Mineralogically, Quartz, Feldspars, silt stone, mud stone, clayey and siliceous materials, ferruginous materials and organic substances have been identified in varying composition. These materials first of all reflect properties of local geology of place of pottery production, but also potters' habits, which largely determined use of particular kinds of temper and addition of artificial components, in an effort to improve properties of clay and to impart particular properties to pottery. Presumably, each distinguished fabric groups and subgroups revealed that manufactured in the different workshop. The results of this work support the fact that these analyzed potsherds have chosen from different sites, trench and layer and produced in different period. Thin section analysis revealed that sand, organic substances and Quartz grit might have been used as tempering materials.
- The FTIR result confirmed that two type of clay are used for pottery production. AMB-01, 06, 08, 09, 10, 12, 15, 17, 23, 25, 26, 28, 29 and SPR-01 and SPR-05 are made of white clay origin of Kaolinite. While, AMB-02, 03, 04, 05, 07, 11-14, 16, 18-22, 24, 27, 30-31; BMH-01-05 and KGF-01-03 are made of red clay origin of Kaolinite.
- FTIR analysis also revealed that all potsherds would have been fired in between 700–900 °C under oxidizing atmosphere (except AMB-17, 23 and SPR-01). AMB-17, 23 and SPR-01 showed presence of magnetite in their composition which reflects reducing atmosphere and these potsherds might have been fired around 800-900 °C.
- FTIR analysis also revealed that Microcline is present in analysed potsherds of AMB-01, 03, 17, 21, 26, 27, 30; SPR-01, 03, 05 and 08. The presence of Microcline indicates that these potsherds might be fired around 800 °C.

- Organic materials have been identified using FTIR in AMB-01, 02, 04, 06, 09, 15, 17, 18, 20, 22, 24, 25, 28, 30, 31, 32, 33; SPR-04, 07, 08; BMH-02, -05; KGF-02 and KGF-03. While, AMB-01, 03, 08, 10, 17, 18, 19, 20, 21, 22, 23, 24, 25, 28, 28; SPR-02 and SPR-08 showed carbon overtone in their compositions.
- The presence of exothermic and endothermic peaks in TG–DSC analysis of all three potsherds confirmed that firing temperatures are in range of 650–800 °C. This result also showed that more weight loss occurred at dehydroxylation in comparison to dehydration, and organic materials were used for manufacturing pottery by ancient artisan.
- The micro-Raman spectroscopy revealed that all analysed historical potsherds might have been fired in range of 650-900 °C under oxidizing atmosphere. Mineralogically, Quartz found abundantly in all historical potsherds. Anatase is also found almost in all potsherds. Other minerals are also detected in historical potsherds. For examples, plagioclase is detected in AMB-21, AMB-26, and SPR-08. The carbon black materials are detected in AMB-01, 05, 06, 09, 14, 15, 28, 32, KGF-02 and 03. Hematite is detected only in AMB-05, SPR-02, 08 and BMH-01. The presence of Anatase, carbonate and Hematite indicate firing range between 650-900 °C and oxidizing atmosphere.
- Scanning electron micrographs yields information on firing processes of analysed pottery. SEM analyse revealed that –

i. AMB-06, AMB-11, AMB-15, AMB-17, AMB-25, AMB-29, AMB-31, AMB-33, BMH-01, BMH-02, KGF-01, KGF-02 and KGF-03 are fired around 800 °C under oxidizing condition due to no occurrence of vitrification (NV).

ii. AMB-02, AMB-05, and AMB-08 are fired in between 850-950 °C under oxidizing atmosphere due to occurrence of extensive vitrification (EV) during firing.

iii. AMB-01, AMB-03, AMB-07, AMB-10, AMB-13, AMB-21, AMB-25, AMB-27, AMB-28, SPR-01, SPR-02 and SPR-07 are fired under oxidizing atmosphere in range of 800-850 °C during manufacturing pottery due to occurrence of initial stage of vitrification (IV).

- EDX analysis revealed SiO_2, Al_2O_3, Fe_2O_3 and K_2O are higher than other elements in all samples and these are varying in all pottery samples. Present of titanium oxide is confirmed by EDX analysis. EDX and LIBS analysis revealed divergent concentrations of Ca, Na, Mg, Ti, Cl, S, and P. EDX result reveals that high amount of silica and alumina rich constituent in composition of historical pottery. The high content of silica and alumina rich constituent indicates high quantity of Kaolinite is used to manufacture historical pottery. Powder XRD results also revealed good amount of Kaolinite in composition of analysed pottery.

It is concluded that historical potters of studied region are used open firing, oven firing and sometimes kiln firing techniques. As discussed earlier, all methods of open firing differ from approximately 400-700 °C. Oven firing is differs in range of 700-800 °C. Above 750 °C temperature can be achieved in kiln firing. The different firing techniques of all analysed potsherds revealed that different workshops have been used to produce pottery. The firing temperature of historical pottery has been determined using various analytical methods. Combining results of applied various techniques revealed that all potsherds were fired in range of 650-900 °C firing temperature under oxidizing atmosphere.

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Chapter 4
Ethnographical Investigation of Pottery Tradition in Assam

4.1 Introduction

Ceramic ethnoarchaeological study mainly focused on research of pottery production system in small-scale societies and described by domestic industries (Stark 2003: 196). Studies have focused more on ceramic as object of business, hierarchy, societal status and skills. To investigate basic issues in archaeological record such as pottery is whether handmade or wheel-made, strength of product, porosity level, an investigator can deal with limited experimental methods and use ethnoarchaeological approach as research laboratory for scientific analysis. In present research work author acted as an observer and documented entire pottery production technology.

According to Costin (2000), for archaeologists, ethnoarchaeological approach is useful for interpreting and tracking pottery production using comparative data, descriptive frameworks and hypotheses of source of ceramics. Costin (2000) stated that there are several key goals of archaeological investigation on ceramic manufacturing processes such as: production techniques (craftsperson, means of production-raw materials and equipment, principles of spatial and social group, finished product, principles and mechanisms of distribution and users), changes in technological and organizational aspects and to detect and describe cross-cultural regularities and variability in manufacturing process of ceramic production.

The key objectives of physicochemical methods are to reconstruct manufacturing techniques of archaeological pottery. Also, physicochemical methods are used to determine provenance of archaeological pottery by characterizing composition and it can compare with known origin of pottery or with raw resources. In previous chapters (chapter 2 and 3), manufacturing processes of Neolithic and Historical pottery samples from Northeast India have been investigated using various scientific techniques. Mineralogical and physiochemical analysis revealed information about clay type, organic and inorganic inclusions and firing process. Furthermore, results indicate that Quartz, Kaolinite, Hematite and Feldspar mineral group is present in properties of clay composition of all analyzed pottery samples Elemental investigation also confirmed that SiO_2, Al_2O_3 and FeO are present in clay composition of all analyzed pottery samples. Petrographic analysis also reveals that sand and organic matters might have been used as tempering materials pottery production. Scientific analysis also revealed firing temperature and condition of Neolithic and Historical pottery samples. Results demonstrated that all analyzed neolithic potsherds fired in between 400-700 °C under oxidizing atmosphere. While, all historical pottery fired in between 600-900 °C. Most of historical potsherds fired under oxidizing atmosphere but few pottery samples fired in reducing condition.

Physicochemical study of archaeological pottery is greatly informative, but, this analysis can reveal only one side of ceramic studies. In addition, ethnographic and ethnoarchaeological approaches to study the modern potters' groups and their pottery production processes such as forming, decorating, firing, marketing of finished product and uses of their societies may extract very useful information about archaeological pottery.

Generally, ethnographic and ethnoarchaeological approaches of pottery assemblages focused on surveys, number of pots in certain households and on use life or durability of ceramics (Rice, 1987, p. 293). Ethnographical studies of particular household pottery are also very useful to generalize its use, discard, replacements which are vital to investigate and understand archaeological pottery assemblages or record. This may be useful to reveal how clay procurement and preparation reflects composition of pottery and behavior through investigation of materials from living potter communities.

The modern pottery is a contemporary phenomenon knowledge which can be used for interpreting past. Archaeological relevance of contemporary phenomena has influenced researchers to document traditional technologies. This provides data for developing stronger archaeological inferences for explaining site formation, depositional processes, settlement pattern etc.

According to Arnold, *et al.* (1991), several analytical methods are useful to highlight problems regarding human activities to its materials remains more than compositional examination of ceramic. For archaeologists, issue of compositional study is relating to chemical constituent of an artefact and its manufacturers' behavior. Arnold (1971 p. 28) stated that there are several problems complicating potter behavior and elemental composition of pottery. Selection of raw materials depends on physical properties – color of clay, plasticity, presence of non-plastic inclusions or tempering materials etc. These physical properties of raw materials are vaguely indicated in chemical elements of pottery composition.

Ethnographic approach for analyzing known provenance will help to identify chemical properties of pottery and draw assumption about methodology and concepts about ancient ceramic technology. It is permitted to decode precisely information about trade, exchange, and manufacture.

In Assam, Hira and Kumar are two traditional potter communities who produce handmade and wheel-made pottery respectively. Clay used by Hira potters is locally called "Hiramati" and is collected from surface of certain area while clay used by Kumar potter is locally called "Kumarmati" and it is collected from five to ten feet depth depending on availability of clay. Both are household industries and is practiced by a particularly marginalized caste group only. Studies have revealed that tradition is dying because younger members of community are no more interested in learning art as it is a time consuming and low-income profession.

This work investigates relationship between potters' behavior in procuring and using raw materials of traditional potters of Assam. It also investigates manufacturing technology and mineralogical, physical and chemical constituent of modern pottery using analytical techniques. Furthermore, application of scientific techniques of ethnographic pottery samples helps to make assumptions about manufacturing techniques of archaeological pottery. Objective of study is to develop measures for conserving this intangible cultural heritage. Present study will document whole manufacturing process of Hira and Kumar pottery by using ethnographic methods. To achieve above stated objectives, field work have been conducted in Hirapara, Khehenipara - Kamrup district and Kumargaon- Dergaon, Kumargaon- Dhekial – Golaghat district, Assam (Appendix I). Present study also deals with analytical techniques such as Powder XRD, FTIR, SEM-EDX, MHT and thin section petrography.

4.1.1 Alipub’ Hirapara (26°19'25.05" N, 91°44'40.34" E)

‘Alipub’ Hirapara is a Hira village situated at foothill of ancient Madan Kamdev Hill in rural Kamrup district, Assam. Among 32 households, 30 families belong to Hira community. Subsequently, only 5 Hira households still manufacturing pottery.

4.1.2 Khehenipara (26°20'47.06"N, 91°46'50.38"E)

Khehenipara is situated in Kamalpur of Kamrup district, Assam. Khehenipara is a medium size village with total 331 families and has population of 1655 (835-males and 820-females) as per Population Census 2011.

4.1.3 Kumargaon, Dergaon, Golaghat, Assam:

Kumargaon is located at Dergaon Tehsil in District Golaghat. illage is situated in a distance of 30/35 kilometers from Golaghat City. This village is inhabited by more than 4o Kumar households. But there is only one family in village practicing pottery making tradition as a source of earning up till now.

4.1.4 Kumargaon, Dhekial (26°36'4.30"N, 93°59'1.72"E)

Kumargaon is located at Dhekial in Golaghat District. The village is situated in a distance of 15/20 kilometers from Golaghat City.

4.2 Manufacturing Processes

The whole process can be classified into five parts and it can be explained as follows –

4.2.1 Sources of Clay & Selection of the suitable clay for making pots:

Hira potters (Hirapara ‘Ali Pub’) collect clay from an open and moist field situated in Bamunigaon about 1 km south of their village. The field lies between approximately 100 bighas (0.024711 Acres) in area. Procurement of clay from Bamunigaon is since unknown past. Selection of clay is depending on their colour, texture and plasticity. Hira potters use to collect clay from surface.

Kumar potters (Kumargaon-Dergaon) collects clay from bed of Gelabil River which is flowing about 500 meters away from village. In particular village, only one Kumar potter is continuing pottery making tradition. Kumar potters (Kumarpara-Dhekial) collect clay about 4-5 km away from their village.

4.2.2 Storage of clay

Hira potters (Hirapara 'Ali Pub') stored procured clay in courtyard. For this, they dig trench under tree or shed. In bottom, they place banana leafs and covered with a thin layer of sand. Then placed clay and cover with sand. To retain moisture, they covered with banana leafs or jute bag. Although, Kumar potters (Kumargaon-Dergaon) prefer to keep procured clay under shadow of tree or shed and covered with jute bag. Kumar potters (Khehenipara) also keep clay under shed.

4.2.3 Processing of clay

The cleaning is first stage of processing of clay. Potters of Assam (both- Hira and Kumar) remove pebbles, gravels, roots and other impediment materials from clay. Kumar potters mix two or more varieties of clay. Sand, ash, cattle-dung are add to improve quality of clay. But, Hira potters do not add any other type of clay.

The clay processing technique of Hira potters is very simple. During kneading process, they add only sand as tempers. Hira potters knead clay on a stone slab. They sprinkle sand on stone-slab before kneading. Then, they knead clay with both foot and a long cylindrical wooden bat. Water is added time to time to make it soft. In meantime, unwanted elements are removed from clay. Whole process of preparing clay takes 20-30 minutes. Only, Hira women are involved in kneading of clay.

The clay processing of Kumar potters of Khehenipara is same as Hira potters. Although, clay processing of Kumar potters from Kumargaon-Dergaon and Kumarpara-Dhekial (Golaghat) is different from potters of Hirapara and Kehenipara (Kamrup district). The first stage is to make dry clay into small pieces using spade. After that, they keep clay into water for 30-50 minute to make it smooth and then cut it using bamboo slicer (locally known as *'Katoni'*). Then, clay is kneaded by both foot and hand. During kneading, they mix sand as temper according to requirement. Prepared clay is ready for making pots in wheel.

4.2.4 Equipment of Hira and Kumar potters (with local term)

The Hira potter's equipment (figure 4.1) and its functions are following –

1. **Big stone slab:** for kneading clay.
2. **Beater** (locally known as Gayen): cylindrical in shape and made of wood.
3. **Different sizes of Anvil** (locally known as Xoru xil and Dangor xil): use for giving shape to pots.
4. **Beater** (locally known as Pitani): used for giving shape.

The Kumar potter's equipment (figure 4.2 to 4.7) and its functions are following –

1. **Wooden flat slab** (locally known as *'Pira'*)**:** A big flat wooden board use for kneading clay.
2. **Point** (locally known as *'Sak nail'*)**:** Point in its pointed edge wheel is placed, because of this pointed wood wheel can rotate)
3. **Potters' wheel** (Locally known as '*Sak'*)**:** The wheel is made of iron, wood and clay. The rim of wheel is plastered with clay.
4. **Bamboo stick** (locally known as '*Sak ghurua mari*')**:** bamboo stick on wheel is use to rotate wheel.
5. **Wooden Beater (**locally known as *'Pitani'***):** using to give shape to wares.
6. **Wooden Plate** (locally known as *'Athali'*)**:** use for shaping earthen wares.
7. **Clay article** (locally known as *'Majani'*)**:** use to make outer surface of pots smoother.
8. **Tool made of cement or stone** (locally known as '*Bolia'*)**:** it is used for giving shape to pots and also makes surfaces smoother.
9. **Earthen bowl** (locally known as *'Aphori'*)**:** use for shaping xaneki/saru and base of other pots.
10. **Hearth** (locally known as '*Peghali'*)**:** Firing place or kiln

Figure 4.1 Tools of Hira potters: 1. Big stone slab: for kneading clay 2. Beater (Gayen): cylindrical shaped made of wood 3. Different sizes of Anvil (Xoru xil and Dangor xil): use for giving shape to pots 4. Beater (Pitani): used for giving shape.

Figure 4.2 Various Tools: 1) Point (Sak Nali, in its pointed edge the wheel is placed, because of this pointed wood the wheel rotate) 2) Bamboo stick (Sak Gurua Mari, the bamboo stick on the wheel is use to rotate the wheel)

Figure 4.3 Kumar Potters’ wheel (Sak) 1) Khehenipara 2) Khehenipara (metal wheel) 3) Kumargaon-Dergaon 4) Kumargaon-Dhekial

Figure 4.4 Various tools: 1) Earthen bowl (Aphori, use for shaping xaneki/saru and the base of other pots) 2) Clay item (it is known as "majani", use to make the outer surface of the pots more mooth.) 3) Wooden Plate (Local term is "Aathali", use for shaping the earthen wares) 4) Tools made of cement or stone, it is use to give shape to the pots and also use to make the surfaces smooth. In local term it is known as “Bolia”.

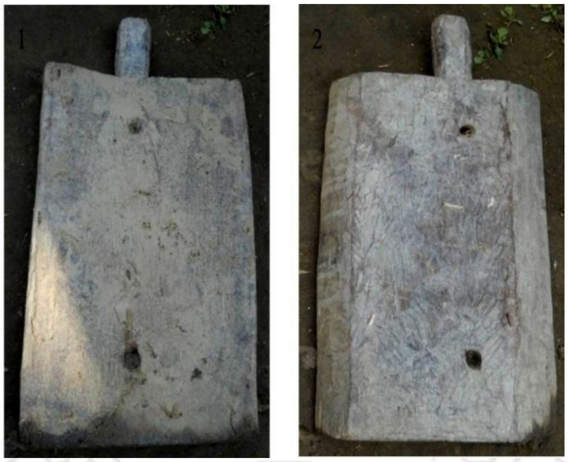

Figure 4.5 Wooden platforms (Pira): used for kneading clay

Figure 4.6 Oven (Peghali): 1) Firing place or kiln (Kumargaon-Dergaon) big hole is use to place the fire wood and 2) other hole is the place where pots are keep for firing. 3) and 4) Kumargaon-Dhekial

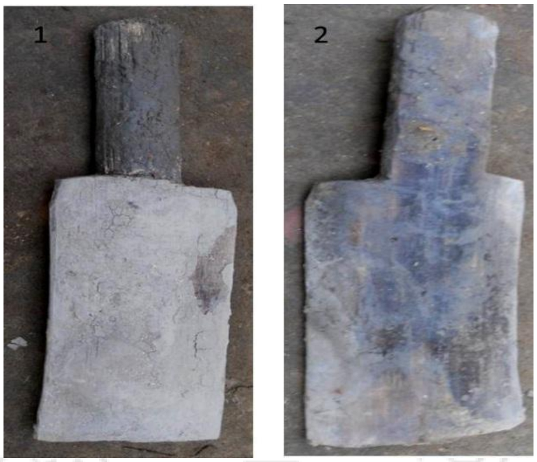

Figure 4.7 Wooden beater: front and Back view (it is lacally known as Pitani, use to give shape to the wares)

4.3 Forming, Finishing and Decorating of pottery

In hand-moulding, Hira potters initially make circular flat base (like a disc) of approximate 15-20 cm diameter (figure 4.8). Then, they dry base for whole night (approximately 12 hours). After this, they pressed in between thumb and fingers. Further, they keep base on anvil and it is beaten anti-clockwise by beater. Results of this, it expands in size. The edges are lifted up to give it a bowl shape (figure 4.8) for making lower part of pot. After lower part is done, they make coils of clay and by joining coils in lower portion neck and rim part of pot is made.

In next steps, they polish pottery by a piece of cloth (wet with mixed of water and clay). Sometimes, Hira potters engrave, perforate and cut facets as per to get desired design. Engraving is made by pointed tools on leather-hard pottery. Cut facets are done on half-dried pottery using sharp tool which has a knife-like blade. Perforation is made on unique type of vessel (locally called *'Jap Saru'*). This is used for cooking, especially for making cakes (Pithas) and to steam rice.

Figure 4.8 Hira potters' Manufacturing process 1. Base 2. Some finish products and bases 3. Coloring pots with Geru before firing 4. Unfired finished products stored in shed

The Kumar potters use wheel for making pottery. Only, menfolk of Kumar community is throwing pottery by using wheel, while other members of family including female help them to give product to final shape. In throwing, Kumar potter placed a lump of processed clay on rotating wheel head. Then, they manipulate lump of clay with his fingers to shaping an object into hollow ware. Duration of throwing pots depend on several humans and mechanical factors, such as speed of wheel, quality of clay and size of wares.

Both communities dry products in sunlight and sheds and time required for drying depends on weather conditions. But within a month, pots have to be fired. Both communities use common red ochrous clay (geru) for coloring wares. Generally, coloring is done just before firing pottery.

4.4 Firing Technique of Hira and Kumar potters of Assam

The simplest form of firing techniques is used by Hira and Kumar potters of Hirapara and Khehenipara respectively. Both potter communities use open firing method and without any fire-channel. The bottom layer is made with paddy straws or dry grass and leafs of eight-nine inch thick. Then husk is spread evenly on it and after this dry cake of cow-dung is piled. After that, they make a layer of firewood. If dung is not available then only firewood is used. Upon this, pots are arranged in circular fashion. Above pots, they again make few layers of husk, paddy straw, and ash and again husk respectively. These layers are made in such a way that all pots should be covered up. Then, pile is covered by mud paste or any other such materials subjected to protect lacks of cool air and to keep heat properly for enough time. The heap is burnt from all sides at once. The entire firing duration takes approximately one and half hour. And cooling is of two-three hours.

The firing technique of Kumar potters from Khehenipara is same as Hira potters from Hirapara. But, firing process of Kumar potters of Kumargaon-Dergaon and Kumargaon-Dhekial is different from Hira and Kumar potters of Hirapara and Khehenipara. Generally, potters of Kumargaon use oven firing technique. For this, they make a platform of soil or brick and it is circular, rectangular or oval in shape. In middle of platform, they make a hole and connect towards one end (Figure 4.6). In local term, it is called *'Peghali'*. The straws are placed on surface of oven and then layers of husk, woods, and dry plants are placed. After this, wares are piled and husk is spread in between. Again straws are spread to cover up wares. In last stage, heap is sealed with clay or mud and makes few stake-holes towards rear in rows and fired. Duration of firing is varied from one to two days.

Figure 4.9 Hira Pottery: 1) Piled for firing 2) Firing process 3) After firing

4.5 Mineralogical and Physicochemical analysis of Modern Pottery

In previous chapters (chapters 2 and 3), I have studied mineralogical and physiochemical properties of ancient pottery. In addition, source of raw materials and manufacturing techniques – processing of clay, forming, decorating and firing processes of ancient pottery were unknown.

While, in present chapter, contemporary pottery (known source of raw materials and known manufacturing techniques) and its' composition of raw materials such as clay and temper have been investigated by using scientific techniques. Results will be compared with ethnographical information.

This work investigates fabric of ethnographic pottery samples by using thin section petrography. FTIR, SEM-EDX and Powder XRD have been applied to determine firing temperature and atmosphere. Physical characteristics of analysed ceramics, such as hardness have been determined by using Mohs' scale and Micro Hardness Tester.

4.5.1 Petrographic Analysis

i. The description of thin section matrix of modern pottery

Five modern pottery samples have been used for thin section analysis. These modern samples were collected during field visit. Thin section photomicrographs of modern potsherds are given in figure 4.10. The details of thin sections' matrices of all pottery samples are following:

a. HIRAP-01

The matrix is composed of fine grain micaceous matrix. There are streaks of carbonaceous materials at some places. There is reddish irregular deposit that might be iron junk. Few irregular squarish colorless grains with a cloudy appearance are also found. It might be weathered Feldspar grains. Voids are also seen which may be found due to removal/decay of carbonaceous materials.

b. KKUP-01

Matrix of KKUP-01 contains numerous air bubbles. The central portion of slide contains irregular patches of fine opaque material and may be deposits of carborundum powder (abrasive). Voids spaces are observed; probably voids spaces impart porosity to materials. In general, material is composed of pale brownish micaceous fragments. Under cross nicol these fragments show high ochre interference color.

c. KKUP-02

The slide of KKUP-02 is composed of fine brownish micaceous matrix with numerous irregular voids spaces. Some of these contain irregular opaque impurities. There is extensive deposition of black carbonaceous materials and may be a remnant of charcoal or burnt vegetative materials. There is minor amount of Quartz distributed unevenly over matrix.

d. DKUP-04

Matrix is composed of deep brown color materials which contains irregular and discontinuous light brown micaceous streaks. There is extensive distribution of angular Quartz grains. Few rounded opaque bodies are also observed, it may be due to present of carbonized vegetative remains.

e. DKKUP-07

The slide is composed of very fine opaque materials and contain fine Quartz particle. There are few irregular voids spaces which have a black internal lining.

ii. Mineralogical analysis of contemporary pottery

Mineralogical composition of potsherds depends on composition of the raw material and on firing conditions. Minerals identified by Powder XRD are (Table 4.1) Quartz, Kaolinite, Goethite, Hematite Biotite, Rutile and Orthoclase in all ethnographic pottery samples. Powder-XRD indicates that Quartz is predominant in all analyzed pottery samples. Kaolinite is second dominant minerals identified in all pottery. Pottery samples from Khehenipara (KKUP-01 and KKUP-02) have good amount of Kaolinite in their constituent. Goethite is present in all pottery samples. Tabulated data (table 4.1) indicates that Hematite is present only in KKUP-01. Biotite is presnt in KKUP-01 and KKUP-02. Rutile is identified in KKUP-02, DKUP-04 and DKUP-07. Orthoclase is present in HIRAP-01 and KKUP-02. Furthermore, minerals identified by FTIR are Quartz, Hematite and Microcline in all samples.

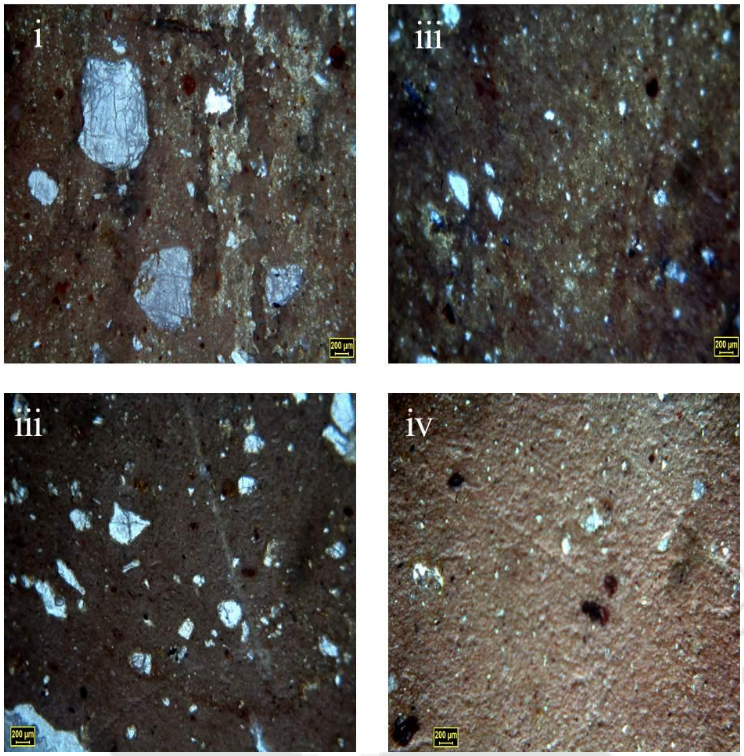

Figure 4.10 Thin section photomicrographs of (i) Hira-01 (ii) KKUP-01 (iii) DKUP-04 and (iv) DHKUP-07

Table 4.1 Minerals Identified by Powder X-Ray Diffraction of modern pottery (+++: abundance, ++ present in good amount, + present, -: absent)

Minerals Identified by XRD analysis of Modern pottery samples from Assam								
Si. No.	**Sample name**	**Quartz**	**Kaolinite**	**Hematite**	**Goethite**	**Biotite**	**Rutile**	**Orthoclase**
1	HIRAP-1	+++++++++	+	+	+	-	-	+
2	KKUP-01	++++++++	++++	++	+	+	-	-
3	KKUP-02	++++++	++++	+	+	+	+	+
4	DKUP-04	++++++++++	++	+	+	-	+	-
5	DKUP-07	++++++++	++	+	+	-	+	-

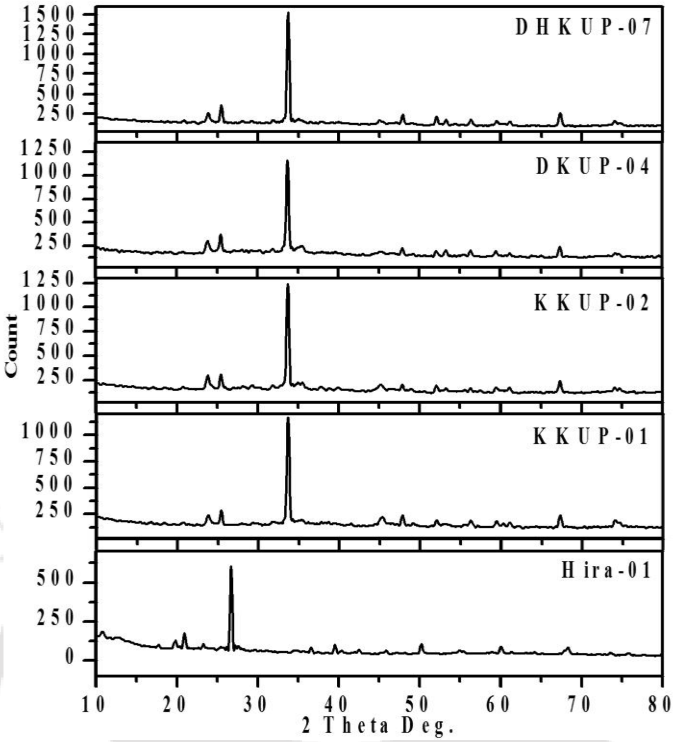

Figure 4.11 XRD Pattern of Modern Pottery: Hira-01, KKUP-01, KKUP-02, DKUP-04 and DHKUP-07

4.6 Infrared analysis of Modern Pottery

The FTIR spectra of pottery samples of five modern pottery shown in figure 4.12. Peak positions of various bands of varied intensity and tentative assignments are given in Table 4.2.

Medium to strong spectra around 3426 cm^{-1} and a weak to medium spectra around 1630 cm^{-1} (observed in all samples) is due to O-H stretching and O-H bending of absorbed water molecule in samples.

Weak spectrum around 2950-2850 cm^{-1} is probably due to aliphatic C–H stretching bands as observed in organic materials.

Infrared bands around 1400 cm^{-1} and 1300 cm^{-1} are due to carbonate overtone/ combinations detected in specimens, such as KKUP-01, 02; DKUP-04 and DHKUP-07.

Very strong bands around 1040 cm^{-1} are due to red clay origin which has been detected in all modern potsherds.

Quartz has been identified due to weak bands around 798 cm^{-1}, 778 cm^{-1} and 694 cm^{-1} present in all examined pottery.

A very weak peak at 558 cm^{-1} and weak to medium spectra around 475 cm^{-1} is attributing to presence of Hematite. Hematite is present in all analyzed pottery samples indicate that pottery are fired in oxidizing condition (Velraj *et. al.* 2009).

Presence of absorption band in pottery samples (Hira_P and DKUP-04) around 460 cm^{-1} is due to presence of Microcline.

Table 4.2 The IR spectra and probable assignment of modern pottery samples (Hira_P, KKUP-01, KKUP-02, DKUP-04 and DHKUP-07) were recorded in ARS (As Received State).

Hira-01 (Hira_P)		
Peak position (cm^{-1})	**As received State**	**Tentative Vibrational Assignments**
3471	Medium	O-H stretching of absorbed water molecule
1640	Weak	H-O-H bending of water
1047	Very Strong	Si-O-Si (Kaolinite)
797, 773	Weak	Si-O of Quartz
694	Very Weak	Si-O of Quartz
461	Very Strong	Microcline
KKUP-01		
3439	Medium	O-H stretching of absorbed water molecule
2923, 2850	Very Weak	C–H stretching
1653	Weak	H-O-H bending of water
1451	Very Weak	Carbonate overtone/combinations
1033	Very Strong	Si-O-Si (Kaolinite)
798, 774	Very Weak	Si-O of Quartz
694	Very Weak	Si-O of Quartz
477	Weak	Fe_2O_3 (hematite)
KKUP-02		
3439	Medium	O-H stretching of absorbed water molecule
2923, 2850	Very Weak	C–H stretching
1684	Medium	H-O-H bending of water
1447	Very Weak	Carbonate overtone/combinations
1025	Very Strong	Si-O-Si (Kaolinite)
798, 772	Very Weak	Si-O of Quartz
475	Medium	Fe_2O_3 (hematite)
DKUP-04		
3439	Strong	O-H stretching of absorbed water molecule
2923, 2853	Very Weak	C–H stretching
1633	Medium	H-O-H bending of water
1454, 1339	Very Weak	Carbonate overtone/combinations
1025	Very Strong	Si-O-Si (Kaolinite)
797, 675	Very Weak	Si-O of Quartz
460	Very Weak	Microcline
DHKUP-07		
3439	Strong	O-H stretching of absorbed water molecule
2923, 2853	Very Weak	C–H stretching
1637	Medium	H-O-H bending of water
1460	Very weak	Carbonate overtone/combinations
1020	Very strong	Si-O-Si (Kaolinite)
798, 694	Very weak	Si-O of Quartz
558	Very weak	Fe_2O_3 (hematite)
475	Weak	Fe_2O_3 (hematite)

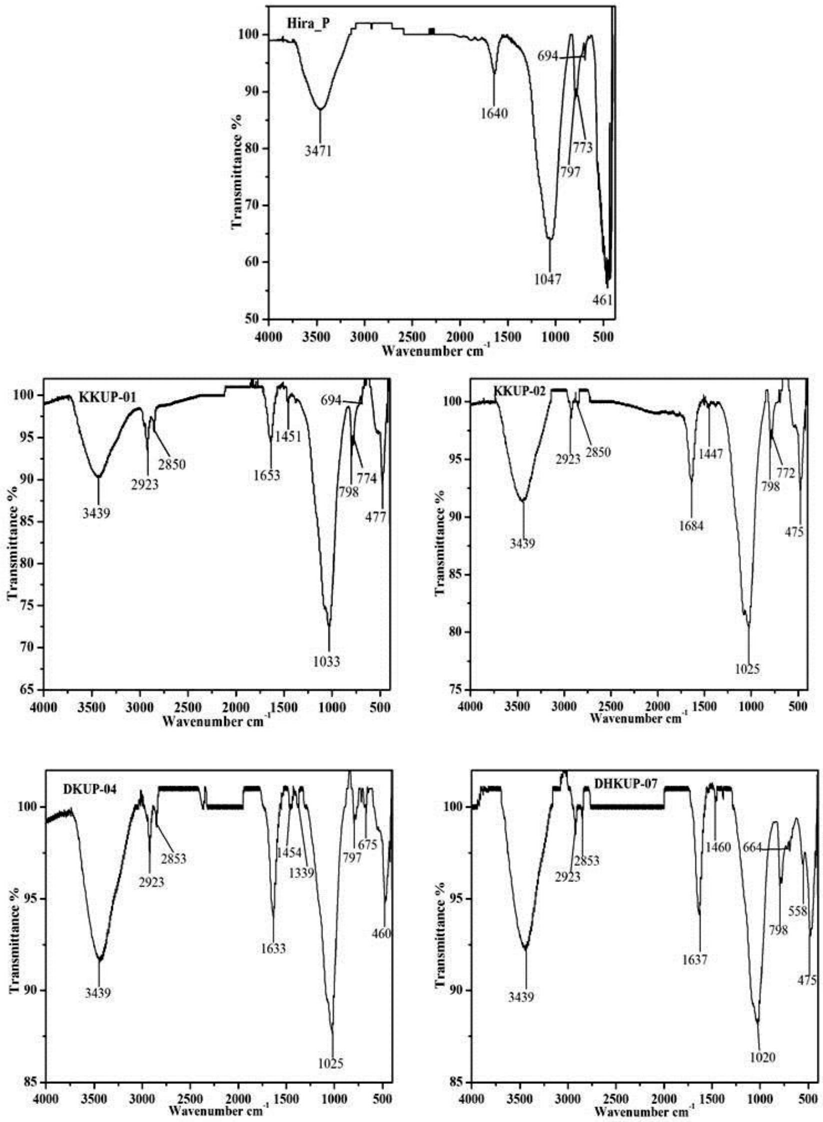

Figure 4.12 FTIR graph of Modern pottery: HIRAP-01, KKUP-01, KKUP-02, DKUP-04, DHKUP-07

4.7 Elemental analysis of Modern pottery by means of EDX

4.7.1 Energy-dispersive X-ray analysis

According to tabulated data of EDX (table 4.3), amount of SiO_2 varies between 48.91 and 55.57 oxide wt. % for modern pottery. From EDX analysis, it is evident that DHKUP-07 has highest amount of Silicon oxide in their composition and KKUP-01 has lowest weight percentage in their composition. Amount of Al_2O_3 contain approximately similar in all pottery samples ranging from 26.88 to 29.09 oxide wt. %. Table showed that iron oxide is present in good amount in all modern pottery which is ranging from 5.84 to 11.50 oxide wt. percentage.

Table 4.3 EDX results of Modern pottery

Sample Name	**Oxide (Weight %)**							
	Na_2O	**MgO**	**Al_2O_3**	**SiO_2**	**TiO_2**	**CaO**	**K_2O**	**Fe_2O_3**
HIRAP-01	0.59	1.17	26.81	52.40	3.49	1.08	3.58	10.88
KKUP-01	1.01	2.46	26.88	48.91	2.82	1.36	5.06	11.50
DKUP-04	1.02	2.26	29.09	55.11	1.40	0.97	2.97	7.18
DHKUP-07	1.44	2.15	27.99	55.57	2.51	0.55	3.96	5.84

Amounts of Na_2O present in most of analyzed modern pottery samples ranging from 0.59 to 1.44 oxide wt. % have very low quantity in their composition respectively. Tabulated data show that presence of MgO in modern pottery is in low amount, ranging from 0.17 to 2.46 oxide weight %.

Presence of K_2O in all pottery samples ranging from 3.58 to 5.06 oxide wt. %. TiO_2 substances ranging from 1.40 to 3.49 oxide wt. % is present in all pottery samples. Presence of CaO in pottery samples is very low varies from 0.55 to 1.36 oxide wt. percentage.

4.8 Micro-indentation Hardness Testing (MHT)

Five pottery samples (1x1 cm^2 approx.) are used for micro hardness test. Table no. 4.4 and figure 4.14 show that pottery has a small and medium hardness value. From tabulated data, it is evident that most of analyzed pottery samples show medium hardness value. It varies from 46.33 HV to 40.50 HV. Microindentation test demonstrated that DHKUP-07 has hardest surface with 46.33 Hardness value and KKUP-02 has softest surface with 40.50 Hardness Value.

Table 4.4 Micro Hardness of pottery from Modern Pottery

Si. No.	**Sites Name**	**Standard deviation Hardness Value (HV) (500 gf. load)**	**Mohs' Scale (1-9)**
1	HIRAP-01	40.89	3
2	KKUP-01	41.13	3
3	KKUP-02	40.50	3
4	DKUP-04	45.00	3
5	DHKUP-07	46.33	3

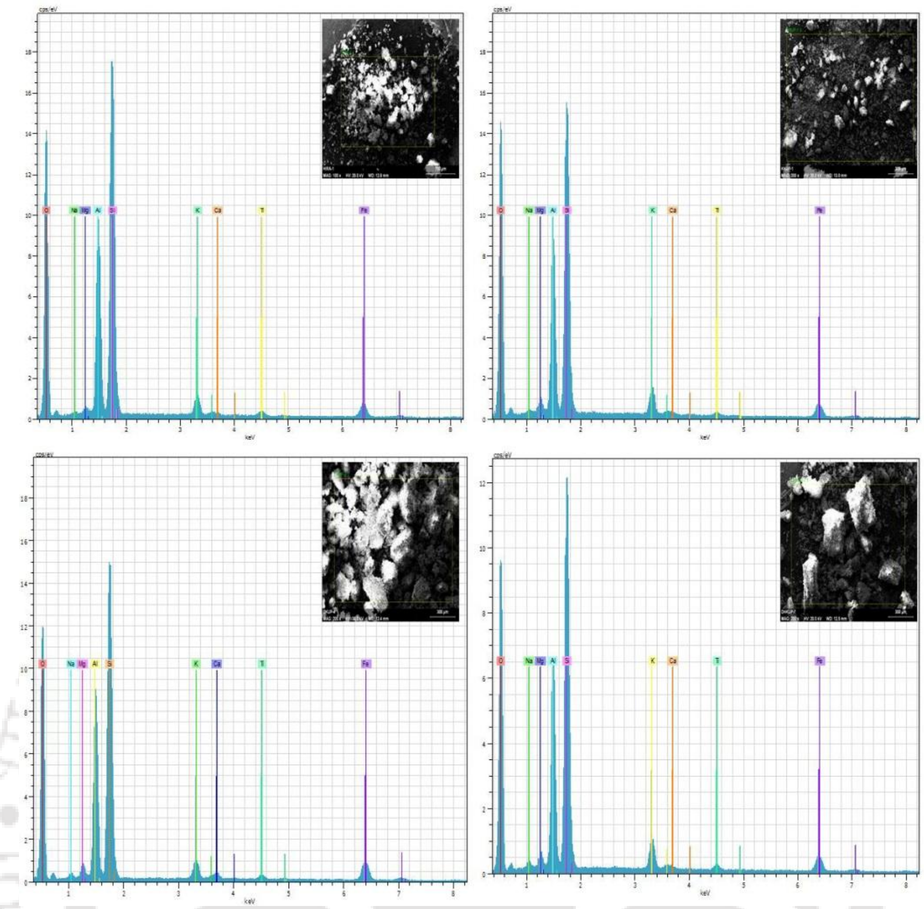

Figure 4.13 EDX Results of HIRAP-01, KKUP-01, DKUP-04 and DHKUP-07

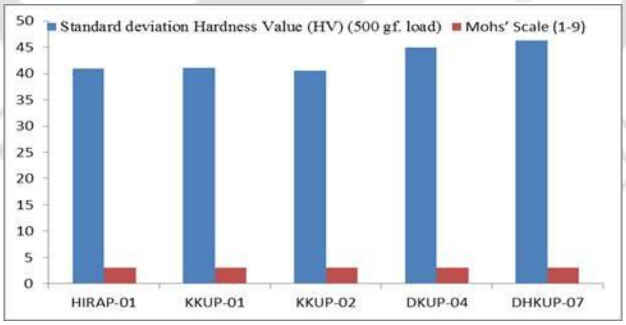

Figure 4.14 Variations of Micro Hardness Test and Mohs' Scale

4.9 Observation on technology of modern pottery

Observation on manufacturing technology of modern pottery has been obtained from fabric examination by petrographic microscope; chemical analysis FTIR, EDX; mineralogical study by Powder-XRD.

Petrographic results confirm that Quartz is primarily present in all samples. Besides Quartz, Feldspar is observed in all analyzed pottery samples. Organic material has been also observed in their composition. The results reflect that potters of both communities are adding sand as temper to improve properties of clay. Grains are mostly coarse in size and angular to sub-angular in shape. Matrices of all pottery are showing micaceous habits. Angularity of grains indicates that primary clay have been used for pottery production. The matrices of these pottery (figure 10) show poor sorting because size of grains is not uniform.

Thin section result indicates that all analyzed modern pottery samples show lighter red to reddish and brown color matrices. This indicates that these pottery might have been fired above 600 °C under oxidizing environment. Ethnographic study also indicates that both potter communities use open/oven firing methods. In open firing, air passes throughout firing process. Presence of organic matter and brown color matrices of pottery samples indicate that pottery was fired in short duration under oxidizing atmosphere above 600 °C. This is also confirmed by blackish to light greyish color core of all pottery samples. Matrices showed organic material is present in composition of pottery in irregular shape. It indicates that it occurs naturally because field report confirmed that traditional potter groups of Assam do not use any organic compounds during processing of clay. However, organic materials could be removed if pottery was fired for longer-duration in oxidizing condition. Ethnographic report reveals that entire process of firing of pottery in both communities is short time of period and not more than one and half hours. Often, they use quick-burning fuels such as straw, cattle-dung, husk, or brushwood.

Infrared results indicate that spectrum around 3700 cm^{-1} is absent in all pottery samples. As discussed earlier, this band is reflects destruction of clay structure when subjected to fire above 450-500 °C. Absence of this band indicates that all modern pottery samples are fired above 450-

500 °C. The very strong bands around 1040 cm^{-1} are present in all pottery suggests that firing temperature should be around 650 °C.

Presence of Hematite is due to IR band around 561 cm^{-1}-535 cm^{-1} and 475 cm^{-1} in KKUP-01, KKUP-02 and DHKUP-07 respectively. It is suggests that all pottery were fired in open air or oxidizing atmosphere during manufacturing. Presence of Hematite reveals existence of iron oxide which also confirms firing temperature as above 600 ºC. This is also confirmed by ethnographic investigation that air passes throughout firing duration. Infrared band around 460 cm^{-1} is assigned to Microcline that frees from any temperature effects and stable up to 500-800 °C. Presence of Microcline that is also confirmed by Powder-XRD, suggest that firing temperature of these analyzed pottery, would not been exceed 800 °C.

In addition, Powder-XRD, FTIR and thin section analysis have identified Quartz, feldspars mineral group and iron oxide as main composition of analyzed pottery.

SEM investigation has good agreement with mineralogical analysis obtained by Powder-XRD and thus confirming FTIR results. All investigated Scanning electron photomicrograph (figure 4.15) indicate that no sign of vitrification. SEM images (figure 4.15) of HIRAP-01, KKUP-01 and DHKUP-01 showed that clay has marginally rounding of edges. It is indicates that stage of no-vitrification and initial vitrification which occurred at temperature of 700-800 °C (Matau F. *et al.* 2013, p. 3). This is indicating that firing temperature of HIRAP-01, KKUP-01 and DHKUP-01 did not exceed above 800 °C, which is confirmed by thin section, XRD and FTIR investigation. While, DKUP-04 shows stage initial vitrification, which means it is fired in between 700-800 °C.

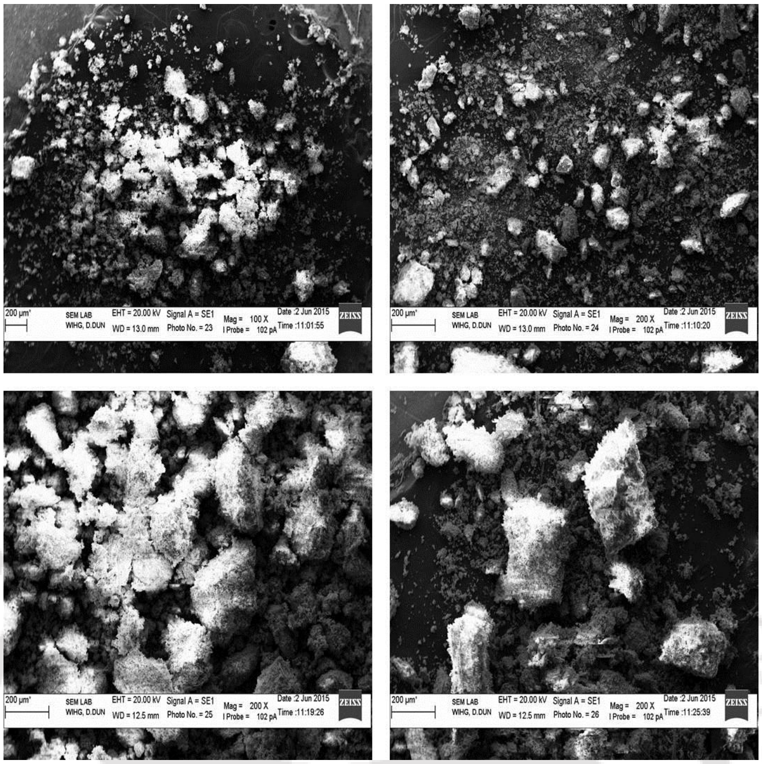

Figure 4.15 Scanning Electron micrographs of HIRAP-01 (no vitrification), KKUP-01(no vitrification), DKUP-04 (initial vitrification) and DHKUP-07 (no vitrification)

4.10 Conclusion

The earliest handmade pottery in Assam is known from Neolithic site of Daojali Hading is (dated to 2.7±0.3 ka, LD1728) two thousand seven hundred years old. Clay is not well levigated in Daojali Hading pottery and it showed a Standard deviation hardness value (HV) (500 gf Load) of 23.99. Minerals identified by XRD, FTIR and Raman Spectroscopy in Daojali Hading clay are Quartz, Kaolinite, Goethite, Hematite, Biotite and Hornblende. Clay mineral Kaolinite is detected in all pottery samples. Also, this is confirmed by presence of Hematite and Anatase in Neolithic pottery samples. Presence of Hematite also confirmed that all potsherds are fired in oxidizing atmosphere.

The earliest date of wheel made pottery tradition in Assam comes from Ambari, Guwahati. The material dated was charcoal from Historical Levels Trench AGXI-2161; layer 3; depth 1.2m The date is 1030 ± 105 A.D (TF-1019). Pottery types reported from dated layer were kaolin white; (ii) buff; (iii) red; (iv) grey and (v) dark grey to black ware.

Minerals identified in Ambari pottery are Quartz, Kaolinite, Goethite, Hematite, Biotite, Hornblende, Orthoclase, K-Feldspars (Microcline/Orthoclase).

Matrix shows reddish color and is composed of a fine siliceous matrix together with appreciable amount of sub angular to rounded coarse siliceous Quartz particles. Larger grains are fractured and contain deposition of ferruginous materials within fractures. Numerous rounded to irregular deposits are seen and these have a prominent rim surrounding them.

In comparison to handmade Daojali Hading pottery clay is well levigated in modern handmade Hira pottery and it gave a Standard deviation hardness value (HV) (500 gf. Load) of 40.89. Minerals identified in Hira clay, using same techniques are Quartz, Kaolinite, Goethite, Hematite Biotite, Rutile and Orthoclase. The modern Hira potters too fire pots in oxidizing condition. Presence of same type of minerals in neolithic Daojali Hading clay and modern Hira clay means that clay used by potters of both the periods is same. But technique of processing clay has improved as is understood from description of thin section matrices of Daojali Hading Neolithic pottery and modern Hira handmade pots.

In Daojali Hading pottery matrix is formed by large sub rounded and fractured Quartz grains floating in a matrix which is composed irregularly and is distributed in reddish and opaque materials. Mineralogically larger fragments are composed of a mixture of fine Quartz and clayey materials indicating that these particles are derived from preexisting rock fragments. Whereas, matrix of modern Hira pottery is composed of fine grain micaceous material. There are streaks of carbonaceous materials at some places. There is a reddish irregular deposit, few irregular squarish colorless grains with a cloudy appearance which might be weathered Feldspar grains. The firing technique in both periods is same. Also not much change is noticed in design and decoration of pots. In Daojali Hading there are 3 types of pottery Cord-impressed, Stamped Dull-Red, Plain Brick-Red ware. The modern day potters no longer make cord-impressed ware but stamped dull red ware and plain brick-red wares are common even now. Thus, we see that craft has not developed technologically in last two thousand and seven hundred years.

Minerals identified in clay used by Kumar potters are Quartz, Kaolinite, Goethite, Hematite Biotite, Rutile and Orthoclase. The same sets of minerals were identified in Ambari pottery too.

Matrix is composed of fine brownish micaceous material with extensive deposition of black carbonaceous materials. There is minor amount of Quartz distributed unevenly over matrix. The firing temperature ranges between 650° to 950° in oxidizing atmosphere. There are no painted wares.

Here, we see in case of wheel made pottery too same type of clay has been used approximately one thousand years before. Changes are only noticed in technique of processing clay. In Ambari matrix sub-rounded to rounded Quartz grains are visible while in modern pottery Quartz grains are less but more organic remains are present. Firing temperature ranging between 650° to 950° in oxidizing atmosphere is true for potteries of both periods. Design and decoration have remained almost same in all these years.

Individual inventiveness is essential to technological innovation. Social and economic forces strongly influence what technologies will be undertaken, paid attention to, invested in, and used. Such decisions occur as a consequence of circumstances and values of a society at any particular time. In case of Assam we see that in no particular time there was any pressure on potter's community to improve or innovate. Most technological innovations spread or disappear on the basis of how people respond to such innovations. For any tradition to evolve it must be relevant to the community, it has to be continuously recreated and transmitted from one generation to another. Did this fail to happen in Assam?

The most wide-spread explanation in evolutionary biology about fact that life goes on but evolution does not happen is stabilising selection. It suggests that advantage for a species which is already well adapted to its environment will be to avoid changing much. It is a type of natural selection that favours the average individuals in a population because changes are disadvantageous. Changes are a drawback and stabilising selection will discard deviations from the well-functioning norm. A similar situation in history of pottery craft in Assam can be visualized. Change was avoided. Change was avoided or not required because maybe there were other materials like bamboo and wood which were used for the same purpose.

From the modern context one exception to this rule was marked. Kumars of Dhekial village, Golaghat district prepare a clay oven for firing pottery. This pottery shows initial vitrification, which means fired in between 800-850 °C.

Dhekial is especially known for jaggery or molasses sold in long necked ‘Dhekial Koloh’ or pot. Dhekial was famous for its good quality jaggery from historical times. In recent years as sugarcane plantations have been converted too tea gardens molasses manufacturing in area has come down. Otherwise this item was traded all over Northeast India in these long necked globular jars. For this, they make a circular boundary of clay or brick. It is circular, rectangular or oval in shape. In middle of circle, they keep a hole in local term, called *‘Peghali’* for letting smoke out. Then straws are placed on surface of oven and then layers of husk, woods, and dry plants are placed. After this, wares are piled and husk is spread in between. Again straws are spread to cover up wares. In last stage, heap is sealed with clay or mud. Few holes are made at the bottom and in rows and fired. Duration of firing is varied from one to two days. This is only instance of firing pottery using a kiln in Assam. The antiquity of this technique is not known but references of molasses from Dhekial sold in these special long necked pots are found in oral traditions of Assam tracing back to a historical past. No evidence of any amount of vitrification is known from archaeological pottery of Assam recovered till date.

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Chapter 5
Conclusion

The application of scientific techniques or archaeometric techniques or more precisely analytical methods in studying archaeological pottery to understand its manufacture, use and provenance is developed as a methodology in archaeological studies today. The mineralogical and physiochemical studies of pottery are greatly enlightening to the scholars. The ethnographic and ethnoarchaeological studies of modern pottery and potter communities have greatly helped the researchers to understand the technological aspects of ancient pottery production techniques. For example, hand moulding, throwing, decorating, colouring the pot, firing processes, and marketing of finished product and usage of pottery in the societies. However, compositional study of materials from prehistoric reference has developed as a significant module of the most systematically oriented archaeological analysis.

One of the main contributions of the thesis is the methodology developed for understanding provenance of archaeological pottery. For understanding the matrix of clay thin section petrography has been used. For mineral characterisation XRD, FTIR and Raman Spectroscopy has been used. For elementary analysis EDX and LIBS has been used. For ascertaining the firing range FTIR, SEM, Raman Spectroscopy and TG-DSC has been used and for ascertaining the hardness of the potsherds MHT and Moh's scale has been used.

From the Northeastern part of India and especially from Assam, the only pottery type known is the Neolithic Chord marked pottery from Daojali Hading dated to 2.7±0.3 ka (LD1728), . The 'Ambari ware' is the next type known dated to 1030 ± 105 A.D (TF-1019). But in all these archaeological sites there are no sturdy storage jars or prolific occurrence of a particular design or shape. Pottery found is mostly plain. Colors are different shades of red and black. Medium sized globular pots, 20-30 cm in height with a diameter of 15-20 cm in the mouth are most common in both the Neolithic, Megalithic and Historical period sites. Another feature of this pottery finds is that they are highly fragmented. The handmade and the wheel made tradition existed parallely but certain points of convergence were also marked. The thesis attempted to map the development of the craft of pottery in Assam starting from a known past.

This was done by using provenance analysis. The results of the provenance analysis indicated that starting from a known past both the wheelmade and the handmade tradition have not changed. Techniques of manufacture, shape, design have remained almost same for the last 3000 yrs. Why has the craft not evolved? For any tradition to evolve it must be relevant to the community, it has to be continuously recreated and transmitted from one generation to another. Did this fail to happen in Assam?

5.1 The Neolithic Pottery

The combination of Petrography, Powder XRD, FTIR, OSL, micro-Raman Spectroscopy, SEM-EDX, LIBS, MHT and Scratch hardness (Mohs' Scale) have been applied to mineralogical and physiochemical investigation of Neolithic pottery of Northeast India.

The chronology of the Neolithic sites studied ranges between Cal BP 6410 to 6290 from Ranyek Khen, Nagaland to 2.3 ka of potsherds from Gawak Abri, Meghalaya. This is a timespan of four thousand years during which Neolithic dwellers of Northeast India used pottery together with ground stone implements which includes adzes, axes, double shouldered celts, grinding stones, querns and mullers. As per the chronology ascertained Ranyek Khen is the oldest site followed by Nongpok Keithelmanbi, Manipur (4460±120BP), followed by Law Nongthroh, Meghalaya (BP 3170 to 2990), Daojali Hading, Assam (2.7 ka) and Gawak Abri, Meghalaya (2.3 ka).

Thin section petrography coupled with polarizing microscope has revealed information regarding wide variety of natural and artificial clay minerals, inorganic and organic, materials which occurred naturally or inclusions as temper in neolithic potsherds from Northeast India. Mineralogically, Quartz, Feldspars, silt stone, clayey and siliceous materials, ferruginous materials and organic substances have been identified. These materials first of all reflects properties of local geology of place of manufacture of pottery, but also potters' habits, which largely determined use of particular kinds of temper and addition of artificial components, in an effort to improves properties of clay and to impart particular properties to pottery. Use of organic substances, Quartz grit and sand as tempering materials are also confirmed by past studies on neolithic pottery (Sharma, 1967).

Micro Raman analysis revealed that Quartz and Anatase are present in almost all potsherds. Other minerals such as Plagioclase, Hematite, Wollastonite, organic materials, Apatite and Gypsum have been identified. The presence of Anatase, carbonate and Hematite indicate the firing range between 500-700 °C and oxidizing atmosphere. Raman spectroscopy results have good agreement with other experiments such as FT-IR, XRD, thin section and SEM-EDX.

SEM analysis is revealed that no vitrification occurred in all Neolithic potsherds which indicate that all samples might have been fired below 800 °C. In addition, EDX data showed SiO_2, Al_2O_3 and Fe_2O_3 are occurred as primary constituent in all samples. EDX analyses are also revealed that in divergent concentrations of Ca, Na, Mg, Ti, Cl, K, S, and P occurred in all Neolithic pottery. The high content of silica and alumina rich constituent indicates high quantity of Kaolinite is used to manufacture analysed Neolithic potsherds. Powder XRD results also revealed good amount of Kaolinite in composition of analysed pottery. The presence of Feldspars mineral group is confirmed by thin section and Powder XRD analysis. Report of Geological survey of India (GIS) supports above findings (Kesari, 2011). In studied region, deposition of both minerals Kaolinite and weathered Feldspars are well known. The deposition of Kaolinite and weathered Feldspars indicate that analysed pottery manufactured locally. It is also confirmed by high content of Magnetite, Hematite and goethite in samples (Kesari, 2011).

It is also concluded that Neolithic potters of studied region used open firing techniques. As discussed earlier all methods of open firing differ from approximately 400-700 °C. The firing temperature of Neolithic pottery has been determined using various analytical methods. Combining results of applied various techniques revealed that all potsherds were fired in range of 500-700 °C.

5.2 The Historical Pottery

The same analytical techniques have been used for analysing the historical pottery from Ambari, Sri Sri Suryapahar, Bamuni Hills and Kangla Fort. Thin section petrography analysis revealed that both type of clay (primary and residual clay) might have been used to produce analyzed potsherds. On the basis of mineralogical content, all analyzed potsherds have been characterized into fifteen distinguished fabric groups and sub-groups. Mineralogically, Quartz, Feldspars, silt stone, mud stone, clayey and siliceous materials, ferruginous materials and organic substances have been identified in varying composition. Presumably, each distinguished fabric groups and subgroups revealed that the pottery was manufactured in the different workshops. The results of this work support the fact that these analyzed potsherds have been chosen from different sites, trench and layer and produced in different periods.

Thin section analysis revealed that sand, organic substances and Quartz grit might have been used as tempering materials. The FTIR result confirmed that two type of clay are used for pottery production. Kaolin of white clay origin and kaolin of red clay origin have been used. FTIR, TG–DSC analysis, micro-Raman spectroscopy and SEM analysis revealed that all potsherds have been fired in between 650°–900 °C under oxidizing atmosphere.

It is concluded that historical potters of studied region are used open firing, oven firing and sometimes kiln firing techniques.

5.3 Ethnographic Data

Documentation of contemporary potters of Assam, both handmade and wheelmade technique of manufacture indicated strong similarities between the archaeological data and the modern data.

In comparison to handmade Daojali Hading pottery clay is well levigated in modern handmade Hira pottery and it gave a Standard deviation hardness value (HV) (500 gf. Load) of 40.89. Minerals identified in Hira clay, using same techniques are Quartz, Kaolinite, Goethite, Hematite Biotite, Rutile and Orthoclase (table 5.1). The modern Hira potters too fire pots in oxidizing condition. Presence of same type of minerals in neolithic Daojali Hading clay and modern Hira clay means that clay used by potters or the handmade pottery of both the periods is same. But technique of processing clay has improved as is understood from description of thin section matrices of Daojali Hading Neolithic pottery and modern Hira handmade pots.

In Daojali Hading pottery matrix is formed by large sub rounded and fractured Quartz grains floating in a matrix which is composed irregularly whereas, matrix of modern Hira pottery is composed of fine grain micaceous material. The firing technique in both periods is same. Also not much change is noticed in design and decoration of pots. In Daojali Hading there are 3 types of pottery Cord-impressed, Stamped Dull-Red, Plain Brick-Red ware. The modern day potters no longer make cord-impressed ware but stamped dull red ware and plain brick-red wares are common even now. Thus, we see that craft has not developed technologically in last two thousand and seven hundred years.

Minerals identified in clay used by Kumar potters are Quartz, Kaolinite, Goethite, Hematite Biotite, Rutile and Orthoclase (table 5.1). The same sets of minerals were identified in Ambari pottery too.

Matrix is composed of fine brownish micaceous material with extensive deposition of black carbonaceous materials. There is minor amount of Quartz distributed unevenly over matrix. The firing temperature ranges between 650° to 950° in oxidizing atmosphere. There are no painted wares.

Table 5.1 Minerals Identified by Powder X-Ray Diffraction of selected potsherds (+++: abundance, ++ present in good amount, + present, -: absent)

Minerals Identified by XRD									
Si. No.	**Sample name**	**Quartz**	**Kaolinite**	**Hematite**	**Goethite**	**Microcline/ Orthoclase**	**Microcline**	**Anorthite**	**Biotite**
1	DJL (Handmade)	++++++	++	-	+	-	-	-	-
2	HIRAP-1 (Handmade)	+++++++++	+	+	+	+	-	-	-
3	AMB-01 (Wheelmade)	+++++++	+	++	+	-	-	-	-
4	KKUP-01 (Wheelmade)	++++++++	++++	++	+	-	-	-	+

Here, we see in case of wheel made pottery too same type of clay has been used approximately one thousand years before. Changes are only noticed in technique of processing clay. In Ambari matrix sub-rounded to rounded Quartz grains are visible while in modern pottery Quartz grains are less but more organic remains are present. Firing temperature ranging between 650° to 950° in oxidizing atmosphere is true for potteries of both periods. Design and decoration have remained almost same in all these years.

For any tradition to evolve it must be relevant to the community, it has to be continuously recreated and transmitted from one generation to another. Change was avoided or not required in the pottery craft of Assam because maybe there were other materials like bamboo and wood which were used for the same purpose.

The most wide-spread explanation in evolutionary biology about the fact that life goes on but evolution does not happen is stabilising selection. It suggests that the advantage for a species which is already well adapted to its environment will be to avoid changing much. It is a type of natural selection that favours the average individuals in a population because changes are disadvantageous. Changes are a drawback and stabilising selection will discard deviations from the well-functioning norm. A similar situation in the history of the pottery craft in Assam can be visualized. Change was avoided or not required because maybe there were other materials like bamboo and wood which were used for the same purpose.

Lignic is a term coined by Solheim II in 1969 for a period in Southeast Asian prehistory beginning with the early Hoabinhian, at about 42,000 B.P. It is hypothesized that during this period wood/ bamboo is the most important material for making tools rather than stone. This theory has not been proven, nor even tested archaeologically (Solheim 1970). Existence of such a stage in the prehistory of Northeast India was hypothetically stated based on the observations made on the contemporary Garo society (Sharma 2007). Using the same hypothesis the slow development of the craft of pottery making in Northeast India can be explained. Pottery traditions in Assam have always existed with the bamboo and wood craft tradition in the past as well as in the present.

The local environmental condition seems to have influenced the development of the distinctive character of the craft of pottery in this region. The sturdy storage jars, the thick walled cooking pots are all missing from the archaeological context here together with paintings and glazes. Mapping the evolution of the pottery craft in Assam was important for understanding the technological as well as the socio-economic development of the craft. Few technological changes have been marked but there have been no fundamental innovation or invention in its 3000 year (approx.) old history to bring in effective changes.

5.4 Limitations

The study claims to provide a complete picture of the provenance of the Neolithic and historical pottery of Northeast India. But this claim is truer for the Neolithic period only while for the historical period only one sample from outside Assam could be collected. But there have been successful excavations in historic sites of Manipur (Sekta) and Meghalaya (Bhaitbari). Our attempts to collect samples from these sites were unsuccessful. Similarly, ethnographic documentation of contemporary potters could only be done in Assam from where primary data (clay and pottery) were collected. Similar documentation could not be carried out in other parts of Northeast India due to the lack of resources. For future researchers this is a potential area of research which can throw further light on the evolution of the pottery tradition in Northeast India.

The present study is able to draw attention of scholars to the way in which studies of this type can provide facts which is valuable and vital for understanding pottery. As highlighted by the present study, the application of archaeometric methods provided here will expose new paths for the analysis of ceramic technology and the archaeology of Northeast India.

An application of a combination of archaeometric and ethnoarchaeological approach to traditional potters groups and tribes may provide an explanation of other archaeological issues such as site formation, depositional processes and settlement patterns in Northeast India which is outside the scope of this thesis.

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Appendix I
Map of archaeological sites of North East India

Appendix I

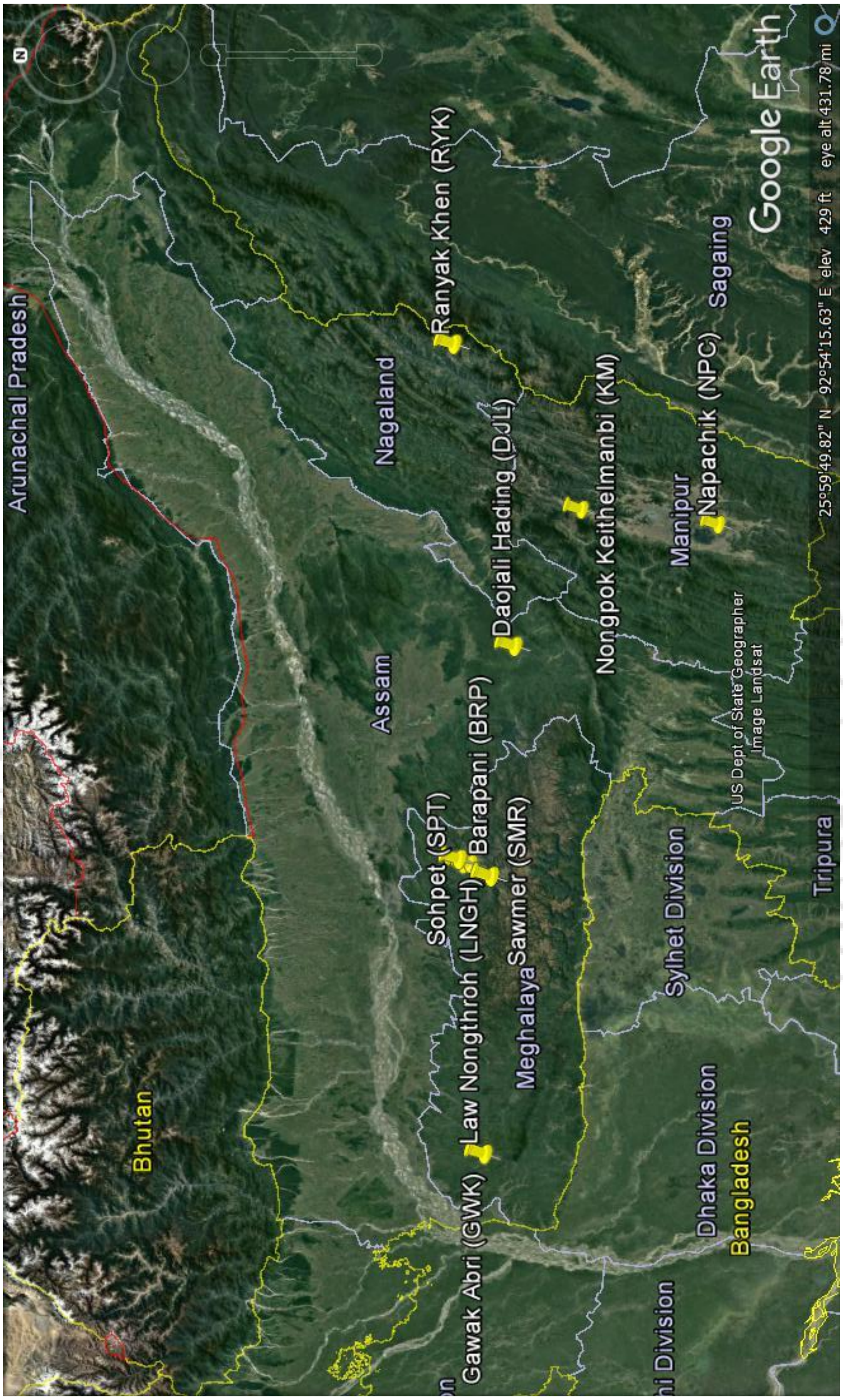


Plate 1 Map of Neolithic sites of Northeast India

Appendix I

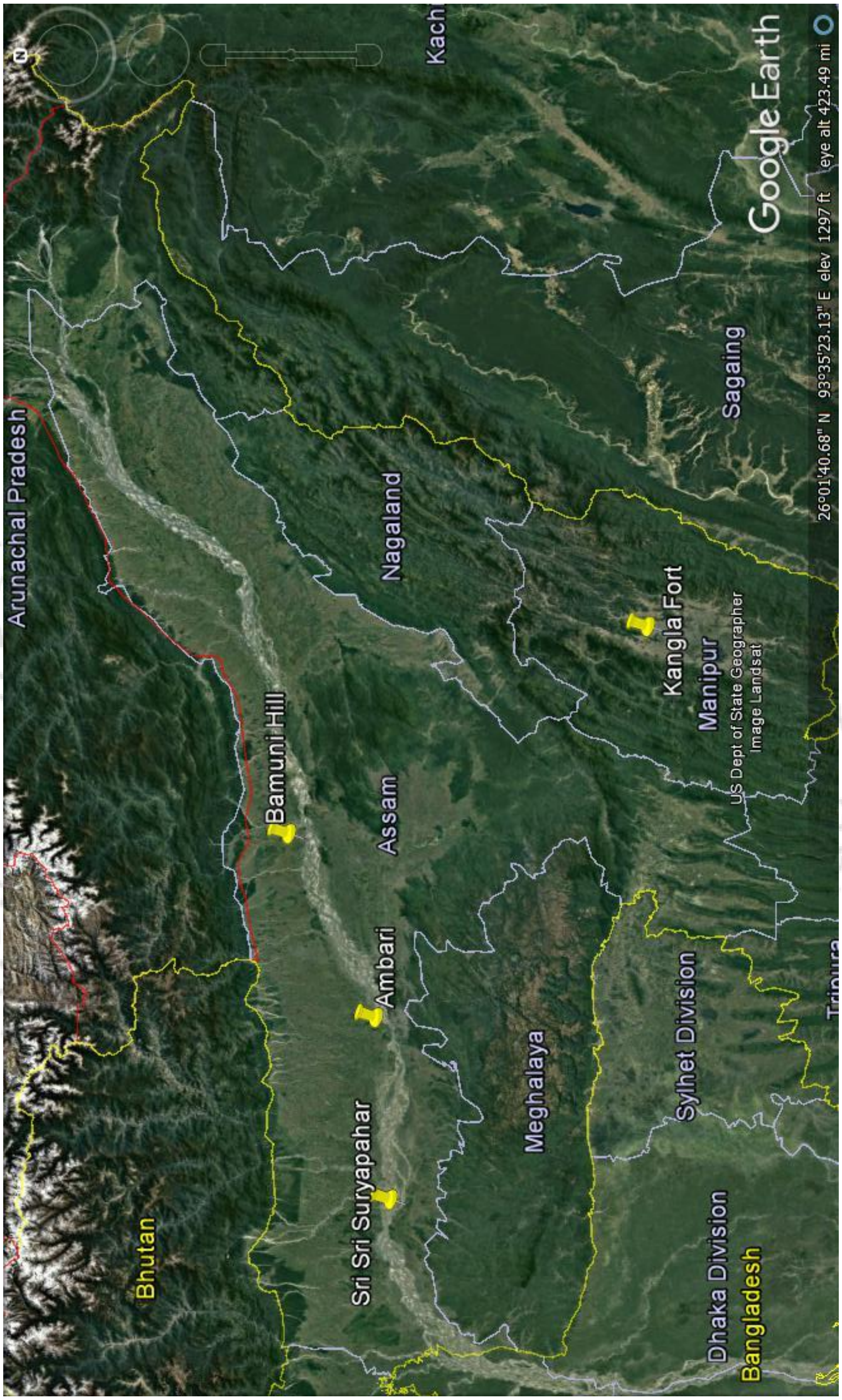


Plate 2 Map of Historical sites of Assam and Manipur

Appendix I

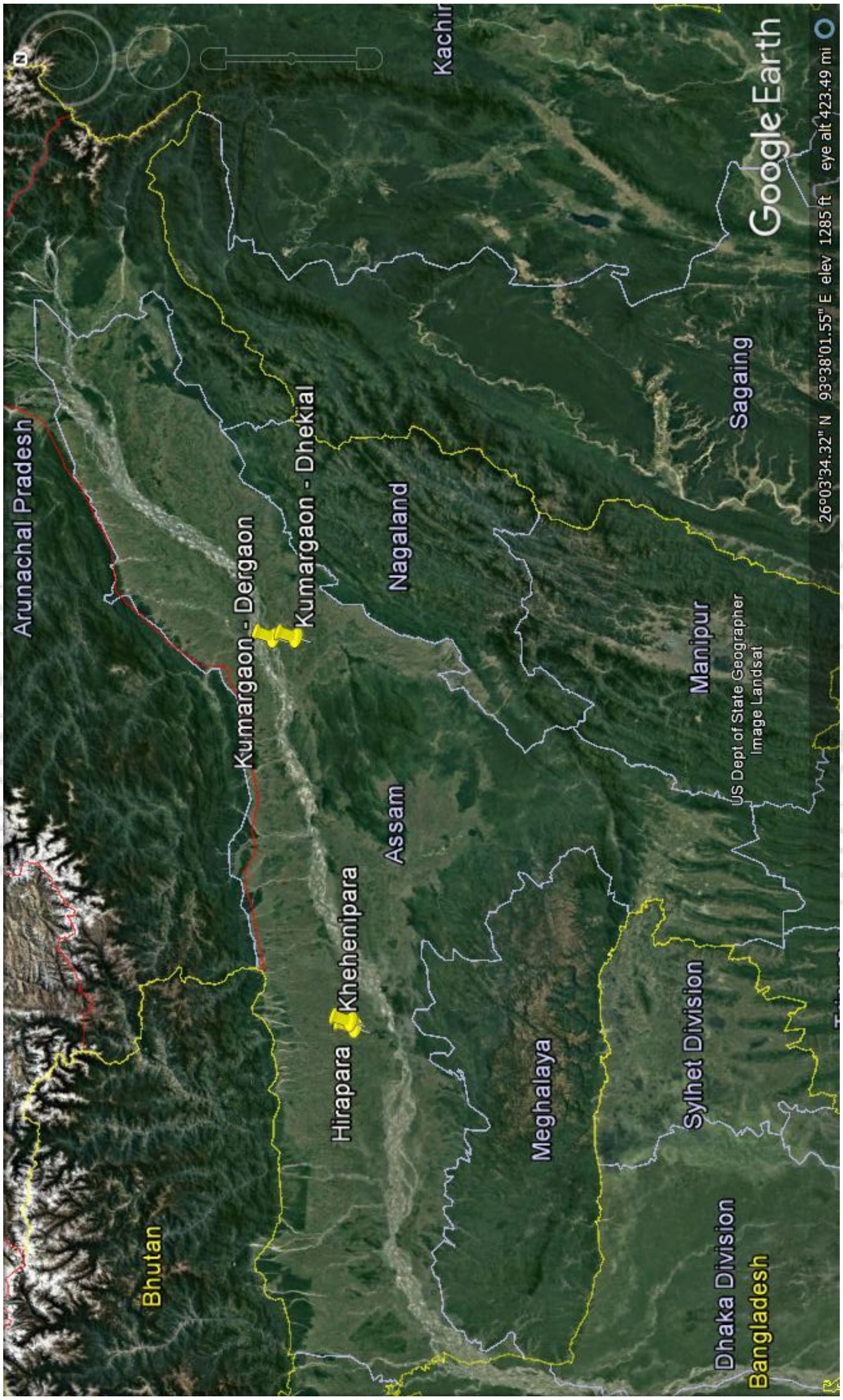


Plate 3 Map of Modern villages of Assam (field work conducted villages)

Appendix II

Morphology of Neolithic Potsherds

Appendix II

Table 1 Morphological Details of Neolithic potsherds

Si. N.	Sample No.	Technique	Texture	Color	Features
01	DJL	Handmade	Medium	Plain red	
02	BRP-01	Handmade	Medium	Red	
03	BRP-02	Handmade	Medium	Plain red	Groove pattern on outer surface
04	KM-01	Handmade	Very Coarse	Red	Groove pattern on outer surface
05	KM-02	Handmade	Medium	Red	Groove pattern on outer surface
06	KM-03	Handmade	Very Coarse	Red	Groove pattern on outer surface
07	LNGH-01	Handmade	Very Coarse	Light gray	
08	LNGH-02	Handmade	Very Coarse	Red	
9	LNGH-03	Handmade	Coarse	Pale red	
10	NPC-01	Handmade	Medium	Red	Grid pattern
11	NPC-02	Handmade	Medium	Dark gray	Grid pattern
12	NPC-03	Handmade	Medium	Pale red	
13	NPC-04	Handmade	Fine	Pale red	
14	NPC-05	Handmade	Fine	Pale red	
15	RYK-01	Handmade	Medium	Red	
16	RYK 02	Handmade	Medium	Red	Groove pattern
17	RYK-03, RYK-04, RYK-05	Handmade	Coarse	Red	
18	GWKC	Handmade	Thick body, very Coarse	Plain red	
19	SPT-01, SPT-02	Handmade	Very Coarse	Plain red	
20	GWKF	Handmade	fine, Thick body	Plain red	
21	SMR-01	Handmade	Coarse, thick body	Dark brown	Groove pattern
22	SMR-02	Handmade	Coarse, thick body	Plain red	
23	SMR-03	Handmade	Coarse, Thin body	Pale red	
24	SMR-04	Handmade	Coarse, Thin body	Red	Groove pattern
25	SMR-05, SMR-06	Handmade	Very Coarse	Plain red	
26	SMR-07	Handmade	Medium	Light brown	
27	SMR-08	Handmade	Medium	Light brown	Groove and Grid pattern
28	SMR-09	Handmade	Very Coarse, thin	Plain red	

Appendix II

Plate 4

Neolithic Potsherds: Daojali Hading (DJL), Gwak Abri (GWKC), Gawak Abri (GWKF), Nongpok Keithelmanbi (KM), Napachik (NPC) and Ranyak Khen (RYK)

Appendix II

Plate 5

Neolithic Potsherds: Barapani (BRP), Law Longthroh (LNGH), Sohpet Bneng (SPT) and Sawmer (SMR)

Appendix III

Morphology of Historical Potsherds

Appendix III

Table 1 Morphological Details of Historical potsherds: Ambari (AMB)

Si. No.	Sample No.	Technique	Texture	Color	Features
01	AMB-01, AMB-13	Wheelmade	Very fine, well levigated	Buff	Kaolin ware, Rim sherd
02	AMB-02	Wheelmade	Medium, thick body	Pale red	
03	AMB-03	Wheelmade	Fine	Plain red	Two concentric line inner the neck
04	AMB-04	Wheelmade	Medium	Plain red	Knob
05	AMB-05, AMB-06	Wheelmade	Very fine, well levigated	Plain red	Rim
06	AMB-07	Wheelmade	Very fine, well levigated	Buff	Kaolin ware, everted rim sherd
07	AMB-08	Wheelmade	Very fine, well levigated	Buff	Kaolin ware, Rim sherd
08	AMB-09	Wheelmade	Very fine, well levigated	Plain red	Body
09	AMB-10, AMB 18	Wheelmade	Coarser than sample no. 1 and 7	Buff	Kaolin ware, Rim
10	AMB-11, AMB-19, AMB-22	Wheelmade	Fine, thick	Red	Tile
11	AMB-12	Wheelmade	Fine, thick	Buff	Tile
12	AMB-14, AMB-16	Wheelmade	Fine, well levigated	Plain red	Body
13	AMB-15, AMB-25	Wheelmade	Very fine, well levigated	Buff	Kaolin ware, Body
14	AMB-17	Wheelmade	Fine, well levigated	Plain red	Everted rim sherd
15	AMB-20	Wheelmade	Fine, thick body	Plain red	
16	AMB-21	Wheelmade	Fine	Red	Everted rim, double concentric line around rim
17	AMB 23	Wheelmade	Very fine, well levigated	Plain red,	Body, concentric lines
18	AMB 24	Wheelmade	Fine	Plain red	Base
19	AMB-26	Wheelmade	Very fine, well levigated	Buff	Kaolin ware, Rim sherd, concentric lines outer surface of rim
20	AMB-27	Wheelmade	Medium	Red	Base
21	AMB-28	Wheelmade	Fine	Gray	Base
22	AMB-29	Wheelmade	Fine	Dark gray	Rim
23	AMB-30	Wheelmade	Fine	Plain red	
24	AMB-31	Wheelmade	Fine	Plain red	Rim
25	AMB-32	Wheelmade	Fine	Plain red	Knob
26	AMB-33	Wheelmade	Fine	Plain red	Base

Appendix III

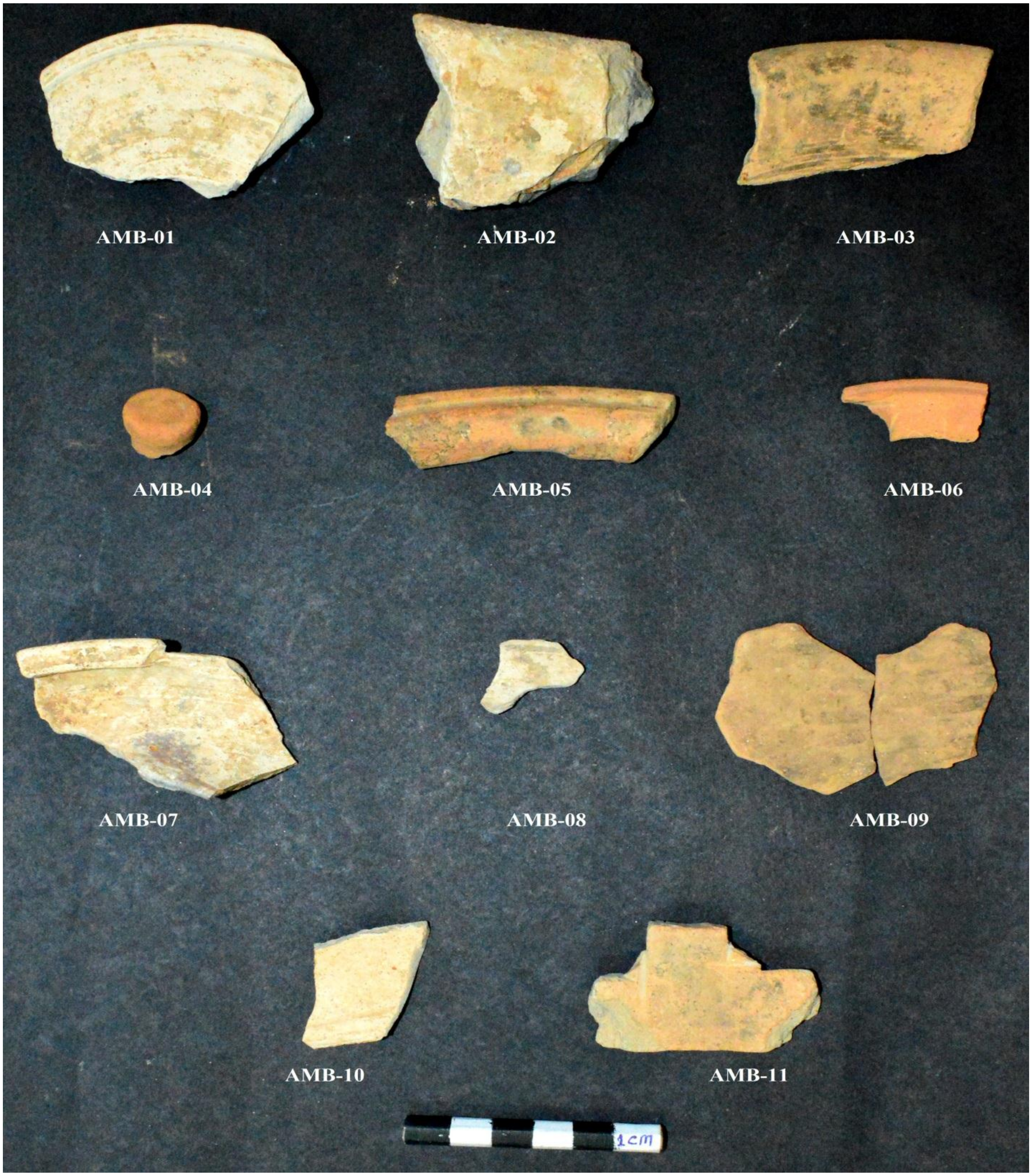
Table 2 Morphological Details of Historical potsherds: Bamuni Hill (BMH) and Sri Sri Suryapahar (SPR)

Si. No.	Sample No.	Technique	Texture	Color	Features
01	BMH-01, BMH-02, BMH-04, BMH-05	Wheelmade	Very fine	Plain red	
02	BMH-03	Wheelmade	Very fine, well levigated clay	Plain red	
03	SPR-01, SPR-02, SPR-0-3	Wheelmade	Medium	Plain red	
04	SPR-04	Wheelmade	Medium	Plain red	Concentric parallel line on rim
05	SPR-05	Wheelmade	Medium	Plain red	Parallel lines on body
06	SPR-06	Wheelmade	Coarse	Light brown on outer and Red in inner surface	
07	SPR-07	Wheelmade	Coarse, very thick	Plain red	
08	SPR-08	Wheelmade	Very fine, well lavigate		Everted Rim sherd
09	KGF-01	Wheelmade	Medium	Plain Red	Grooved pattern on body
10	KGF-02	Wheelmade	Medium	Red	Leaf pattern on body
11	KGF-03	Wheelmade	Medium	Black	Leaf pattern on body

Appendix III

Plate 6

Historical Potsherds: Ambari (AMB)



Appendix III

Plate 7

Historical Potsherds: Ambari (AMB)

Appendix III

Plate 8

Historical Potsherds: Ambari (AMB)

Plate 9

Historical Potsherds: Bamuni Hill (BMH), Kangla Fort (KGF) and Sri Sri Suryapahar (SPR)

Publication

1. **Singh, P**., Sharma, S., (2016) "**Thermal and Spectroscopic characterization of archaeological pottery from Ambari, Assam"** *Journal of Archaeological Science: Report*-5, pp. 557-563 (ISSN: 2352-409X).

2. **Singh P. (2015) "Ancient Pottery of Assam: A Study"** '*Anusilana: Research Journal of Indian Cultural, Social & Philosophical Stream'* Vol. LXV, PP. 117-124 (ISSN 0973 – 8762).

3. **Singh P. (2015) "Thin Section Petrographic Investigation of Ceramic in Archaeology",** in *'Recent Field Researches in Indian Archaeology & Literature'*, ed. By AK Dubey *et al*. Bookwell publication, Delhi, pp. 1-7 (ISBN**:** 978-93-80574-95-0).

4. Sharma S., **Singh P., "Using Luminescence Dating to Address issues with the Neolithic Pottery of Northeast India",** *communicated in Current Science.*

Conferences &Workshop

International Conferences

1. 28-30 December 2010: presented a paper entitled **"Cultural Adaptation: An analysis of the prehistoric sites of Northeast India"** in international conference organized by **Department of Ancient Indian History, Culture and Archaeology, University of Lucknow** and **International Research Institute of Buddhist Studies, Lucknow,** joint annual conference of **IAS, ISPQS and IHCS.**

2. 15-18 February 2014: paper presented entitled **"Neolithic Pottery of North-eastern Indian: A Study by using Thin Section Petrography, Micro Hardness Tester and Furrier Transform Infrared (FT-IR)"** in International conference of **Society of South Asian Archaeology** (**SOSAA)**, organized by **Directorate of Culture and Archaeology, Govt. of Chhattisgarh** in collaboration with the **School of Studies in Anthropology, Pandit Ravi Shankar Shukla University, Raipur.**

3. 06-09 October 2014: presented a paper entitled **"Handmade Pottery Tradition of Hira Potters of Assam"** in International Seminar on **Archaeology and** language the joint annual conference of **IAS, ISPQS and IHCS** held at **Deccan College post-Graduate & research Institute, (Deemed University), Pune**

National Conferences

1. 22-24 November 2012: Paper presented entitled **"Ancient Pottery of Assam: A Study"** in joint annual conference of **IAS, ISPQS Department of Archaeology and Ancient History, Faculty of Arts, the Maharaja Sayajirao University of Baroda**, Vadodara.

2. October-November, 2014: presented a paper entitled **"Ancient Pottery of Assam: A Study"** in Graduate Research Meet, organized by **Department Humanities and Social Sciences, Indian Institute of Technology Guwahati.**

3. April 24th to 26th 2015: paper presented entitled **"The Scientific Analysis of Artefacts in Archaeological Science"** in the national seminar organized by **Centre of Advanced Study**, **Department of AIHC & Archaeology; Banaras Hindu University with main theme, '*Recent Field Researches in Indian Archaeology & Literature'*.**

4. March 17th to 20th 2016: participated in poster presentation competition in **Research Conclave 2016**, organized by **Students' Academic Board, Indian Institute of technology Guwahati.**

Workshop

1. 18-24 March 2013: Attended a workshop on "**Analytical Strategy: An Experimental Approach**' organized by **Department of Ancient History, Culture & Archaeology, University of Allahabad.**

2. 15-22 March 2014: Attended a workshop on "**Experimental Lithic Knapping & Micro-wear Studies, a Regional Workshop for Archaeologists of Northeast India** jointly organised by the **Department of History & Archaeology, Nagaland University, Kohima Campus, & the Indian Council of Historical Research (ICHR), New Delhi**.

“Pottery tends to arouse strong emotions in archaeologists: they either love it or hate it… it has an indefinable fascination, and potentially full of information, which has to be teased out of it by careful and painstaking study”.

‘Pottery in Archaeology’ Orton, C., Tyers, P., & Vince, A. (1993), p. 3.

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