Microchemical Journal 95 (2010) 311–314

Contents lists available at ScienceDirect

Microchemical Journal

journal homepage: www.elsevier.com/locate/microc

Microscopy, porosimetry and chemical analysis to estimate the firing temperature of some archaeological pottery shreds from India

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ARTICLE INFO

Article history:
Received 17 November 2009
Received in revised form 18 January 2010
Accepted 19 January 2010
Available online 28 January 2010

Keywords:
SEM-EDS
Chemical analysis
Porosimetry
Firing temperature
Production technology
Pottery shreds

ABSTRACT

In recent times, science and technology have been applied to the world of cultural heritage, preservation and conservation. In the present study, the pottery samples belonging to 4th century were collected from Tandikkudi in Dindugul district of Tamilnadu, India and were subjected to chemical analysis to outline the information about the raw materials and technological–productive aspect such as firing temperature. Investigations such as Scanning Electron Microscope and Energy Dispersive Spectrometer (SEM/EDS) are also done for the accurate observation of the morphology and the qualitative and Semi-quantitative determination of the chemical elements present in the sample. The firing temperature of the samples at the time of manufacturing is also estimated from apparent porosity of the samples which agrees well with the SEM analysis. The results obtained from different analytical techniques on pottery shreds provide information of the firing temperature of the pottery which lies in the range of 800 °C–1000 °C in the oxidizing atmosphere. Moreover it was observed that the samples collected from this site are low refractory in nature and the artisans of Tandikkudi have used both calcareous and non-calcareous clays for their household utilities, but were unaware of firing their artifacts at reduced atmosphere or closed kiln. Hence this paper is a useful analytical tool for predicting the firing atmosphere, type of clay (calcareous, non calcareous) and its nature (fine and coarse). Hence this paper is suitable for estimating the firing temperature of ancient potteries.

1. Introduction

Archaeology, the study of ancient human society by reconstructing environmental settings and cultural systems, typically from their physical remains (masonry, pottery, coins, engravings, etc.). Archaeology often interacts with history to provide a broad view of the human experience. More knowledge about the great civilization is brought out with archaeological excavations in recent times. History of ancient civilizations cannot be written without archaeological evidence and the tradition [1]. Pottery is one of the most enduring materials known to mankind. In most places it is the oldest and most widespread art since pottery is a durable artifact, which was utilized by various cultures around the world. It has proven to be a boon for archaeologists. The study of pottery helps to identify the different cultural groups and their influence on one another [7]. Archaeological pottery throws ample light on nature, culture and the development of the civilization of the people. In the present study, archaeological pottery sample collected from Tandikudi 12°35′N, 76°53′ E site in Dindugul district, Tamilnadu, India. Various methods like Chemical analysis, SEM-EDAX, and Porosimetry have been employed to estimate the firing temperature and firing atmosphere.

2. Sample detail

Tandikkudi (12°35′N, 76°53′ E) is located in Dindigul district, Kodaikanal taluk at 47 km north-east of Vathalagundu at down station of Palani Hills in Tamilnadu, India. This site was excavated by the Department of Epigraphy and Archaeology, Tamil University, Thanjavur. This place has number of archaeological evidences belonging to the Iron Age. In addition to red and black wares beads made of Pavalam were also found in this site.

3. Methods

Water absorption method [12–14] was adopted to measure the porosity values of the pottery samples. The microphotographs of the sample were recorded using SEM JSM 561OLV JEOL. The maximum magnification possible in the equipment is 3, 00,000 times with the resolution of 3 nm. The elemental analysis was done using the OXFORD INCA Energy Dispersive X-ray spectrometer (EDS). The fresh fracture surfaces of the potteries in the received state (ARS) that were coated with the thin layer of platinum were examined using SEM, typically setting at a magnification of ×2000 for all the sample of study. The microphotographs were recorded with the attached accessories of high-resolution cathode ray tube and a role film camera [8]. Titration method was adopted to find the chemical composition of potteries for chemical analysis [4].

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doi:10.1016/j.microc.2010.01.006

4. Results and discussion

The archaeological pottery samples collected from the site are named TNK1, TNKK2, TNK3, TTK4 and TNK5 respectively. The samples were analyzed and its firing atmosphere and firing temperature was determined.

4.1. Firing temperature estimation using chemical analysis

Chemical analysis is a well established procedure and the chemical composition of potteries have been estimated for the samples collected from Tandikkudi archaeological sites using Titration method. The chemical composition can be used to define the pottery of a particular area and people by determining the raw materials used.

To know the type of clay minerals (Calcareous/Non-Calcareous and either low or high refractory) and to determine the firing atmosphere adopted by the artisans at the time of manufacture, the chemical analysis was performed on the samples. The clays mainly have the concentrations of silica, (SiO_2), alumina (Al_2O_3) and fluxes (K_2O, Fe_2O_3, CaO, MgO and TiO_2) as the composition. The nature of clay minerals whether calcareous or non calcareous clay can be identified from the percentage of Calcium Oxide (CaO). According to [5] the clays containing CaO greater than 6%, are known as Calcareous clays and CaO less than 6% are known as Non-Calcareous clays. If the fluxes concentration (K_2O, Fe_2O_3, CaO, MgO and TiO_2) are more than 9% the clays are classified as low refractory and classified as high refractory [5] if the fluxes in the sample are less than 9%. From the ratio of iron oxides (Magnetite–Fe_2O_3 and Hematite–Fe_3O_4) present in the sample the atmospheric condition of the sample fired at the time of manufacture can also be identified. As suggested by [2] if the amount of magnetite present in the sample is large then it may be fired at reducing atmosphere. If the amount of hematite is high it may be fired at oxidizing atmosphere. With these characteristic features, the results of the archaeological potteries under the present study are analyzed with a view to correlate the SEM pattern with firing temperature ranges attained by the excavated potteries at the time of manufacture [5].

From Table 1, it is obvious that, the concentration of Silica is more due to the presence of various amounts of Quartz in all the samples of interest. A part from silica, the most abundant impurity elements are Al, Ti, K, Mg, Fe and Ca. The composition of Fe and Ca determines the nature of clay minerals and firing atmosphere adopted by the artisans. The percentage of CaO ranges from 1.40–3.38% in TNK1–TNK3, are of Non-Calcareous type. The remaining Tandikudi samples TNK4 and TNK5 have CaO% of 6.92 and 6.96 respectively hence these two samples alone are of Calcareous type [5] and the variation in concentration of CaO and Magnetite and Hematite is shown in Figs. 1 and 2. The percentage and distribution of the iron oxides are the principal cause for color of the potteries. Red color of the pottery is due to the presence of hematite and black color is due to more amount of magnetite in the samples [8,10,15]. Here an attempt has been made to correlate the color of the potteries with the state of iron content. The samples TNK1 and TNK5 alone Red and Black ware which may be due to the presence of carbonaceous material and all other remaining samples are Red/Red slipped ware, hence the atmosphere prevailed by the artisans at the time of manufacture may be Oxidizing atmosphere [2].

The samples collected are low refractory in nature which implies that the composition of fluxes is more. The artisans of Tandikkudi site have utilized the potteries of both calcareous and non-calcareous type. Hence the chemical analysis provided useful information about the analytical composition of the specimens, and the manufacturing technique.

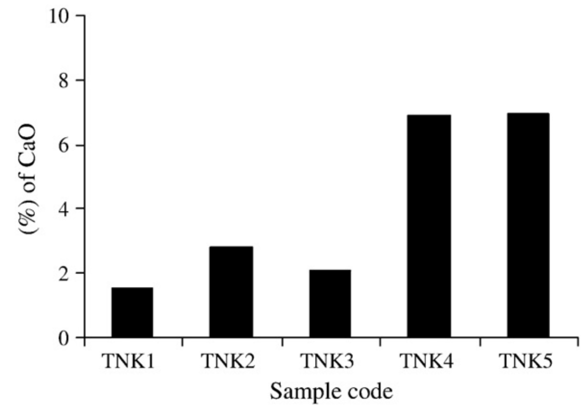

Fig. 1. Variation of CaO in the pottery samples.

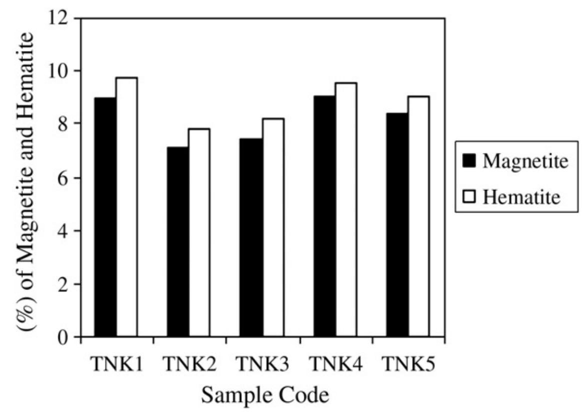

Fig. 2. Variation of hematite and magnetite in the pottery samples.

4.2. Firing temperature estimation using SEM with EDS analysis

Scanning Electron Microscope (SEM) for an accurate observation of the morphology and Energy Dispersive X-ray (EDX) microanalysis for the qualitative and Semi-quantitative determination of the chemical elements present, provide information about complete characterization of artifacts [14]. Moreover, examination of the fresh fracture surface and polished section provided some technological

Table 1
Chemical composition in percentage of Archaeological pottery shreds of Tandikkudi site using chemical analysis.

Chemical composition	TNK1	TNK2	TNK3	TNK4	TNK5
SiO_2	40.52	49.64	46.28	59.99	33.12
Al_2O_3	20.37	16.85	19.46	12.06	20.64
K_2O	4.21	2.16	3.21	1.32	5.38
Fe_2O_3	9.77	7.82	8.20	9.54	9.08
CaO	1.54	2.80	2.10	6.92	6.96
MgO	2.48	2.80	0.76	4.06	1.26
TiO_2	2.35	1.31	7.24	1.09	0.54
Fe_3O_4	8.99	7.11	7.45	9.05	8.39

Table 2
Chemical composition in percentage of archaeological pottery shreds of Tandikkudi sites using EDX analysis.

Chemical composition	TNK1	TNK2	TNK3	TNK4	TNK5
MgO	2.08	2.63	0.67	5.66	1.71
Al_2O_3	20.78	17.19	19.64	13.97	21.46
SiO_2	44.24	45.49	36.92	53.80	39.08
K_2O	4.40	2.98	3.38	1.69	5.85
CaO	0.62	1.27	4.71	6.88	6.84
TiO_2	2.89	1.58	7.69	1.02	0.60
FeO	24.98	23.29	26.98	16.97	30.03