

MICROSTRUCTURAL AND MICROANALYTICAL STUDY – (SEM) OF ARCHAEOLOGICAL POTTERY ARTEFACTS

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Investigations of archaeological pottery artefacts using the potentialities of Scanning Electron Microscope (SEM) along with Energy Dispersive Spectrum (EDS) provide characteristic information on the mineral composition and the percentage variation of minerals present in the fired samples of shred studied in the received state (ARS). It becomes possible to establish categorically the characteristic nature of the pottery shreds from the micrographs respectively. It is also observed that the size and shape of the particles comprising the shreds are heterogeneous in nature. The distribution of pores is not uniform and their difference in diameter in the respective shreds are indicative of the difference in the firing temperature achieved and firing conditions adopted by the artisans at the time of manufacture of the pottery products. The result showed that pottery samples AKM 1 and AKM 2 were fired in between 800–850°C and AKM 3 in the range of 1050–1100°C.

Key words: Archaeological artefacts, firing temperature, microstructure, Pottery shred, SEM-EDS, vitrification, coiling.

1. INTRODUCTION

The archaeology pottery artefacts are the most important sources of information about the daily life and cultural aspects of the people lived at the time of manufacture of the pottery. Further to understand the life style of the people lived in the past, clay technology is assessed to identify the technical sophistication and techniques of firing methods adopted to manufacture the pottery products of varied utilization aspects by the ancients.

The characterization of ancient artefacts is very important not only for archaeologist but also professionals working in the field of physical sciences. The role of physicist is to help in prospecting the archaeological artefacts with the help of scientific methods employing spectroscopic tools. The chemical and physical characterization of artefacts in terms of its mineralogical composition provides significant technological information regarding its methods of manufacture, specifically the firing techniques adopted.

The instrument and techniques used in the present study are the *Scanning Electron Microscope* (SEM) equipped with *Energy Dispersive Spectrum* (EDS) microanalysis detector. It is an excellent tool and it has been found useful in many applications to analyse the archaeological potteries (Goldstein *et al.*, 2003; Tite *et al.*, 1982; Tite *et al.*, 1992). The SEM with EDS analysis is non-destructive and capable of offering precise elemental composition information towards the characterization of archaeological pottery artefacts. The analysis of the composition is mainly aiming to identify the techniques involved in their manufacture. Further the provenances may also be inferred from the mineralogical composition of reference groups.

2. GEOLOGICAL SETTING OF EXCAVATION SITE

Alagankulam is an ancient port city located at 24 km east of Ramanathapuram and 3 km from the coast of Bay of Bengal on the northern bank of river Vaigai, Ramanathapuram district, Tamilnadu, India. Alagankulam is far-famed ancient Pandia's port, played significant role in trading with Romans around 500 BCE to 1200 CE (Srither, 2005). The geological site map of Alagankulam is given in Fig. 2.

3. CHRONICLES OF ALAGANKULAM

The word Argalou of Ptolemy of Erythrean Sea suggests the location of Alagankulam (Mc Crindle, 1887). The word Argeirou in Geography of Ptolemy also comprises the location of Alagankulam in Orgalic Gulf, which is suggested to be Rameswaram area (Mc Crindle, 1885). It had substantial contribution on commercial and economic growth of an ancient Tamilnadu. Roman merchants with wine in amphorae jars laden here in late Roman period (Rajan, 1988). Puranuru, an anthology explains about the Pandya kings consumed the delicious wine from Rome. The inordinate love of importation from Romans might have led to the establishment of a Roman colony and a sea port at Alagankulam. An ancient compendium comprises about the Roman guards at the palace of the Pandya king Nedunchezian (Srither, 2005). It is referred that a few Roman coins belong to AD 383–408 also were found in the excavation site (Kasinathan, 1992).

4. SAMPLES

The three representative pottery shreds found in Alagankulam have been designated by the authors as AKM 1, AKM 2, and AKM 3 respectively. They were procured with the courtesy of State Archaeology Department, Government of Tamilnadu, Chennai, India from the excavation conducted at Alagankulam recently. The



Fig. 1 – Face (1) & Face (2) of the Pottery shreds AKM 1, AKM 2 and AKM 3.

pottery shreds were collected from layers of different depth. It is stated by the archaeological department that the variation in depth of the layers at the excavation can also represent the age of the shred buried chronologically in the past.

The shreds are bifaces and labelled by the authors as face (1) and face (2) arbitrarily. The nature of the bifaces is not similar in appearance. The face (1) of the shreds AKM 1 and AKM 3 is glazed whereas the face (1) of AKM 2 is line embossed. The face (2) in all three specimens is glazed in visual estimation. The physical attributes of pottery shreds are given Table. 1.

Table 1

Physical attributes of designated pottery shreds

Pottery Designation	Trench m	Thickness $\times 10^{-3}$ m	Colour/Surface	
			Face (1)	Face (2)
AKM 1	1.90	3.614	Red/Glazed	Grey black/Glazed
AKM 2	5.10	4.632	Black/line embossed	Grey black/Glazed
AKM 3	6.40	14.622	Brownish/Glazed	Cream grey/Glazed

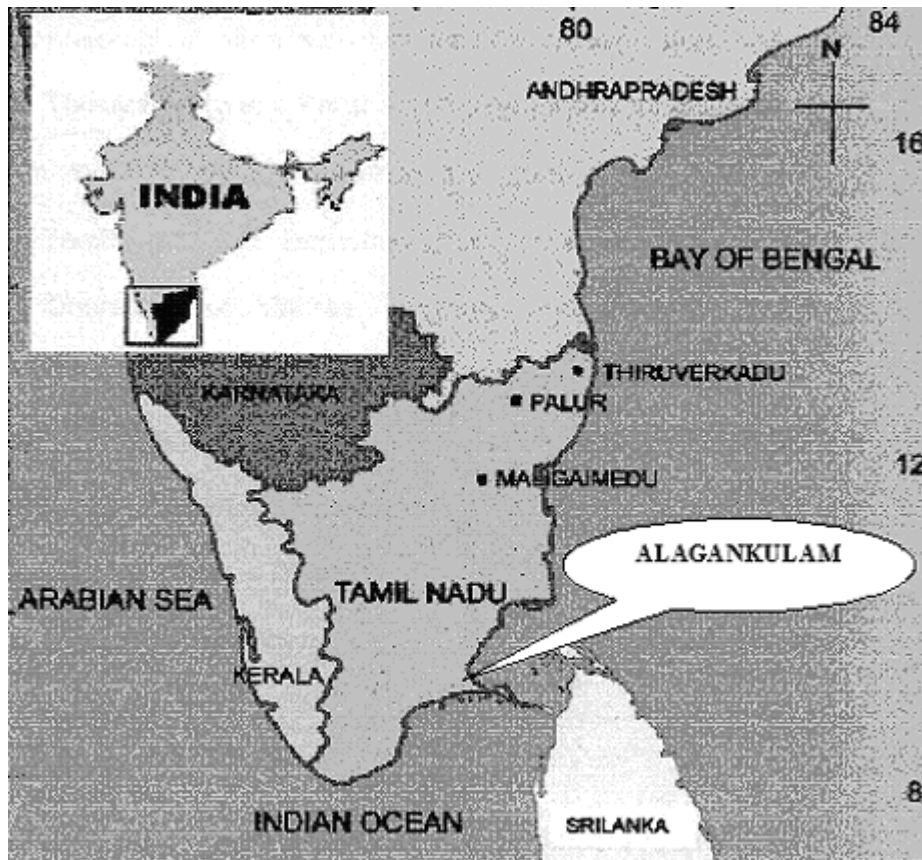


Fig. 2 – Geographical map of the excavation site, Alagankulam (Tamilnadu).

5. EXPERIMENTAL

The microstructure and elemental composition of all the three pottery shreds in the received state (ARS) were evaluated from their micrograph by *scanning electron microscope* (SEM) of model JSM-5610 LV JEOL along with the energy dispersive spectral analysis (EDS). The received state (ARS) examination of the potteries provides information on the internal morphology developed during the firing at the time of the manufacture. The maximum magnification possible in the equipment is 3,00,000 times with a resolution of 3nm. The fresh fractured surfaces of the potteries in the received state (ARS) were coated with thin layer of gold and examined using SEM, by typically setting at same magnification $\times 2000$ for all the three samples of study respectively. The quantitative analysis was carried out using the energy dispersive X-ray at an accelerating voltage of 20 kV for major and minor elements respectively (Na, Mg, Al, Si, K, Ca, Fe, W, S, Cl, and Ti).

6. RESULTS AND DISCUSSION

The energy of characteristic X-ray that is emitted when electrons strike a solid specimen enabled to identify the main elements that were present in the pottery samples (Krapukaityte *et al.*, 2006). The EDS spectra of the three shreds are shown in Fig. 3 – Fig. 5 respectively. The EDS spectrum of each sample exhibit the presence of elements Silicon, Aluminium, Potassium, Iron, Magnesium and Sodium as the predominant constituents of the shreds but variation in the percentage of the composition of the minerals respectively.

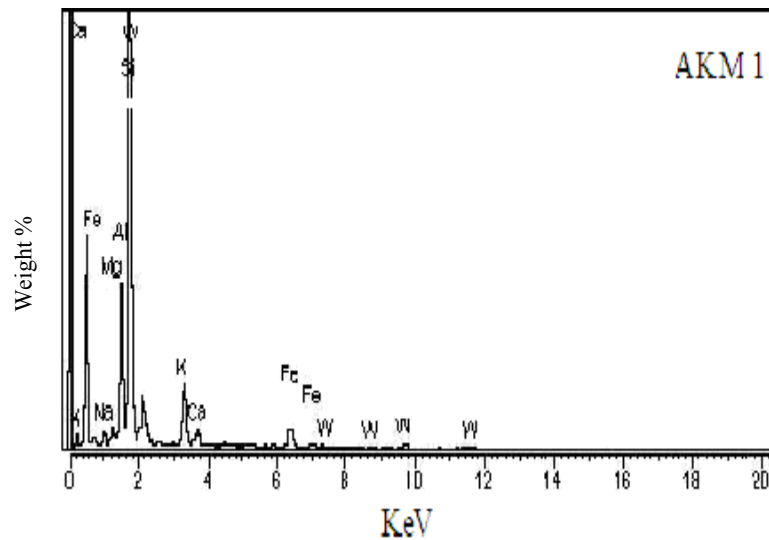


Fig. 3 – The EDS spectrum of the fractured shred AKM 1.

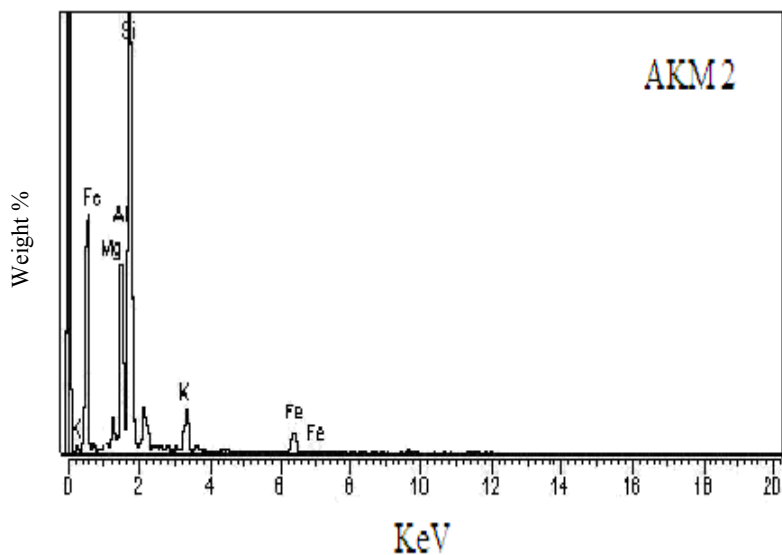


Fig. 4 – The EDS spectrum of the fractured shred AKM 2.

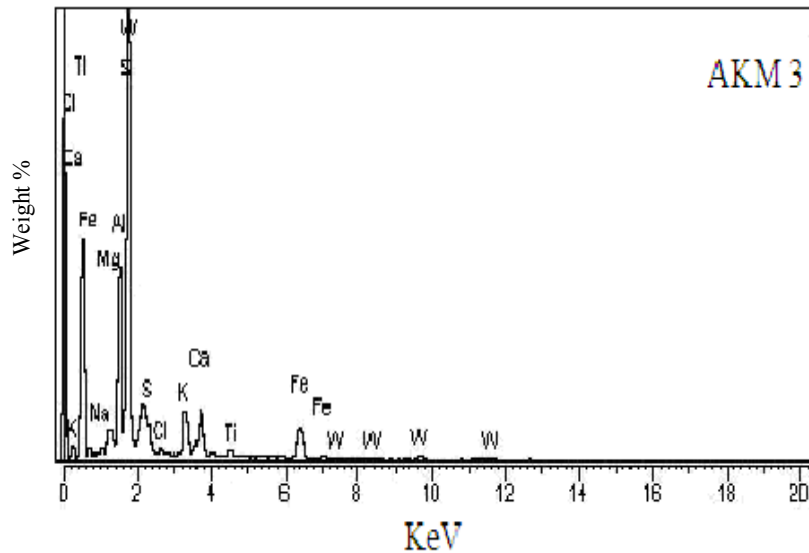


Fig. 5 – The EDS spectrum of the fractured shred AKM 3.

Table 2

Elements present in the pottery shreds of study

Element	Elemental concentration weight (%)		
	AKM 1	AKM 2	AKM 3
Sodium Na	----	1.54	1.36
Magnesium Mg	2.78	1.38	2.73
Aluminium Al	18.72	14.61	18.54
Silicon Si	59.72	49.9	52.55
Potassium K	7.30	11.33	7.12
Calcium Ca	----	3.04	6.95
Iron Fe	11.48	8.85	12.63
Tungsten W	----	9.35	6.93
Sulphur S	----	----	1.82
Chlorine Cl	----	----	0.85
Titanium Ti	----	----	1.88

The varied percentage of composition of the elements present in the specimens AKM 1, AKM 2 and AKM 3 show that the shreds are of characteristically different in nature. The amount of Potassium present in the samples AKM 1 and AKM 3 is about 7% and 11.33% in AKM 2. Potassium compounds disassemble as fluxes during firing, encouraging the initial sintering and extensive vitrification (Peri Mitri *et al.*, 1999). The presence of Ca in the AKM 2 and AKM 3 and its absence in AKM 1 indicates that AKM 2 and AKM 3 were

fired relatively in the lower range of temperature than the AKM 1, but it is difficult to appraise the exact temperature from the mineralogical data alone. Presence of same elements found in AKM 2 and AKM 3 is the practical evidence that the clays might be procured from the same area for making the pottery products. Though the silicon is found as the major element in all the three shreds but its percentage is varying from 49.9% to 59.72%.

Fig. 6 – SEM Micrograph of the fractured surface AKM 1.

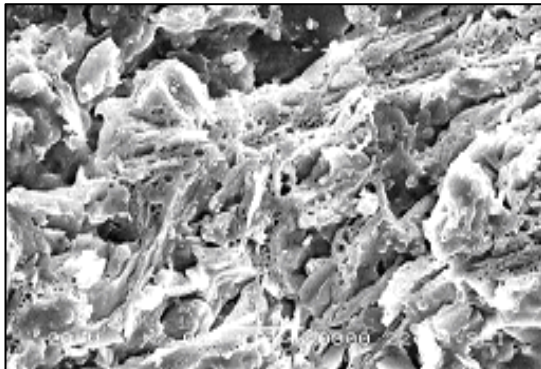
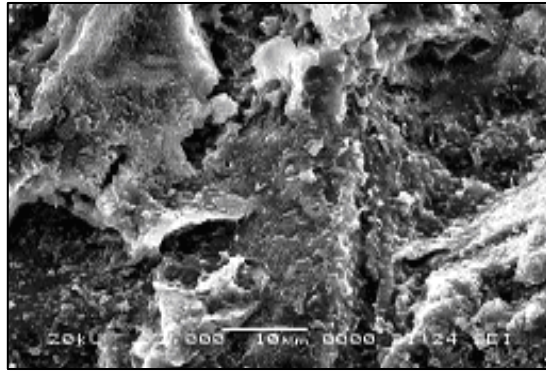


Fig. 7 – SEM Micrograph of the fractured surface AKM 2.

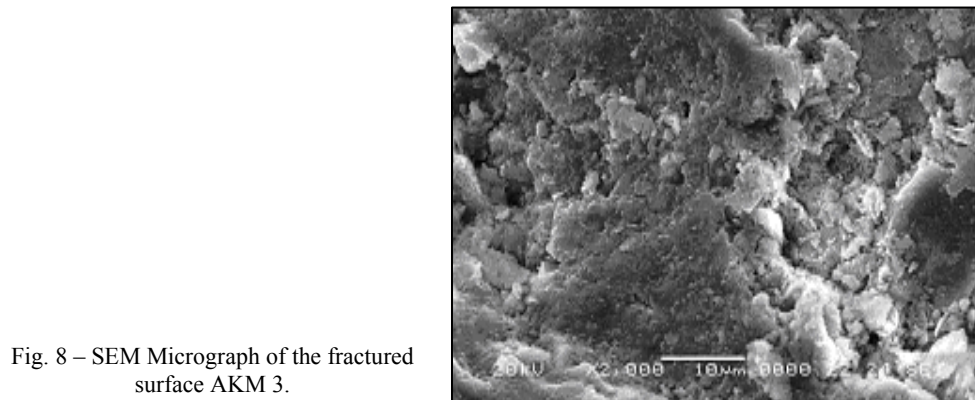


Fig. 8 – SEM Micrograph of the fractured surface AKM 3.

If the pottery shred have less than 6% of Ca, the clay used for the make is of non calcareous in nature (Maniatis and Tite, 1981). Based on this and from EDS data it is inferred that the samples AKM 1 and AKM 2 have been made of non calcareous clay since the percentage of Ca found in AKM 1 is zero and in AKM 2 is 3.04%. But the percentage of Ca in AKM 3 is found to be more than 6% (6.95%). So it is possible to infer that the clay used in the pottery shred AKM 3 was calcareous in nature.

The Scanning Electron Micrographs of AKM 1 and AKM 2 show that the particles are irregularly shaped and varied in sizes. As it is seen in AKM 1 and AKM 3 the surface of the pottery comprises of volumetric plate like grains with a different crystallite sizes ranging from 10 μm to 20 μm . It allows to interpret that they were fired relatively at low temperature (Krapukaitypte *et al.*, 2006). The observation of smooth surface observed in AKM 1 and AKM 3 is due to the reflective nature caused by early sintering (Rye, 1981). The appearance of smooth surface spots of area in the respective micrographs of AKM 1 and AKM 3 is adverted to initial vitrification stage for both non calcareous AKM 1 and calcareous AKM 3. So it is considered that the range of temperature fired the samples is typically between 800 to 850°C in an oxidising atmosphere (Maniatis and Tite, 1981).

The careful examinations of micrographs of AKM 2 reveal that the particles were heterogeneously shaped with a lot of incisions. In some parts of its micrograph horizontally aligned pores were present which may be attributed to coiling of pottery (James Feathers, 2006). An encompassing distribution of bloating pores in AKM 2 reveals that this specimen was fired in multi-step technology at relatively high temperature (Colomban *et al.*, 2004 & Colombian *et al.*, 2003). The presence of fine bloating pores with diameter of 0.2 to 4 μm were due to the continuous vitrification during firing in reduced atmosphere (Molera *et al.*, 2001). As it was reported that the size of pores would become 10–50 μm around 1050–1100°C (Maniatis and Tite, 1981), the presence of small sized pores in AKM 2 indicate that the samples might have been fired at temperature below 1050°C.

7. CONCLUSIONS

The SEM-EDS study of pottery shreds AKM1, AKM2 and AKM3, excavated in Alagankulam showed different morphology and elemental composition. The data obtained by EDS allowed to differentiate the samples into the types by its calcareous nature. The samples AKM 1 and AKM 2 were characterized as non-calcareous and AKM 3 as calcareous. The appropriate range of firing temperature of the shreds was identified from the respective morphology. The SEM pattern of the samples suggest that the shreds AKM 1 and AKM 2 were fired in between 800–850°C and AKM 3 fired in the range at 1050–1100°C.

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