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Analysis of ancient potteries of Tamilnadu, India by spectroscopic techniques

R.Ravisankar¹, S.Kiruba², A. Chandrasekaran³, G. Senthilkumar⁴ and C. Maheswaran⁵

¹Post Graduate & Research Dept. of Physics, Government Arts College, Tiruvanamalai-606601, TN, India ²Department of Physics, St.Joseph's College of Engineering, Chennai-119, Tamilnadu, India ³Department of Physics, Sacred Heart College, Thirupattur-635601, Tamilnadu, India ⁴Department of Physics, University College of Engineering, Arani-632317, Tamilnadu, India ⁵Anthropology Section, Government Museum, Egmore, Chennai-600008, Tamilnadu, India rrs_phy@rediffmaill.com, rrs_phy@yahoo.co.in

Abstract

We report the spectroscopic investigation of ancient potteries excavated in Tamilnadu. Qualitative analyses were carried out to determine the major and minor constituent minerals present in the samples based on the band position or location of IR absorption peaks. From the prominent i.r. absorption peaks, the minerals were identified from the available literature. The constituents of minerals present in the potteries are further confirmed by XRD technique. Both FT-IR and XRD techniques reveals the mineral composition of ancient potteries. Energy dispersive X-ray fluorescence (XRF) was used to determine the major, minor and trace element composition of potsherds of ancient potteries. The concentration of trace elements is helpful for the characterization of pottery produced in a given region. The combined techniques are very much useful for the characterization of potteries.

Keywords: Ancient pottery, mineral analysis, FT-IR, XRD, EDXRF, elemental analysis

Introduction

One of the most important areas of archeology is the identification of the source and origin of ancient artifacts. Knowing the origin is very important for tracking correctly the ancient civilization and history. Shards from pottery vessels are the most common artifacts found during excavation of archeological sites, so archaeologist are interested to in the studies of pottery fragments. Most archaeologists have classified ancient artifacts (pottery) by their shapes and uses. The pottery is capable of revealing many aspects concerning a prehistoric culture including probable place of manufacture, the origin of the raw material, trade etc. The mineral study and elemental composition of the pottery analysis gives an idea about the firing temperature and also yield the information about the potshards. The mineral analysis is carried out by FT-IR and XRD technique and elemental analysis is done by EDXRF technique.

Infrared spectra act as "finger print" technique and yield information about the atomic groupings present in the samples. Infrared spectroscopy, which acts as a research tool in mineralogy, is most powerful if used in conjunction with X-ray diffraction and other techniques. Using the infrared -spectra, unique information about the group of minerals in which the specimen belongs, the degree of crystalline and non-crystalline impurities and reactions of minerals with chemicals in their environment can also inferred.

. Energy dispersive X-ray fluorescence (XRF) was chosen for the quantitative analysis of potsherds because of its accurate, relatively cheap and easy to use. It has short processing times and very low detection limits. The fundamental principle behind XRF is that when electrons of particular elements are excited by X-rays they emit or fluorescence a spectrum of X-rays that is specific to that element. XRF is frequently used for sourcing archeological artifacts.

In the present study, ancient potteries from Tamilnadu was obtained local archeological authorities and subjected to several analytical methodologists. FT-IR and XRD was used for mineral characterization, EDXRF for the determination of the chemical elements characterization. The combined results of the analysis may be used for the characterization of the ancient potteries.

Material and methods

Sample collection

Eight potsherds (P1-Nilagri, P2-Sangumugam, P3-Perumanal, P4-Tholvazangu, P5-Panjal, P6-Suthuvalai, P7-Rasakamangalam & P8-Stoneage) were provided from the Government Museum, Egmore, Chennai, Tamilnadu, India covering a period of prehistoric period. The samples were taken at depth from soil surface to about 6m. After removal of surface layers, the pottery shards were oven dried for 48h at 100°c and ground into fine powder using an agate mortar. They were sieved using a 90µm mesh to maintain uniform particle size for analysis.

FT-IR Technique

The KBr pellet technique was followed for the mineral analysis. Sample of 2 mg is mixed with 40 mg of spectroscopic KBr in the ratio 1:20 using a mortar and pestle. Before mixing, necessary amount of KBr powder is dried at 120°C for six hours in an oven. Otherwise the broad spectral peak due to free OH will seriously affect the interpretation on the bound hydroxyls associated, with any of the minerals. The major and minor minerals are gualitatively determined by FT-IR technique. The ABB



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Vol. 3 No. 8 (Aug 2010)

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BOMEM 104.MB series available in Dr.Ceeal Analytical Laboratory, Thorappakkam, Chennai, Tamilnadu, India is made use of in the present work for recording the i.r. spectra of the samples at room temperature. For each samples, five to six pellet specimens are prepared and the spectra were taken in the mid region of 4000-400cm⁻¹. The instrument scans the spectra 16 times in 1 minute and the resolution is 5cm⁻¹. This instrument is calibrated for its accuracy with the spectrum of a standard polystyrene film. Every time, before the spectrum of sample is obtained; the spectrum of the polystyrene film is taken and checked for the accuracy and transmittance. The best spectrum for each site was considered as a representative spectrum of the site. The typical i.r. spectrum is shown in Fig.1.





XRD Technique

Samples for X-ray powder diffraction (XRD) studies were packed in shallow cavities in glass slides to minimize preferred orientation. The X-ray patterns of pottery samples were recorded at room temperature by using X-ray diffractometer (D500, Siemens) having a curved graphite crystal diffracted monochromator, with a source of CuKa radiation and Nal(TI) scintillation detector from Material Science Division (MSD), Indira Gandhi Centre for Atomic Research (IGCAR), Kalpakkam, Tamil Nadu, India. Qualitative mineralogy of the soil samples is determined with the standard interpretation procedures of XRD. Major mineral in the studied sample is guartz. Typical X-ray diffractrogram of pottery samples are shown in Fig. 2.

EDXRF Technique

One gram of the fine ground (sieved through 100 mesh) sample and 0.5g of the boric acid were mixed. The mixture was thoroughly ground and pressed to a pellet of 30 mm diameter using a 15 ton hydraulic press. The samples were analyzed using the EDXRF available at

Environmental and Industrial Safety Section, Safety Group, Indira Gandhi Centre for Atomic Research (IGCAR), Kalpakkam, Tamilnadu. The instrument used for this study is a PW 4025 Minipal supplied by M/s Philips, Netherlands. The spectrometer is fitted with a side window X-ray tube (9 W) that has Rhodium as anode. The power specifications of the tube are 4-30 kV; 1 µA- 1mA. Removable sample changer of the instrument accommodates 12 samples at a time. Selection of filters, tube voltage, sample position and current are fully computer controlled. Beam spot area (elliptical) for the instrument is 81.7 mm^2 . The instrument has the features of Multi Channel Analyzer (MCA) test, standardless determination and automatic gain correction. Gain correction is performed when the beam stop is in the



reference position. Beam stop contains a reference sample (an alloy of aluminum and copper). Copper is used for gain correction. Al and Cu are used for instrument energy calibration. The stream sediment standard (GBW 7305) was used as P2-Sangumugam reference material. The certified value and the present study of the standard analysis is given in Table1.

Results and discussion

FT-IR analysis

By comparing the observed frequencies with available literature, the minerals such as quartz, feldspar, calcite, kaolinite, montmorllinite, magnetite and heamatie have been identified. The mineral and tentative assignment of the potshards is reported in Table 2.

The quartz mineral in the samples were detected with the bands at 795,775, 695 and 455cm⁻¹ and the the IR bsorption peaks of quartz were reported by many workers (Lyon, 1967; Chester & Green, 1968; Farmer, 1974; Clearance Karr, 1974; Coates, 1977; Hlavay et al. 1978; Russell, 1987; Reig et al., 2002; Ko & Chu, 2005; Ravisankar et al., 2006; Ravisankar, 2009). Out of the



four peaks observed for quartz, the bands at 795cm⁻¹ and 775cm⁻¹ may be assigned symmetrical stretching modes and those at 695cm⁻¹ and at 455cm⁻¹ may be due to symmetrical bending mode and asymmetrical bending respectively. The presence of quartz gives information about the origin of the potteries; quartz was used in piedmont for covering of engobed pottery, while kaolin was used elsewhere for the same purpose. The presence of quartz in the largest part of the samples confirms thus the common origin of these samples.

The feldspar groups of minerals have been analyzed

by IR technique and reported by (White. workers manv 1971: Farmer, 1974; Clerence Karr, 1974; Ghosh,1978; Hlavay et al., 1978; Russell, 1987; Neog et al., 1999; Xu, et al., 2001; Ravisankar et al., 2006; Ravisankar, 2009). The i.r. absorption peaks appearing at 635, 545, 435 & 405cm^{-1} were assigned to feldspar in the samples. The peaks at 635 & 545cm⁻¹ indicated the presence of orthoclase. Albite was observed in the samples by the presence of peaks at 435 & 405cm⁻ . The presence of feldspar, as a common component of potteries, does not give relevant information about the techniques used for their production.

The presence of IR absorption peaks at 715 & 875cm⁻¹ is identified by calcite in the samples (Chester & Elderfield, 1967; Lyon, 1967; Farmer, 1974; Clearance Karr, 1974; Ghosh, 1978; Russel, 1987; Senthilkumar *et al.*, 2001; Reig *et al.*, 2002; Ravisankar *et al.*, 2006; Ravisankar, 2009). The presence of calcite (CaCo₃) in the finds may occur essentially for two reasons; a low firing temperature or a post burial deposition processes. Calcite

Table 1. Stream sediment standard values and the EDXRF results (in ppm unless % is indicated)

Elements	Certified	EDXRF analysis			
	Values	Value			
AI%	7.84	7.46			
Ca%	3.17	3.18			
K%	1.62	1.63			
Fe%	4.32	4.28			
Ti%	0.52	0.52			
Si%	31.92	32.37			
V	109	109.7			
Co	18.5	18			
Cu	137	138.9			
Ba	440	633.9			
Zn	243	261.3			
Pb	112	159.6			
Cd	0.82	0.88			
Mn	1160	1137			

Vol. 3 No. 8 (Aug 2010)

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exits up to 800°c, when the CaO formation is promoted, followed by the formation of the so-called "hightemperature crystalline phase" made of Ca-silicates or Ca-Al-silicates such as gehlenite (CaAl₂ SiO₇), diopside $(CaMgSi_2O_6)$ and anorthite (CaAl₂-Si₂O₈). The simultaneous presence of calcareous clays of calcite and high temperature minerals clearly rules out any hypothesis of primary calcite, strengthening the assumption of a secondary origin due to deposition induced by water of the burial soil. Clearly, in noncalcareous clays, calcite is not expected.

> The presence of kaolinte and montmorilinte indicates clav minerals in samples. The IR absorption peaks appear at 3690, 3630, 1030 & 930cm⁻¹ in the samples, which indicates presence of kaolinite. The i.r. absorption peaks of kaolinte are reported by (White, 1971; many workers Clarence Karr, 1974; Farmer, 1979; Russell, 1987; Crowley & Vergo,1988; Clark et al., 1990: Summer, 1995; Xu et al., 2001; Madejova & Komadel, 2001; Ko & Chu, 2005; Ravisankar et al., 2006; Ravisankar, 2009). Kaolinite shows characteristic IR absorption а sequence in the range of 3700-3600 cm⁻¹. These absorbances

correspond to stretching vibrations of inner surface OHs 3696 cm⁻¹ and inner OH 3620 cm⁻¹. Absorbance at 1105 and 1034 cm⁻¹ are attributed to anti-symmetric Si-O-Si stretching, whereas those at 1018 cm⁻¹ are attributed to stretching of an O atom bound to both Si and tetrahedral AI (Summer, 1995).

The IR absorption bands around 1635 and 3445cm⁻¹ show the presence of montmorillonite in the samples

Table 2. Infrared ab	sorption fred	quencies and	Probable assig	gnment of	potteries of	Tamilnadu
		/			/	

Frequency (cm ⁻¹)	Mineral Name	Tentative Assignment	Reference
3690	Kaolinte	Inner surface OH Stretching vibration	Russell,1987; Summer,1995
3620	Kaolinte	O-H- Stretching of inner hydroxyl	Russell, 1987; Summer, 1995
3445	Montmorillonite	O-H- Stretching of absorbed water molecule	Russell, 1987
1035	Kaolinte	Si-O-Stretching of clay mineral	Russell, 1987; Summer, 1995
935	Kaolinte	O-H deformation	Russell, 1987
875	Calcite	Fe ³⁺ (AI-OH)	
795	Quartz	Si-O of Quartz	Hlavay <i>et al</i> ., 1978; Coates,1977
775	Quartz	Si-O of Quartz	Hlavay et al., 1978; Coates, 1977
715	Calcite	-	
695	Quartz	Si-O of Quartz	Hlavay et al., 1978; Coates, 1977
635	Orthoclase	AI-O coordination vibration	Hlavay et al., 1977; Coates, 1977
585	Magnetie	Fe-O of Fe ₃ O ₄	
545	Orthoclase	Si - O asymmetrical bending vibration	Coates, 1977; White, 1971
535	Hematite	Fe-O of Fe ₃ O ₄	
475	Hematite	Si-O-Si bending of silicates	
455	Quartz	Si-O asymmetrical bending vibration	Hlavay et al., 1978; Coates, 1977
435	Albite	Si-O of mixed vibration	
405	Albite	-	



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(Clarence Karr,1975; Ghosh, 1978; Summer,1985; Clark *et al.*, 1990; Ravisanka *et al.*, 2006; Ravisankar, 2009). The most distinguishing feature of the montmorillonite spectrum is the broad absorption band that ranges from 3,300 to 3,500 cm⁻¹. This band typically centered about 3400 cm⁻¹ is due to H-O-H stretching of water molecule present in the interlayer region of montmorillonite (Summer, 1985).

Hematite and magnetite is identified by the presence of the peaks at 540 & 475cm⁻¹ and 585cm⁻¹ respectively (Clarence Karr, 1975; Ghosh, 1978; Russell, 1987; De Benedetto et al., 2002). The presence of magnetite and hematite provides interesting information about the firing atmosphere. Iron oxides are very influenced by the firing atmosphere, in fact they differ in colour according to firing conditions: when oxygen is almost absent in the kiln i.e. under reducing conditions, reduced compounds as magnetite (black colored) form: on the contrary, under the influence oxidizing conditions, the oxidized form as hematite (reddish) prevail. The presence of hematite in some finds could give further information about the provenance of the clay used for the mixture. Our results of the i.r analysis of mineral findings in potteries are agreement with those of De Benedetto et al. (2002).

The IR spectra indicated that all the samples contained calcite in different amounts. Calcite exists as impurity of local clays. Observation of calcite (CaCo₃) bands allows us to draw conclusion on firing temperature. The processing temperature should be below the decomposition temperature of calcite that is about 700° - 900° C reported by Ham *et al.* (2007).

The presence or absence of specific mineral assemblage is often used for the estimation of firing temperature of the pottery. Quartz and feldspar are abundant in all potshards samples. The presence of quartz and feldspar may indicate temperature of at least 900°C.

XRD analysis

Qualitative mineralogy of the potshards is determined with the standard interpretation procedures of XRD. Major mineral in the studied sample is quartz. The mineral wise discussion is as follows:

Quartz: Quartz is ubiquitous and forms one of the most abundant mineral in all the samples. It is identified by the distinctive reflection at 4.26A°, 3.34A°, 2.46A°, 2.28A°, 2.24A°, 1.85A°, 1.67A°, 1.54A°, 1.45A°, 1.38A° and 1.18A°.

Feldspar: The feldspar mineral is invariably present in all samples. Albite is identified by basal reflection at 4.03A°, 3.64A°, 3.24A°, 3.13A°, 3.18A°, 3.14A°, 2.93A°, 2.84A°, 2.27A° and 1.98A°. The basal reflection at 3.21A°, 2.98A° and 1.44A° indicate the presence of orthoclase.

Clay Mineral. The clay mineral kaolinte is identified in the samples by basal reflection at 1.66A°, 1.78A°, 3.54A° and 3.59A°.

Calcite: Only a trace amount is identified by the three distinct reflections in the d spacing of $3.03A^\circ$, $2.820A^\circ$ and $2.82A^\circ$.

The presence or absence of specific mineral assemblage is often used for the estimation of firing temperature of the pottery. Quartz and feldspar are abundant in all potshards samples. The presence of quartz and feldspar may indicate temperature of at least 900°c. These two minerals persist on firing up to 1000°c and thus they are obliviously constituents of a silica-rich raw clay mineral. Quartz may be an indigenous mineral of natural clays or clay or may be intentionally added temper.

The mineralogical composition depends on the regional geology and the potters habits and experience. Adding non plastic materials, known as temper, the raw clay is improved to workability and allow water to evaporate more smoothly minimizing shrinkage and preventing cracking. Potters have used a variety of temper including quartz, limestone, shells, volcanic ash

and even crushed potshards. The quartz rich raw clay or quartz tempered potshards of our study are less resistant to mechanical and thermal stresses during use, compared to calcite rich pottery.

EDXRF analysis

Table 3 lists the major, minor and trace elements found in the potsherds. Abundant amount of Si, Al, Ca, Mg, Fe, K and Ti was found. Mn, Cu, Zn, Pb, La, Co, V, Cd and Cr were founded to be minor constituents. The variations in the trace element concentrations refer geological diversity. The chemical composition of the potsherds is strongly related to the source of clay and other materials used for its production. The highest content of Si, Al, Ca Mg, Fe, K

 Table 3. Elemental composition of potteries of Tamilnadu (in ppm unless % indicated)

			laleated)				
P1	P2	P3	P4	P5	P6	P7	P8
21.86	26.20	24.96	28.77	24.03	26.49	26.69	25.82
4.87	7.82	4.91	5.00	6.35	7.09	5.91	4.21
0.42	0.65	1.16	1.00	1.45	1.31	0.46	0.51
0.31	0.67	0.62	0.59	0.57	0.69	0.54	0.46
6.36	5.81	4.79	4.99	5.22	4.60	5.08	4.26
0.49	1.41	2.12	1.60	2.14	1.84	1.07	1.46
1.13	0.64	0.59	0.47	0.41	0.46	0.77	0.53
321.2	361.3	338.9	1040.8	434.4	334.2	452.2	434.4
31.0	25.5	23.3	15.0	18.8	22.9	15.3	38.5
24.8	79.2	47.7	65.7	60.5	71.3	68.1	63.7
27.5	25.5	25.6	24.8	15.2	27.4	30.4	35.3
9.6	46.5	41.9	31.9	44.4	27.3	46.4	20.7
121.6	96.3	21.9	24.4	24.5	20.4	23.9	19.4
51.4	56.0	29.6	111.4	47.5	35.9	54.8	63.8
0.1	0.6	1.0	1.0	1.0	0.8	0.4	0.6
121.6	96.3	88.8	88.5	83.3	82.0	93.0	102.0
	P1 21.86 4.87 0.42 0.31 6.36 0.49 1.13 321.2 31.0 24.8 27.5 9.6 121.6 51.4 0.1 121.6	P1 P2 21.86 26.20 4.87 7.82 0.42 0.65 0.31 0.67 6.36 5.81 0.49 1.41 1.13 0.64 321.2 361.3 31.0 25.5 24.8 79.2 27.5 25.5 9.6 46.5 121.6 96.3 51.4 56.0 0.1 0.6 121.6 96.3	P1 P2 P3 21.86 26.20 24.96 4.87 7.82 4.91 0.42 0.65 1.16 0.31 0.67 0.62 6.36 5.81 4.79 0.49 1.41 2.12 1.13 0.64 0.59 321.2 361.3 338.9 31.0 25.5 23.3 24.8 79.2 47.7 27.5 25.5 25.6 9.6 46.5 41.9 121.6 96.3 21.9 51.4 56.0 29.6 0.1 0.6 1.0	P1 P2 P3 P4 21.86 26.20 24.96 28.77 4.87 7.82 4.91 5.00 0.42 0.65 1.16 1.00 0.31 0.67 0.62 0.59 6.36 5.81 4.79 4.99 0.49 1.41 2.12 1.60 1.13 0.64 0.59 0.47 321.2 361.3 338.9 1040.8 31.0 25.5 23.3 15.0 24.8 79.2 47.7 65.7 27.5 25.5 25.6 24.8 9.6 46.5 41.9 31.9 121.6 96.3 21.9 24.4 51.4 56.0 29.6 111.4 0.1 0.6 1.0 1.0 121.6 96.3 88.8 88.5	P1 P2 P3 P4 P5 21.86 26.20 24.96 28.77 24.03 4.87 7.82 4.91 5.00 6.35 0.42 0.65 1.16 1.00 1.45 0.31 0.67 0.62 0.59 0.57 6.36 5.81 4.79 4.99 5.22 0.49 1.41 2.12 1.60 2.14 1.13 0.64 0.59 0.47 0.41 321.2 361.3 338.9 1040.8 434.4 31.0 25.5 23.3 15.0 18.8 24.8 79.2 47.7 65.7 60.5 27.5 25.5 25.6 24.8 15.2 9.6 46.5 41.9 31.9 44.4 121.6 96.3 21.9 24.4 24.5 51.4 56.0 29.6 111.4 47.5 0.1 0.6 1.0 1.0 1.	P1P2P3P4P5P621.8626.2024.9628.7724.0326.494.877.824.915.006.357.090.420.651.161.001.451.310.310.670.620.590.570.696.365.814.794.995.224.600.491.412.121.602.141.841.130.640.590.470.410.46321.2361.3338.91040.8434.4334.231.025.523.315.018.822.924.879.247.765.760.571.327.525.525.624.815.227.49.646.541.931.944.427.3121.696.321.924.424.520.451.456.029.6111.447.535.90.10.61.01.01.00.8121.696.388.888.583.382.0	P1 P2 P3 P4 P5 P6 P7 21.86 26.20 24.96 28.77 24.03 26.49 26.69 4.87 7.82 4.91 5.00 6.35 7.09 5.91 0.42 0.65 1.16 1.00 1.45 1.31 0.46 0.31 0.67 0.62 0.59 0.57 0.69 0.54 6.36 5.81 4.79 4.99 5.22 4.60 5.08 0.49 1.41 2.12 1.60 2.14 1.84 1.07 1.13 0.64 0.59 0.47 0.41 0.46 0.77 321.2 361.3 338.9 1040.8 434.4 334.2 452.2 31.0 25.5 23.3 15.0 18.8 22.9 15.3 24.8 79.2 47.7 65.7 60.5 71.3 68.1 27.5 25.5 25.6 24.8 15.2 27.4

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From the EDXRF analysis, the abundant amount of Si, Al, Ca, Mg, Fe, K and Ti was founded and it supports the vibrational spectroscopic findings; the presence of aluminosilicates, quartz, iron oxidies (Haematite and magnetite) and feldspar (Sevim Akyuz et al., 2008).

Conclusion

Application of FT-IR and XRD techniques together with the EDXRF for the study of ancient potshards shows the technological conditions implemented for the production of pottery and found to give useful information about the analytical composition of potteries. Spectroscopic results indicated that the potteries were fired to a temperature less than 900°C.

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Vol. 3 No. 8 (Aug 2010)

- ISSN: 0974-6846
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