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CHARACTERIZATION OF REPRESENTATIVE ANCIENT POTTERIES: CHEMICAL, MINERALOGICAL AND MORPHOLOGICAL STUDIES

Siva Sankar PANDA[1*], Gopi Nath JENA[2], Dibishada B. GARNAYAK[3]

[1] National Research Laboratory for Conservation of Cultural Property, Lucknow, Uttar Pradesh, India
[2] Hazardwari palace Museum, Archaeological Survey of India, Murshidabad, West Bengal, India.
[3] Excavation Branch IV, Archaeological Survey of India, Bhubaneswar, Odisha, India

Abstract

This paper explores the characterization of six potsherd samples from Udayagiri, India. X-ra Fluorescence (XRF), Fourier Transform Infrared (FTIR) and Scanning Electron Microscop coupled with Energy Dispersive X-ray system (SEM-EDX) were used for the determination of th mineralogical, chemical and microstructural analysis of potsherd samples. RO, R_2O_3 and RC were deduced from chemical composition data using Seger molecular formula. Present study als emphasis on the investigation of different methodical approach in terms of firing temperature clay mineral type and tempering material in the pottery making process of that era. The uses of siliceous or non-calcareous raw material are probably related to the specific utilization of th pottery in ancient time. The presence of specific minerals in the pottery matrix indicates th practice of local clay mineral. The flux amount reflects of higher firing temperatures, while cla composition provides the information to understand production methodology. The temperin materials in all the samples can be observed in morphological images. The obtained results als showed that non- calcareous clay minerals were used for pottery production and potsherds wer firing between the temperatures from 600 to 900^0C.

***Keywords**: Ancient pottery; XRF; FTIR; SEM-EDX; Firing temperature;*

Introduction

Men emerged in earth through evolution. Pottery being the most abundant tracer in all archaeological excavations, the classification at any level of such manufactures has a key role in historical studies. Archaeologists, historians explore the past through the pottery and establish the cultural age and provide a sequence of human history from pre-history to historical age. Clay materials like pottery, brick, and tiles are major innovative designs since Neolithic period with the development of human race. Its use, ranging from functional to decorative purposes in everyday life makes it answerable to cultural linkage, technological advance and methodology of artisans during which these were produced. In order to know better about the presence of foreign element or exotic influence present in the pottery, or to determine the relative chronological sequence viable with other historical sites, or again, to understand communication of trade network etc., the study of the ceramics is quite indispensable. The identity of the product depends on initial composition of clay, additives used, the firing temperature, atmosphere and duration of heat treatment [1-2]. 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

* Corresponding author: Pandasivasankar@gmail.com

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. Systematic and multidimensional scientific approaches are always essential to study these very complexes and challenging archaeological artifacts [3-8]. The chemical, mineralogical, morphological, porosimetry characterization of ancient pottery can unlock evidence to raw materials used for production, firing temperature and determine the technological development related to its manufacture [4, 9-11]. A comparison study within various groups of locally produced fine ware [12-14], replica study [15-18], using different instruments [19] and color measurement [20] were well explored for ancient potteries. Several authors studied firing conditions [21-22] and influence of organic matter in the methodology [22-28]. Numerous excavations were carried out by Archaeological Survey of India (ASI) of Odisha circle since 1985 to 2004 at Udayagiri (1st century A.D to 12th Century A.D); a Buddhist site situated 102 km north east from Odishan capital city Bhubaneswar. The site was identified as "Madhavapura Mahavihara" on the basis of epigraphical findings. The excavation had partially revealed a double storied monastic complex datable to 8th Cent. A.D and an important antiquities images of Buddha, Tara, Manjusri, Avalokitesvara, Jatamukuta Lokesvara and terracotta sealings. A good number of stone inscriptions datable from 5th -13th cent A.D are also recovered. The researches of the ancient potsherds of various period and places have been carried out and reported globally [3-5]. Yet, there is lack of scientific research and methodological approach on the potsherd.

In the present study, fragmented potsherds from Buddhist site Udayagiri from India were approached by techniques such as XRF, FTIR and SEM-EDX. The outcome from this analysis suggests the methodologies behind pottery production along with chemical and mineral composition. FTIR spectroscopy is having several advantages over the XRD mineral identification as absolutely no need for sample preparation, less analysis time moreover disordered structures can easily point out [5-9]. The aim of this research is to provide an insight into the technology of the pottery made, and the constituents that used for the manufacturing of potteries.

Materials and analytical methods

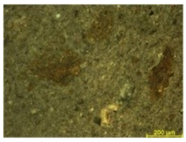
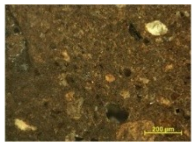
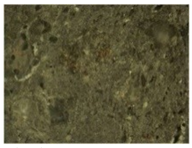
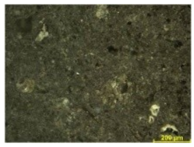
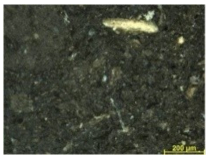
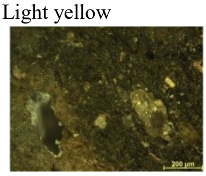
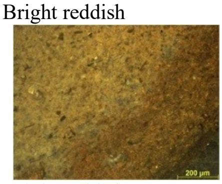
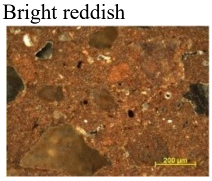
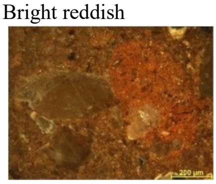
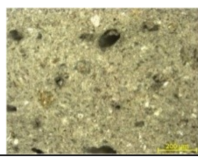
Sampling

Six samples from excavated Buddhist site at Udayagiri (20°38′30″N 86°16′09″E) India, at different depth were selected for the present investigation. Figure 1 shows representative images of selected potsherds.

Fig. 1. Representative fragment Images of some selected potsherds from Udayagiri, Odisha: (a) UDG-1; (b) UDG-2; (c) UDG-3; (d) UDG-4; (e) UDG-5 and (f) UDG-6

Numerous excavations were carried out by Archaeological Survey of India (ASI) Bhubaneswar circle, Odisha, since 1985 to 2004 at Udayagiri (1st century A.D to 12th Century A.D); a Buddhist site situated 102 km north east from Odishan capital city Bhubaneswar. The samples are labeled as UDG-1, UDG-2, UDG-3, UDG-4, UDG-5 and UDG-6 for Udayagiri potsherd (UDG-Udayagiri). After removal of surface impurities, the potsherds were used for instrumental analysis. Table 1 shows the colour of each pottery fragment from rear and front view.

Table 1. Description of potsherd samples from Udayagiri, India.

Sample Id	Location	Analytical methods employed	Colour (front) / Microscopic image	Colour (Rear) / Microscopic image	Texture
UDG-1	Udayagiri	XRF, FTIR, SEM-EDX	Grayish yellow 	Dark gray yellow 	Medium grained
UDG-2	Udayagiri	XRF, FTIR , SEM-EDX	Grayish yellow 	Grey 	Medium – coarse grained
UDG-3	Udayagiri	XRF, FTIR , SEM-EDX	Black 	Light yellow 	Medium grained
UDG-4	Udayagiri	XRF, FTIR , SEM-EDX	Bright reddish 	grey reddish 	Fine grained
UDG-5	Udayagiri	XRF, FTIR , SEM-EDX	Bright reddish 	Bright reddish 	Coarse grained
UDG-6	Udayagiri	XRF, FTIR , SEM-EDX	Gray 	Gray 	Medium – coarse grained

FTIR technique

The infrared spectra were recorded in the mid region 400-4000cm^{-1} using PERKIN ELMER FTIR interferometer. The KBr pressed pellet technique was used to record the spectrum. A sample is mixed with KBr in the ratio on 1:20 using a mortar and pestle and pressed to 5 tons for one minute in preparing the disc. The spectra were obtained at $4cm^{-1}$ instrument scans the resolution and the number of scans is 32 within the standard wave number. The best spectrum for each sample was considered as a representative spectrum and provided in Figure 2. To get more detailed view and analyses of the spectrum another region $2000\text{-}400cm^{-1}$ was selected shown in Figure 3.

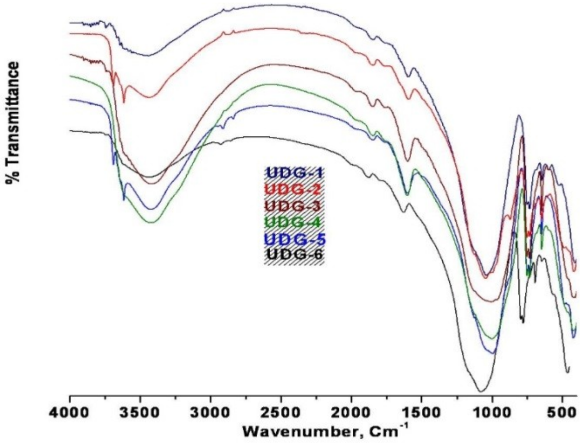


Fig. 2. FTIR spectra of pottery shreds within the region $4000\text{-}400cm^{-1}$
(a) UDG-1; (b) UDG-2; (c) UDG-3; (d) UDG-4; (e) UDG-5 and (f) UDG-6 respectively

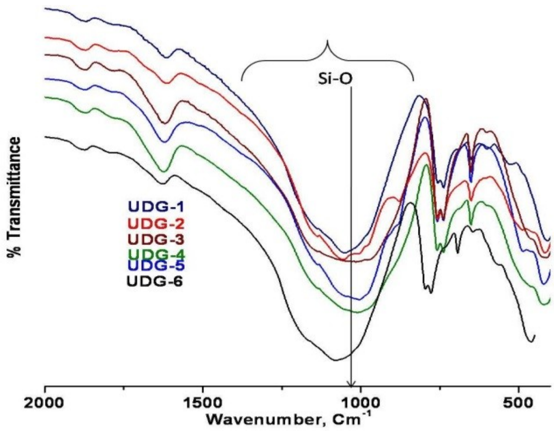


Fig. 3. FTIR spectra of pottery shreds within the region $2000\text{-}400cm^{-1}$
(a) UDG-1; (b) UDG-2; (c) UDG-3; (d) UDG-4; (e) UDG-5 and (f) UDG-6 respectively

XRF techniques

The pottery samples were analyzed for major chemical composition using XRF. The finely grinded powder samples were dried in the oven at a temperature of 105°C for 24 hours to make it moisture free. Glassy beads were prepared by fusing the flux (Merck A12, $Li_2B_2O_7$) 5.5000gm with the powered sample 0.5000gm at an ambient temperature of 1050°C for 30 minutes. The total weights of each bead are 6 gms. A Panalytical 4500 X-ray Fluorescence (XRF) instrument was used for the chemical analyses of the prepared glass beads. The XRF data for chemical composition for major elements are provided in the Table 2, with percentage in their oxide form. By calculating the mole number based on Seger formula the value of RO_2/R_2O_3 and $RO+R_2O/R_2O_3$ were calculated and provided in the Table 2.

Table 2. Chemical analysis (XRF) of characteristic potsherds from excavated Buddhist site, Udayagiri, Odisha, India.

Sample	SiO_2	Al_2O_3	Fe_2O_3	CaO	K_2O	Na_2O	MgO	MnO	TiO_2	P_2O_5	LOI	Total	Mole ratio	
													RO2/R2O3	**RO+R2O/ R2O3**
UDG-1	70.67	15.08	6.44	0.95	2.71	0.45	1.04	0.07	0.88	0.58	1.13	100.00	6.29802	0.42154
UDG-2	64.05	18.87	10.03	0.76	2.62	0.35	1.31	0.13	1.08	0.32	0.48	100.00	4.33352	0.32577
UDG-3	60.91	18.97	8.42	0.70	2.15	0.39	0.83	0.07	1.06	0.97	5.53	100.00	4.31333	0.26351
UDG-4	63.40	18.83	9.73	0.79	2.88	0.36	1.56	0.22	1.12	0.27	0.84	100.00	4.34748	0.37442
UDG-5	68.31	14.98	7.78	0.65	2.34	0.43	0.85	0.08	0.87	0.73	2.98	100.00	5.86137	0.33333
UDG-6	63.19	20.17	9.49	0.81	2.72	0.33	1.47	1.19	1.18	0.27	0.17	100.00	4.04336	0.38550

SEM-EDX analysis

The freshly fractured polished cross sections were sputtered with gold (Au) in order to obtain conductivity to prevent charging under electron beam. Backscattered electron images (BMI) and secondary electron images (SCI) are obtained by Zeiss Evo 50 EP Scanning Electron Microscope (SEM). The semi-quantitative chemical analysis of the slip layers were obtained with an Oxford INCA Energy 200 Energy Dispersive System (EDX). The EDX spectra were gained from 10mm working distance between 5 and 10 kcps at least 40 s. Quantification results were given in the form of oxides. The typical SEM and their EDX images of representative potsherds for UDG-3and 6 are shown in Figures 4 and 5 respectively.

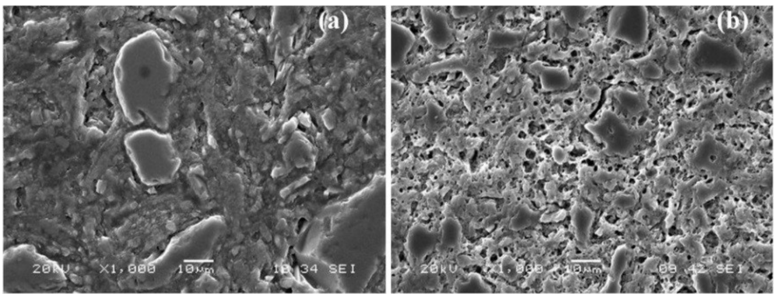


Fig. 4. Representative SEM Image of the sample (a) UDG-3; (b) UDG-6: 1000X

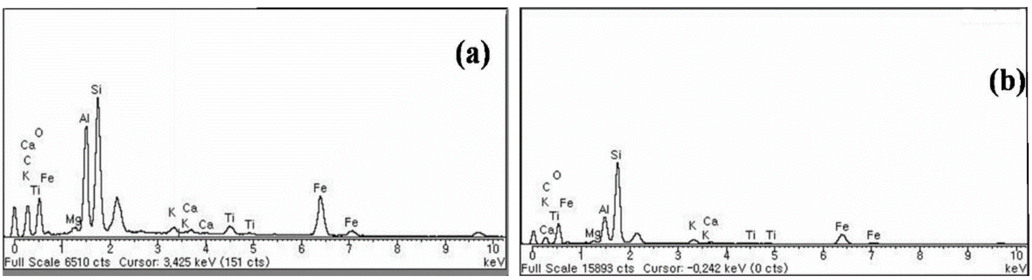


Fig. 5. EDX spectrum of (a) UDG-3; (b) UDG-6

Result and discussion

FTIR analysis

FTIR is very powerful tool to predict about the mineral composition and investigate their transformation with thermal effect. FTIR spectrums of six pottery fragments are shown in Figures 2 and 3 and their vibrational assignment for six pottery samples are provided in the Table 3.

Table 3. Observed adsorption frequency in the region of 400-4000cm^{-1} of the ancient potteries of Excavated Buddhist site Lalitgiri together with mineral identification.

Sample	SILICATE MINERAL	CLAY MINERALS			Hydroxyl (-OH) group stretching vibration from H-O-H			Iron oxides
	QUARTZ	Orthoclase	Feldspar	Kaoilinite				
UDG-1	797,694,778,661,830	-	728,661	1043	1635	-	3428	471
UDG-2	1080,778,796,712	545	-	-	1624	3694	3442	467
UDG-3	796,778,660,714,831	543	728,660	1046	1631	-	3419	462,524
UDG-4	1080,778,795,669,694	545	728,645	-	1630	-	3445	469
UDG-5	796,778,694,714,,834	543	714,658	1035	1631	3694	3431	471,524
UDG-6	1080,795,778,788,693	530	--	-	1626	-	3440	469

An intense peak at 3700cm^{-1} is due to asymmetric stretching vibration of the vibration hydroxyl group. According to *G. Velraj et al.,* [6, 11] the absorption band at 1629cm^{-1} are mainly due to H-O-H bending of water molecule and that may be from the moisture associated with potteries. A medium spectrum in all six spectrums appearing at 1630-1640cm^{-1} are due to H-O-H bending of water may be due to moisture present in the sample. Two weak peaks at 2860 and 2910cm^{-1} belong to C-H stretching vibrations and indicate the presence of organic materials [1-2, 13-16]. These organic materials can also be used by the porters during preparation of the pottery recipe, in order to improve plasticity [15, 24, 26-28]. A strong and broad band at 1000-1100 cm^{-1} region centered at 1040 and 1080cm^{-1} indicates the presence of Silicate (Si-O) in clay minerals. Since the bands present in this region were broad multiple peaks a Gussian band fitting procedure was employed in order to clarify the type, center and nature of peak present. After deconvolution of the IR band in the broad region of 1450-840cm^{-1} (as shown in representative potsherd UDG-1, Figure 6 (spectrum a and b) three peaks can be assign near 948, 1040 and 1148cm^{-1}. The band corresponding to 948cm^{-1} may be due to Muscovite which is also confirmed from SEM spectrum shown in Figure 4. Similarly, the band near 1040 and 1145cm^{-1} may be due to Orthoclase [12, 29]. According to *R. Palanivel* and *Rajesh Kumar* [9] and *R. Palaivel and G. Velraj* [13] intense peak at 1030 and 1080cm^{-1} are due to red clay or white clay origin Kaolinite respectively. The presence of absorption band at 795, 695 and 778cm^{-1} are due to presence of (Si-O) quartz. Other peaks of interest are 475, 580cm^{-1} due to iron oxides. Our results are in agreement with reported values of mineral composition of pottery by *C.E. De Benedetto et al.* [12].

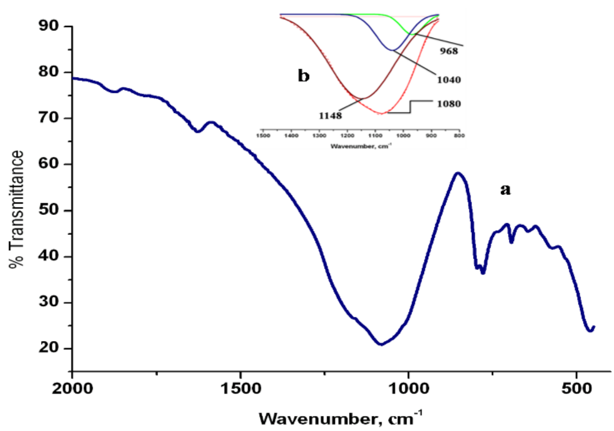


Fig. 6. Representative FTIR spectra of (a) UDG-1 within the region 2000-400cm^{-1} (b) deconvoluted IR frequency region 1440-8504cm^{-1} of UDG-1 spectrum.

Mineral and firing temperature analyses

FTIR study can be useful in determining the level of firing temperature and firing conditions of the clay artifacts. The sheet structure of clay mineral upon heating effect gets collapsed which can be monitored easily by FTIR investigation. The symmetric stretching vibration band at 3700cm^{-1} is mainly due to hydroxyl group. On firing clay mineral, the octahedral layer of clay mineral, which has a set of hydroxyl groups of, has been reflected by marked alternation and disappearance of the above bands. Broad Si-O stretching bands at about 1000cm^{-1} in all FTIR spectra are due to contributions from various silicate minerals. The thermal effect can be fairly judged form shifting or broadening of silicate band in the infrared spectrum. Structural changes that occur during the firing process will (including destruction of some minerals and formation of others) effects the position of Si-O stretching and deformation bands in FTIR spectra. According to the studies by *L. Damjanović et al.,* [1] the maximum of the Si-O band shifted systematically with thermal exposure of clay mineral. At 600°C the maximum peak will detected at 1036cm^{-1}; at 700°C, a single peak with maximum at 1042cm^{-1} was detected; at 800°C, the band split and two maxima were observed at 1050 and 1078cm^{-1}; similarly at 900°C, a single band appearing at 1082cm^{-1}. In our case the peak maxima at 1035cm^{-1} is obtained at for UDG-5, similarly 1043 and 1046cm^{-1} maxima is obtained for UDG-1 and UDG-3 respectively. But a broad peak centered at 1080cm^{-1} is obtained for UDG-2, UDG-4 and UDG-6. Comparing these positions, with the literature available it is possible to estimate the firing temperature range of Udayagiri samples as between 600-900°C. The presence of band at 1035 or 1080cm^{-1} in all the samples indicates the clay artifacts fired above 600°C and made up of disordered clay. A band near 3600cm^{-1} in samples UDG-2 and 5 are due to hydroxyl (-O-H) group for Al(OH) vibration in the Octahedral sheet stretching presence of which proves that the samples fired below 800°C. The mineralogical composition for final product of this clay material depends upon the local geology, firing atmosphere and how the pottery was used. Semi quantitative mineralogical results obtained from FTIR are provided in Table 3 showed that the main identified minerals of the same samples are quartz, feldspars, mica group (except UDG-2 and UDG-6) and magnetite in Udayagiri pottery. The presence or absence of specific mineral often used for the estimation of firing temperature of potteries [24-26, 29]. The absence of clay mineral like Kaolin indicates the firing temperature more than 500^{0}C. The presence of non-clay mineral components like quartz, muscovite and other mineral additive play an important role in the firing characteristics. These two minerals quartz and muscovite undergoes decomposition between 950°C and formation of magnetite formed at a temperature more than 850°C. The amorphous arrangement of silica and alumina is retained until a temperature of about 980°C. In the present studies, decrease of crystalinity of muscovite and formation of magnetite (Fe_2O_3) that is the replacement of Fe with Al with -OH in case of UDG-2 and UDG-6, indicates the firing temperature is more than the 850°C but presence of feldspar group indicates that the firing temperature did not exceed 900°C similarly the presence muscovite mineral indicates a temperature of less than 700°C. The production methodology of the artifacts containing magnetite shows that fired in a reducing atmosphere [4, 9,18-22, 25-29]. Table 4 provides the estimation of firing temperature of ancient pottery shreds. This estimation of firing temperature from FTIR and SEM-EDS are well correlating to each other.

Table 4. Clay type identified for the representative fragments along with the firing temperature estimate

Sample code	Clay type	Firing temp. (Estimated)
UDG-1	Non-Calcareous,Low refractory	750-800^{0}C
UDG-2	Non-Calcareous,Low refractory	850-900^{0}C
UDG-3	Non-Calcareous,Low refractory	700-800^{0}C
UDG-4	Non-Calcareous,Low refractory	850-900^{0}C
UDG-5	Non-Calcareous,Low refractory	600-700^{0}C
UDG-6	Non-Calcareous,Low refractory	850-900^{0}C

Chemical analysis

Chemical composition of the fabric strongly related to the actual clay source and other materials used for its manufacture. The data shows the represented fragments of pottery samples both from Udayagiri has high SiO_2 content and low CaO content are typical of non-calcareous clays with relatively simple mineral assemblage. The calcium deficiency in the above mentioned pots may indicate that either the raw clays were extracted from a non-calcareous deposit or were not refined with calcite temper. Alkalis have moderate concentrations 0.33%-0.45% for Na_2O and 2.15%-2.88% for K_2O. This sodium and potassium contents may be related to feldspar content. The composition of fluxes (K_2O, CaO, MgO and Na_2O) indicates more than 9% in all the pottery wares, hence the fragments collected for the present studies are of low refractory [4-7, 9]. The fluxes may helpful in high firing temperature and promoting sintering. Comparatively a high organic matter may be added to enhance the plasticity to the clayey material. *R. Ravisankar et al.* [4] suggests that existing of non-calcareous type (Ca<5%) clay reveals the samples fired below 900^0C. The value of RO_2/R_2O_3 is relatively high that is within the range of 4.09 to 6.29 and while $RO+R_2O/R_2O_3$ is low which is in between 0.26 to 0.42. The consistence values of fluxes and stabilizers shows that, similarity in surface characteristic, firing range, and the recipe for the pottery preparation.

SEM/EDX Analysis

The micrographs about the type of clay used [15, 22] tempering materials, morphology [16] and chemistry [20] of the final product can be observed from micro-morphology of pottery. The characteristic micrographs for potsherd samples of Udayagiri are shown in Fig.4 and also in Table 1**.** SEM observation confirms the results found through the mineralogical (FTIR) and Chemical (XRF) analyses. As stated by *C. Grifa et al. [*20] when the pottery is fired a temperature higher 850°C the sheet-like texture is completely disappeared. Similarly, the cellular structure and development of larger pores in the clay matrix shows its presence when the samples are fired above 950°C. Abundant phyllosilicates in the pottery matrix shown in Figure 5 suggests a lower firing temperature that is a temperature between 700 to 750°C. According to *S. Kramar et al.* [25] the presence of many small pores with a vitrified surface as in UDG-6 indicates a higher firing temperature which resembles the estimated firing temperature in case of FTIR. The occurrence of elongated pores with no grain to grain contact could be seen in UDG-3 suggesting a firing temperature lower than 800°C [4].

Conclusion

Following conclusion could be drawn from overall analysis of the represented fragments collected from excavated sites, Udayagiri, India. The combined analytical technique of FTIR with XRF and together with SEM-EDX provides useful information about the mineral and chemical composition. From the analysis firing temperature of the potteries were established. From the mineralogical and chemical results it is found that the pottery samples were having tempering material Quartz (SiO_2) which reduces the propagation of shrinkage crack during initial firing and prevent the pottery from cracking during firing. The images from SEX-EDX provide the use tempering material in the final products. The artisans of that period have sufficient knowledge how to use tempering minerals according to the demand and use of the pottery. Last but not the least the use of combination modern instruments rather than a particular one; provide more authentic and reliable information about the ancient artifacts.

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