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FTIR Investigations of the Selected Pottery Sample Excavated From Korkai-Tamil Nadu, South India

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Abstract:
The present work investigates the presence of minerals, raw materials used and ceramic technologies adopted in making pottery during the ancient period belonging to one of the important archeological site Korkai of Tamil Nadu, India. One pottery sample of the period 800BC is used for the present work. Fourier Transform Infra Red (FTIR) spectroscopy has been used for the studies. The presence of quartz, kaolinite, montmorillonite, haematite and magnetite in the selected pottery sample has been identified. The classification of the firing temperatures and firing features of pottery sample is also presented. The firing of pottery under reduced condition is judged from the magnetite and haematite contents establishing the ability of the ancient artisans of Tamilnadu to achieve controlled firing.

Keywords: *FTIR, Kaolinite, quartz, haematite, magnetite, montmorillonite*

1. Introduction
The study of pottery through the ages is considered to be an essential requirement to identify, understand and characterize civilizations. The style of pottery, the clay minerals used, the firing temperature and firing techniques employed by ancient artisans, if known, can all be helpful in unraveling the cultural sequence of a particular archeological site, the trade and cultural links it had in the past, the technological advancement made and the historical periods involved. The characteristics of the ancient pottery sample obtained from excavations are mostly not known. So, investigations have to be made to assess the characteristics of ancient potteries so that the technological knowledge, skills of the ancient artisans, the age of civilizations, cultural links, etc., can be understood. Extensive investigations have been made on the various aspects of ancient materials excavated from different sites in countries like Greece, Rome, Bulgaria, Egypt, Iraq and China. But in India such studies are scanty and this is more so in South India. So in this work an attempt has been made to study the pottery sample obtained from Korkai which is one of the important archeological site of Tamilnadu with a view to analyze the characteristics of the selected pottery sample which belongs to the period of 800 BC.
A brief account of the available history of the selected site and their importance is given below.

2. Korkai
Korkai is situated at a distance of three kilometers to the north of the Tamaraparani river of latitude $8^{o}38'20''$ and longitude $78^{o}03'34''$ in Srivaikundam taluk of Tuticorin District in Tamil Nadu. It was a famous ancient port city in the Pandya Kingdom during Sangam period. The Archaeological Survey of India conducted excavations and the excavations revealed a large number of urn burials of pre Christian era dating back to 8^{th} century B.C, potteries, terro cotta objects, coins etc.

3. Method
The selected Pottery sample has been analyzed by recording its vibrational spectra with the aid of FTIR spectroscopy using Perkin Elmer FTIR spectrophotometer model 1600 instrument in the range 4000-400 cm^{-1} in the 16 scan mode as potassium bromide pellet.

4. Results and Discussions
The selected pottery sample has been studied in the "as received state"(ARS) and the FTIR spectrum obtained is shown in figure 1. Infrared frequencies in cm^{-1} of the pottery sample (as received condition) from Korkai along with vibrational assignments are given in table1.
The spectrum shows that the sample has a broad band in the hydroxyl stretching vibrational region of 3700-3400 cm^{-1} which is the characteristic clay mineral region. The existence of a wide band in the 3700-3400 cm^{-1} region in the pottery sample may be taken to

indicate the highly disordered nature of the clay minerals present in them. Since the pottery is essentially a fired sample, it is but quite natural that the clay minerals are in a highly disordered state.

In the pottery sample a broad band appears around 3430 cm^{-1}. This band taken together with the weak band around 1630 cm^{-1} represent the O-H stretching and O-H bending due to adsorbed water. Miller has shown that in the case of kaolinite, the 3718 cm^{-1} peak is broadened and is shifted to around 3650 cm^{-1}on heating the kaolinite to temperatures of the order of 820 ^{0}C and beyond when it is in the completely disordered form. The pure kaolinite has four bands in the O-H stretching region 3718 cm^{-1}, 3686 cm^{-1}, 3672 cm^{-1}, and 3636 cm^{-1}. Pure montmorilonite has bands in the O-H stretching region at 3650 cm^{-1} and 3520 cm^{-1}.

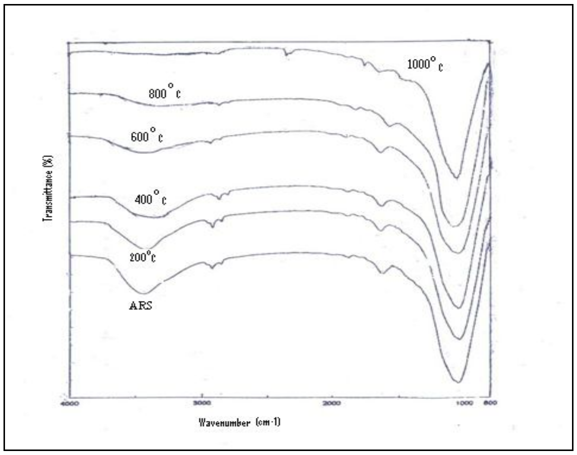

Figure 1

Wave number (cm^{-1})	Assignments
3856.93	O-H Stretching of clay mineral
3436.53	O-H Stretching of adsorbed water
2364.3	Si-O of quartz
2337.3	Si-O of quartz
1887.0	Si-O of quartz of fine particle
1461.7	SI-O Stretching of clay minerals
728.96	Si-O of quartz
682.00	Si-O of SiO_4
641.69	Si-O bending mode in Si-O-Si
571.00	Fe-O of Fe_3O_4
535.90	Fe-O of Fe_2O_3
478.26	Si-O-Si bending of silicate

Table 1

Similarly, the weak bands in the range of 3525-3570 cm^{-1}obtained for the sample may be taken to indicate the possibility of the presence of some highly disordered montmorillonite or calcined montmorillonite in the sample. So a mixture of non-plastic kaolinite and more plastic montmorillonite appears to have been used as raw clay minerals in the manufacturing of pottery by the ancient artisans. According to Miller, the bands at 1100 cm^{-1} and 915 cm^{-1} are due to the vibration of aluminum hydroxyl in the octahedral sheet structures.In highly disordered kaolinite the bands at 1100 cm^{-1} and 1035 cm^{-1} merge to give a broad absorption. It is also found that there is progressive destruction of the octahedral layer on account of heating accompanied by weakening of the 915 cm^{-1} band.

The sample shows a doublet absorption bands around 795 cm^{-1} and 780 cm^{-1} and a peak around 693 cm^{-1}. This doublet and the band at 693cm^{-1} are attributed to the Si-O modes of crystalline quartz. Farmer and Russell have shown that the presence of a sharp band at 694 cm^{-1} can be attributed thin particles/fine grains and in the case of thick particles/coarse grains this band would shift to 689 cm^{-1}. The sample has hematite as indicated by the peak around 535 cm^{-1} which is the characteristic peak of haematite. It has also a peak around 571 cm^{-1} indicating the presence of magnetite.

It has been found that the sample of Korkai indicates the presence of strong well defined haematite and magnetite peaks. So it is likely that the sample would have been fired under reduced conditions.

Potteries are essentially fired clay materials. So an understanding of the thermal transformation occurring in clay materials when they are heated may help to infer the firing temperatures.

To get a clear idea about the firing temperature that could have been used in firing the selected pottery sample, a refiring study has been conducted. The refired pottery sample has been studied using the FTIR spectroscopic technique. It is an established fact that once a ceramic is fired at a certain temperature and altered, it “freezes” at this stage, which cannot be altered by subsequent refiring unless the original firing temperature is exceeded. This property of the ceramic provides a method of assessing the firing temperature used in making a certain ceramic in antiquity For estimating the firing FTIR spectroscopy through the refiring test, the sample has been refired in the laboratory in a furnace at the temperatures of 200 ^{0}C, 400 ^{0}C, 600 ^{0}C, 800 ^{0}C and 1000 ^{0}C. The FTIR spectra obtained for the refired sample are shown in figure 1 along with the spectra for the ‘as received state’ sample. Comparison of these spectra obtained at different firing temperatures for sample indicates that the peaks around 3650cm^{-1} and 3550 cm^{-1} and the wide band in the hydroxyl region exist up to 800 ^{0}C and disappears at 1000^{0}C, the Si-O peak and wide band in the 1100-900 cm^{-1} region remain more or less unaltered upto 800 ^{0}C and vanish at 1000 ^{0}C.

5. Conclusion

The FTIR analysis shows that the pottery sample contains kaolinite, quartz, haematite and magnetite. The clayey matrix appears to be composed of highly disordered calcined montmorillonite.

The study has shown that the artisans used mixer of kaolinite and montmorillonite clays for making pottery and that in addition to the clay, quartz also formed raw materials. The study has also shown the technological capabilities of the artisans with respect to their ability to achieve the necessary firing temperature, their ability to adopt controlled firing and their understanding of the properties of clay and other materials they used in making potteries. The study has brought to light the interesting fact that the ancient artisans of Korkai region of Tamilnadu during the ancient period it self have adopted almost the same technologies which are being used in the modern 21st centaury for making potteries.

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