FT-IR spectroscopic analysis of archaeological pottery from Arikamedu, Puducherry, India

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ABSTRACT

The present work deals with the FTIR spectroscopy study of the ancient pottery from excavated from Arikamedu, Puducherry, India. This technique is a powerful tool for assessing both chemical and mineralogical composition of the archaeological materials. The firing methods and firing conditions of potsherds at the time of manufacture by a potter were inferred from the characteristic absorption peaks and the bands observed due to the presence of magnetite and hematite in the samples. FT-IR spectroscopic results revealed that archaeological samples were fired to a temperature greater than 800°C.

Keywords: Ancient Pottery, FT-IR, Firing Temperature

INTRODUCTION

The artistic and archaeological approach gives a guideline to the classification of an artwork, analyzing the manufacture’s stylistic features to place it in a correct geographical and historical framework. Many papers about multidisciplinary studies on artistic objects were published so underlying the importance of the cooperation between humanistic and scientific efforts [1]. The identification of the excavated material origin is by no means easy and straightforward. Only the power of combined analytical techniques allows for confident and detailed establishment of the geographical origin of the raw material used to manufacture the investigated object. In all types of ceramic manufacturing and other types of applications, clay is used as a main material. Kaolinite and illite are the most widely used clay mixture in the ceramic industry [2]. FT-IR spectroscopy is common and well established tool for the ceramic body identifications, also reveal the type of clay and temperature of firing and firing condition. Many researchers have applied FT-IR spectroscopy in archaeological potteries [3-5]. Based on the available literature, it is found that the mineralogical composition and firing condition are inferred from the FT-IR spectrum.

Hence FT-IR technique is employed in the present work to analyze the chemical compositions and estimate the firing temperatures achieved by the artisans of ancient times and the firing techniques adopted for the pottery shreds collected from the archeological site Arikamedu, Puducherry, India.

MATERIALS AND METHODS

The pottery shreds were recently excavated from the site Arikamedu (11°53’26”N; 79°48’32”E) of Puducherry, India. The pottery shreds of Arikamedu belonging to 5th century BC. Black and Red ware and Red ware were collected in the site. The samples are labeled as AM1, AM2 & AM3. After removal of surface layers, the pottery shreds were grounded into fine powder using agate mortar. They were sieved using an 85µm mesh.
The infrared spectra were recorded in the mid IR region 400–4000 cm\(^{-1}\) using PERKIN ELMER FTIR interferometer in Centralized Instrumentation Sophisticated Laboratory, Annamalai University equipped with Glober source. The KBr pressed pellet technique was used to record the spectrum. The crushed samples were grounded before making the KBr pellet. The samples were mixed with KBr in the proportion of 1:20 and pressed to 5 tones for one minute in preparing the disc. The accuracy of the measurement is ±5 in 4000 to 400 cm\(^{-1}\) region. The best spectrum was considered as a representative spectrum of the samples. Fig-1 shows the FT-IR spectrum of the Arikamedu potsherds.

**RESULTS AND DISCUSSION**


The great advantage of FTIR spectroscopy is high sensibility that permits the determination of many components, even in very small amount. Frequencies (cm\(^{-1}\)) obtained from recorded spectra of two clay samples are tabulated with corresponding minerals in Table 1. The different types of minerals were identified in the potsherds such as quartz, feldspar, clay minerals, hematite, magnetite and organic carbon from the IR absorption bands. Quartz is one of the crystalline minerals. The presence of the bands around at 460, 695, 775 cm\(^{-1}\) along with 1080 cm\(^{-1}\) in the pottery fragments is due to quartz [6]. The presence of the sharp band at 695 cm\(^{-1}\) indicates thin particles. The presence of broad absorption band in all samples around at 3440 cm\(^{-1}\) along with the weak band at around 1640 cm\(^{-1}\) are due to absorbed water [7]. Kaolinite is the major constituent of clays mineral is identified by presence of the band at 1030. The absorption band positioned around at 645 and 730 cm\(^{-1}\) may be assigned to Al-O-Si stretching of feldspar (orthoclase). The IR absorption bands at 460-465 and 585-590 in the samples indicates microcline. The presence of peak at 720-725 cm\(^{-1}\) in the samples indicates the presence of albite [8].

<table>
<thead>
<tr>
<th>S.No</th>
<th>Observed wave numbers (cm(^{-1}))</th>
<th>Mineral Name</th>
<th>Sample ID</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.</td>
<td>458, 777, 695, 1080</td>
<td>Quartz</td>
<td>+ + +</td>
</tr>
<tr>
<td>2.</td>
<td>466, 590, 645, 723, 728</td>
<td>Feldspar</td>
<td>+ + +</td>
</tr>
<tr>
<td>3.</td>
<td>1033, 1642, 3441</td>
<td>Clay Minerals</td>
<td>+ + +</td>
</tr>
<tr>
<td>4.</td>
<td>540</td>
<td>Hematite</td>
<td>+ + +</td>
</tr>
<tr>
<td>5.</td>
<td>580</td>
<td>Magnetite</td>
<td>- - +</td>
</tr>
<tr>
<td>6.</td>
<td>2925, 2855</td>
<td>Organic matter</td>
<td>+ + +</td>
</tr>
</tbody>
</table>

Absorption band around 540 cm\(^{-1}\) is due to hematite present in the the pottery samples was reported by Velraj et al [4]. This band was observed in all the three samples. The band 580 cm\(^{-1}\) indicates that the presence of magnetite was observed in the sample AM3. This band does not appear in AM2 and AM3 indicates the absence of magnetite. Lara Maritan et al and Colombini et al have reported that the two weak peaks observed at 2852 cm\(^{-1}\) and 2922 cm\(^{-1}\) are due to C-H stretching mode and reveal the presence of some organic contribution [9-10]. The above two bands were observed in all the pottery fragments of Arikamedu. The organic matters have a high specific surface and good molding capacity, improving the plasticity of the clays during fabrication.
3.2. Firing Temperature Determination using FT-IR study

The spectra of the all samples in the received state shows no characteristics absorption bands for the inner hydroxyl water bands at 3700 and 3620 cm\(^{-1}\) indicating the firing temperature of the sample might be more than 450°C. Ramaswamy and Kamalakannan have reported that the IR absorption band at 915 cm\(^{-1}\) is due to Al(OH) vibrations in octahedral sheet structure which begins to disappear at the temperature 500°C [11]. But in all the samples chosen for the study have no such band. It is indication that all the samples were fired above 500°C. A broad symmetry band centered at 1030 cm\(^{-1}\) indicates that the samples might have been subjected to firing temperature of 650°C. The presence of the band at 1030 cm\(^{-1}\) in the all the spectra indicates that the clay is disordered conditions, possibly due to iron substitution which leads to red clay type [12]. At 650°C, silicate structure collapses and a broad symmetry band is observed at 1030 cm\(^{-1}\) for red clay and 1080 cm\(^{-1}\) for white clay. These two bands were observed in the samples AM1 and AM3 reveal that pottery made by both the clays during manufacturing by artisan. The presence of hematite in all the samples and magnetite in AM3 indicate that the samples were fired above 800°C. The appearance of absorption peak at 535 cm\(^{-1}\) in all samples in received state implying that the potteries were fired in an oxidizing condition, the reason for the red colour of the pottery [4]. This indicates that the artisans of Arikamedu were well aware of technique of firing the potteries in an oxidizing atmosphere.

From the above discussions one can conclude that the samples was subjected to firing temperature around 800°C under oxidising atmospheric condition.

CONCLUSION

Application of FT-IR spectroscopic for the study of the archeological pottery provided useful information about the mineral composition, the firing temperature and firing atmosphere of the pottery. The different types of minerals were identified in the potsherds such as quartz, microcline, orthoclase, kaolinite, montmorillonite, hematite and magnetite. From the FT-IR technique it is focused that all the samples of Arikamedu have been fired above 800°C and confirmed that the samples were fired in the oxidizing atmosphere.

REFERENCES