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Firing Temperature Analysis of Ancient potsherds from Odugathur, Vellore Dist, Tamilnadu, India by TG-DTA Technique

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Abstract : Firing is the key process in pottery production. Firing temperature analysis of the potteries may be used to assess the basic properties of the matrix and its temper. TGA-DTA study is commonly employed to thermal studies of potsherds to estimate the firing temperature. In the present work, thermogravimetry (TG) and differential thermal analysis were carried out for pottery samples recovered from the ancient settlements of Odugathur. The TG–DTG curves were obtained in the temperature range 0–1000°C. Thermal analysis results showed larger mass loss at dehydration than dehydroxylation and the organic material is used in the making of the pottery. From the analysis Odugathur potsherds were fired below 800°C.

Keywords : Ancient Pottery, Firing Temperature, DTA-TG analysis.

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1. Introduction

Archeological artifacts like potteries, bricks and tiles are of the earliest and most significant innovation of mankind. Ancient potteries are the most common artifacts found during excavation of archeological sites, so archaeologists are interested in the studies of pottery fragments. The systematic scientific study of the above artifacts reflects the technological development, civilization and trade links between different countries during the past .Various spectroscopic and magnetic studies are used for the study of archeological artifacts like potteries, bricks and tiles.

Pottery is capable of revealing many aspects concerning a prehistoric culture including probable place of manufacture, the origin of raw material, trade etc. Firing is the key process in pottery production. The quality of firing undoubtedly influenced the properties of vessels. We can assume that firing could be a value criterion for ceramic products. Today it provides a valuable source of information for archaeologists, helping us to understand the level of a potter's skill in a particular workshop, at a given time. It also allows us to judge the quality of the product. So it is important to determine the approximate firing temperature and assess the basic properties of the matrix and its temper. Using laboratory analysis should also answer the question of how pottery may contribute to our knowledge of pottery production and whether pottery can be taken as an indicator of significant changes in society (e.g.by changing type of temper or preparing of ceramic mass).

The temperature analysis results obtained in laboratory differ greatly between laboratories. The firing temperature range is generally accepted among archaeologists and confirmed by thermal analysis. TGA-DTA study is commonly employed to thermal studies of potsherds which may give degradation temperatures, absorbed moisture content of materials, reaction processes and combustion of organic materials in the ancient artifacts. Several authors had estimated the firing temperature of the potteries using TG-DTA techniques [2, 6, 7, 8 & 10]. This paper aims the firing temperature analysis of potsherds recovered from the ancient settlement of Odugathur, Vellore Dist, Tamilnadu by TG-DTA technique.

2. Materials and Methods

2.1. Sample Collection

The pottery samples were recovered from the site Odugathur village of Vellore Dist (lat.13º 48'N; long 80º 10"E). The samples were collected at 8m from the surface of the soil. The pottery shreds of Odakathur village belonging between 100BC and 300 AD in South India. Red slipped ware was collected in the site. All the fragments collection is different in variety and colors. The typical collection of pottery samples is shown in Figure 1. The samples are labelled as OD1, 2, 3 & 4. After removal of surface layers, the pottery shreds were ground into fine powder using agate mortar. This fine powder is used for different analyses.

Figure 1. The ancient potteries of Odugathur, Vellore dist

2.2. Thermal Analysis (DTA-TG)

Thermal analysis that includes thermogravimetric (TG) investigations and differential thermal analysis (DTA) has been performed using DERIVATOGRAPH 1500Q. The data were collected up to 1000°C with increments of 10° C/min.

3. Results and Discussions

3.1. DTA-TG Analysis

Differential thermal analysis is used to study how individual minerals or substances change during a gradual increase in temperature (sample is heated to approximately 1,000 °C). DTA has served primarily to determine the firing temperature of the samples. The results were evaluated on the basis of a thermogram, where it is demonstrated by a loss of mass, together with endothermic and exothermic reactions. The TG-DTA curves of two ancient potteries coded as OD1 & OD2 from odugathur is shown in Figure 2 & 3. Differential thermal analytical data of odugathur shreds is given in Table 1 and the weight loss percentage during their firing from room temperature to 1000°C is presented in Table 2.

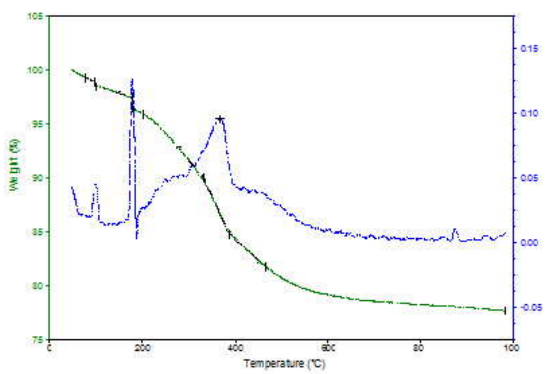

Figure 2. TG-DTA Cuves of OD1

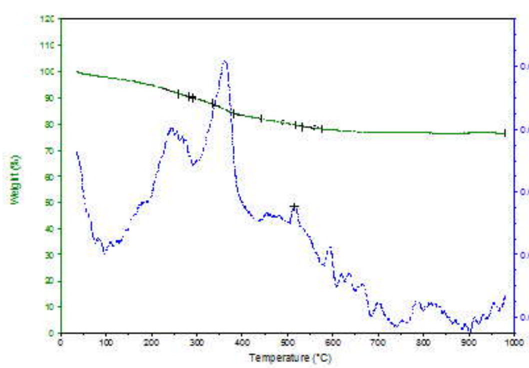

Figure 3. TG-DTA Cuves of OD2

Moropoulou et al [5] and Franquelo et al [3] stated that the endothermic peak around 100°C to 200°C is due to the adsorbed water. The endothermic peak observed between 100-200°C in DTA curves characterize the adsorbed water in the samples of OD1 & OD2. The presence of an exothermic peak from 250°C–500°C is due to combustion of organic material in the pottery shreds as reported by Shoval [9] and Clark [1]. In our study, a small exothermic peak found in OD1 & OD2 in the temperature range of 250-500°C indicates combustion of organic material present in the samples.

Shred Code	Peak Nos	Peak Temperatures
OD1	1st	101
	2nd	180
	3rd	360
OD2	1st	90
	2nd	260
	3rd	530
	4th	650

Table -1

DTA data of odugathur shreds

Shred ID	Weight Loss (%)				Total Weight Loss (%)
	Dehydration (30 - 200 °C)	Decomposition of hydroxyls (400 - 600° C)	Decomposition of calcite (700 - 800° C)	Residual mass (%)	
OD1	1.08	1.48	0	77.65	22.35
OD2	3.0	2.80	0	76.66	23.34

Table-2

TG data of odugathur shreds

Mackenize et al. [4] have stated that the endothermic peak appearing from 550°C–650°C is due to the decomposition of kaolinite and it is due to its dehydroxylation. The presence of dehydroxylation kaolinite peak is the indication that the pottery is not fired above 650°C. The absence of this endothermic peak in OD1 indicates that the shred is fired at temperature above 650 °C. In OD2, the presence of kaolinite is well evidenced by endotherm between 550 and 650° C. It indicates that the kaolanite mineral has survived in the firing process applied by the ancient potter. The presence of dehydroxylation kaolinite peak is the indication that the pottery is not fired above 650 °C. Further the absence of exothermic peak between 900 and 1000 °C in OD1 & OD2 indicating that all the samples were fired at 900ºC or below this temperature.

According to Drebushchak et al [2] the weight loss of the pottery in thermogravimetry can be explained in three steps. The thermal loss from temperature ranging from room temperature to 100 ºC is due to dehydration, from 400-500 ºC is due to decomposition of hydroxyls and from 700-800 ºC is due to the decomposition of carbonates, mainly calcite.

Based on the above statement, the weight loss between room temperature to 100 ºC is due to dehydration and it is noticed in the TGA curves of OD1 & OD2. The weight loss (%) of OD1 and OD2 of dehydration are 1.08 % and 3.0% respectively. In our analysis, the samples OD1 & OD2 showed less than 3% of weight loss during 400-600°C as a result of decomposition of hydroxyls. The absence of peak in the region of 700-800 ºC indicates the absence of calcite in the samples. The total weight loss through the entire thermal analysis of OD1 & OD2 is 22.35% and 23.34% respectively. The residual mass in TG analysis of OD1 & OD2 in our study is 77.65 % and 76.66%.

From the analysis, the samples OD1 & OD2 are considered to be fired in between 700-800 °C as evidenced by the absence of calcite peak in this respective region of temperature. Further the organic material might be added intestinally as a binder in the preparation of pottery.

3.2. Archimedes Method

For a more comprehensive understanding of the properties of pottery from odugathur, the Archimedes method was used. This involves weighing samples (which were dried under temperature of 110 °C) immersed in water and, after another drying, determining their water absorption from the differences of their weight. This provides us the basic information concerning the vessels and their possible use, and also expands our knowledge about the quality of the firing process and the matrix. Higher the results are, the lower quality of firing process is expected (Table 3).

Shred ID	**Absorbability [%] in water**
OD1	13.60%
OD2	10.47%
OD3	16.02%
OD4	14.57%

Table-3

Measuring of water absorption results

Conclusion

The thermal reactions associated with the linear temperature ramp between room temperatue to 1000°C in an inert atmosphere were also realized by TG-DTA analysis of the potsherds from odugathur. The presence of the exo and endothermic peaks in DTA analysis, shows that the firing temperature of the samples OD1 & OD2 must be below 800°C. TG analysis also reveals the percentage of mass loss due to dehydration, dehydroxylation, and the combustion of organic material contained in the clay used to make the pottery sample studied in the present investigation. Further Thermal analysis results showed larger mass loss at dehydration than dehydroxylation and organic material is used in the making of the pottery. Finally the odugathur, potsherd was fired below 800°C from the TG-DTA analysis.

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