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# A view of microstructure with technological behavior of waste incorporated ceramic bricks

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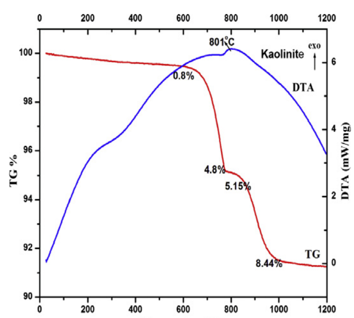
## HIGHLIGHTS

- TG–DTA analysis of raw clay (Kaolinite).
- Study of technological properties for recycled ceramic bricks.
- Supportive microscopic observation through SEM analysis.
- Provides an optimum temperature and composition for the recycling of ceramic rejects.

## GRAPHICAL ABSTRACT

TG–DTA curve for raw clay (Kaolinite).




**TG-DTA curve for raw clay (Kaolinite)**

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## ABSTRACT

Production of ceramic bricks from mixtures of ceramic industry wastes (up to 50 wt%) from the area of Vriddhachalam, Cuddalore district, Tamilnadu, India and kaolinitic clay from Thiruvananthapuram district, Kerala were investigated. The firing behavior of the ceramic mixtures was studied by determining their changes in mineralogy and basic ceramic properties such as water absorption, porosity, compressive strength and firing shrinkage at temperatures ranging from 900 to 1200 °C in short firing cycles. The effect of the rejects addition gradually up to 50 wt% was analyzed with the variation of temperature on the mechanical properties and microstructure of the bricks. The highest compressive strength and lowest water absorption is observed for the sample with 40% rejects at 1100 °C which is supported by the results of SEM analysis. The resulting ceramic bricks exhibit features that suggest possibilities of using the ceramic rejects in the conventional brick making methods.



## Introduction

Alternative methods for the final disposal of wastes from various industries are the need of the hour. The business conscience of recycling stimulates both small and big enterprises to look for alternative solutions aiming at recycling and adding commercial value to the final products taking into consideration the environmental legislation.

An environmentally correct solution, which is nowadays being investigated, is the incorporation of these wastes in cement and clayey bodies [1–3]. In particular, the recycling of different solid wastes, both from the city and industrial origins, through its addition into ceramic products is worldwide being studied [4–6].

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The high temperature firing stage typical of the brick manufacturing is fundamental to the sludge/clay particles consolidation. The firing stages also make it possible to immobilize the contaminants within the silicate phases [7,8].

Crushed powder obtained from fired ceramic wastes is a non plastic material used in the clay mixture to produce ceramic materials. The recycling of the ceramic rejects in the same industry leads to the lower production cost due to the lesser usage of raw materials. The previous works show the prospective benefits of using fired ceramic wastes as the brick or tile additive in the fired matrix in which the wastes from bricks was added up in quantities to 20 wt% that were fired at 970 °C [9,10]. The impact of the addition of the rejects on the porosity and mechanical strength of the final product should be studied in order to estimate the limited amount of wastes to be added into clayey body for the processing and improved quality of the ceramic product. Ceramic waste powder when incorporated into a mixture acts as fluxing agent in replacement of traditional feldspar to obtain a vitreous microstructure during sintering of ceramic materials. In the present paper, the effect of the fired ceramic frits (CF), the ceramic wastes from the ceramic materials, added into a clayey body (up to 50 wt%) was studied with the sintering temperatures of 900–1200 °C in order to correlate the properties and microstructure of the industrial ceramic body with the addition of the wastes.

## Materials and methods

### *Preparation of specimens*

The ceramic frits, a waste material generated while producing any type of ceramic material, is obtained from the Government ceramic industries of Vriddhachalam (located in the state of Tamilnadu, India) and made into a fine powder in a pulvariser. The basic raw material (Kaolinitic clay) is obtained from Thiruvananthapuram district, Kerala, India. The raw material and CF were mixed with water and the slurry was dried at 100 °C in a rotating drier until 8–10% humidity. The dried material was then crushed and sieved to pass through a 150 mesh (100 μm) to obtain suitable powders for pressing. Unfired rectangular (90 mm × 30 mm × 30 mm) specimens in lots of 10 for each mixture were moulded using an extrusion apparatus. Six batches of samples were made in which the percentages of CF are as in Table 1.

Firing was carried out in a laboratory electric furnace reaching different maximum temperatures in the range of 900–1200 °C at regular temperature intervals of 100 °C with a soaking time of 1 h at the maximum temperature needed. Cooling occurred by natural convection after it was turned off.

### *Experimental techniques*

The knowledge of chemical and mineralogical compositions is mandatory in characterization studies of clay mixtures. The elemental composition of the clay material and CF was analyzed through XRF (PW 1400 Philips).

For FTIR analysis, dry grinding was carried out by placing 50 mg of the sample in an agate mortar. Using KBr pellet technique, the sample is mixed with KBr at 1:30 ratio since it gives rise to maximum transmittance [11] and the mixture is then pressed into a transparent disc in an evocable dye at sufficiently high pressure. Using the Nicolat–Avatar 330 series FTIR spectrometer, the infrared spectra for all the samples were recorded in the region 4000–400 cm$^{-1}$. The resolution of the instrument is 4 cm$^{-1}$ and the accuracy ±0.01 cm$^{-1}$.

Thermal analysis was performed on raw clay (kaolinite). Measurements were run by coupled Differential Thermal Analysis (DTA) and Thermal gravimetry (TG).

Microphotographs of the samples were recorded with a JEOL JSM 5610LV SEM.

After sintering at selected temperatures, the specimens were subjected to compressive strength, firing shrinkage and water absorption tests. Compressive strength was determined using a universal testing machine by dividing the maximum load with the applied load area of the brick samples. Water absorption was determined according to the formula,

$$\text{Water absorption}\% = 100(W_d - W_i)/W_d$$

$W_d$ – Weight of the dry sample; $W_i$ – Weight of the sample after 24 h of immersion in water.

The firing shrinkage of the fired samples was determined as

$$FS\% = 100(L_R - L_F)/L_R,$$

$L_R$ and $L_F$ are the lengths of the raw and fired specimen respectively.

To determine the apparent porosity, the samples were heated continuously in boiling water for above 6 h and left to cool overnight which enables the pores to get filled up with water to saturation. The saturated specimens were then weighed by immersing in water as ($W_1$) and in air as ($W_2$). The samples were then placed in hot air oven at 200 °C and dried for about 6 h to remove the water contents completely and then weighed as ($W_3$). To standardize the values of the results the percentage of porosity was calculated using the relation,

$$\text{Porosity}\% = 100(W_2 - W_3)/(W_2 - W_1)$$

## Results and discussion

The chemical composition of the kaolinitic clay and the reject material is presented as in Table 2. The clay material values show the expected typical composition: rich in silica and alumina with minor amounts of CaO, $K_2O$, Mg, Fe and Na oxides. The CF material basically contains $SiO_2$ and $Al_2O_3$ in a proportion like kaolinite, indicating its aluminosilicate origin. The major difference in terms of chemical composition of the two raw materials lies in the alumina content of CF. The significant difference in the amount of iron oxide will be responsible for minor coloring of the fired samples. Considering these resulted compositions of the clay and the rejects, the properties of the fired mixture should be those expected for a conventional ceramic material.

In TG–DTA curve, the clay showed an endothermic peak at 60 °C, with a mass loss of 1.4%. The mass loss of 0.8% between

**Table 1**
Composition of the test samples (in wt%).

| Sample | CF0 | CF1 | CF2 | CF3 | CF4 | CF5 |
|---|---|---|---|---|---|---|
| Kaolinitic clay | 100 | 90 | 80 | 70 | 60 | 50 |
| Ceramic frits | 0 | 10 | 20 | 30 | 40 | 50 |

**Table 2**
Chemical composition of the clay material and CF (in wt%).

| Oxides | Kaolinitic clay | CF |
|---|---|---|
| $SiO_2$ | 51.93 | 51.94 |
| $Al_2O_3$ | 45.55 | 42.77 |
| $Fe_2O_3$ | 0.647 | 1.943 |
| CaO | 0.048 | 0.436 |
| $K_2O$ | 0.059 | 0.300 |
| MgO | – | 0.142 |
| MnO | – | 0.028 |
| $Na_2O$ | 0.342 | 0.354 |
| $P_2O_5$ | 0.524 | 0.438 |
| $TiO_2$ | 0.453 | 1.132 |