



SPECTRAL STUDY OF WASTE INCORPORATED CERAMIC BRICKS WITH GRANITE WASTE AT JEGADEV, KRISHNAGIRI DISTRICT IN TAMIL NADU, INDIA

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ABSTRACT

A production of ceramic bricks from mixtures of ceramic industry wastes (up to 50 wt%) from the area of Jegadevi, Krishnagiri district, Tamilnadu, India. 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.

Key word: ceramic bricks, ftir, sem, porosity

1. INTRODUCTION

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]. 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 granite 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 granite 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. Granite 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 granite 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.

2.MATERIALS AND METHODS

2.1 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 pulveriser. The basic raw material (Kaolinitic clay) is obtained from Nellur district, AP, 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 μ m) to obtain suitable powders for pressing. Unfired rectangular (90 mm \times 30 mm \times 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.

Table 1.Composition of the samples

sample	GF0	GF1	GF2	GF3	GF4	GF5
CLAY	100	90	80	70	60	50
GRANITE WASTE	0	10	20	30	40	50

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.

3.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 $\pm 0.01 \text{ cm}^{-1}$. 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, $\text{Water absorption\%} = \frac{W_i - W_d}{W_d} \times 100$; W_i – Weight of the sample after 24 h of immersion in water. The firing shrinkage of the fired samples was determined as $\text{FS\%} = \frac{L_R - L_F}{L_R} \times 100$; 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, $\text{Porosity\%} = \frac{W_2 - W_3}{W_1 - W_3} \times 100$.

4.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, K₂O, Mg, Fe and Na oxides. The CF material basically contains SiO₂ and Al₂O₃ 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.

Table 2 chemical composition of pure and additive as granites

oxides	Granite	Clay
SiO ₂	52.93	50.94
Al ₂ O ₃	48.55	41.77
Fe ₂ O ₃	0.547	1.843
CaO	0.038	0.336
K ₂ O	0.049	0.200
MgO	0.002	0.242
MnO	0.001	0.038
Na ₂ O	0.442	0.454
P ₂ O ₅	0.624	0.538
TiO ₂	0.353	0.932

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 250 °C and 550 °C can be attributed to the combustion of organic matter, which creates an endothermic effect [12]. Silicate dehydroxylation was observed between 450 °C and 650 °C. The DTA curve showed a slightly endothermic peak centered at 500

°C. Between 700 and 820 °C the decomposition of CaCO_3 with the release of CO_2 was indicated by the endothermic effect centered at 780 °C with the mass loss of 5.15% (Fig. 1).

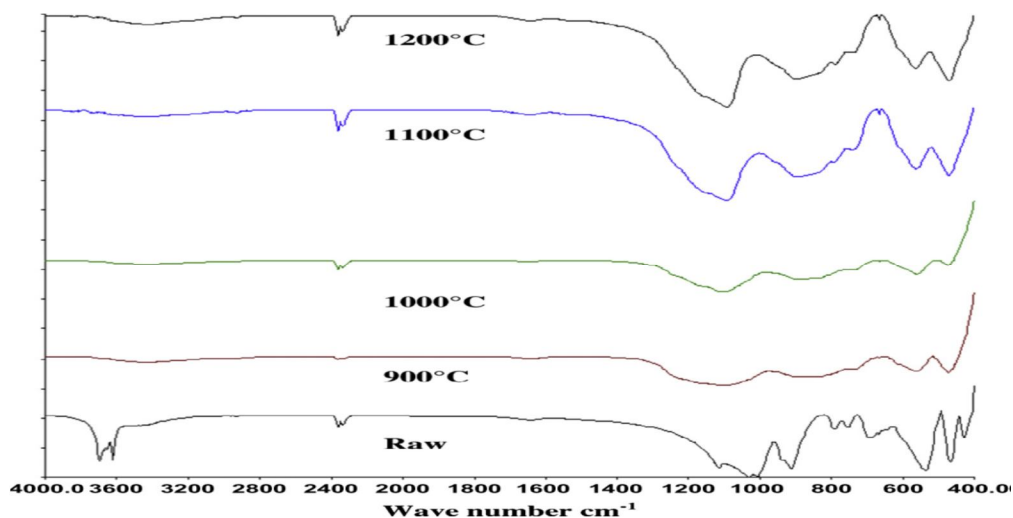
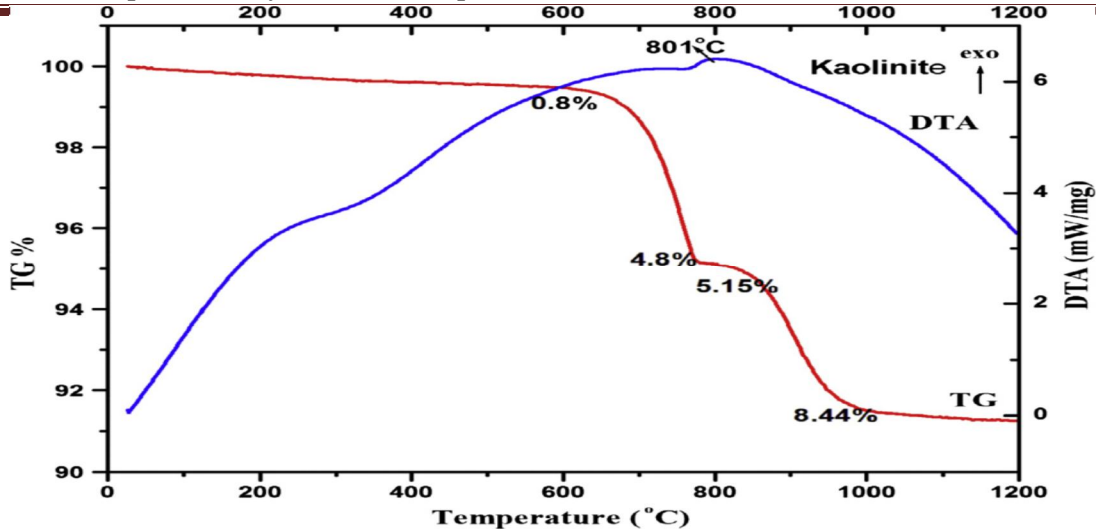


Figure. 5. TG-DTA curve for raw clay

Hence the starting sintering temperature of the ceramic bricks is 900 °C in the present study. As Kaolinite is the most widely used clay in the ceramic industry, the effect of heat on kaolinite mixtures has been widely studied [13,14]. Kaolinite with almost Al in the octahedral positions has four absorption bands in the O-H stretching region (Fig. 2a).

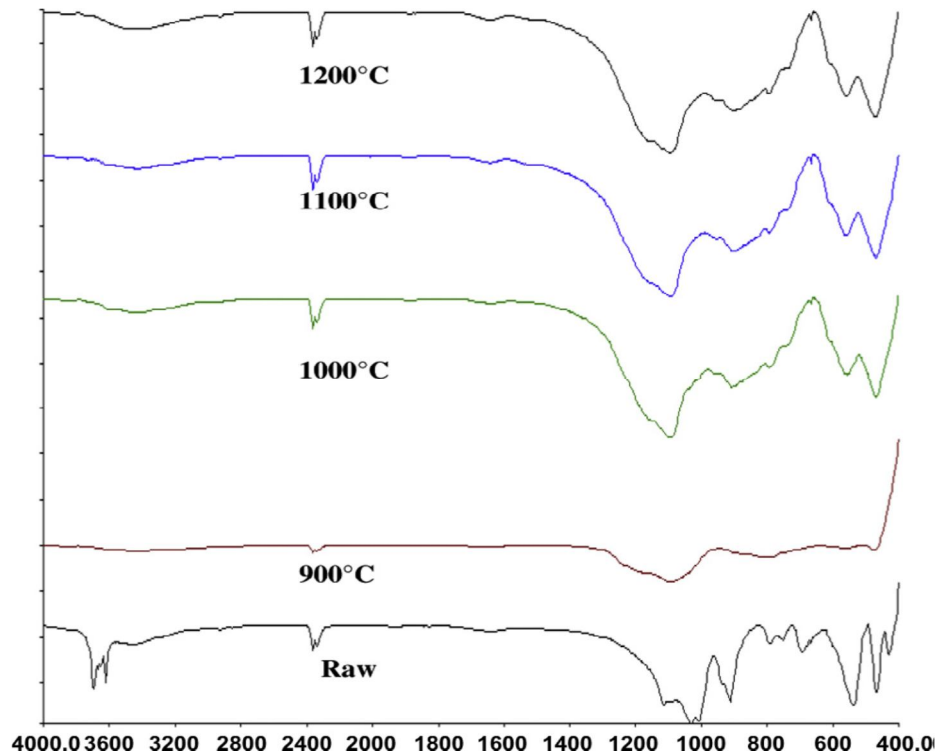
The O-H stretching of inner hydroxyl group lying between the tetrahedral and octahedral sheets give the absorption band at 3620 cm^{-1} . The characteristic band appearing at 3694 cm^{-1} corresponds to in-phase symmetric stretching vibration whereas two weak absorptions at 3669 and 3652 cm^{-1} are assigned to out of plane stretching vibration [15].

The IR absorption bands in the region 3800–3600 cm^{-1} in the spectra of clay minerals are due to the stretching vibrations of free hydroxyl groups present in them. The weak absorption bands at 3446 and 1636 cm^{-1} are attributed to O-H stretching and H-O-H bending vibrations of adsorbed water molecules [16,17]. The dehydroxylation is partly followed by the crystal framework collapse and the tetrahedral sheet disorder can be seen from the broadening of Si-O stretching band in the region 1100–1000 cm^{-1} . The vibrations of aluminum hydroxyl in the



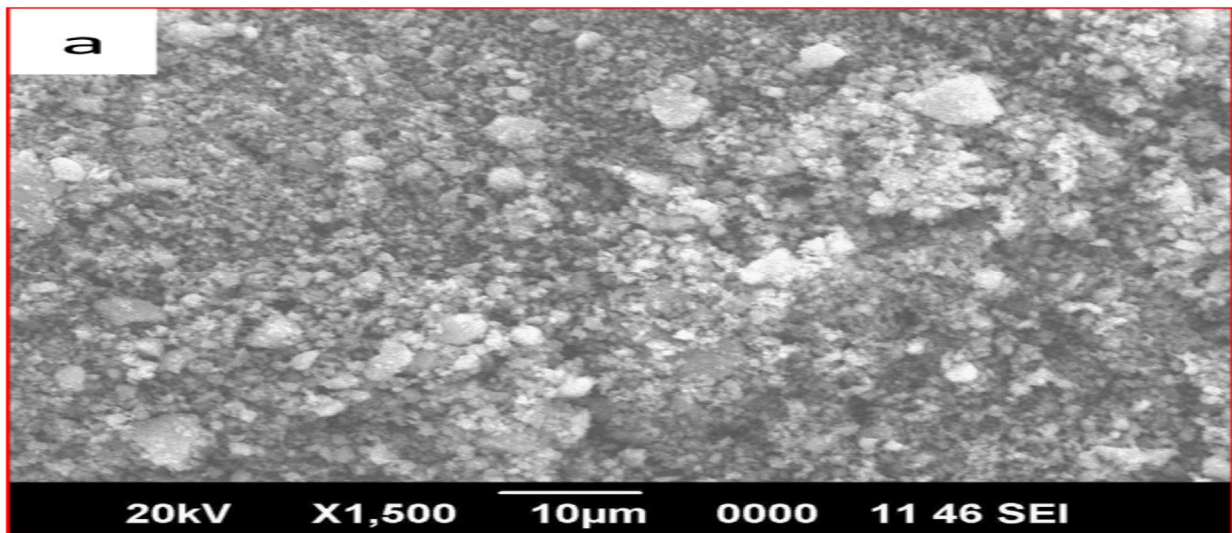
octahedral sheet structure give rise to strong absorption at 913 cm^{-1} [18]. The absorption band at 469 cm^{-1} decreased to lower frequency with temperature increasing from $900\text{ }^{\circ}\text{C}$ to $1200\text{ }^{\circ}\text{C}$. The absorption band at 797 cm^{-1} decreased gradually which indicate the level of quartz decreased gradually.

The variations in the values of OH stretching and bending wave numbers from sample to sample are usually attributed to varying strength of hydrogen bonding between OH and H_2O molecules and some oxygen in the structure. A peak in the fired sample spectra at 1093 cm^{-1} arises from asymmetric Si–O–Si stretching vibration [19] and the particle size is believed to be directly proportional to the intensity of this peak [20] (Fig. 2b).

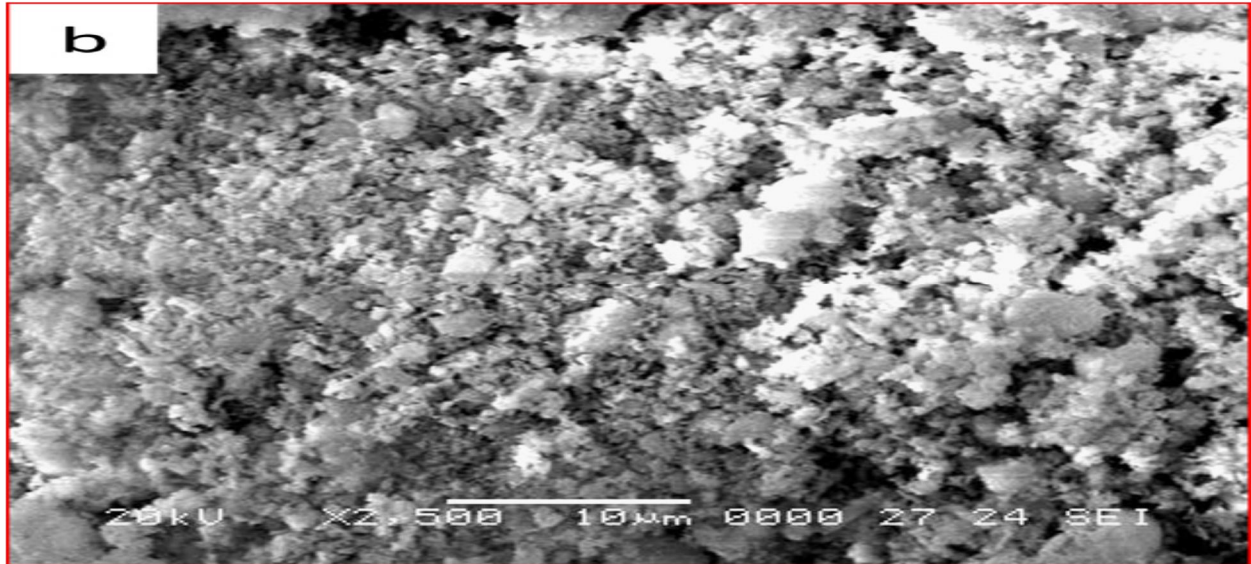


The impact of sintering temperature and waste additions on the technological properties of the test samples. The inference is clear that the properties are modified for higher sintering temperatures. Water absorption and firing shrinkage are the key factors determining the durability of the ceramic bricks. The dimensional factor of the finished ceramic products is mainly controlled by the linear shrinkage which indicates the degree of densification during sintering. The firing shrinkage is mainly affected by the firing temperature [21,22]. In our study the firing shrinkage increases linearly with temperature from 4.13 to 7.22 for the GF4 samples. The shrinkage in bricks occurs due to the loss of chemically and mechanically bound water.

The higher the temperature fired, the more moisture quickly evaporates under the same controlled conditions. However, the shrinkage results lie in the good quality category (<8%) for industrial production of ceramics. The variation of water absorption of the studied samples with different temperatures. The liquid phase formed above 1000 °C penetrates and isolates the adjacent pores causing a fall in water absorption. As far as the porosity is concerned, the values reduce significantly from 56.3% to 42.08% (GF4) from 900–1100 °C which was approximately a reduction of 25%. The purpose of the solid state sintering process is to develop atomic bonding between particles by a diffusion mechanism [23]. This is also the temperature at which vitrification was first detected by SEM [24]. The compressive strength increased as the firing temperature increased from 900 to 1100 °C [25]. The enhanced vitrification causes a sharp increase in the compressive strength at 1100 °C with decrease in porosity.



The amorphous/glassy phase, crystalline phase, porosity and overall structure of the products were investigated by SEM studies. The SEM studies show that high K-bodies have a considerable amount of amorphous/glassy phase. Figs. 4a–4b show the typical microstructures of fired ceramic bricks observed at the grained powder after the diametral compressive strength test. The microstructure of the ceramic bricks with 40% rejects (CF4) indicated a denser more sintered microstructure. When compared with GFO, the sintered sample CF4 shows a dense



matrix (Figs. 4a–4b).

At 1100 °C (Fig. 4b), the ceramic bricks presented advanced sintering stage. In this stage, the elimination of large amount of pores that exist within the structure occurred. In addition, the vitrification is in progress. In fact, the glassy phase infiltrates the pores of the structure and cause densification via liquid phase sintering. This justifies the improved technological properties such as lower water absorption and higher mechanical strength of the ceramic bricks.

5. CONCLUSION

The present work, it can be concluded that the incorporation of ceramic rejects up to 50 wt% into clayey bodies is likely the potential recycling way for the wastes into the Granite industry.

The results indicate that no changes in the brick production process are needed as no major changes in the final properties of the bricks were observed. This is due to minor difference of alumina content and the absence of higher amounts of alkali ions in the rejects.

The optimum composition of 40% Granite rejects with the sintering temperature of 1100 °C is most suitable for recycling in the ceramic industries as it has the maximum compressive strength with lowest water absorption.

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