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## RESEARCH ARTICLE

### INFRARED SPECTROSCOPIC STUDY OF ARTICULATED CERAMIC BRICKS

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#### ABSTRACT

The IR vibrational spectra of the ceramic test samples were recorded and discussed which serves as the molecular fingerprint to give proof of identity without recourse to any other analytical method and yields basic information on the changes in the interatomic bonding. The possible use of ceramic rejects in the clayey body draws particular attention. The IR assignments of the raw samples are almost alike despite the different compositions and show stretching and bending modes of kaolinite at different absorption frequencies of 3694 cm<sup>-1</sup> and 913 cm<sup>-1</sup> respectively. The structural changes take place at different calcination temperatures which are interpreted in depth. An analysis of the mineralogical composition during a firing cycle up to 1200 °C has demonstrated that they possess satisfactory characteristics for the desired applications in the ceramic industry.

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#### INTRODUCTION

For vast majority of clay usage only empirical knowledge is used to process the clay which frequently results in products below specifications. Since the microstructure and properties of any ceramics depends on the characteristics of the raw materials and processing parameters to assure the quality of the ceramic products (Kingery et al., 1976), a complete characterization of the precursor clays as a function of the firing temperature is needed. The objective of the present work is to characterize the ceramic test samples imbedded with ceramic rejects. The re-use of such industrial wastes involves economy of deficient raw materials and permits improvement of ecological situation in metallurgical and refractory works.

The knowledge of chemical and mineralogical compositions is mandatory in clay characterization studies: the former mainly depends on the raw materials used to produce the wares and also on processing changes, the latter on both the initial composition and the processing, as minerals are the fingerprints of the stable and also the metastable solid phases formed during firing (Rice, 1987). The ceramic wastes show non-plastic behavior due to their major constituents, expressed as oxides, silica (SiO<sub>2</sub>) and alumina (Al<sub>2</sub>O<sub>3</sub>). These wastes generated after the firing stage of ceramics can eventually be added to clayey body in the proper ceramic processing. This non-plastic type of waste can be advantageous for the processing and quality of bricks fired at temperatures above that of the original production (Vieira and Monteiro, 2007).

#### MATERIALS AND METHODS

##### Sample labeling and preparation

The basic raw material (Kaolinitic clay) is obtained from Thiruvananthapuram district, Kerala, India. The ceramic rejects which is a waste material generated while producing any type of ceramic material is obtained from the Government Ceramic industry of Vriddhachalam, Tamilnadu, India and made into a fine powder in a pulveriser. 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 (93mm x 29mm x 29mm) specimens in lots of 10 for each mixture were moulded using an extrusion apparatus. The composition and naming of the ceramic test samples 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 hr at the maximum temperature needed. Cooling occurred by natural convection after it was turned off.

Table 1. Composition of the samples (%)

Sample	Kaolinitic clay	Ceramic rejects
S0	100	0
S10	90	10
S20	80	20
S30	70	30
S40	60	40
S50	50	50

##### Infrared analysis

FTIR spectroscopy is a powerful tool in assessing both chemical and mineralogical composition of the ceramic

bodies. 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 and the mixture is then pressed into a transparent disc in an evocable dye at sufficiently high pressure. Using the Nicolat-Avatar 360 series FTIR spectrometer, the infrared spectra for all the samples were recorded in the region 4000-400  $\text{cm}^{-1}$ . The resolution of the instrument is 4  $\text{cm}^{-1}$  and the accuracy  $\pm 0.01 \text{ cm}^{-1}$ . At each and every time, this instrument was calibrated for its accuracy with the spectrum of a standard polystyrene film.

## RESULTS AND DISCUSSION

### Mineralogy of the raw samples

The FTIR spectra of raw samples (R.M) appear in Fig. 1a and they provide qualitative and quantitative information about the order or the disorder of the structure, depending on the relative intensity of the OH band in the 3700-3600  $\text{cm}^{-1}$  region (Madejova 2003). Fig. 1a shows the spectra of the samples S0-S50 in the raw state (unfired) from which it is clear that before sintering, the spectral characteristics of all the six samples are almost similar. This indicates the fact that any of the compositional wt% (S10-S50) can be assigned for the production of ceramic briquettes as these spectra correspond to well ordered kaolinite structures (Farmer, 1979). Kaolinite with almost Al in the octahedral positions has four absorption bands in the O-H stretching region (Fig. 1a).

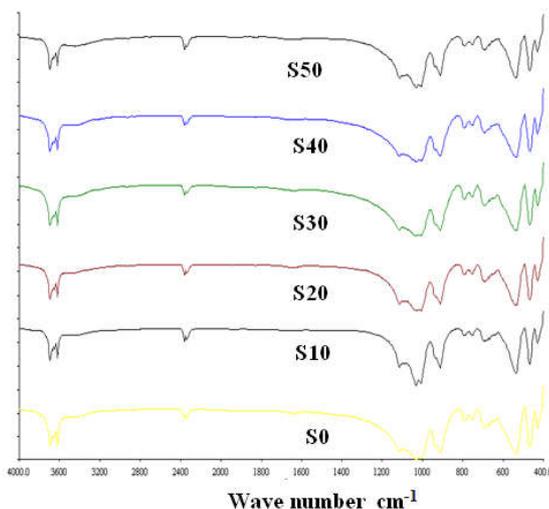


Fig 1(a) FT-IR Spectra of raw (unfired) ceramic brick samples

The O-H stretching of inner hydroxyl group lying between the tetrahedral and octahedral sheets give the absorption band at 3620  $\text{cm}^{-1}$ . The characteristic band appearing at 3694  $\text{cm}^{-1}$  corresponds to in-phase symmetric stretching vibration whereas two weak absorptions at 3669 and 3652  $\text{cm}^{-1}$  are assigned to out of phase stretching vibration. A combined Al-O and Si-O deformation mode is observed at about 468  $\text{cm}^{-1}$  in the spectra (Freud and Farmer, 1974). Three spectral deformation bands at 431, 468 and 537  $\text{cm}^{-1}$  are showed in this spectral range which were assigned as Si-O, Si-O-Si and Si-O-Al deformation vibrations respectively. It is found that all the samples have quartz as secondary mineral.

### Mineralogy of the fired samples

The IR absorption bands in the region 3700-3600  $\text{cm}^{-1}$  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  $\text{cm}^{-1}$  are attributed to O-H stretching and H-O-H bending vibrations of adsorbed water molecules (Sankaran and Ramasamy, 2000), (Wolff, 1963), (Venkatachalapathy et al., 2003). The dehydroxylation is partly followed by the crystal framework collapse (Elsaas and Olivier, 1978) and the tetrahedral sheet disorder can be seen from the broadening of Si-O stretching band in the region 1100-1000  $\text{cm}^{-1}$ . The vibrations of aluminium hydroxyl in the octahedral sheet structure give rise to strong absorption at 913  $\text{cm}^{-1}$ . 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<sub>2</sub>O molecules and some oxygen in the structure. A peak in the fired sample spectra at 1093  $\text{cm}^{-1}$  arises from asymmetric Si-O-Si stretching vibration. The strong distinct absorption bands present at 1115  $\text{cm}^{-1}$  and 913  $\text{cm}^{-1}$  in all the spectra of the studied samples indicate the well ordered kaolinite structure. Well ordered clays are characterized by the presence of bands at 910, 1030 and 1120  $\text{cm}^{-1}$  in which 910 and 1120  $\text{cm}^{-1}$  are due to Al-(OH). They are replaced by a single band at 1030  $\text{cm}^{-1}$  as the disorder increases (Percival et al., 1974). (Figs 1b-1g).

The presence of quartz can be explained by Si-O asymmetric bending vibrations at 468  $\text{cm}^{-1}$  and Si-O symmetric bending vibrations at 695  $\text{cm}^{-1}$ . These assignments are in good agreement with that reported for quartz (Ramasamy et al., 2009). The peak at 795  $\text{cm}^{-1}$  in the fired samples provides the evidence for the presence of dickite (Cassagnere et al., 1999), (Ip et al., 2008). The bands associated to 'condensed' AlO<sub>4</sub> tetrahedra expected in the range of 850-750  $\text{cm}^{-1}$  are not observable probably because of their lower intensity and broadening of the strong band belonging to SiO<sub>4</sub> tetrahedra of the remaining phases present in the samples. The absence of the bands at 539 and 913  $\text{cm}^{-1}$  in the spectra of the fired samples can be related to the change of octahedral coordination of Al<sup>3+</sup> in kaolinite (Biljana R. Ilic et al., 2010). The major change in the spectra in all the thermally treated samples corresponds to the disappearance of the band at 538  $\text{cm}^{-1}$  absorption and the evolution of a band at 563-567  $\text{cm}^{-1}$  for different compositions in all the studied temperatures. These could be confidently assigned to anorthite (Kobayashi and Kato, 1994) as a result of solid state reaction between CaO and clay. The rest of the spectrum is similar in its general features because the same phases are maintained.

### Conclusion

From the mineralogical characterization of the ceramic test samples through FTIR, it can be observed that they have the characteristics similar to the conventional raw materials. The effectiveness of the rejects addition is shown by thermal activation of the minerals. The presence of kaolinite, quartz and anorthite in all the studied samples is a notable factor for the production of ceramic bricks with addition of the rejects. The comparison of the spectra of the kaolinitic clay with other compositional mixtures indicates that it is suitable to produce ceramic bricks with the studied compositions of the mixtures at the sintering temperatures of 1100 and 1200 °C.

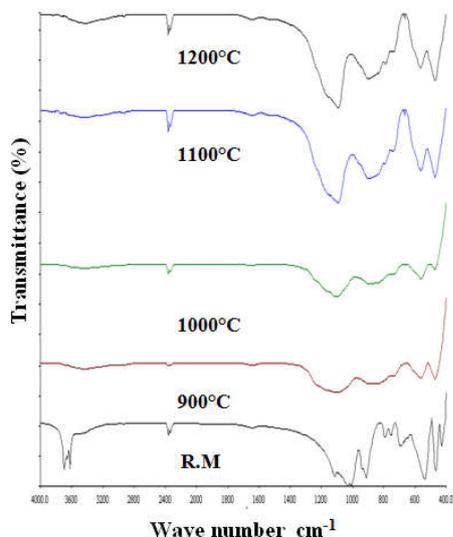


Fig. 1(b) FT-IR spectra of S0 samples

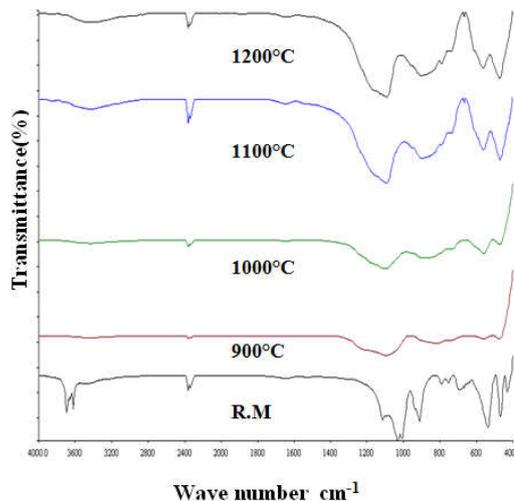


Fig. 1(c) FT-IR spectra of S10 samples

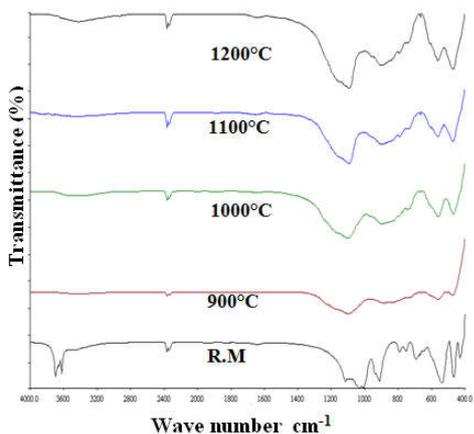


Fig. 1(d) FT-IR spectra S20 samples

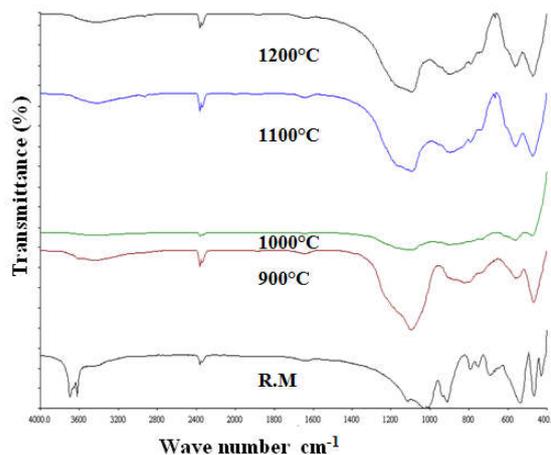


Fig. 1(e) FT-IR spectra of S30 samples

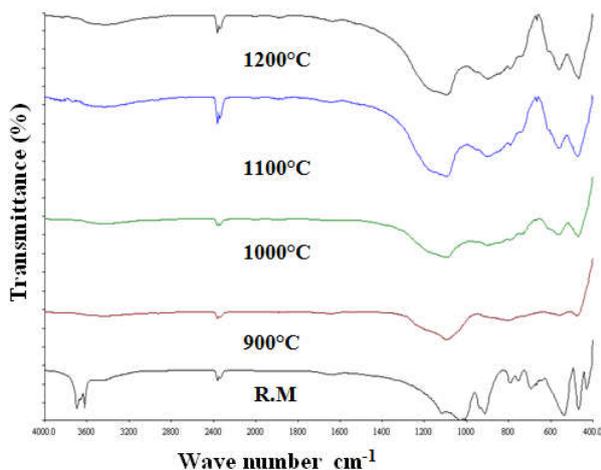


Fig 1(f) FT-IR spectra of S40 samples

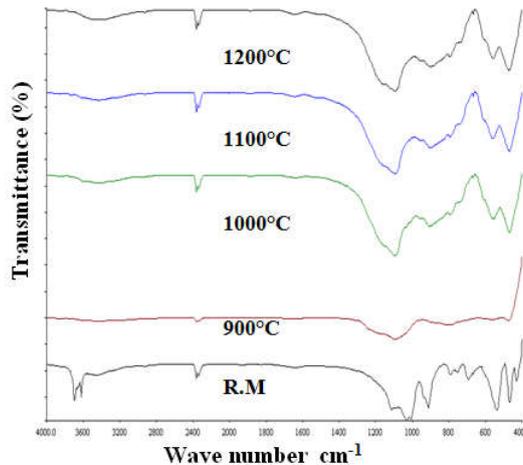


Fig 1(g) FT-IR spectra of S50 samples

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