



Structural, dielectric and surface morphological properties of ball clay with wet grinding for ceramic electrical insulators



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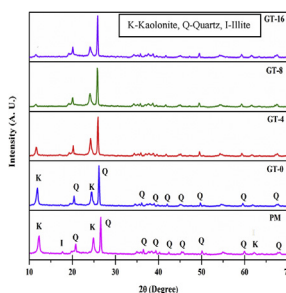
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HIGHLIGHTS

- The physical properties of ball clay with wet grinding are carried out.
- The XRD pattern reveals that the quartz and kaolinite are main clay materials.
- The dielectric parameters are found to be varied with frequency and temperature.
- The SEM studies show the grains size of ball clay is reduced with grinding.
- The EDS pattern reveals silica-rich glassy phase of ball clay material.

GRAPHICAL ABSTRACT



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ABSTRACT

In this work, a study on structural, dielectric and surface morphological properties of ball clay with wet grinding is undertaken. The wet grinding treatment was performed using ball and vibro mills for different time spells of 2, 4, 8 and 16 h. The raw material and ground ball clay samples were subjected to the XRD, LCR meter and SEM to investigate the structural, dielectric and morphological properties respectively. The compositional analysis of the raw material was also carried out using energy dispersive spectroscopy (EDS). The XRD patterns reveal that the kaolinite is the main clay mineral along with quartz and the samples become amorphous in nature with grinding treatment. The dielectric constant and dissipation factors are also calculated with frequency and temperature. The ground sample for 8 h shows maximum dielectric constant and dissipation factor at 100 kHz and 1 MHz frequencies respectively while ground sample for 16 h shows maximum dielectric constant at 1 MHz. An increase in dielectric constant at the high frequency range (~MHz) is observed with grinding treatment. The SEM studies reveal that the grains are densely packed, well defined and having continuous solid phase. The grain size is observed to be reduced with wet grinding due to increase in surface area.

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1. Introduction

Ceramic materials have received considerable attention in the last few decades due to their relatively easy approach and low cost [1]. Nowadays, these materials are generally used to fabricate pottery items, ceramic electrical insulators, refractory bricks,

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decorative products, wall or floor tiles, many industrial processes such as paper making, cement production and chemical filtering etc. Clays are essentially aluminosilicates with many important components, several oxides and many other compounds. Though the ball clay is very rare mineral and found in very few places yet stocks are available in Northwestern Rajasthan of India which is fine-grained and highly plastic as well as mainly kaolinitic sedimentary clay. The grinding treatment is subjected by friction and impact forces such as planetary, vibratory and oscillating mills, the physicochemical changes are obviously occurred in material [2–7]. The applications of ceramic products are evaluated by their physical properties which depend on their mineralogy, chemical compositions and grain size [8]. In clay minerals, grain size reduction is produced either by wet or dry grinding. Recently, an attention is given to the study of structural change due to dry and wet grinding using ball and vibro mills. The physical and chemical properties are changed due to grinding and heating treatments which can be exploited either to new application purposes or to enhance known properties [3,9–14]. The grain size distribution in a body mixture has a significant impact on packing efficiency which affects the shape and size of pores, microstructure development and shrinkage behavior [15].

The effect of mechanochemical treatment on structure and electrical properties of montmorillonite is investigated by Abbas and Srasra [16] who found that the crystallinity of montmorillonite was decreased with grinding. They also observed an increment in dc conductivity and dielectric constant in the range of MHz with grinding. A depth study on the physico-chemical properties of shales of illitic clays is carried out by Wilson et al. [17]. The influence of milling on sintering and technological properties of anorthite based porcelainized stoneware is reported by Taskiran et al. [15]. They analyzed that the milling has a major impact on the particle size distribution and the densification was increased with high degree of close particle packing. Amin et al. [18] reported the preparation and evaluation of hyper branched *p*-chloromethyl styrene polymers/montmorillonite clay nano-composites as dielectric materials. The effect of clay addition on broadband dielectric properties of multi-walled carbon nanotube/polyvinylidene fluoride (MWCNT/PVDF) composites in frequency range of 10^1 – 10^6 Hz is investigated by Khajepour et al. [19]. They found that the permittivity and dissipation factor both depend on the clay content and dissipation factor considerably decreased by clay addition as a result of the hindrance role. Sengwa et al. [20] studied the dielectric properties of some minerals of western Rajasthan in the frequency range 100 Hz –100 kHz. They found that the dielectric constant was decreased with frequency and the minerals followed the Cole-Cole dielectric dispersion relation. The electrical and dielectric properties of Poly (Ethylene glycol)-polyurethane/poly (Methylmethacrylate)/Montmorillonite (MMT) composite are reported by Chilaka et al. [21]. They found that the conductivity was increased with MMT concentration up to 5 wt % and thereafter it decreased. They also observed that the dielectric permittivity and dielectric loss were decreased with frequency. A study on composition, origin and industrial suitability of the Aswan Egyptian clays is undertaken by Baioumy and Ismael [22] who observed that the clays had Fe_2O_3 and TiO_2 as major contents. The grinding of clay materials affects their leaching behavior and the heating treatment is responsible for the formation of new phases and even the dry grinding treatment leads to a progressive collapse of the structure [23–26]. It is a crucial step in ceramic processing as it creates an improvement in the physical and chemical uniformity in the powder mixture [15].

The grinding depended study of properties of ball clay is not understood and the above facts invite an attention to undertake a study on the structural, dielectric and morphological properties of

ball clay with wet grinding. The wet grinding treatment was performed using ball and vibro mills for different time spells of 2, 4, 8 and 16 hours (h). The raw material and ground ball clay samples were subjected to the XRD, LCR meter and SEM to find the structural, dielectric and morphological properties respectively. The compositional analysis of the raw material was also carried out using EDS.

2. Experimental details

2.1. Sample preparation

The ball clay raw material was taken from Daga Mines of Bikaner, Rajasthan state of India comprised mainly compounds SiO_2 , Al_2O_3 , TiO_2 , Fe_2O_3 and K_2O . The chemical composition was undertaken employing X-ray fluorescence (PAN alytical Mini Pal4) technique. The raw material was named as PM which is pure and used after manually grinding without any treatment like heating and grinding.

An amount of 8 Kg ball clay raw material and 8 L water were taken for grinding treatment with 50 Kg porcelain balls as grinding media in the ball mill (SINCO Model 465) which was operated at 440 V and 50 Hz. The grinding treatment was performed for 2 h in ball mill and was considered equivalent to null in vibro mill and the sample was identified as GT-0. For further treatment, the vibro mill (Spankler M-45) was used as grinder and the same amount of ball clay and water was taken in ball mill with 98 Kg cylindrical alumina pebbles as grinding media. The vibro mill was driven by 960 rpm, 246.08 Watt and vibrator motor working on 440 V, 50 Hz. The treatment was performed for 4, 8 and 16 h of time period and the samples were identified as GT-4, GT-8 and GT-16 respectively. The resultant which was obtained from the ball and vibro mills after grinding treatment was passed through a sieve cloth which was able to remove about 85% water contents. In order to remove the air contents, the yield was passed through a pug mill (VSN 515) which graded casting with 745.7 Watt motor of rpm 1440. The pug mill resultant was having cylindrical shape with 2.15 cm diameter and 10–14 cm of length which was then dried in sunlight for 24 h to remove moisture contents. Thereafter, all samples were annealed in a programmable controlled furnace (EIE 1502) at temperature 110 °C for half an hour to remove moisture contents fully. In furnace, the heating was made by silicon carbide rods with temperature rate 3–4 °C/minute. Finally, the samples were manually ground and used to perform XRD and SEM measurements. The block diagram of sample preparation sequence is shown in Fig. 1.

To carry out dielectric measurements, the raw and ground samples were pelletized with a pressure 5 ton/cm² using hydraulic machine followed by heating at temperature 600 °C for 1 h in a muffle furnace (Sonar HTF 1225) to keep pellets in a bound shape. The mass of pellets was measured by high precision electronic balance (Precisa ES225SM-DR) of sensitivity 0.001 mg and the thickness as well as areas of pellets were also calculated. These pellets then were coated with silver paste on both sides for electroding and kept in hot air oven (Mac MSW 211) for 30 minutes at temperature 100 °C as well as subjected to LCR meter for dielectric measurements.

2.2. Characterization

The structural properties of raw material and ground ball clay samples were carried out employing an X-ray diffractometer (Rigaku Miniflex). The XRD of $\text{CuK}\alpha_1$ and $\text{CuK}\alpha_2$ both radiation of wavelength 1.54184 Å was used with a scan speed 2°/minute and scanning step size 0.02°. The powder samples of ball clay were tightly filled in the sample holder and the measurements were

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