Abstract: This work focuses on the application of β‒Wollastonite content with different particle sizes on the bulk density and weight loss of clay based ceramics. The ceramic bodies were prepared by mixing clay with a synthesized β‒wollastonite (5%, 15%, and 25 %) using a solid slip casting method. The clay (75 µm) and β‒wollastonite were (63, 75 and 125 µm) mixed with different particle size and fired at 950 °C, 1000 °C, and 1050 °C of firing temperatures. The fabricated ceramic material was exhibited a maximum bulk density of 2.35 g/cm³ on 25% content of β‒wollastonite, 63 µm particle size of β‒wollastonite and 1050 °C of firing temperature. However, a minimum bulk density of 1.87 g/cm³ was found on 25% content of β‒wollastonite, 125 µm particle size of β‒wollastonite and 950 °C firing temperature. A minimum of 2.32% ignition loss was recorded on a ceramic specimen that contained 25% of β‒wollastonite with a particle size of 125 µm and fired at 950 °C of firing temperature. In contrast, a maximum of 4.37% ignition loss was observed at a ceramic body made up of 5% of β‒wollastonite content with 63 µm particle sizes and fired at 1050 °C. In general, the result shows that the firing temperature, the particle size of β‒wollastonite, and the addition of β‒wollastonite have a great effect on the bulk density and ignition loss of the clay ceramic materials.

Keywords: β‒Wollastonite, Clay, Ceramic, Firing Temperature, Particle Size, Bulk Density, Weight Loss.

1. Introduction

Ceramic materials are mainly made of clays and/or other inorganic raw materials and an important construction material employed in most construction sectors. The fabrication of ceramic starts from the material, grinding and mixing, and typically shaped at atmospheric temperature, then dried and subsequently fired at a temperature sufficient to develop the desired properties [1]. The quality and price of the final product of ceramic material rely on the composition of raw materials, firing cycle [2], mixing proportions [3, 4], and particle size of the starting materials [5]. The ceramic industry could be a dynamic sector whose technological innovation and market trends have drawn a complex picture of products and processes. This technological evolution has directly entailed with raw material formulations. For this study, the ceramic material specimens were made up of clay and synthesized β‒Wollastonite.

Clay is the name usually given to all or more or less compacted sedimentary rocks deriving from changes of primary rocks, predominantly containing "clayey minerals" of the micronic dimension. Clay-based minerals are the main component of ceramic bodies; their amount usually ranges from 40-60%. They confer plasticity and workability in the
green state and furnish the main oxides involved, with fluxes and sintering aids, in the consolidation mechanism of the body during firing. [6]. Clays consisting of pyrophyllite (Al₂Si₂O₅(OH)₄), talc (Mg₃Si₄O₁₀(OH)₂), kaolinite (Al₂Si₂O₅(OH)₄) and montmorillonite (Al₂Si₂O₅(OH)₂·nH₂O) are essential for ceramics characteristics [7]. Clay minerals are among the maximum ample minerals on earth and are of substantial interest due to their low cost [8]. Natural clay deposits are the main supplying materials for the manufacture of light structural ceramic products with good mechanical properties such as low shrinkage and heat transfer [9].

β-Wollastonite (β-CaSiO₃) is a naturally occurring form of calcium silicate with acicular crystal addiction [10]. Naturally, calcium silicate has two polymorphic forms; wollastonite, low-temperature phase β-Wollastonite and pseudo-wollastonite, high-temperature phase α-Wollastonite [11]. Wollastonite usually occurs as a common constituent of a thermally metamorphosed impure limestone, it additionally could arise when the silicon is because of metamorphism in contact altered calcareous sediments, or contamination in the invading igneous rock [12]. The β-wollastonite mineral phase is produced at lower temperatures and the phase transition temperature to α-wollastonite is more than 1125 °C [13].

β-Wollastonite is an extremely interesting but little-studied material which has a combination of properties, such as lack of volatile constituents, fluxing characteristics, low dielectric constant, low dielectric loss, thermal stability, low thermal expansion, low loss of ignition and low thermal conductivity, hence is used in ceramic fabrication, medical material for artificial bones and dental roots, high-frequency insulator, filler material in resins and plastics, paper, civil construction, ceramic glazes, metallurgy, paint, and frictional products [14-17].

Ethiopia’s geological formation revealed nothing about β-wollastonite. Ethiopian ceramic and paint factories are imported β-wollastonite from overseas international locations [18]. That was the reason, the previous work focused on the synthesis of β-wollastonite from rice husk and limestone to maximize the utilization of local resources. It was characterized using Fourier Transforms Infrared Spectroscopy (FTIR) and Powder X-ray Diffraction (XRD) and also used as a reinforcement filler for the fabrication of ceramic tiles. The results revealed a tremendous effect on linear shrinkage, water absorption, acid resistance, and compressive strength of ceramic tiles [19]. The present work intends to apply the synthesized β-wollastonite to investigate its effect on the ignition weight loss and bulk density of clay ceramic materials.

2. Materials and Methods

2.1 Materials

The raw materials were β-wollastonite and Clay. β-wollastonite was synthesized and characterized in previous work [19]. Clay was obtained from Kalu Woreda, South Wollo, Ethiopia. Disk Mill and Ultra-Centrifugal were used to mill clay and β-wollastonite. Fourier transforms infrared spectrometer was used for the qualitative characterization of surface functional groups present in the clay mineral. Likewise, Sieves were used to sieve clay and β-wollastonite. The compactor was used for compacting a green body during casting into a rectangular mold. A digital balance and Standard ruler were used to weigh and measure green body dimensions and ceramic materials, respectively. Electric Oven was used for drying clay and green body samples. The electric furnace was used for firing the green body, to transform into ceramic materials.

2.2 Methods

2.2.1 Manufacturing of ceramic sample

Clay was separated manually from its impurities and soaked in water for 3 days and washed to take away undesirable matters. Then, it was dried overnight in an oven at 105 °C and then added into disk mill then handover to the ultra-centrifugal mill. The powdered clay was then passed through a sieve with a nominal aperture of 75 µm whereas the β-wollastonite was sieved in three different sieves with nominal apertures of 63 µm, 75 µm, and 125 µm. β-wollastonite was mixed to clay in three different weight proportions of 5%, 15%, and 25%. Then, tap water (10% by mass) was poured into the mixture under vigorous mixing until the mixtures were easy to work by hand and allowed to stand for sixteen hours. The mixtures were cast into rectangular (120 × 65 × 8 mm³) formwork (mold) and compacted by applied uniform pressure (20 MPa). The green bodies formed were then covered and kept in the cabinet for two days in open-air to slowly lose its moisture content. After that, the green bodies were opened and then dried at 105 °C in an oven for twenty-four hours. The dried bodies were loaded into the furnace and heated at 5 °C/min until 250 °C and held at this temperature for a half-hour. Then, the temperature was gradually increased to 950 °C, 1000 °C, and 1050 °C at a heating rate of 10 °C/min and allowed for an hour to make sure complete firing. The ceramic materials were then allowed to cool in the furnace to room temperature.

2.2.2 Fourier Transforms Infrared Spectroscopy (FTIR)

FTIR spectroscopic tool is employed to identify the key functional groups of the clay mineral. It uses infrared radiation (a band of frequencies below the visible portion of the spectrum) to investigate a sample. A sample of 1 mg (<63 µm) was mixed with 100 mg of KBr and then pressed to prepare the pellets. The FTIR spectrum was recorded over a wavenumber range of 4000–400 cm⁻¹ with a 4 cm⁻¹ resolution and ordinate unit of transmittance (%) [20].

2.2.3 Determination of Bulk Density (BD)

The Bulk Density (BD) of the ceramic material was calculated by dividing the fired weight by the bulk volume of the ceramic material. The bulk volume was calculated from ceramic body dimensions after firing. The formula is given by equation 1 [21]:

\[ BD = \frac{\text{Fired Weight (g)}}{\text{Bulk Volume (cm}^3\text{)}} \]  

2.2.4 Determination of Loss On Ignition (LOI)

The LOI was determined by the ignition of all of the volatile constituents. The difference between the Dried weight (Dₖ) of...
the material and its weight after firing ($F_w$) is the LOI and determined using equation 2:

$$\text{LOI} (%) = \frac{D - F}{D} \times 100$$

(2)

3. Results and Discussions

3.1 FTIR spectra analysis of Clay

The major functional groups present in the clay mineral were identified by the FTIR spectra as shown in Fig. 1. Region 3800-3000 cm$^{-1}$ (O-H stretching region) which identified Kaolinite associated with octahedral stretch vibrations from OH [22]. The absorption band at 3620 cm$^{-1}$, assigned to H-O-H stretching vibrations of water molecules weakly hydrogen-bonded to the AlAl-OH. Broadband at 3434 cm$^{-1}$ and the band at 1637 cm$^{-1}$ observed due to H-O-H symmetric stretching vibration and O-H bending vibration, respectively. The peak observed at 1033 cm$^{-1}$ and 789 cm$^{-1}$ are attributed to the Si-O symmetric vibrational stretching. The other peaks observed at 540 and 468 cm$^{-1}$ are assigned to Si-O-Al (octahedral Al) and Si–O–Si bending vibrations, respectively [23].

![Fig 1: FTIR spectra of clay](image)

3.2 Effect of operating conditions

Twenty-Seven groups of specimens containing different combinations of clay and β-wollastonite, and particle sizes were fired according to three different firing temperatures. Then, the bulk density and loss on ignition of the ceramic bodies were determined. To investigate the effect of β-wollastonite content the firing temperature must be kept below 1125 °C (changed to α–wollastonite at 1125 °C). And also, to obtain high-strength ceramic bodies, adsorbed and crystalline water must be removed. Similarly, the volatile components must be completely removed or decomposed. Therefore, 950 °C, 1000 °C, and 1050 °C were selected as firing temperatures of the ceramic bodies.

3.2.1 Effect of operating conditions on Bulk Density (BD)

The influence of Firing Temperature (FT), amount of β-Wollastonite Added (AW), and Particle Size of β-Wollastonite (PS) on the Bulk Density (BD) of ceramic materials were studied.

![Fig 2a: Effect of firing temperature on the bulk density of ceramic materials](image)

Table 1: Average values of Bulk Density (BD)

<table>
<thead>
<tr>
<th>Factors</th>
<th>FT (950 °C)</th>
<th>FT (1000 °C)</th>
<th>FT (1050 °C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>PS (µm)</td>
<td>AW (%)</td>
<td>AW (%)</td>
<td>AW (%)</td>
</tr>
<tr>
<td>5</td>
<td>1.96</td>
<td>2.08</td>
<td>2.07</td>
</tr>
<tr>
<td>15</td>
<td>1.91</td>
<td>2.03</td>
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<tr>
<td>25</td>
<td>1.89</td>
<td>1.87</td>
<td>1.93</td>
</tr>
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</table>

Fig. 2a illustrates the effect of firing temperature on the bulk density of ceramic materials. It is observed that the bulk density of ceramic material is increased with firing temperature. The average bulk densities are 1.94, 2, and 2.15 g/cm$^3$ for firing temperature of 950, 1000, and 1050 °C, respectively. When the firing temperature raised from 950 °C to 1050 °C, the pore present in green bodies are filled due to the grain starts growing in the hole part of the bodies and the bulk densities increased correspondingly. A similar result has been reported elsewhere [24, 25] that the bulk density of ceramic materials increased as the firing temperature increased. The lower temperature was insufficient to promote densification and consequently, promote substantial closing the pore formed during green body casting [26].

As can be seen in Fig. 2a, bulk density also varies due to the variation of the amount of β-wollastonite incorporated into ceramic materials. The average bulk density for the addition of 5%, 15%, and 25% of β-Wollastonite are 2.01, 2.03, and 2.05 g/cm$^3$, respectively. As the amount of β-Wollastonite increased from 5 to 25%, the bulk density of ceramic materials increased since β-wollastonite is denser than clay soils [27]. Previous research showed that the ceramic bodies made up of pure clay were exhibited a low bulk density (1.64 g/cm$^3$) [28]. Firing temperature and amount of β-Wollastonite have no synergetic effect (p>0.05) on the bulk density of ceramic material. However, the bulk density of ceramic materials is increased with both firing temperature and the amount of β-wollastonite increased as shown in Fig. 2a.

The bulk density of ceramic materials is decreased when the particle size of β-wollastonite increased as shown in Fig. 2b. This is due to that the fine particles are easy to compact during green ceramic body casting and also have a big tendency of flowability during firing compared to the coarse particles. It is possible to increase the bulk density and decrease the porosity of the ceramic body with the addition of fine particles to larger granule blends. This can be explained by the fact that the small particles fill void spaces between the large particles without dilating the overall system volume [29].
3.2.2 Effect of operating conditions on Loss On Ignition (LOI)

The main and synergetic effect of Firing Temperature (FT), the amount of β-Wollastonite Added (AW), and Particle Size of β-wollastonite (PS) on the loss on ignition of ceramic materials were investigated.

Table 2: Average value of LOI

<table>
<thead>
<tr>
<th>Factors</th>
<th>FT (950 °C)</th>
<th>FT (1000 °C)</th>
<th>FT (1050 °C)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>AW (%)</td>
<td>AW (%)</td>
<td>AW (%)</td>
</tr>
<tr>
<td>PS (µm)</td>
<td>5</td>
<td>15</td>
<td>25</td>
</tr>
<tr>
<td>63</td>
<td>3.99</td>
<td>3.08</td>
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<td>75</td>
<td>3.97</td>
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<tr>
<td>125</td>
<td>3.88</td>
<td>2.97</td>
<td>2.32</td>
</tr>
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</table>

Fig. 3a shows the effect of firing temperature on the LOI of ceramic materials. The average values are 3.15, 3.46, and 3.52% for firing temperature of 950, 1000, and 1050 °C, respectively. The LOI is increased by 9.85% and 1.58% when the firing temperature raised from 950 to 1000 °C and then to 1050 °C, respectively. Similar research [30] revealed that the LOI of the ceramic materials increased with the firing temperature. The maximum variation of the LOI was recorded between the temperature of 950 °C and 1000 °C. This is due to the volatile components present in the materials which are responsible for weight loss during ignition were removed or decomposed at temperatures below 1000 °C. Moreover, the most organic matter and carbonates are ignited below 1000 °C [31]. On the other hand, the less organic matter could be detected for the materials burned between 1000 °C and 1050 °C temperatures. Higher temperatures can also drive off structural water from clays and β-wollastonite [30].

The LOI of ceramic materials is decreased as the quantity of β-Wollastonite added increased (see Fig. 3a). Because, the amount of organic matter, inorganic carbon, and minor organic residue existing in the clay are substantially replaced by β-wollastonite. Besides, the LOI of β-wollastonite is low compared to clay. A comparable result has been reported elsewhere [32, 33] that the presence of β-wollastonite decreases the gas exchange during firing which is a low loss on ignition. The average results of the LOI of ceramic materials are 4.16, 3.19, and 2.79% for the addition of 5, 15, and 25% of β-Wollastonite. Ceramic bodies were prepared from pure clay exhibited high weight loss on ignition (9.7%) [28]. Fig. 3a also shows the synergetic effect of firing temperature and the amount of β-Wollastonite on the LOI of ceramic materials. There is no interaction effect between firing temperature and the addition of β-Wollastonite (p>0.05) on the LOI of ceramic materials. When the composition of β-Wollastonite increased from 5 to 25% the loss on ignition of ceramic materials is decreased by 37.16% (3.95 to 2.48%), 31.21% (4.24 to 2.92%) and 31% (4.29 to 2.96%) for firing temperature of 950 °C, 1000 °C, and 1050 °C, respectively.

Fig. 3b shows the effect of particle size of β-Wollastonite on the LOI of ceramic materials. It is observed that the LOI is decreased with an increase in particle size of β-Wollastonite. Because of the presence of a larger specific surface area and larger exposure for heat, the finer particles were completely ignited than a coarse particle [34]. The weight loss due to the gases off during ignition is the main cause for the formation of...
porous and cracks. The average values are 3.46, 3.4, and 3.27%.

Fig. 3b also shows the interaction effect between the firing temperature and particle size of β–wollastonite on the LOI of ceramic materials (p>0.05). The LOI of ceramic materials shows a small variation between firing temperature 1000 and 1050°C when the particle size of β–wollastonite increased from 63 to 125 µm. On the other hand, the ceramic material fired at 950°C has a low LOI. The average values of loss on ignition of ceramic materials for each particle size of β–wollastonite (63, 75 and 125 µm) are 3.24, 3.17 and 3.06%, respectively.

β‒wollastonite (36.62% (4.08 to 2.58) for 63, 75 and 125 µm of particle size of β–wollastonite, respectively.

4. Conclusion

The bulk density and loss on ignition of ceramic materials were increased with firing temperature. However, both bulk density and loss on ignition of ceramic materials were decreased when the particle size of β–wollastonite increased. Similarly, the bulk density of ceramic materials is increased with the composition of β–wollastonite. The loss on ignition of ceramic materials is decreased when the amount of β–wollastonite increased. In general, it was observed that firing temperature, particle size, and percentage of β–wollastonite have an important effect on the quality of the ceramic materials.

5. References


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