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The Potential of Waste Eggshells Used in the Ceramic Industry: Enhancing Morphological, Structural, and Mechanical Properties of Whiteware Body Prepared through the Solid-State Sintering Process

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Abstract

This paper aims to investigate the utilization of waste eggshells to minimize the production cost of whiteware bodies and ultimately enhance properties such as whitish color, phase stability, compositional variation, and mechanical strength. This work prepared samples of whiteware bodies by a solid-state sintering process with a 5%, 10%, and 15% addition of waste eggshell powder. The powder was prepared by calcinating the eggshell at 800 °C to obtain calcium carbonate. The batch samples were prepared by the dry pressing method, adding different percentages of eggshell powder and then firing at 1100°C. SEM can characterize the addition of eggshells to the whiteware body for morphology and XRD for structural evaluation. The Modulus of Rupture and Impact test determined the mechanical properties of the final fired body. The tests determined eggshells in 5%, 10%, and 15% whiteware bodies to improve morphological, structural, and mechanical properties and their experimental result is 1.8MPa, 11.2MPa, and 7.03MPa in flexural strength, 7.35psi, 9.42psi, 8.86psi in Impact strength and 3.08%, 7.07%, 9.11% in water absorption. The Average particle size is 942.34, 367.185, and 481.79 nm in 5%, 10%, and 15% waste eggshell bodies. *It can be suggested that waste eggshells are a potential substitute for Calcium carbonate in the ceramic industry that can reduce material costs and enhance their properties.*

Keywords: Waste Eggshell, Whiteware Body, Mechanical Properties, Calcination, Solid State Sintering Process.

1. Introduction

In this paper, the whiteware bodies' effect on adding the calcium Carbonate source of waste eggshells that are connected to the poultry farm in 5%, 10%, and 15% and correlation with the whiteware body test results of SEM, XRD, modulus of rupture (MOR), and impact strength. Calcium Carbonate ($CaCO_3$) is used in whiteware bodies for an extended period, initially applied to tableware and artware due to its desirable properties. These include a white color, a smooth finish, an elegant appearance, and in some instances, a degree of clarity [1]. In whiteware, bodies are triaxle formations in raw materials such as silica, feldspar, and clay, which are the main components of the body [1], [2]. Industrial wastes like calcium carbonate ($CaCO_3$) pose a significant threat to our nation and country [3]. Fertilizers, waste eggshells, fish bones, and animal bones have polluted our land, and that directly or indirectly mixes our food and worse reflects on the body. Therefore, many scientists and NGO companies have to promote not to use directly or indirectly this waste and collect this waste at a particular point that is easy to remove [4]. Utilization and recycling of industrial or agricultural waste are regarded as eco-friendly and reduce our environment's negative effects [5]. Recycling and using raw waste materials are not new, but they have been established to reduce exhaustion and improve pollution's health [2], [6]. Waste eggshells were collected, washed, then calcined at 800C temperature, ground into fine particles, and mixed with the batch composition body [7]. They used calcined Eggshells in 0%, 5%, 10%, and 15% for a porcelain body prepared at 1100C temperature by a solid-state sintering process through examined morphological, structural, and mechanical properties[8], [9].

2. Experimental procedure

2.1. Materials

Waste eggshell powder is used in the whiteware body through a solid-state sintering process. Solid-state sintering is a facile process for fabricating ceramic products like whiteware. Whiteware bodies are triaxial composition. These are clay, quartz, and feldspar. Quartz was mainly used for gaining body strength. Feldspar is used as a fluxing agent to reduce the firing temperature and mature the body [10]. Clay is the main skeleton of the whitewares. Waste eggshells are a source of calcium oxide. In the solid-state sintering (SSS), Calcium Oxide and silica make the secondary phase $CaSiO_3$ [11]. Here is the chemical formation of these materials 1. Quartz (SiO_2) 2. Potash Feldspar (K_2O. Al_2O_3.$6SiO_2$) 3. Kaolinite ($Al_2Si2O_5(OH)_4$) 4. $CaCO_3$ source of waste eggshells in the local market 5. Carboxyl Methyl Calluses (CMC). CMC was used for binding materials. A batch body consisting of eggshell powder of 5%, 10%, and 15% composition was prepared in 100 grams of total powder mixture. The uniform and flawless mixture preparation creates a powerful, ideal body.

2.2. Methodology and sample preparation

The Solid State Sintering calcined process prepares the compositional variation of the whiteware bodies. The main ingredients used to manufacture whiteware bodies are Feldspar, clay, and quartz. The body enhances its application, mechanical strength, and other properties based on the inclusion of materials. Eggshell waste was calcined at 800 °C. To prepare the body for ball milling, quartz, and feldspar, two stone minerals, had to be crushed and combined with all other ingredients. The compositional variance among three bodies employed in SiO_2 (51%), feldspar (3%), and the variance formed in kaolinite and waste eggshell percentage. The inclusions of waste eggshell CaO, which behaves like a fluxing agent that reduces the firing temperature. Weighed in 100-gram batches in compositions of 5%, 10%, and 15% discarded eggshell, followed by a 24-hour ball milling process. The slurry was collected in a beaker after it formed, and any remaining water was taken out of the oven to be dried at 80 °C. The sample is prepared for testing after forming the powder into a solid body and firing it at 1100 °C.

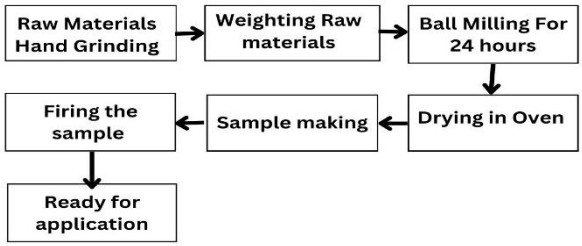


Fig 1. Flow Diagram in sample preparation

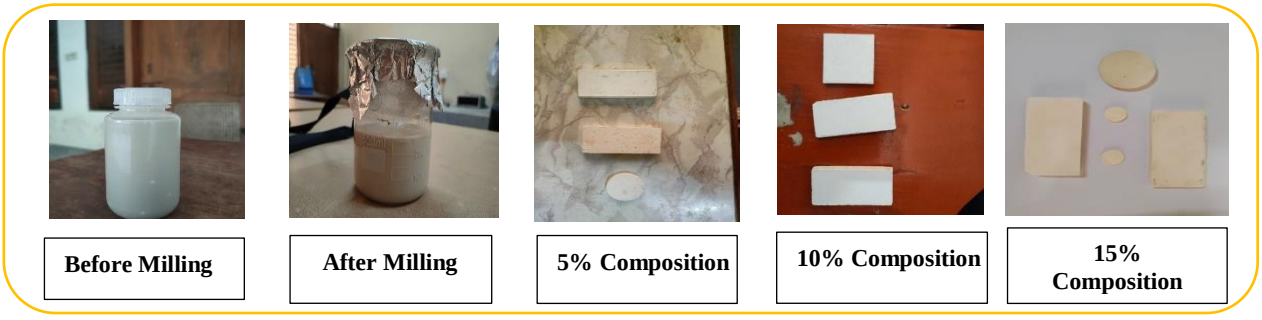


Fig. 2. Sample preparation in 5%, 10%, and 15% waste eggshells batch compositions.

3. Characterization

3.1. XRD

X-ray diffraction (XRD) is a process for understanding a sample's crystal structure, chemical composition, and physical properties of that sample. Crystal samples are mainly a group of atoms and X-rays contain electromagnetic waves. The atom's electrons scatter the incoming electromagnetic waves. The scattered electromagnetic waves provide informative data for the sample's surface. The sample's shape, stress, strain, etc. can affect the XRD results. XRD is measured by Bragg's equation,

$$2d \sin \emptyset = n\lambda \quad (1)$$

Where ∅ is the angle between the sample and the electromagnetic wave, d is the distance between the samples & its range around (2-6) Å, and **λ** is the wavelength of the scattered electromagnetic wave & its range around (1-100) Å.

3.2. SEM

A scanning electron microscope(SEM) is a kind of electron microscope that scans the sample surfaces and makes informative images for the surface's topography. The SEM machine creates electrons that interact with the sample's atoms and produce high-resolution & informative images that are collected by the SEM machine. According to De Broglie equation,

$$\lambda = \frac{h}{\sqrt{2meV}} \tag{2}$$

Where, **λ**=wavelength of the electron & its average wavelength is 550nm,h= Planck's constant(6.626×10^{-34}Js), m=mass of the electron, e= electron charge, V=accelerating voltage & it works in (1-30)Kv.

3.3. Modulus of Rupture (MOR)

Modulus of rupture (MOR) determines the ultimate strength of a material before yields. It defines the tensile strength & compressive stress of the specimen. A three-point load-applying setup is needed to measure the MOR, and the prepared sample must be carefully placed in it. After that force is applied until the sample is yielded. Then the yielding point of the sample & finally MOR value are measured by using the maximum loading force equation, that is

$$\mathrm{MOR} = \frac{3\mathrm{Fl}}{2\mathrm{bh}^2} \tag{3}$$

Where F is the maximum loading force (N), l is the distance between two supports (mm), b is the width of the sample (mm) & h is the thickness of the sample (mm).

3.4. Impact Strength

An Impact test is a material testing method for determining the energy absorbed by the testing material before fracture when the load is applied. It identifies a material's toughness and ductility. Impact tests are (i) the Izod Impact Test and (ii) the Charpy Impact Test. In Izod testing, the sample is placed vertically with the applied pendulum in the testing machine. The pendulum falls from a known height at the opposite point of the notch. In a charpy test, the prepared sample is placed horizontally & notched towards the impact direction in the testing machine. Then a pendulum hammer falls from a known height and hits the sample.

$$\text{Impact strength} = \frac{12\mathrm{i}}{\mathrm{t}^2} \tag{4}$$

Where i is the maximum loading force (ft. - pound), t is the distance between two supports (cm).

3.5. Water Absorption

Water absorption is a material's property that defines the ability of a testing material to absorb water when the testing material is immersed in water in a given situation. For water absorption testing, the testing sample must be dried before testing & measure its weight (w2). After drying, the sample is immersed in water for 24 hours at 25°C. Then sample take-out from water & measured the sample's weight (w1). Finally, Water absorption is determined by the most well-known formula, which is,

$$\text{Water absorption} = \frac{\mathrm{w1}-\mathrm{w2}}{\mathrm{w1}}100\% \tag{5}$$

Here, w_1 is the Wet Sample and w_2 is the Dry sample of batch bodies.

4. Result & Discussion

4.1. XRD analysis

X-ray diffraction (XRD) analysis to investigate the crystalline phases in the whiteware bodies containing varying amounts of waste eggshell powder (5%, 10%, and 15%) subjected to solid-state sintering. The XRD pattern of the

whiteware body exhibits a peak corresponding to the crystalline phase found in triaxial whiteware compositions, such as quartz, clay, and feldspar. Upon heating at 1100°C, a new whiteware body forms calcium silicate ($CaSiO_3$), also known as wollastonite, through the reaction between calcined eggshell and silica. This phase helps to increase the mechanical properties of the ceramic body. For 5% of eggshell content, this graph shows that lower intensity wollastonite peaks indicate the limited amount of calcium oxide present, which reduced the $CaO\text{-}SiO_2$ reaction. With the higher content of eggshell (10% and 15%), the intensity of the wollastonite peak increased, indicating a higher degree of phase transformation and incorporation of calcium from the eggshell into the main ceramic body. This XRD result suggests the incorporation of waste eggshell powder in whiteware bodies leads to new phases like wollastonite and anorthite, which helps enhance the structural and mechanical properties of whiteware bodies. The observed intensity peak represents the corresponding plane for all the samples from the JCPDS card no 96-900-1941.

The average crystalline size of the sample was found out by the Debye-Scherrer equation,

$$d = \left(\frac{k\lambda}{\beta cos\Theta}\right) \quad (6)$$

Where d is the average crystalline size,k is the Debye-Scherrer constant(0.98), λ is the wavelength, β denotes the full width at half maximum (FWHM) and Θ is the Bragg angle. The average crystalline size is shown in the table.

Table 1. Different parameters were achieved from XRD data.

Sample	Average size	Lattice parameter (A^0)		c/a ratio	The volume of the cell
	D (nm)	a	c		($Å^3$)
5%	942.34	13.3587	15.2244	1.139	2328.96
10%	367.185	13.3390	15.1120	1.132	2328.62
15%	841.78	13.3276	15.1222	1.134	2328.35

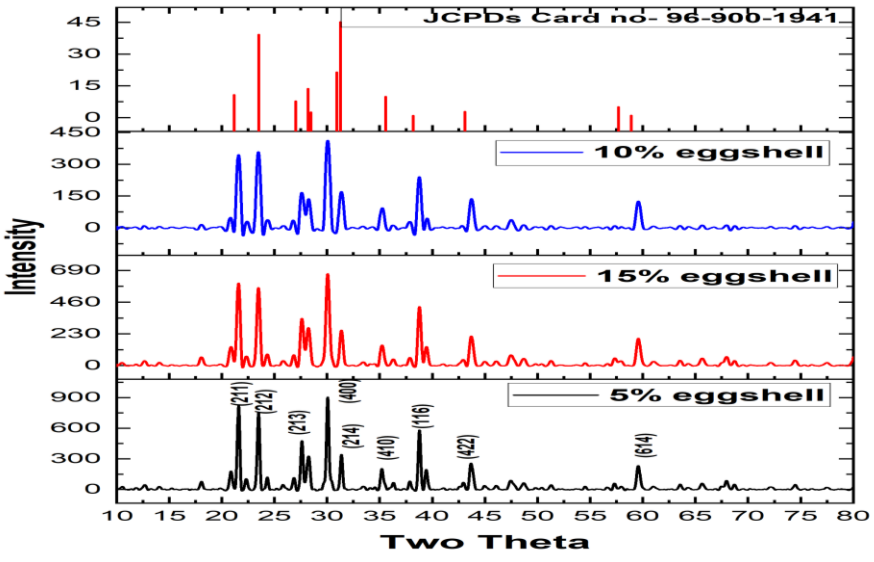


Fig. 3. XRD analysis of 5%, 10%, and 15% waste eggshell sample peak position and miller indices.

4.2. SEM Analysis

The surface Morphology of the sample whiteware body was observed through SEM studies. The figure SEM shows the microstructure of the whiteware sample in 5%, 10%, and 15% waste eggshells. In a 5% sample, the microstructure shows the particles with irregular shapes and have some porosity in the structure. .in the 10% waste eggshell sample the partials are also irregular in shape but the particles are small grain size and have a porous structure. The 15% waste eggshell has a more porous structure than the 5% and 10% also has an irregular shape of the particles, due to the high amount of calcium carbonate that produces carbon dioxide gas. Therefore, the

density of 5% is higher than the 10% and 15% samples. So addition to the increasing eggshell in the whiteware body the density of the whiteware body is decreasing. The SEM image and the particle size distribution are below

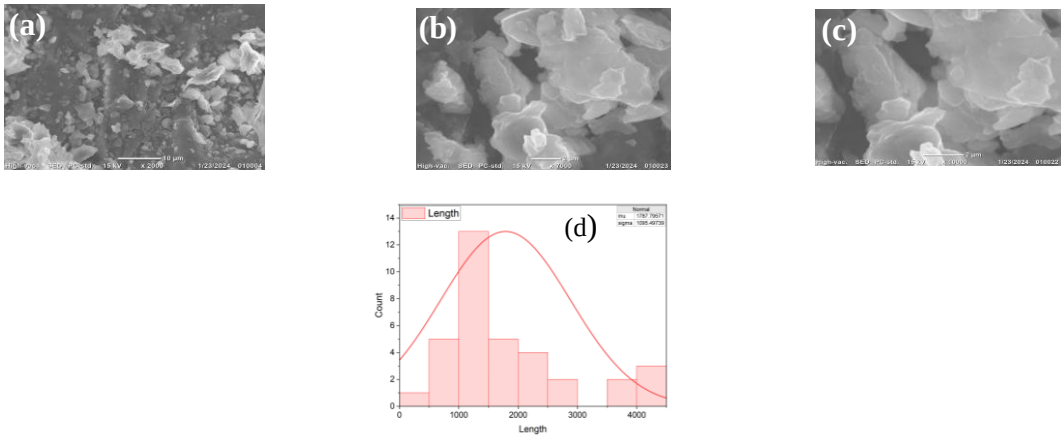


Fig4. SEM image of a) 5%eggshell sample)10%eggshell sample, c)15% eggshell sample, and d) particle size distribution

4.3. MOR Analysis

The mechanical strength of the whiteware body made using varied amounts of leftover eggshells as a partial substitute for traditional raw materials was evaluated using the Modulus of Rupture (MOR) test. The results of the test yielded MOR values that clearly show how waste eggshells affect the ceramic bodies' flexural strength. In plot shows that the 5%, 10%, and 15% batch composition of waste eggshells' Flexural strength values vary. In 5%, 10%, and 15% values are 1.8 MPa, 11.12 MPa, and 5.73 MPa. The value of 10% compositions shows better performance than others 15% has less strength gain because $CaCO_3$ are high percentage of CO_2 to create high. Porosity and amount of use feldspar.

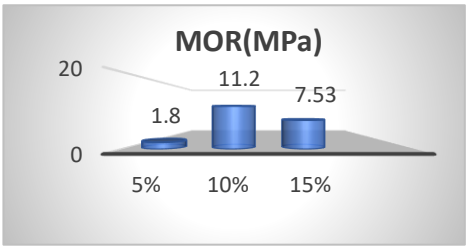


Fig. 5. MOR data analysis and comparison between 5%eggshell, 10% eggshell and 15% eggshell samples.

4.4. Impact Analysis

Impact strength of mechanical properties of the whiteware body using varied amounts of waste eggshells 5%, 10%, and 15% batch compositions to determine the sudden load on the sample. The results vary in compositions 7.75psi, 9.42psi, and 8.86psi shown in the bellow plot below. 10% of compositions show better results for utilizing proper use of other raw materials and waste eggshells. Because of a high percentage of $CaCO_3$, the maturing temperature is high enough because of the low percentage of using feldspar. These effects are affected by the impact strength results.

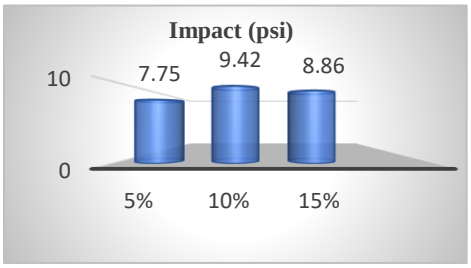


Fig. 6. Impact data analysis and comparison between 5% eggshell, 10% eggshell and 15% eggshell samples.

4.5. Water Absorption

Water Absorption results are shown in the plot 3.08%, 7.07%, and 9.11% in 5%, 10%, and 15% waste eggshells compositions body. Here 5% of compositions have less porosity because less waste eggshells were used, contributing to $CaCO_3$ that less CO_2 produced.

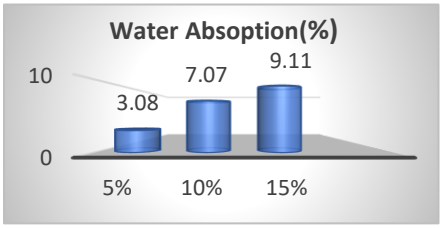


Fig. 7. Water Absorption data analysis and comparison between 5% eggshell, 10% eggshell and 15% eggshell samples.

5. Conclusion

In the experiment, the eggshell waste was used as a favorable alternative source of calcium for the fabrication of porous whiteware ceramic. Increasing the waste eggshell percentage increases the porosity of the whiteware ceramic body. Increasing the porosity led to a decrease in the bulk density and the thermal conductivity of the ceramic. Due to the low bulk density of the whiteware, the whiteware ceramic is lightweight. The compressive strength also decreases due to an increase the porosity. In the whiteware ceramic body composition waste eggshell 10% sample to 15% sample the compressive strength changes from 11.2 Mpa to 5.73 Mpa. According to the research, the waste eggshell used in the whiteware body enhances the morphological, structural, and mechanical properties. The use of 5%, 10%, and 15% waste eggshell in the whiteware body shows that 10% is the optimum batch composition This batch has a higher value of mechanical, structural, and morphological properties than the other batch compositions. The 10% batch composition has a preferable MOR and IMPACT value than the other batch composition. Therefore, utilization of waste eggshell in whiteware body 10% of waste eggshell batch composition is preferable.

6. Reference

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