Green Synthesis of Calcium Oxide Nanoparticles from Pigeon Eggshells for Cement Composites

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ABSTRACT

Solid waste management is one of the challenging issues faced by developing countries. India ranks 3rd in the production of eggs, generating about 3.8 billion kilograms annually. This growth in production at a larger rate is mainly due to the increase in domestic consumption. This could lead to a larger generation of solid waste. To overcome these issues, eggshells could be effectively incorporated into concrete production as cementitious blends. This paper investigates the suitability of pulverized eggshell powder having antibacterial activity, as a partial substitute for cement, and it enhances properties. The material characterization techniques like Scanning Electron Microscope (SEM) X-Ray Diffraction (XRD) were used to investigate the feasibility of using eggshell powder. Then the cement was partially replaced by pulverized eggshell at 5%- 20% by its weight. The fresh property was assessed using the flow table test, while the hardened property was determined using the compressive strength of the cement mortar for up to 30 days. From the test results, 10% pulverized eggshell powder when utilized in mortar gives optimum compressive strength. The microstructural investigations proved to be evident for pulverized eggshells when replaced at 10% delivered good strength and make smart cement.

Keywords: Smart Cement, Compressive strength, Solid waste, Pigeon eggshell, Antibacterial activity

INTRODUCTION

Eggshells are made almost entirely of calcium carbonate ($CaCO_3$) crystals. Eggshell waste is fundamentally composed of calcium carbonate and has the potential to be used as raw material in the production of lime [1-4]. In the modern day, waste management is a critical issue. Birds, fishes, and reptiles produce hard eggshells. The food processing industry, hatcheries, and poultry farms produce chicken eggs for everyday consumption across homes and bakeries. At the same time, with the increase in consumer demand, poultry is rapidly increasing. [5,6]

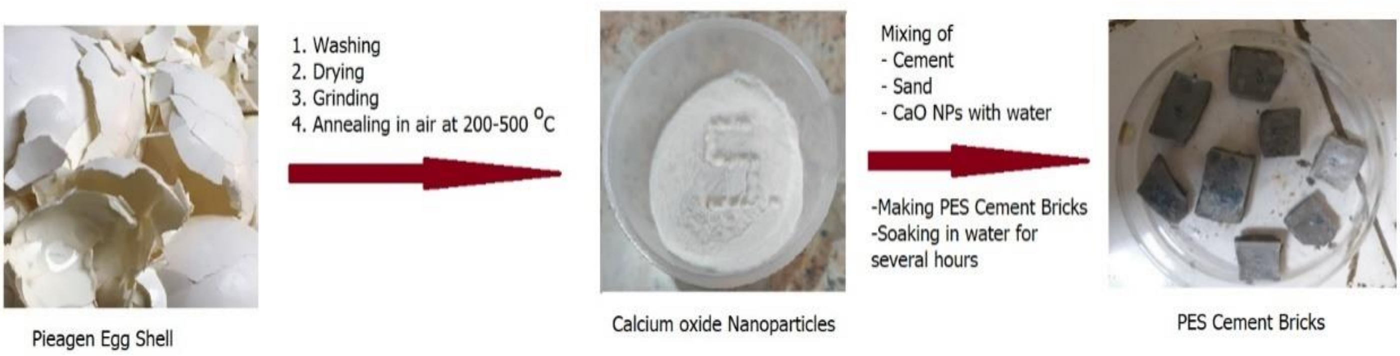
This regular consumption of eggs and disposal of eggshell waste are also adding as a filler to environmental pollution. In the global scenario, a total of 8.4 billion kilograms of chicken eggshell waste is generated every year [7]. All of it is majorly deposited into landfills with high management costs. When these chicken eggshell waste materials are properly used, it can add a step toward sustainability [8]. Researchers from across the globe have identified multiple potential uses for eggshells. Approximately 94% of the eggshell contains calcium carbonate, $CaCO_3$ [9]. Micro-elements of magnesium, iron, copper, boron, sulfur, zinc, silicon, and molybdenum are also contained in the eggshells. Magnesium carbonate (1%) and calcium phosphate (1%) along with organic matter and water are present in the eggshells. Calcium extracted from the shell is a potent source of calcium in the food industry and is used as a diluent in dental preparations [10]. CaO derived from the eggshell was reported to be efficient in the photo-degradation of organic pollutants. The eggshell powder was mixed at 30% by weight of cement to develop soil–cement bricks. Eggshells in soil-cement bricks are an efficient means of utilizing eggshells in the production of sustainable material and thereby reducing the amount of landfill waste disposal [11]. The eggshell membrane is used as a catalyst. Bioactive compounds present in eggshells are beneficial in the cosmetic and pharmaceutical industries. Another study predicted that heated eggshell powder could prove to be a potential source of antimicrobial activity. The effectiveness can vary depending on the bacterial species, with some studies showing higher activity against specific types. When CaO nanoparticles are in contact with the water present in the bacterial growth medium, they react to form calcium hydroxide ($Ca(OH)_2$). This reaction significantly increases the pH of the surrounding environment, creating an extremely alkaline condition that is toxic to bacteria, leading to cell death [12]. In preparing polymeric composites of polyamide and nylon black, the incorporation of eggshell powder has greatly influenced the tensile strength, flexural strength, and impact strength of the composites [13]. Waste eggshell impregnated on Ag nanoparticles was used as an efficient catalyst due to its good-low temperature reducibility [14]. The eggshell powder has been reported as a part replacement in cement with improvement in compressive strength. With an increase in the percentage of eggshell powder in concrete, the density is reduced. The splitting tensile strength was seen to increase with eggshell powder concentrations by the weight of cement. This study highlights the green synthesis of eggshell-derived CaO and its applications in cement. Increased carbonation depth (42%) and compressive strength (58.4%) were seen in the replacement of CaO (up to 40%) obtained from calcined eggshells as a fine aggregate in cement. Replacing heated eggshell powder increased the compressive strength of the mortar by 29% and 15% respectively [15-17]. Most studies on ESP-cement composites focus on macro-level properties like compressive strength and workability, often finding an optimal replacement range of 5-15% by weight. The long-term durability of concrete containing ESP is a major research gap. Some studies suggest a potential decrease in resistance to chemical attacks, such as sulfate and acid exposure, particularly at higher replacement levels. The impact of different processing methods, such as calcination temperatures and grinding fineness, on the pozzolanic activity and performance of ESP is not consistently explored [18]. This paper discusses the synthesis of eggshell powder using Hen egg Shell and Pigeon eggshell (PES), properties of eggshell powder and its correlation with slump, compressive strength, and split tensile strength, along with its utilization in cement concrete. The emerging research on eggshell powder opens new avenues for developing optimal concrete mixtures while minimizing adverse environmental impacts.

Experimental

Methods of Synthesis

Calcium oxide nanoparticles were prepared from chicken eggshells. The collected egg shells were thoroughly washed with water and air-dried for 48 hours. They were then crushed into fine powder using a mortar and pestle for 10 minutes. The fine powder was subjected to heat in a Muffle furnace at 500°C for 3 hours. During this process, carbon dioxide was released, leading to the formation of calcium oxide nanoparticles. The formation of Cao NPs is confirmed using SEM and XRD Study.

Figure 1: Sequence of CaO NPs and PES Concrete preparation



Concrete Mix Procedure and Measurement of Compressive strength

The general procedure for adding pulverized eggshells to a concrete mix. Take the 100g amount of ordinary cement and the 1 g of CaO NPs into the mixer. Mix the dry materials thoroughly to ensure a homogenous blend. Slowly add the specified amount of water while the mixer is running. Continue mixing for several minutes until the concrete mixture achieves a uniform consistency and the desired workability is reached. The addition of eggshell powder should result in a notable increase in the slump compared to a control mix without eggshell powder.

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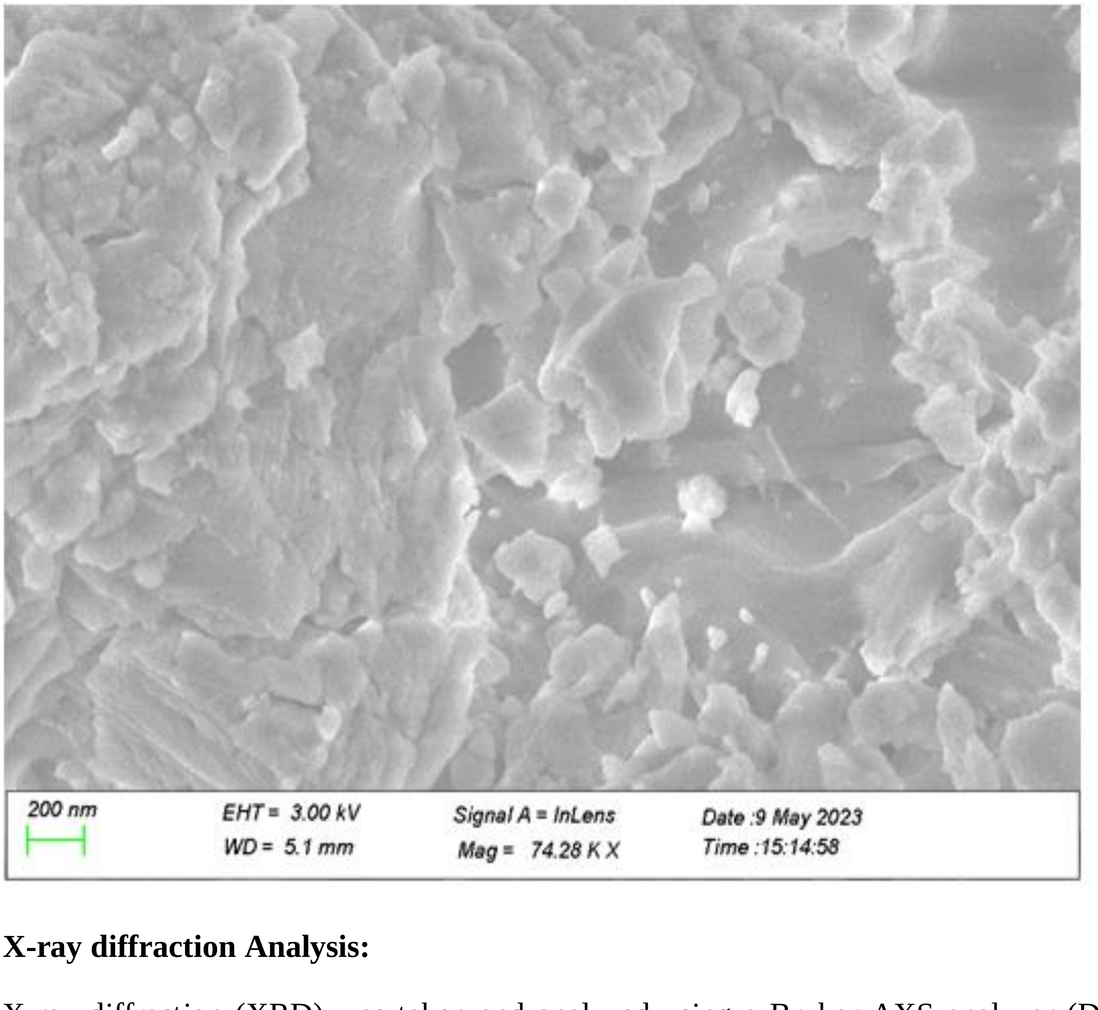
The compressive strength of the cement brick was determined after 10 days of curing. The compressive strength of the brick contains 1% of eggshell ash. The specimens are removed from the curing tank, and their surfaces are wiped clean. The dimensions of the specimens are measured to calculate the cross-sectional area. The specimen is placed in a compression testing machine. The load is applied gradually and without shock at a controlled rate until the specimen breaks. The maximum load at which the specimen breaks is recorded. A minimum of three specimens were tested for each mix to ensure reliable results.

RESULTS AND DISCUSSION

SEM Analysis

Under the SEM, pigeon eggshell powder particles are typically seen as irregularly shaped, angular fragments with sharp edges, a result of the crushing and grinding process. The degree of fineness, which is dependent on the grinding method, dictates the particle size. Finer particles appear more uniform, while coarser powders show a greater variation in size (80-100 nm) and shape shown in figure 2. The surface of the powder particles often appears rough and porous. At higher magnifications, the remnants of the shell's original structure—including the pores that facilitate gas exchange during incubation—are visible.

Figure 2. SEM Image of Pigeon egg shell Powder



X-ray diffraction Analysis:

X-ray diffraction (XRD) was taken and analyzed using a Bruker AXS analyzer (D2 Phaser). Samples were analyzed using a double-crystal wide-angle goniometry with the 2θ scan from 10°–80° at a scan speed of 5° 2θ/min in 0.05° 2θ increments using CuKα radiation (λ =0.15406 nm). The detected peak positions were compared with those of the International Center for Diffraction Data Standard (JCPDS) card no. 77-2376, patterns to identify the crystalline phases. Figure 3 extended peaks represent the dimension of the nano range particles. The XRD results of CaO nanoparticles peaks were observed at 32.24°, 37.40°, 53.92°, 64.21° and 67.45° with corresponding (h k l) values (1 0 4), (1 1 0), (1 2 2), (2 0 2), and (3 0 0) respectively. The average crystallite size was calculated by Debye-Scherrer’s formula and found to 85.97 nm.

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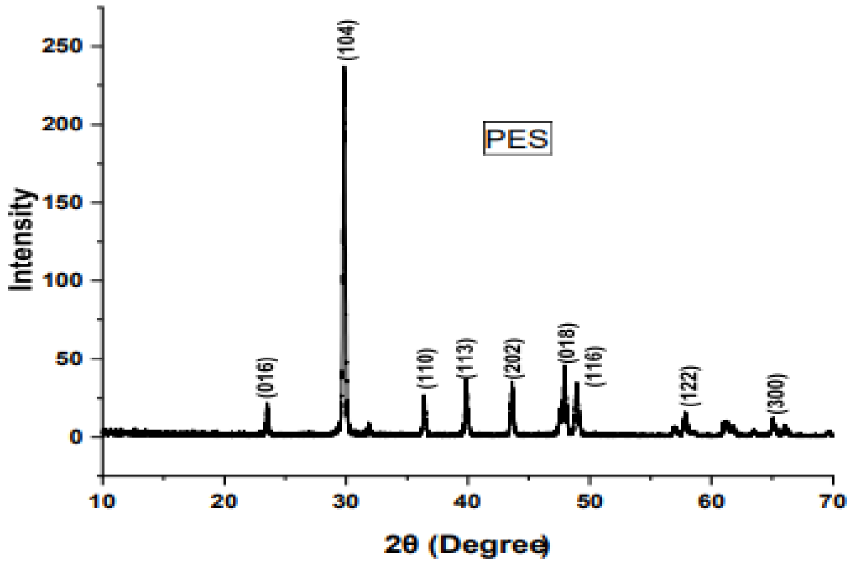
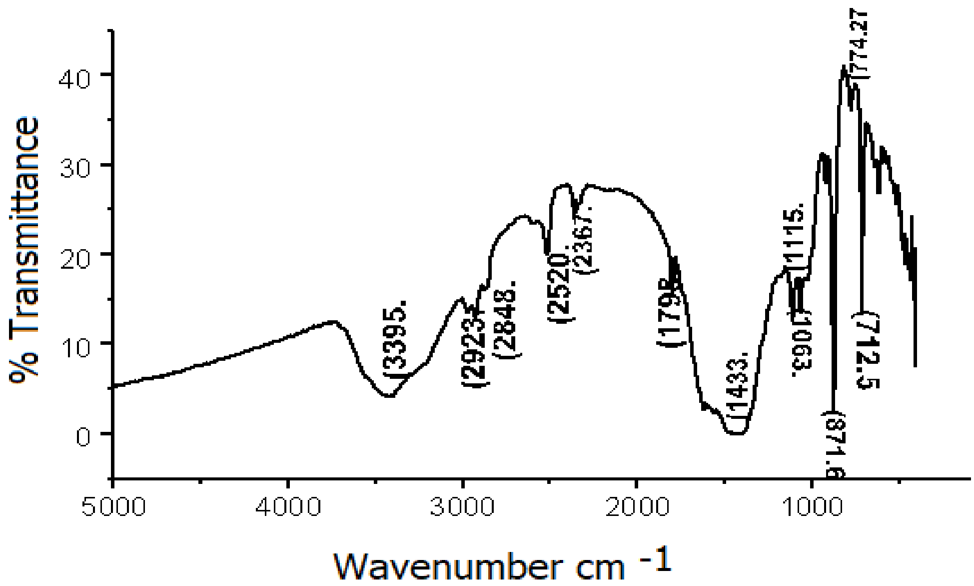
Figure 3: X-ray image of Cao NPS obtained from Pigeon eggshell



FT-IR Analysis

Figure 4 shows presence of asymmetric stretch, out-of-plane bend and in-plane bend vibration modes, respectively, for CO_2^{-3} molecules as expressed. The band at around 1020 cm^{-1} is associated with the bending vibrations of the C-O group in**.** The absorption bands of the organic matter visible at 2520 cm^{-1} and 1798 cm^{-1} disappeared after calcination of the raw eggshell samples. During thermal treatment by calcination, the carbonate in the pigeon eggshells is broken down to CaO and the absorption bands of carbonate molecules can be seen to have migrated to higher energy as represented by 1463 cm^{-1}, 1063 cm^{-1}, 878 cm^{-1}, and 529 cm^{-1}. A sharp peak in this region is often present, indicating the stretching vibration of free hydroxyl groups (O-H). This suggests the presence of residual moisture or calcium hydroxide ($Ca(OH)_2$) on the surface of the nanoparticles. The reduction in the mass of the functional group attached to the CO_2^{-3} ions is believed to be responsible for this development. Available data from literature showed that commercial Ca-O witnessed spectral bands at 867 cm^{-1} and 1483 cm^{-1} comparable to that witnessed by CaO NPs synthesized from waste Pigeon eggshell which was assigned to the vibration modes of mono and bidentate carbonates.

Figure 4: FTIR graph of CaO NPs obtained from Pigeon Eggshell



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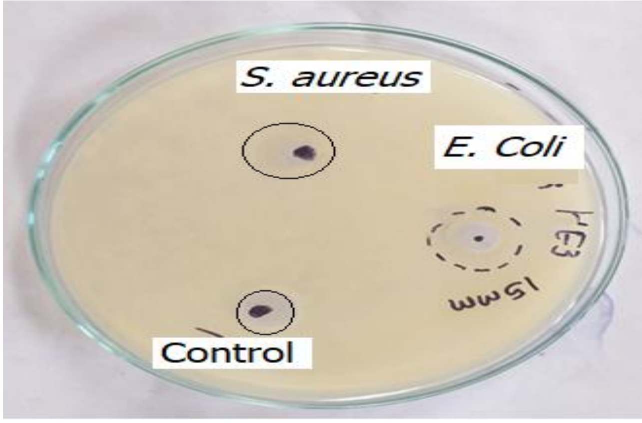
Antimicrobial Activity of CaO Nps

Studies have shown that CaO nanoparticles derived from eggshells are effective against a wide range of bacteria, including both Gram-positive (e.g., Staphylococcus aureus) and Gram-negative (e.g., Escherichia coli, Pseudomonas aeruginosa) strains shown in figure 5. As the concentration of the nanoparticles in the medium increases, the zone of inhibition typically becomes larger. This is due to a higher number of nanoparticles available to interact with and kill the bacteria, leading to a more pronounced antimicrobial effect.

Figure 5: Antimicrobial activity of CaO NPs obtained from Pigeon Eggshell



Effect of CaO NPs on cement:

The addition of pulverized eggshell enhanced the flow ability of concrete. However, the use of pulverized eggshell as a supplementary cementations material has reduced flow ability by making the mix extremely tight. The compression testing equipment was used to assess the effect of pulverized eggshell in cement mortar on the strength property. The composition of bricks sample and its compressive strength of the cement bricks is shown in Table 1.

Table 1: Composition of brick samples with its Compressive strength

Brick sample	Cement	Sand	CaO NPs	Compressive strength (MPa)
Control	**0.9**	**0.1**	**-**	**22**
PES	**0.9**	**-**	**0.1**	**31**
Control	**1.0**	**0.5**	**-**	**25**
PES	**1.0**	**0.5**	**0.1**	**37**

The control sample, with a composition of 0.9 kg of cement and 0.1 kg of sand, achieved a compressive strength of 22 MPa. The "PES" sample, which included 0.1 kg of CaO NPs in a mix of 0.9 kg cement and 0.1 kg of sand, yielded a significantly higher strength of 31 MPa. This represents an approximately 41% increase in strength compared to the control sample. The second control sample, with a mix of 1.0 kg of cement and 0.5 kg of sand, reached a compressive strength of 25 MPa. The corresponding PES sample, containing the same cement and sand quantities plus 0.1 kg of CaO NPs, resulted in a compressive strength of 37 MPa.

CONCLUSION

The synthesis of calcium oxide nanoparticles from waste eggshells offers a sustainable and economically viable approach with potential applications in various fields. A cost-effective and straightforward method was proposed to synthesize calcium oxide particles from eggshells rich in calcium carbonate, presenting a sustainable approach to material development. The synthesized nanoparticles were and revealing the formation of calcium oxide particles with particle sizes ranging from 80 – 90 nm for CaO NPs obtained from Pigeon eggshell. This marks an impressive approximately 48% increase in strength. The data consistently demonstrates that the addition of CaO NPs to the brick mix substantially improves its compressive strength. The study demonstrates that the presence of calcium carbonate within pulverized eggshell enhances its interaction with cement, leading to reduced voids within the cementitious system, thereby contributing to the material's strength and durability. The study contributes to the development of eco-friendly construction materials with potential for widespread application.

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