Characterization of Bio-Cement Reinforced with Eggshell Composites

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ABSTRACT

Eggshells are an interesting inorganic compound with a chemical composition and crystallographic structure similar to that of $CaCO_3$. This study aims to acquire a better understanding of the use of eggshells, which are commercially incorporated into cement mixtures, to increase the mechanical strength of cement-eggshell composite. The cement mixture was prepared by incorporating different compositions (5, 10, and 15 wt.%) of eggshell powder. The raw eggshells were cleaned, crushed, dried, sieved into small pieces, and mixed with the cement according to the desired composition. Analysis of the eggshell powder showed similar peaks to $CaCO_3$, indicating that eggshells can be used as a calcium source. The results indicated that the addition of 10 wt.% finely ground eggshell powder improved the compressive and flexural strength of the cement mix. It was also found that eggshell powder can potentially be used as a filler, as it exhibited the highest water absorption at 15 wt.%. The use of eggshells as a reinforcing component in environmentally friendly cement mixes has the potential to improve the environment by reducing waste, the need for conventional cement resources, and the pollution caused by excessive cement mining.

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Background of study

Recently, there has been a sharp increase in the demand for cement as a key material in the construction industry, particularly in developing countries. Cement production and usage are predominantly concentrated in Asia, accounting for 73% of worldwide production and 81% of global consumption [1]. Consequently, it is imperative to implement measures aimed at reducing energy consumption. Verma et al. [2] have reported that cement production results in the generation of various gaseous emissions, including nitrogen oxides, sulfur dioxide, carbon dioxide, and particulate matter.

In response to the social and environmental challenges related to sustainability and energy conservation, the cement industry is compelled to curtail cement production and partially substitute it with alternative cement ingredients. The majority of eggshell waste, deemed relatively worthless, is currently disposed of in landfills, which exacerbates significant environmental issues. In 2018, global egg production reached 76.7 million tonnes, representing a 14.95% increase compared to the previous decade [3].

Consequently, it is crucial to identify an appropriate substitute to effectively address this waste dilemma. Eggshells, which are composed primarily of calcium carbonate ($CaCO_3$), provide a unique combination of particle strength as a bio-ceramic composite rich in calcium (Ca). They possess numerous beneficial properties. Aside from containing traces of protein and other organic matter, eggshells consist mainly of $CaCO_3$, which is the most abundant form of Ca found in nature, present in shells, coral reefs, and limestone. The hybrid composite incorporates eggshells in the form of calcium oxide (CaO), which is tougher than carbide reinforcement particles. Compared to synthetic materials, this natural composite is inherently stronger and has a lower density. $CaCO_3$ serves as the main component of eggshells, sharing a similar composition with brittle substances such as chalk, limestone, cave stalactites, shells, coral, and pearls. Interestingly, in some countries, large quantities of eggshells are generated as waste every year and deposited in landfills, making them a promising bio-sorbent [4].

Eggshell as reinforcement in cement

Concrete is extensively used in the construction of buildings, bridges, and other structures. It is an indispensable material in construction. Several studies have indicated that replacing cement mixes results in a more workable building material [5] and that a solid mixture of materials, including cement, creates a sturdy structure. Reinforcement in concrete serves as a scaffold, enhancing the strength and toughness of the material.

According to Chandrappa and Kamath [6], eggshells are utilized as reinforcement in composites, thereby increasing their tensile strength. Eggshells are used as fillers in natural fiber composites, synthetic fiber composites, particle boards, laminates, and metal matrix composites. Although there is limited research in this field, some studies have explored various combinations of materials to analyze the properties of resulting composites. The incorporation of eggshell particles improves the interaction between the resin and the material, leading to increased tensile, flexural, hardness, impact, and shear strength of the composites [7]-[8]. The primary constituent of eggshells in this study is calcium carbonate ($CaCO_3$), which shares identical chemical properties with cement. Hence, the scientific endeavour dedicated to the conversion of waste into valuable resources within this project is substantial, thereby rendering the use of eggshells a viable concept worthy of adoption.

Methodology

Aggregate preparation

First, the preparation of chicken eggshell samples was necessary. The collected eggshells were thoroughly cleaned and washed with water, repeating these steps multiple times under running water to ensure the removal of all foreign contaminants, dirt, and dust. The protein layer, also known as the inner shell membrane, was carefully peeled off from the eggshells. Subsequently, the eggshells were dried for approximately 24 hours until completely dry in an oven set at 60 °C for 5 hours.

To further process the cleaned eggshells, they were initially crushed into smaller pieces using a hammer. Then, a mill was used to grind them into a powder with a particle size of 45 µm. A planetary ball mill was employed for the grinding process, continuously reducing the eggshells to smaller fragments. The ball mills effectively crush the eggshells, reducing their size. After grinding, the fine eggshell powder was sieved for 5 minutes using a sieving machine to eliminate any large particles, resulting in a relatively fine powder with a varied particle size distribution.

Cement mixed preparation

The specimens to be produced involve mixing a conventional cement mixture comprising cement, sand, and water. The mixing ratios for the concrete mixtures are presented in Table 1. Subsequently, the fine eggshell powder is added to the mixture in proportions of 5, 10, and 15 wt.% as a substitute for a portion of the cement. The resulting mixture is then poured into molds, with dimensions of 100 x 100 x 20 mm for compression testing and 267 x 212 x 200 mm for flexural testing. All concrete cubes are left to cure for 24 hours.

Once cured, the concrete mixtures are removed from the molds and further cured with water. As a comparative measure, a conventional cement mix is prepared as a control sample to assess both the similarities and differences between the reinforced and normal cement. Three sample blocks are prepared for each specified composition, with additional replicate samples to ensure robustness in the data collection process.

Table 1: Mix proportions of the concrete mixture

Materials (kg/m^3)	Control	1	2	3
Cement	200	180	160	140
Fine aggregate	150	150	150	150
Eggshells (wt.%)	-	5	10	15
Water/cement ratio	0.45	0.45	0.45	0.45

Characterization

X-ray diffraction (XRD) characteristics

X-ray diffraction (XRD) analysis of the eggshell samples is conducted using a Philips X'Pert Pro Diffraction instrument. Before fully mixing the eggshell powder into the concrete, a small amount of the fine powder with a size of 63 µm is set aside in order to examine it using the XRD instrument. The purpose of using the XRD method in this study is to identify the presence of $CaCO_3$, minerals, and other crystalline materials within the eggshells. It allows for the determination of the crystallographic structure, grain size, and preferred orientation of polycrystalline or powdered solid materials. XRD is commonly used for determining crystal size and identifying unknown compounds by comparing the diffraction data with a database maintained by the International Centre for Diffraction Data.

Mechanical characterization

Compression test

The compressive strength test is carried out using a universal testing machine, specifically the Instron 5582 model, and follows the specimen dimensions prepared in advance. The ASTM C109 [9] standard is employed to determine the compressive strength of the concrete. A compressive load is applied to the unsupported center of the specimen. Prior to testing, it is important to ensure that the top and bottom surfaces of the specimen are parallel to prevent bending or deformation at small strains. The specimens are compressed at a standard speed, and the deformation under various loads is recorded. The compressive strength and composition of the eggshell powder are then calculated and plotted on a graph.

Flexural test

In order to predict the load at which concrete members may crack, it is important to measure the flexural strength of the concrete. The flexural test is conducted using the same machine as the compression test, the Instron 5582 universal testing machine, but with a different probe. A midpoint loading test is performed to determine the flexural strength of the concrete [10]. Additionally, a midpoint bending test is conducted to determine the deformation of the concrete mix. To prevent any potential slippage of the specimen, the grips used in the testing machine should be appropriately ribbed.

Physical characterization

Water absorption

The water absorption rate is determined using the ASTM C1585 [11] method, which involves submerging the specimen in water for a specified period. In Figure 1, it can be observed that the cement blocks were immersed in water for 14 days. The specimens are then dried and re-immersed in water for another 14 days. The weight of the specimens is recorded before and after water immersion. After 14 days, the specimens are removed from the water, and their weight is determined. Finally, the percentage of water absorption is calculated using Equation (1).

$$\textit{Water absorption (\%)} = \frac{|m_{final} - m_{initial}|}{m_{initial}} \; x\ 100 \qquad (1)$$

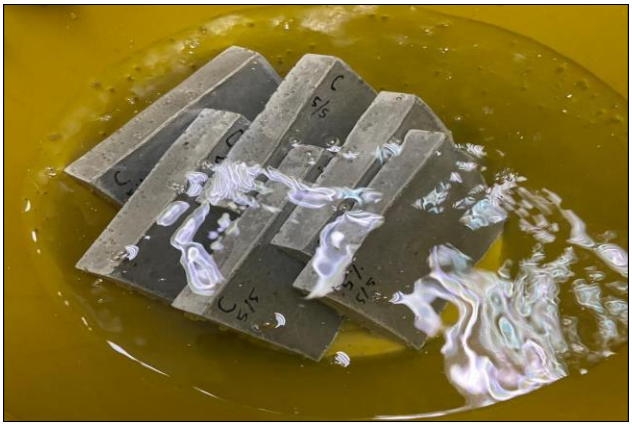

Figure 1: Cement-eggshell composites were immersed in water for 14 days

Results and discussion

Analysis of raw sample

Figure 2 illustrates the XRD pattern of pure eggshell, which contains a substantial amount of $CaCO_3$. From the XRD pattern, it is evident that eggshells are rich in $CaCO_3$. The most prominent diffraction peak occurs at $2\theta=29.4^o$, which has been confirmed by Shiferaw et al. [12] as the peak with the highest intensity.

Furthermore, additional peaks appear at $2\theta=23.1^o$, 39.4^o, and 47.5^o. The strong and sharp peaks in the XRD pattern indicate that the eggshell $CaCO_3$ powder has a well-crystalline structure and is abundant in aragonite. This observation aligns with the findings reported by Kamba et al. [13]. All of the peaks can be attributed to the pure aragonite phase of $CaCO_3$. When comparing the XRD peak information with the JCPDS file No. 85-1108, a close resemblance is observed.

Therefore, eggshells serve as a valuable material for a Ca source. According to Hoque et al. [14], the utilization of organic materials from unconventional sources is particularly advantageous for biomaterial applications.

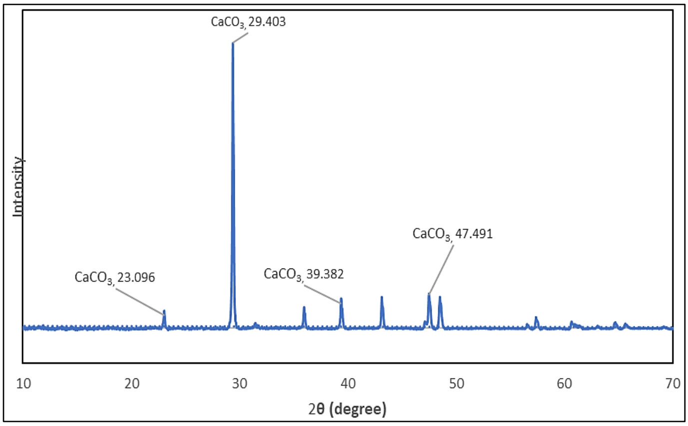

Figure 2: XRD pattern for $CaCO_3$ in natural eggshells powder

Mechanical properties

<u>Compressive strength</u>

The compression test is a crucial method for evaluating the behavior of composites and assessing their mechanical properties. However, measuring these properties using compression testing equipment can be challenging [15]. Saba et al. [16] also emphasized that the compression test allows for the analysis of material behavior under compressive loading, with compressive stress and strain calculated based on measurements of compression and deformation at different loads. According to Parthasarathi et al. [17], compressive strength is the most significant property of concrete, particularly for high-performance concrete.

Figure 3 presents a comparison of the compressive strength between eggshell powder used as a cement replacement and the results obtained in this study for varying concentrations (0, 5, 10, and 15 wt.% eggshells).

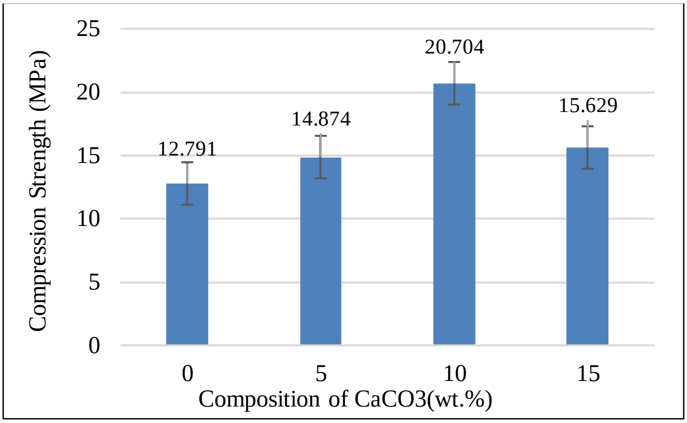

Figure 3: Compressive strength of concrete reinforced by different $CaCO_3$ contents result for 14 days

The findings indicate an increase in the workability of the concrete mixture. From Figure 3, it is apparent that adding finely crushed eggshells in the amount of 10 wt.% (as $CaCO_3$) results in an increased compressive strength compared to the control concrete cube. This enhancement can be attributed to the particle size of the eggshells themselves. Conversely, it was discovered that incorporating 15 wt.% eggshells into the cement decreases the compressive strength of the concrete, with the value nearly equal to that of the control concrete. This decrease can be attributed to the higher water absorption of 15 wt.% eggshell powder compared to 10 wt.% eggshell powder.

The presence of eggshell powder, containing organic matter, has the potential to hinder the chemical reactions that contribute to concrete strength, consequently impacting the bond between cement particles and resulting in a compromised overall structure. In conclusion, the utilization of 10 wt.% eggshells in pozzolanic construction aids in the improvement of concrete compressive strength. However, substituting 15 wt.% eggshells for cement leads to a reduction in the strength of the control concrete. Therefore, the replacement of 10 wt.% eggshells is considered the optimum percentage for achieving the highest compressive strength. However, the addition of these tiny particles as reinforcement has specific limitations. The compressive strength of the concrete decreases significantly once the composition exceeds 10% by weight. It has been observed that using a high proportion of fine eggshell particles impairs the compressive strength.

According to Djamaluddin [18], concrete exhibits high compressive strength but low tensile strength. The tensile strength of concrete is approximately 10-20% of its compressive strength. However, this weakness can be overcome by incorporating reinforcing steel, which is why reinforced concrete is widely used in structures worldwide. Hama et al. [19] studied the relationship between the proportion of eggshell substitutes and compressive strength. They discovered that the compressive strength of eggshell composite concrete (ESP) was greater than that of reference mixes with 3, 5, and 8 wt.% ESP. However, the compressive strength began to decrease at 10, 13, and 15 wt.% ESP. The reduction in compressive strength at 10, 13, and 15 wt.% substitution is mainly due to the formation of pores during the water absorption test.

According to Amin et al. [20], the compressive strength improved by 5.2, 1.0, 4.6, 11.1, and 0.7% at eggshell powder concentrations of 5, 10, 15, 20, and 25 wt.%, respectively, compared to the control sample. According to Imran et al. [21], the compressive strength of specimens with up to 10 wt.% eggshell powder was similar to that of specimens with 0 wt.% eggshell powder. However, the compressive strength decreased with increasing eggshell powder concentration at 15 and 20 wt.%.

Flexural strength

Moving on to flexural strength, it refers to the ability of concrete to resist deformation under a bending moment. The flexural test is performed on a 267 x 212 x 200 mm cube for 14 days to assess the development of flexural strength during curing. In general, the flexural strength of concrete increases with the addition of eggshell powder content up to a certain point. Eggshells act as fillers that can enhance the flexural strength of concrete, resulting in higher flexural strength in eggshell mixes compared to control mixes [22]. Some studies have indicated that the optimum eggshell content for maximum flexural strength is the same as the optimum eggshell content for maximum compressive strength [23].

The flexural strength results are depicted in Figure 4, where the highest flexural strength is achieved with an eggshell content of 10% by weight, exhibiting greater strength than the control sample. In this study, 10% by weight $CaCO_3$ was obtained through a calcination method. This involved weighing the $CaCO_3$ extracted from eggshells by their weight in grams. Therefore, 1 gram of $CaCO_3$ corresponds to 1% by weight $CaCO_3$. Comparing the results reveals that the substitution of eggshells mainly has a positive effect on the development of concrete strength after 14 days for various combinations of eggshells. The superior performance of the 10 wt.% eggshell composite can be attributed to its high $CaCO_3$ content and its ability to effectively fill the concrete. However, when using 15 wt.% eggshells in place of cement, the strength of the control concrete decreases.

Hence, replacing 10 wt.% cement with eggshells is considered the optimum percentage for achieving the highest flexural strength. However, at higher replacement percentages, the performance of the specimens is affected by excessive water absorption of the eggshell powder, indirectly reducing the water-cement ratio of the mixture [24]. The flexural strength of eggshell concrete follows a similar pattern as the compressive strength, as expected since the compressive and flexural strengths of concrete are generally correlated [25].

In general, the flexural strength of eggshell concrete increases to some extent as the eggshell content increases. According to Ramya et al. [26], the ideal eggshell content for maximum flexural strength is the same as the optimum eggshell content for maximum compressive strength. Similarly, Chong et al. [27] state that the ideal eggshell content for maximum flexural strength is between 6 and 10 wt.%.

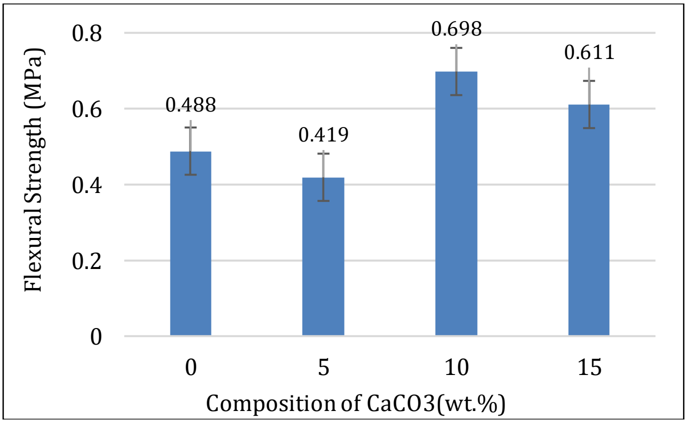

Figure 4: Flexural strength of concrete reinforced by different $CaCO_3$ contents result for 14 days

Physical properties

<u>Water absorption method</u>

The water absorption method was used to measure the total water uptake. Table 2 and Figure 5 demonstrate that the use of eggshells increases the weight loss of the concrete by up to 15 wt.%. The water absorption of the concrete mix can be attributed to the high water absorption capacity of the fine aggregates of eggshells. Generally, adding fine particles like eggshells to concrete can elevate its water absorption. It is evident that as the percentage of eggshell powder increases, the water absorption of all mixes also increases.

This phenomenon occurs because the fine particles create voids in the concrete, allowing water to penetrate more easily. The water absorption of concrete without eggshell powder (0 wt.%) is higher compared to that of eggshell concrete with 5 wt.%. This is because the eggshell powder acts as a filler, reducing pore spaces in the concrete. Ultimately, this helps diminish water absorption and enhance the overall durability of the concrete. If no eggshell powder is added to the concrete (0 wt.%), the concrete may exhibit higher water absorption due to the absence of the filling effect provided by the eggshell powder. The lack of filler can result in more open spaces or pores in the concrete, facilitating easier water penetration. Conversely, the addition of 5 wt.% eggshell powder assists in filling these pores and decreasing water absorption. Therefore, adding eggshell powder can help reduce the water absorption of the concrete.

Table 2: Water absorption of a concrete mix by different compositions of $CaCO_3$ contents

Composition of $CaCO_3$ (%)	Initial weight (g)	Final weight (g)	Water absorption (%)
0	338.39	362.10	7.00
5	353.01	372.74	5.59
10	331.50	353.00	6.49
15	332.90	356.96	7.23

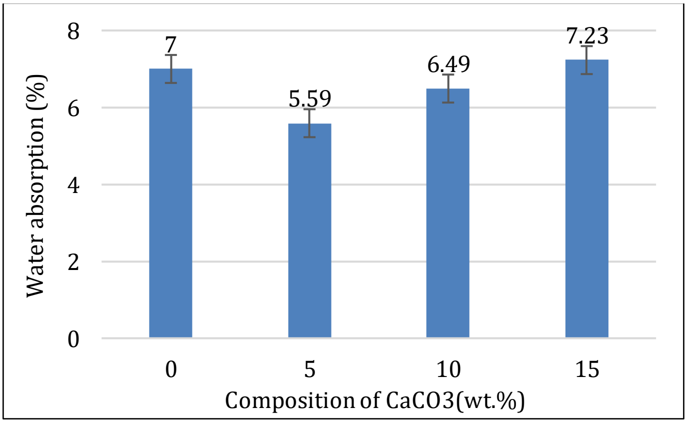

Figure 5: Water absorption of concrete reinforced by different $CaCO_3$ content after 14 days

It is essential to note that water absorption depends on various factors, such as the quantity and size of fines added, the concrete type used, the curing process, and environmental conditions. Thus, carefully considering the mix design and properties of the materials used is crucial when incorporating fine particles into the concrete. The high $CaCO_3$ content of eggshell powder enables it to absorb more water, resulting in increased water absorption with higher filler content [28].

Eggshell powder is naturally impermeable, which contributes to a reduction in the permeability of concrete. Additionally, during the mixing process at the time of pouring, eggshell powder promotes the acceleration of cement hydration by forming mono-carboaluminate [29]. Zhang et al. [30]. noted that the permeability of concrete has a significant impact on its strength, particularly in marine environments [30]. However, it should be noted that as the proportion of eggshell powder in concrete increases, the acid attack resistance of the concrete decreases. Research has shown that an increase in the water-to-cement (W/C) ratio leads to higher carbonation in concrete, primarily due to increased permeability and porosity [31].

Conclusions

From the research conducted, it appears that the use of fine eggshell particles as reinforcement in concrete mixtures was successful. The results show that an optimum percentage, namely 10% eggshell powder, gives the optimum value for the mechanical properties. Eggshell powder in concrete mixes can be used as an alternative disposal strategy for eggshell powder. The existing types of eggshell powder and those expected to be landfilled in the future were used in concrete mixes. In all cases, there was an optimum amount of eggshell powder that could be used without changing the mix preparation and still produce acceptable results.

For the recommendations, the exposure time for 7 and 28 days should also be studied and compared with the current results, of 14 days.

Contribution of Authors

N.A.S.M.R.: Conceptualization, Methodology, Experimental, Writing – original draft, Visualization, R.R.: Supervision, Conceptualization, Methodology, Experimental and Visualization, M.M.M, S.O, M.N. Z. and S.M.Y: Conceptualization and Supervision. All authors reviewed and approved the final version of this work.

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Conflict of Interest Statement

All authors declare that they have no conflicts of interest.

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