JPPIPA 11(11) (2025)

Jurnal Penelitian Pendidikan IPA

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Relationship of Physical Properties and Crystal Structure $CaCO_3$ Purebred Chicken Egg Shells

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Received: July18, 2025
Revised: October 07, 2025
Accepted: November 25, 2025
Published: November 30, 2025

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DOI: 10.29303/jppipa.v11i11.12227

Abstract: Preliminary research has been carried out on purebred chicken egg shells with research linking the physical properties and crystal structure of $CaCO_3$ of purebred chicken egg shells. The XRF results showed the atomic elements Si, P, Ca, Ti, Fe, Zn and Sr, with a Ca content of 95.603%. The use of 3%, 5% and 10% mixing variation methods, at heating temperatures of 900°C, 1000°C, 1100°C and 1200°C. The physical properties of dry shrinkage and water absorption decreased for each mixing variation and heating temperature, while the burn shrinkage values, density, and fracture strength increased for each mixing and heating temperature. The crystal structure formed did not change the mixing variation and heating temperature, namely the result of *MATCH entry number* 96-901-5692 of the hexagonal crystal system, *the space group* R-3C and the cell units a=b= 4.9771Å and c = 17.3369Å.

Keywords: $CaCO_3$; Crystal structure; Physical properties; Purebred chicken egg shell

Introduction

Chicken eggshells are an abundant biomineral waste that is often underutilized despite their high potential (Abdullah et al., 2023; Djayasinga et al., 2022; Tan et al., 2015). Approximately 95% of eggshell composition consists of calcium carbonate (CaCO3), a compound widely used in various fields such as pharmaceuticals, cosmetics, agriculture, and bioceramic materials (Awogbemi et al., 2022; Djayasinga et al., 2022). This makes eggshells a promising source of eco-friendly and low-cost raw materials. However, to optimize their utilization, a deeper understanding of the crystal structure and physical properties of the CaCO3 they contain is essential (Abdullah et al., 2023).

One important approach in characterizing biomineral materials is the analysis of their physical properties and crystal structure (Guo et al., 2021; Saraswati et al., 2023; Sulpis et al., 2018). CaCO3 can exist in three main crystalline phases calcite, aragonite, and vaterite each with distinct physical characteristics and thermal stability (Liang et al., 2023; Sun et al., 2018; Widyastuti et al., 2024). These crystalline forms significantly influence the material's performance in specific applications. Therefore, studying the relationship between the crystal structure and the physical properties of eggshells is crucial for determining their suitability for targeted uses.

X-ray Fluorescence (XRF) is commonly used to determine the elemental composition of materials. In the case of eggshells, XRF analysis typically shows that calcium is the dominant element, comprising up to 95% of the material. However, XRF cannot detect light elements such as carbon and oxygen, which are integral to the CaCO3 structure. As a result, the full composition of calcium carbonate cannot be confirmed through XRF alone. To obtain calcium in its elemental or more reactive forms, thermal reduction methods involving reducing agents such as activated carbon, aluminium, or magnesium are often used to convert CaCO3 into elemental calcium or calcium oxide at high temperatures.

Eggshells from various chicken breeds, including purebred types, generally contain approximately 95%

How to Cite:
Fajrah, M. C., Wardah, A., & Soegijono, B. (2025). Relationship of Physical Properties and Crystal Structure CaCO3 Purebred Chicken Egg Shells. *Jurnal Penelitian Pendidikan IPA*, *11*(11), 433–439. https://doi.org/10.29303/jppipa.v11i11.12227

calcium (Ca), as determined by X-ray Fluorescence (XRF) analysis. However, XRF is unable to detect the presence of light elements such as carbon (C) and oxygen (O), which are essential components of calcium carbonate (CaCO3). This limitation arises because XRF is less sensitive to elements with low atomic numbers. Therefore, although the primary compound in eggshells is CaCO3, only calcium is typically identified in XRF results. To isolate calcium in its elemental or other usable forms from CaCO3, one of the widely used approaches is reduction at high temperatures using reducing agents such as activated carbon, aluminium (Al), or magnesium (Mg). These materials serve as electron donors, enabling the decomposition of calcium carbonate and the formation of elemental calcium or calcium oxide (CaO), depending on the reaction conditions (Veronika & Rohmawati, 2022).

Some studies on CaCO3 mixing are shells using carbon dioxide in the carbonization process (Widyastuti et al., 2024). The following is the effect of calcination temperature variations on the properties of calcium carbonate in rice field snail shells (Nuryoto et al., 2021). Furthermore, the next research is the synthesis of hydroxyapatite from ale-ale shells using graphite-type carbon material (Nida Hamida, 2021). Calcium content of carbonate in the shell of the snail mas antacids (Yuliatun et al., 2023).

This research will conduct basic research with XRF and particle size measurements with an Andersen pipette, and also perform mixing of CaCO3 Purebred Chickens with the addition and variation of 98% purity activated carbon mixing (0%, 3%, 5% and 10%) and heating variations at (900, 1000, 1100 and 1200)oC. This method is carried out to analyze the physical properties , namely, dry shrinkage, water absorption burn, density and fracture strength from the mixing results and temperature variations. To complete this initial analysis, crystal structure analysis was also carried out using X-Ray Diffraction (XRD) (Deepak & Sargunan, 2019; Utami et al., 2022).

Research on the relationship between the physical properties and crystal structure of calcium carbonate (CaCO3) in purebred chicken eggshells holds significant urgency, considering the high potential of eggshell waste, which has been underutilized. Eggshells are composed of up to 95% CaCO3, making them a promising alternative raw material in various fields such as pharmaceuticals, bioceramics, agriculture, and cosmetics. However, the applicability and performance of CaCO3 largely depend on its crystal structure—especially its polymorphic phases such as calcite, aragonite, and vaterite—which influence its physical properties, including hardness, density, and specific surface area. This research is important because most existing studies focus only on commercial laying hens, while purebred chickens possess distinct genetic and physiological traits that are likely to affect the crystal structure and physical quality of their eggshells. By specifically examining purebred chickens, this study not only supports the exploration of new biomaterial sources but also aligns with the concept of circular economy and the sustainable use of organic waste (Ahmed et al., 2022; Pipich et al., 2008).

Although numerous studies have been conducted on the characterization of eggshells, most have focused on commercial laying hens. Purebred chickens, which have distinct genetic profiles and mineralization physiology, have not been extensively studied in this context. This presents an opportunity to explore the unique characteristics of eggshells from purebred chickens, both in terms of their crystal structure and physical properties. Thus, this research is essential to support the development of biomineral-based materials derived from organic waste and to promote sustainable practices aligned with circular economy principles (Diningsih & Rohmawati, 2022; Qiao et al., 2017; Qothrunnada et al., 2023).

In terms of novelty, this research offers a different approach compared to previous studies. The focus on purebred chickens as a source of CaCO3 is still very limited in the scientific literature, making this study a valuable contribution to understanding biomaterial variation based on poultry genetics. Furthermore, this research directly correlates the crystal structure of CaCO3 with the physical properties of the eggshell, which is rarely explored in depth. This approach allows for a more practical analysis, particularly in determining the potential application of eggshell-derived materials as bioceramic components or natural calcium supplements. The characterization techniques used are also comprehensive, including morphological analysis, phase composition, and thermal properties, providing detailed and reliable data. Therefore, this study presents a significant novelty in the field of biomineral-based materials and has the potential to drive the development of environmentally friendly and locally sourced materials that are both economical and sustainable.

Method

The main ingredients of the research are $CaCO_3$ purebred chicken eggshells and Aldrich purity of 98% purity. Supporting materials are pure H_2O and Polyvinyl Alcohol (PVA). Then the test equipment, the PSB0400 fracture test, the density and water absorption of Sartorius 2442, the Andersen pipette tube 500 ml. Process equipment of drying oven, printing equipment, printer press and furnace 46200 type high temperature.

The process methods carried out are the process carried out is that purebred chicken egg shells are

blended until smooth then a variation of 3%, 5% and 10% activated carbon mixing is carried out. Mixing is done using a magnetic stirrer on the stove at 50°C, then heated to 110°C to evaporate the H_2O. After that, the sample is burned at temperatures of 900°C, 1000°C, 1100°C and 1200°C. Samples are ready to be tested by characterization of dry sust, burn sust, density, water absorption and fracture strength. The tested samples were analyzed and concluded.

Result and Discussion

X-ray Fluorescence (XRF) Analysis

The XRF (X-ray fluorescence) test in this study utilizes spectrophotometry to identify and quantify the elemental composition of purebred chicken eggshells. This technique is a non-destructive analytical method that enables the detection of various elements based on the characteristic secondary (or fluorescent) X-rays emitted from a material when it is excited by a primary X-ray source. The primary objective of this XRF test is to determine the elemental constituents present in the eggshells. As shown in Figure 1, the quantitative results of the XRF characterization indicate that calcium (Ca) is the predominant element in the eggshell structure, with a concentration of 95.603% by weight. This value is significantly higher than those of other detected elements such as silicon (Si), phosphorus (P), sulfur (S), titanium (Ti), iron (Fe), zinc (Zn), and strontium (Sr). The dominance of calcium can be explained by its naturally high abundance in biological calcified structures, as well as its high atomic and molecular weight compared to the other trace elements. This confirms that calcium carbonate ($CaCO_3$) is the main constituent of the eggshell matrix, which plays a crucial role in providing structural strength and protection for the developing embryo inside the egg.

This high calcium content plays a crucial role in providing mechanical strength and rigidity to the eggshell, ensuring protection for the developing embryo against physical damage and environmental threats. Meanwhile, the presence of other elements such as silicon (Si), phosphorus (P), sulfur (S), titanium (Ti), iron (Fe), zinc (Zn), and strontium (Sr) in smaller amounts indicates their role as trace elements involved in the biomineralization process. For example, phosphorus (P) may contribute to the regulation of mineral crystallization, while strontium (Sr) is known to substitute calcium in the calcite crystal structure. Although their concentrations are relatively low, these elements can influence the physical and chemical properties of the eggshell, such as hardness, solubility, and resistance to pressure. Therefore, understanding the elemental composition of chicken eggshells is important not only for agricultural and food industry purposes but also for the development of biomimetic materials inspired by natural structures

Atom	Chem.Formula	*weight* (%)	*at/mole* (%)
14	Si	0,693	0,994
15	P	0,277	0,359
16	S	0,338	0,424
20	Ca	95,603	95,988
22	Ti	1,200	1,008
26	Fe	1,238	0,892
30	Zn	0,229	0,141
38	Sr	0,422	0,194

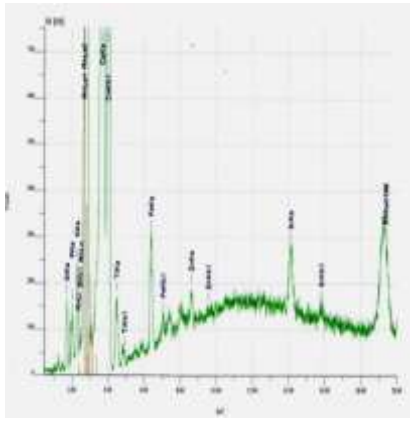

Figure 1. Purebred chicken eggshell XRF pattern chart and XRF results table

Meanwhile, qualitatively, it can be seen in Figure 1 in the form of a graph of the formation of peaks of each element as a result of the XRF characterization.

Dry Shrinkage of Combustion at 110°C

The graph in Figure 2 is describing the length of the sample over time is getting smaller and smaller because the graph state is decreasing, the shrinkage state is constant. Shrinkage without mixing of activated carbon is achieved at 80 minutes while mixing of activated carbon and $CaCO_3$ of 3% and 5% purebred chicken egg shells is achieved at 70 minutes and 60 minutes for 10% mixing. That the addition of activated carbon speeds up the drying time.

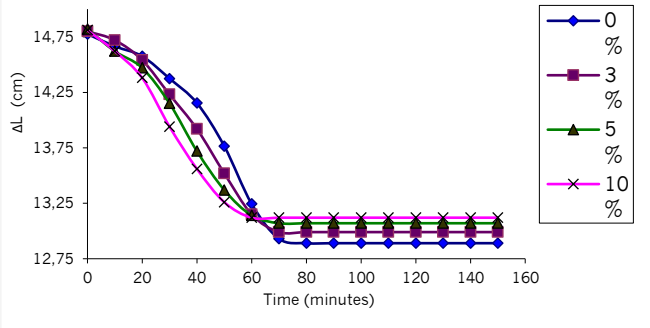

Figure 2. Dry shrinkage value at 110°C per % of mixing

Density

Based on Figure 3, it can be described that the mixing variation affects the mixing density value of activated carbon and CaCO3 of purebred chicken egg shells. The density price of 3%, 5% and 10% with the higher the temperature the density value increases, but there is a tendency at a temperature of 1200°C to be close to constant.

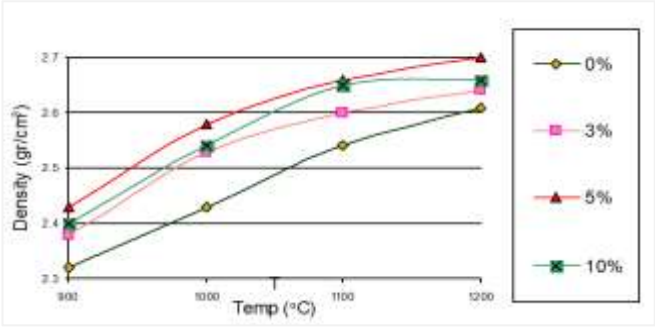

Figure 3. Density dependence (gr/cm³) per % of mixing to combustion temperature

Water Absorption

The mixing variation of 3%, 5% and 10% activated carbon and $CaCO_3$ of purebred chicken eggshells affects the water absorption. However, at a temperature of 1200°C, the water absorption value indicates a more constant direction, in this case, in addition to mixing the heating temperature, it also affects the water absorption value because this is very related to the density of the material.

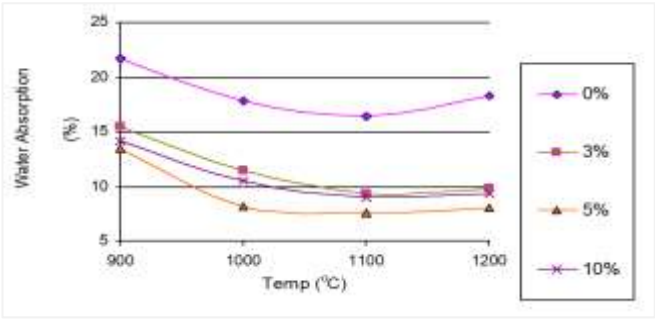

Figure 4. Dependence of water absorption per % of mixing to the combustion temperature

Burn Loss

Based on the results of the graphic description, the value of 0% blending combustion at 1200°C combustion and the lowest combustion shrinkage was found at 10% mixing with a combustion temperature of 900°C. Based on this, it can be analyzed that the addition of activated carbon slightly reduces the risk of pruning or changes in shape and size due to the combustion process.

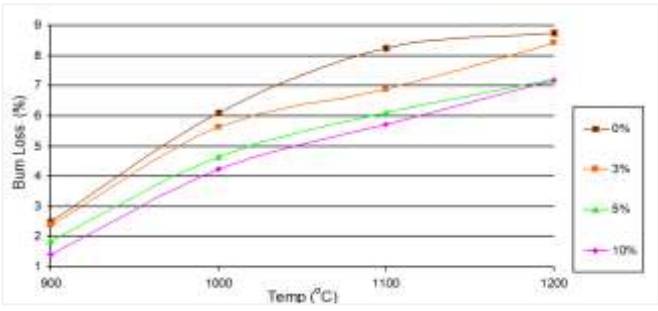

Figure 5. Burn shrinkage value of any % of mixing to the combustion temperature

Bending strength

The analysis in Figure 6 describes the fracture strength values for all 3%, 5% and 10% patches, where the higher the heating temperature the higher the fracture strength value.

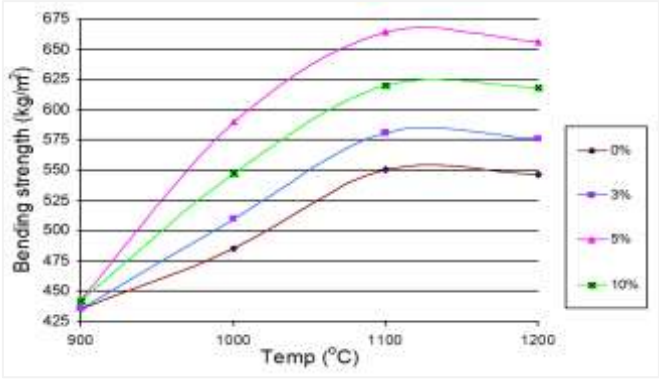

Figure 6. Fracture strength value per % of mixing to combustion temperature

$CaCO_3$ Crystal Structure Purebred Chicken Shell

Proof for all mixing and temperature variation methods for crystal structures, from the three largest peaks matched based on the crystal plane with *the Match software* in Figure 8. The results of the analysis from the description shown in Figure 7 that the crystal structure matched with *the Match* software obtained a matching result at *entry number* 96-901-5692 with a hexagonal crystal system, cell unit a = b ≠ c (a=b=4.9771Å and c = 17.3369Å), space group R-3C and a density of 2.681 g/cm3. When it is related to the results of the physical properties of dry shrinkage, burn shrinkage, density, water absorption and fracture strength in the results without mixing and mixing variations, the results of XRD and XRD analysis for the crystal structure of no mixing (0%) and 3%, 5% and 10% mixing with activated carbon are not visible changes in the crystal structure, even though the heating temperature has exceeded the melting point of $CaCO_3$ but in this case combustion is not carried out with this vacuum which is a factor main. As for the mixing variation, it also does not change the crystal structure and even the carbon added to each mixing is burned out, this is also due to the heating treatment without vacuum. The addition of 0%, 3%, 5% and 10% mixing variations all resulted in a decrease in dry shrinkage and water absorption, an increase in the value of burning, density and fracture strength, this describes that without mixing and mixing determining only the heating temperature, for mixing has not changed because the added carbon is burned out, for subsequent studies special treatment must be added when mixing with activated carbon.

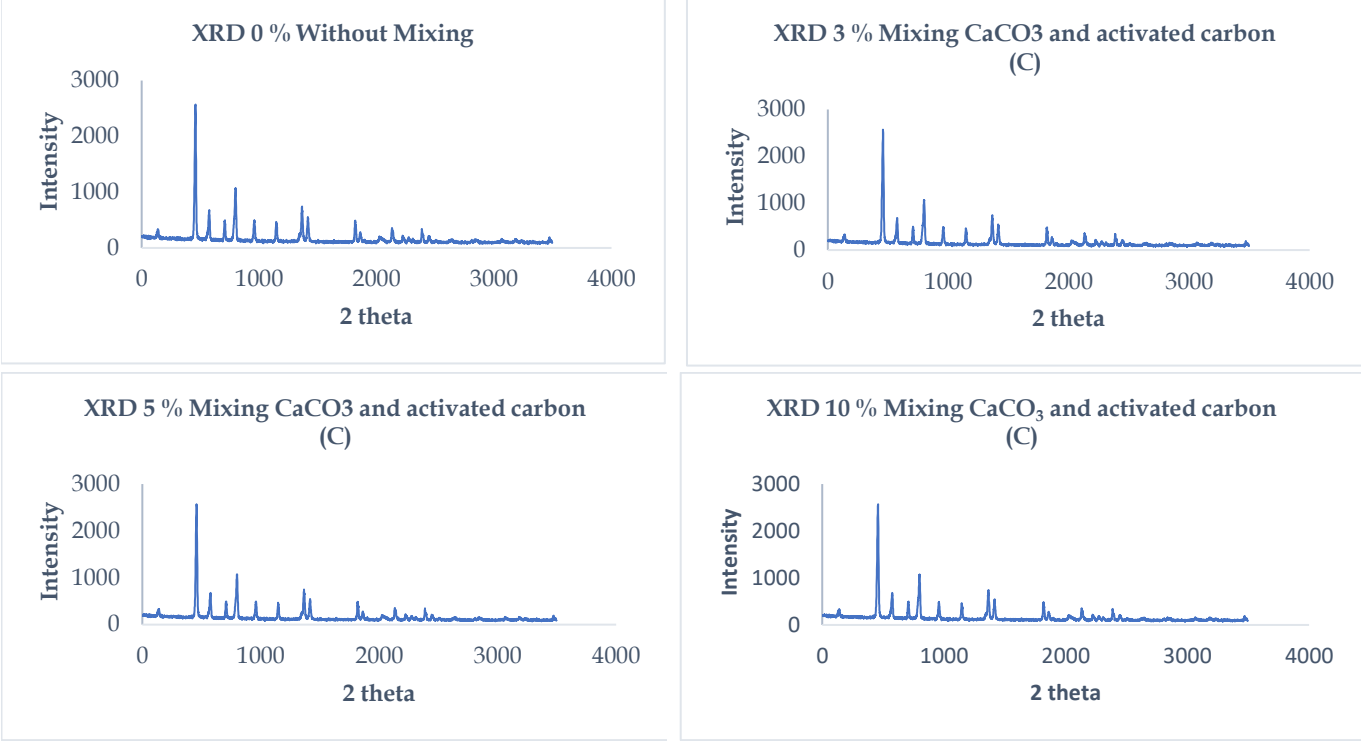

Figure 7. Yield of XRD $CaCO_3$ purebred chicken shells without mixing (0%) and Mixing with activated carbon at 3%, 5% and 10% temperatures of 900°C, 1000°C, 1100°C and 1200°C

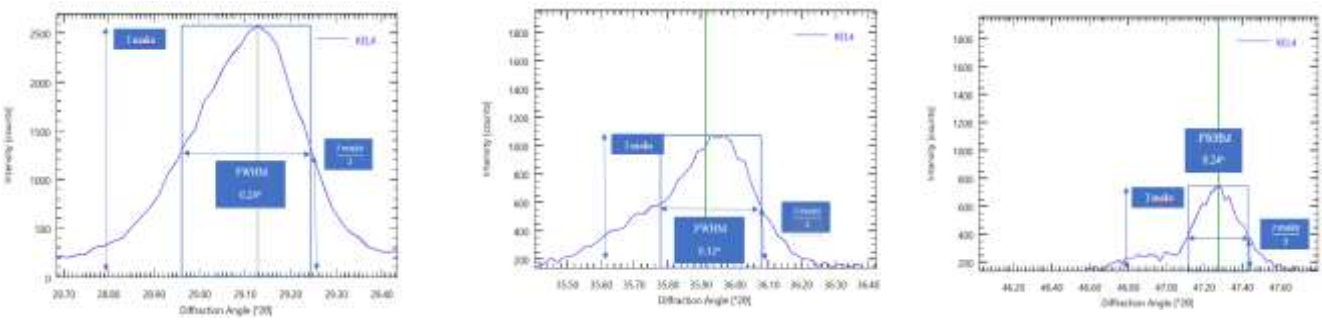

Figure 8. Largest peak by crystal plane [111][112][330]

However, the XRD patterns across all samples whether with or without carbon addition showed no significant changes in crystalline phase. This lack of transformation is likely due to the absence of a vacuum or inert atmosphere during the heating process. The carbon added as a reducing agent is assumed to have combusted rapidly under atmospheric conditions, preventing any effective reduction of $CaCO_3$. Similar results were reported by Huang et al. (2022), who emphasized the need for controlled atmospheres, such as argon or nitrogen, to facilitate reduction and prevent premature oxidation of reducing agents during high-temperature processing of carbonate materials.

Although the crystal structure remained unchanged, notable changes were observed in physical properties. As the percentage of activated carbon increased, dry shrinkage and water absorption decreased, while burn shrinkage, density, and fracture strength improved. These improvements are attributed not to the chemical action of the carbon since it was likely burned off but to the effects of thermal densification. Elevated temperatures can lead to improved particle packing, reduced porosity, and stronger interparticle bonds, ultimately enhancing mechanical properties. This observation aligns with the findings of Kumar et al. (2023), who demonstrated that sintering biomineral-based materials at high temperatures enhances mechanical strength and density, even when no phase transformation occurs.

Interestingly, despite the carbon burning off during the process, its presence before combustion may have contributed to temporary changes in heat distribution or acted as a transient filler, influencing microstructural evolution. A study by Lee et al. (2021) on the use of organic additives in $CaCO_3$-based composites also noted that temporary carbon-based materials can improve the

final compactness of ceramics, even if they are fully eliminated during firing. In summary, the results of this study are consistent with recent literature showing that structural transformation of $CaCO_3$ requires more controlled conditions than were applied here. Nevertheless, physical enhancements in the material were achieved, likely due to thermal effects rather than chemical or crystallographic modifications. Future studies should consider implementing vacuum or inert-atmosphere processing to allow activated carbon to function effectively as a reducing agent, enabling a more direct impact on the crystal structure and chemical composition of $CaCO_3$-based biomaterials.

Conclusion

Based on XRF results the Ca content is 95.603%. The dry shrinkage value and water absorption are further decreased by mixing 3%, 5% and 10% with heating temperatures of 900^{o}C, 1000^{o}C, 1100^{o}C and 1200^{o}C. The Burn Shrinkage value, density and fracture strength are increased by a mixture of 3%, 5% and 10% at 900^{o}C, 1000^{o}C, 1100^{o}C and 1200^{o}C. The crystal structure formed by the mixing of 3%, 5% and 10% at temperatures of 900^{o}C, 1000^{o}C, 1100^{o}C and 1200^{o}C is the result of *MATCH entry number* 96-901-5692 of the hexagonal crystal system, *space group* R-3C and cell units a=b= 4.9771Å and c = 17.3369Å.

Acknowledgments

Thank you to all parties who have supported and assisted me throughout the research process, especially my supervisors, laboratory staff, and my family for their guidance and encouragement.

Author Contributions

Conceptualization, M.C.F, A.W and B.S methodology, M.C.F, A.W and B.S.; software, M.C.F, A.W and B.S; validation, X M.C.F, A.W and B.S.; formal analysis, M.C.F, A.W and B.S; investigation, M.C.F, A.W and B.S.; resources, M.C.F, A.W and B.S.; data curation, M.C.F, A.W and B.S.; writing—original draft preparation, M.C.F, A.W and B.S.; writing—review and editing, M.C.F, A.W and B.S.; visualization, M.C.F, A.W and B.S.; supervision, M.C.F, A.W and B.S.; project administration, M.C.F, A.W and B.S.; funding acquisition, M.C.F, A.W and B.S. All authors have read and agreed to the published version of the manuscript.

Funding

This research received no external funding.

Conflicts of Interest

The authors declare no conflict of interest.

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