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PREPARED EGGSHELL-DERIVED HYDROXYAPATITE FOR BIOMEDICAL APPLICATIONS

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ABSTRACT

In this research, an interesting use of eggshells to create a product with added value, such as nanoscale hydroxyapatite (HA) is investigated. HA must have the appropriate nanoscale characteristics, such as crystallinity, particle size, shape, surface area, porous nature, and so on, because they contain calcium carbonate and biologically required trace elements such as Mg, Si, and others for many biological applications. The same effect can be obtained by adjusting the reaction parameters, selecting the appropriate synthesis mode, and employing organic modifiers. Eggshell bio-waste was used as the calcium source in this study to create hydroxyapatite powder. We investigated its characteristics for application in manufacturing a functionally graded material (NiTi/HA) utilized in bone culture. The powder was tested using XRD, FTIR, SEM, and a particle analyses. Whereas XRD revealed the structure of the resulting powder, which is comparable to the conventional form of HAP powder, FTIR revealed the presence of the primary compound Hydroxyapatite in producing powder, such as ($PO4^{3-}$, CO_2^{2-} and bending OH−). As a result of this research, we can conclude that the synthetic eggshell produced HA has good particle size, bioactivity, and porous nature.

Keywords: hydroxyapatite, eggshells, calculations, biomedical, calcium, natural, phosphoric acid.

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1. INTRODUCTION

The inorganic mineral hydroxyapatite, often known as HAP $Ca_{10}(PO_4)_6(OH)_2$, makes up about 70% of bone. Natural bone contains HAp crystals that are in the nano range in size. Calcium and phosphorus comprise most of the essential components of hydroxyapatite, with a calcium-to-phosphate proportion of 1.667. In this chemical synthesis process, hydrogen ions are removed at extreme temperatures. However, various methods were utilized to examine how to create these nanocrystals. More research is still needed to fully understand how to regulate these substances' crystallinity, shape, and size [1, 2], Hydroxyapatite (HAP) is a perfect material for orthopedic and dental implantation as well as the components of implantation due to its excellent biocompatibility with soft tissues like gums, muscles, and skin. Widespread applications for synthetic HAP in the repair of hard tissues include bone healing, bone boost, covering implantation, functioning as fillers in teeth or bone. On the other hand, traditional H.A.P ceramics' poor mechanical strength often limits their employment to light loadbearing applications. Recent advancements in nanotechnology and nanoscience have rekindled studies nanoscale Hydroxyapatite formation in order to precisely describe the small-scale features of Hydroxyapatite [3]. Hydroxyapatite is the most extensively utilized biomaterial for repairing and reconstructing flaws in bone tissue. It has the defining characteristics of biomaterials, including traits that make it non-inflammatory, non-toxic, osteoconductive, bioactive, biocompatible, and immune-suppressive [4]. (HAP) could be produced from biogenic materials, as shown in Figure-1, like fish bones [5], eggshells [6, 7], seashells [8], and coral [9] by the use of several chemical synthesis processes. These are based on reactions that take place in a solid state[10].

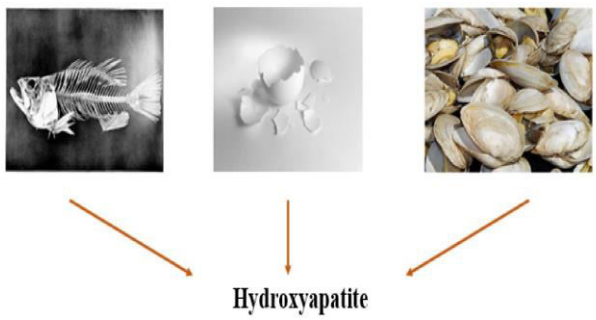

Figure-1. HAP natural sources [11].

Academics are interested in using eggshells to create products with significant added value, like nano-crystalline hydroxyapatite (HA). Calcium phosphate and other elements like Si, mg, and others that are essential for physiology are also found in Eggshells. HA must have the proper nanoscale properties to be helpful in a wide range of biological situations. These characteristics include, among others, their mesoporous nature, surface area, shape, and crystallinity. The formation of eggshell-derived Hydroxyapatite using a variety of organic modifiers in a microwave reactor specifically designed for the work appears to enable the instantaneous production of precursor materials with appropriate nanoscale properties for tissue engineering scaffolds, drug/protein delivery carriers, bone fillers, and other related applications [12].

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2. EXPERIMENTAL PART

Eggshells and phosphoric acid were the experimental starting materials used in this section. The following procedures have been carried out to create HAP powders from eggshells:

- To achieve proper cleaning, eggshells were mechanically cleansed. A toothbrush and purified water are required for cleaning.
- Following meticulous cleaning, the shells were allowed to dry in the open air (for at least twodays).
- The egg shells were sintered (calcined) at 1000 ℃ for three hours using an oven.
- The calcination process has two parts: the first lasts 30 minutes, when almost all organic materials burn, giving off a bad smell and the second is when eggshells turn into calcium oxide.
- Egg shells that had been calcined were crushed and then powdered in a steel kitchen mill.
- Calcium phosphate powder was made by adding phosphoric acid in a 1:1 weight ratio. The powder underwent an exothermic reaction while being stirred constantly with a glass spoon.
- The mixtures were ground in a planetary ball mill at 45 rpm for 22 hours to make them uniform and stop calcined particles from sticking together.
- After milling, the powder was heated in the calcination furnace for two hours at 1000 degrees Celsius in an air atmosphere.
- I passed the resulting powder through a 53μm diameter sieve after grinding.
- The powder produced was HAP.

Figure-2 shows how the experimental process of calcination was used to make HAP from egg shells. Figure-3 depicts the apparatus and instruments used to prepare H.A.P powder from egg shells using the calcination process.

A B C

Figure-2. Depicts the process of producing HAP powder from eggshells. Egg shells were cleaned in A, broken and calcined in B, and crushed and milled in C.

A B C D

Figure-3. Shows the apparatus used to make H.A.P powder from Eggshells: (a) Furnace; (b) Planetary ball; (c) steel kitchen mill; (d) Sieve.

3. RESULTS AND DISCUSSIONS

3.1 X-Ray Diffraction Result

(XRD) is employed to describe solid compounds' quantitative and qualitative characteristics and examine the crystallization of compounds and their phase purity [13].

The relationship that is used:

$$n\lambda = 2d\sin\theta \quad \text{.................. (1)}$$

Where:

N : is a positive integer.

Λ :wavelength,d:interplanar spacing, θ: Bragg's angle.

Bragg's law is used to calculate crystallite size from X-ray diffraction data [14, 15].

Identification of HAP depends significantly on the location, form, width, and strength of the HAP peak in the XRD spectrum. To match the resulting XRD spectrum to the recognized standard pattern analysis, the examination of the HAP, made from powdered egg shells, using X-ray diffraction (XRD) is shown in Figure-4 for the 10o to 80o diffracted angle range. When the peaks are compared to those on (JCPDS) card No., it demonstrates the high purity of the hydroxyapatite phase (09-0432). These outcomes showed raw egg shells' effectiveness in producing H.A.P powder

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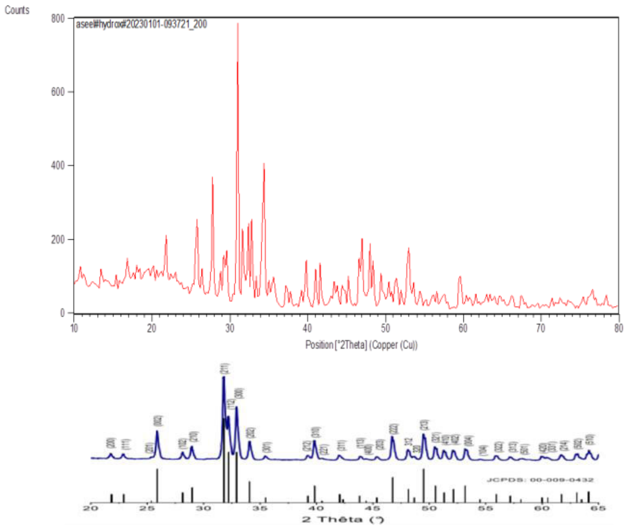

Figure-4. XRD spectrum for H.A.P Egg shells and pure HAP.

3.2 Scanning Electron Microscope (SEM)

A scanning electron microscope was used to examine the surface morphology of hydroxyapatite (SEM). Figure-5depicts SEM images of agglomerated Hydroxyapatite particles. The images also demonstrate the porous nature of the synthetic hydroxyapatite. When used in implants, this porous property is desirable and advantageous because it facilitates interaction between the implant and the biological environment.

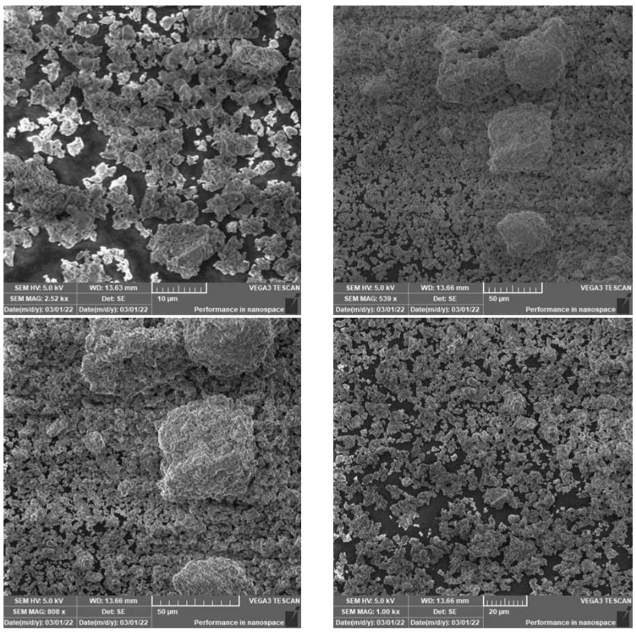

Figure-5. SEM micrographs of HAP of egg shells.

3.3 Analysis of Ftir Spectroscopy

Fourier-transform infrared spectroscopy can identify the amide, phosphate, and carbonate sets that make up the final powder and confirm the production of HAP. According to Fourier-transform infrared spectroscopy formal analysis, transmission mode, the carbonate set is present at about (1410-1450) cm^{-1}, (875) cm^{-1}, and the H-O group is currently at approximately (3500-3200) cm^{-1}. For the P-O set, (10) (1049-1090) cm^{-1}, 1950-2200 cm^{-1}, (962) cm^{-1}, and (560) cm^{-1} are used [13]. As shown in table 1, the energy beams at (3572.29) cm^{-1} and (3643.65) cm^{-1} depict OH-, (1415.80) cm^{-1} represents the amide set of CO3, and (1043.52) cm^{-1}, (972.16) cm^{-1}, (605.67) cm^{-1}, and (551.66) cm^{-1} depict the beams for the PO_4 set. Figure-6 illustrates the constituents of the powder produced by the thermal calcining of eggshells. FT-IR analysis and comparison with reference spectra showed that they were the active sets for HAP powders [16]

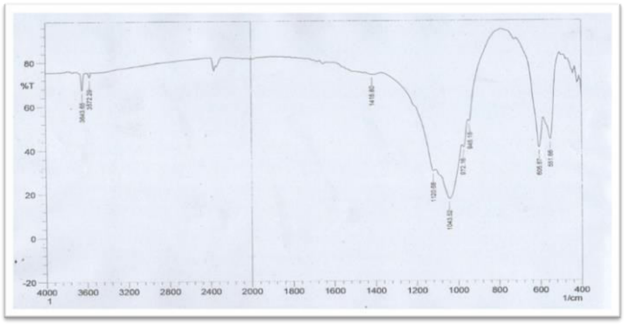

Figure-6. HAP from Egg shells FT-IR spectrum.

Table-1. Shows the significant FT-IR stretching of HAP frequencies from the eggshell.

Bands of Infrared Absorption (cm^{-1})	Description
1043.52	(PO_4^{3-})
972.16	(PO_4^{3-})
605.67	(PO_4^{3-})
551.66	(PO_4^{3-})
1415.80	(CO_3^{2-})
3643.65	OH Bending
3572.29	OH Bending

3.4 Analysis of Particle Size

Figure-7 depicts micro and nano-HAP particle distribution from phosphoric acid and calcined egg shells. The large surface area of the granules led to a high accumulation, As a result, distilled water was used as the screening medium in the absence of dispersants. The size of the particles ranges from 0.301 to 4.759 µm, with an average size of (1.088) µm.

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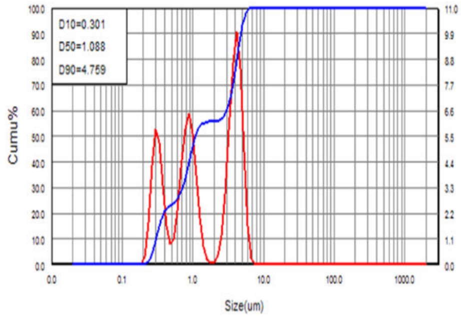

Diam um	Percent
0.100	0.00
0.200	0.06
0.500	23.29
1.000	46.57
2.000	55.78
5.000	93.42
10.000	100.00
20.000	100.00
45.000	100.00
75.000	100.00

Figure-7. Particle size analyses of the HAP/Egg shells.

4. CONCLUSIONS

The results of this study demonstrated that eggshells could be used as a natural source for producing HAP. A detailed structural analysis of HAP produced by a calcining technique and milling by a planetary ball after mixing with phosphoric acid is investigated using the eggshell powder that has been prepared. Using (FT-IR) and other techniques, the powder was described by (XRD). XRD was able to explain the structure of the resulting powder and demonstrate how successfully raw egg shells production Hydroxyapatiteand comparing the result peaks of the XRD pattern with standard Hydroxyapatite. The compounds excitable in pure HAP are present in the eggshell powder that is produced, according to FT-IR analysis. The particle size was also established, with ranges of (0.301 to 4.759) µm and (1.088) µm. But the HA derived from eggshells will undoubtedly be a reasonably priced bio-ceramic material for biomedical purposes. Using egg shells will also be a successful material recycling solution for trash management

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