

ANALYSIS OF DUCK EGGSHELLS AS HYDROXYAPATITE WITH HEAT TREATMENT METHOD

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Abstract

Duck eggshell contains very high calcium, making it a raw material for hydroxyapatite (HA) biomedical applications. The aim of this study was to synthesize natural HA from duck eggshell waste using cleaning, drying, smoothing with a smoothness level of passing is 200 meshes and heat treatment with temperature variations of 850 °C, 900 °C, and 950 °C for one hour. The resulting HA material was characterized using X-ray fluorescence (XRF), X-ray diffraction (XRD) and Fourier transform infrared spectroscopy (FTIR). The results of the physical characteristics of raw duck eggshells without heat treatment are white, slightly bluish in color, the smell and level of smoothness is smooth. However, heated powdered duck eggshells which are heated 950 °C are whiter than the samples which are heated at 850 °C and 900 °C, they have no odor and their smoothness is the smoothest. The small peaks of HA from the non-heat duck eggshell powder were 64.89 % with a CaCO₃ of 23.50 %, then the peak of CaO increased along with the increasing of heat treatment temperature of 900 °C that result HA in 69.31 % and reduce CaCO₃ in 19.44 %. Duck egg shell powder achieved the highest diffraction with the increasing formation of HA at a heat 950 °C by 91.87 % with a very significant decrease of CaCO₃ in 2.25 %. The XRF test results of duck eggshell powder showed that the chemical composition of CaO was 99.50 %, with elemental calcium (Ca) of 99.71 %. The FTIR test results on mesh 200 observed a reduction in the intensity of CaCO₃ absorption, while the intensity of CaO at 364.55 cm⁻¹ was getting stronger, which indicated the formation of CaO at 950 °C. This showed that the CaO formation at a temperature of 950 °C was much more than the formation at a lower temperature. The deformation from the CaO to HA phase was close to perfect with 91.87 % hydroxylapatite.

Keywords: duck eggshell, hydroxyapatite, mesh, calcium carbonate, calcium oxide, heat treatment.

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1. Introduction

The increasing need for ceramic materials in the medical world, especially in dentistry, encourages new findings as an alternative to the use of new materials because denture materials are imported and expensive. Bioceramic is a well-known implant material widely used for repair, replacement, bone and teeth [1, 2]. In addition to ceramics, other materials urgently needed for the reconstruction of decayed, damaged teeth are polymers, metals and composites [3].

Hydroxyapatite (HA) is highly determined by particle size, morphology, crystallinity and composition [4]. However, the particular weakness of hydroxyapatite lies in its poor mechanical properties, especially fracture toughness, which limits its use for non-load bearing applications [5].

Hydroxyapatite (HA) is the main mineral constituent of vertebrate bones and teeth. It has been well documented that HA nanoparticles can significantly increase the biocompatibility and bioactivity of artificial biomaterials [6, 7] and provide support and protection for soft tissues and organs [8]. Hydroxyapatite compounds can be extracted by different techniques such as dry methods

including solid-state methods and chemical mechanics, wet methods including chemical and hydrothermal precipitation, and hydrolysis and high-temperature methods including combustion or heat treatment and pyrolysis [6]. Hydroxyapatite powder is derived from natural materials such as beef bones [7], oyster shells [8], corals [9], fish bones [10], eggshells [11], blood clamshells [12], crab shells [13]. One material that has the potential as a bioceramic material that is very easy to obtain is eggshells [14]. Eggshells have very high calcium, which can be used to develop medical science technology, especially in dentistry [15]. The eggshell consists of a network of protein fibers, linked to crystals of calcium carbonate (CaCO_3) 96 %, magnesium carbonate (MgCO_3) 1 % and calcium phosphate ($\text{Ca}_3(\text{PO}_4)_2$) 1 %, as well as organic matter and water [2, 16, 17]. CaCO_3 , the main constituent of eggshells (96 %), is a naturally occurring amorphous crystal in the form of calcite (hexagonal crystals). Calcium carbonate (CaCO_3) contained in the egg shell by 94 % causes the egg shell to become a potential material in the synthesis of hydroxyapatite then will become a medical application material, such as dental implant material, bone formation and bone replacement [18].

Compound calcium carbonate (CaCO_3) where in everyday life egg shells are abundant and thrown away as waste. Duck egg shells are expected to be a source of biocompatible and bioactive hydroxyapatite for medical applications [19].

In general, eggshells are considered to have no economic value. Even though egg shells have a high calcium content, they can be used as natural hydroxyapatite ingredients [19]. Calcium carbonate is the main component of eggshells that can be used in various applications: dielectric material, gypsum, dental and bone implants, catalysts, and fillers used in multiple industries such as rubber, ink, foodstuffs, cosmetics, pharmaceuticals, and fertilizers [20]. The use of chicken eggshells as a substitute for lime in concrete can provide benefits such as reducing the use of cement, preserving natural lime, and utilizing eggshell waste rich in calcium carbonate (CaCO_3).

Content very high calcium from shell egg duck temporary shell egg duck this abundant all around us and be waste. Alternative solution in utilization waste egg this so that worth economical with process content extract calcium in it as ingredient synthesis hydroxyapatite (HA). Other uses of waste shell egg duck this is environmentally friendly rehabilitation, as well as the development of alternative uses of ceramic biomaterials with method nanoparticles.

Hydroxyapatite (HA) is formed from element main calcium and phosphorus with the formula $\text{Ca}_{10}(\text{PO}_4)_6(\text{OH})_2$. Hydroxyapatite (HA) is a bioceramic material that is a raw material for biomedical applications specifically implant bones and teeth. Research results this can used potency development of ceramic biomaterials method nanoparticles based on medical.

Bone and dental implant bioceramic material technology must character biocompatible where is this material can be accepted by the body in application the medical so needed – technology making hydroxyapatite which results can approach specification hydroxyapatite . Study this use expected heat treatment method produce hydroxyapatite close to or in accordance specification hydroxyapatite.

Potential development of special ceramic biomaterials In meeting the need for bone and dental implants, it is necessary to make new breakthroughs because the number of cases of bone surgery and tooth replacement and tooth decay is quite high, especially in terms of the development of bioceramics in dentistry. The number of cases of orthopedic surgery and tooth replacement is quite high [1, 19]. Thus the need for bioceramics is a recognized implant material and is widely used for repair, replacement, bone and teeth [18].

The use of hydroxyapatite in the medical field in particular Bones and teeth are still being developed through various studies. The uses include: regeneration of bone tissue, fillers in GIC restoration materials, fillers in root canal filling cement, active ingredients for sensitive toothpastes, dental implant coatings. Utilization of duck eggshell waste as one of the breakthroughs and alternative paths in the development of dental biomaterials in biomedical applications. Studies and tests were carried out to determine which hydroxyapatite biomaterial meets the standards by approaching the specifications for a good hydroxyapatite as a denture fabrication material [5].

Several studies have been carried out by researchers and have informed that duck egg shells are potential as dental bioceramic materials. From the research that has been done using duck egg shell characterization technology with the calcination method and a series of chemical tests, (XRD test, XRD test, FTIR test) in getting good hydroxyapatite (HA) and specifications close to standard [17, 19, 20].

Various study has proven addition hydroxyapatite impact significant and take effect good to nature mechanical and biological to the reinforced material, so that said use hydroxyapatite in application biomedical no cause loss or make influence bad at implementation biomedical.

Study more carry on needed for test effectiveness this is below condition clinical in long term as well study about various method synthesis hydroxyapatite nano-sized which will produce optimal properties for application biomedical.

Result of study this that is, get high hydroxyapatite (HA) with a CaO content of 99.50 % of the sintering process with temperature calcination optimal on temperature 950 °C. Process heat treatment with variation temperatures of 850 °C, 900 °C and 950 °C produced pure hydroxyapatite (HA) from CaO as big as 91.87 % at a temperature of 950 °C.

From result study this is what shows that shell egg duck could synthesized for produce hydroxyapatite which can applied ingredient raw biomedical and application in industrial scale.

2. Materials and method

2. 1. Materials

This research used the following stages.

Duck eggshells were processed firstly by cleaning and removing the membranes on the eggshells, then washed and rinsed with Aquades (distilled water), then doused with hot water for 30 minutes at a temperature of 100 °C, and then dried for three days. After drying, the eggshells were crushed and mashed using a blender. A mortar was used to obtain a more delicate powder, then sieved using a 200 mesh sieve. The duck eggshell powder was calcined in a Nabertherm L9/11/SKM electric oven at 850 °C, 900 °C, and 950 °C for an hour. The duck eggshell powder that had been heat-treated was then tested for XRF, XRD, and FTIR.

2. 2. Test Method

2. 2. 1. X-Ray Diffraction Test (XRD)

The crystalline phase of the synthesized powder and analyzed by X-ray diffraction (XRD) was carried out for structural identification of pure duck eggshell powder. XRD patterns were recorded with a Japanese Shimadzu 7000, creating an X-ray diffractometer. The material characterization used CuK radiation and had CuK incident radiation ($= 1.5405 \text{ \AA}$). The phases were identified by comparing the experimental X-ray diffractogram with the standards prepared by the Joint Committee on Powder Diffraction Standards (JCPDS).

2. 2. 2. X-Ray Fluorescence Spectrometry (XRF)

Elemental analysis of hydroxyapatite powder or raw materials from duck eggshell powder was analyzed and synthesized by X-Ray Fluorescence Spectrometry (XRF Thermoscientific). The presence of heavy metals was carried out by inductively coupled atomic plasma emission spectrometry analysis.

2. 2. 3. Fourier Transform Infrared Spectroscopy (FTIR)

Fourier transform infrared spectroscopy (FTIR: Shimadzu type 21). The spectra of the powdered samples were obtained in the wavenumber range of 500–4000 cm^{-1} . Powder compositions were synthesized and analyzed using inductively coupled plasma atomic emission spectroscopy.

3. Results

3. 1. Characteristics of Duck Eggshell Powder Before and After Burning

Heat treatment on duck egg shell powder affects the colour, smell and fineness which explains that the higher the heating temperature, the whiter the colour, which indicates that the content of calcium carbonate (CaCO_3) compounds will deform more completely to calcium oxide compounds will deform more completely to calcium oxide compounds (CaO) so that the formation of more hydroxyapatite nanoparticles are formed, this can be read in **Table 2**. The smell is getting less and less and the fineness is getting better. The overall characteristic test results are shown in **Table 1**.

Table 1
Physical Characteristics of Duck Eggshells Before and After Burning

No.	Code	Physical Characteristics		
		Colour	Smell	Fineness
1	M2	Bluish white	Stink	Fine
2	M2 850	Light gray	Stink	Finer
3	M2 900	White	Not stink	Finest
4	M2 950	White	Not stink	Finest

Table 1 figures out the results of the physical characteristics test (colour, smell, fineness) of duck egg shells show that the higher the heat treatment temperature, the whiter the color, not stink and finest.

3. 2. Characterization of X-Ray Duck Eggshell Powder (XRD)

XRD characterization was used to determine the phase of the compound and the crystallinity of the sample along with the increase in temperature, all samples of duck egg shell powder showed the dominance of the bonding phase of the hydroxyapatite crystalline compound and the highest purity values, the peaks formed were consistent, this is consistent with **Fig. 2**. The XRD diffractography pattern of duck egg shells in **Fig. 1**.

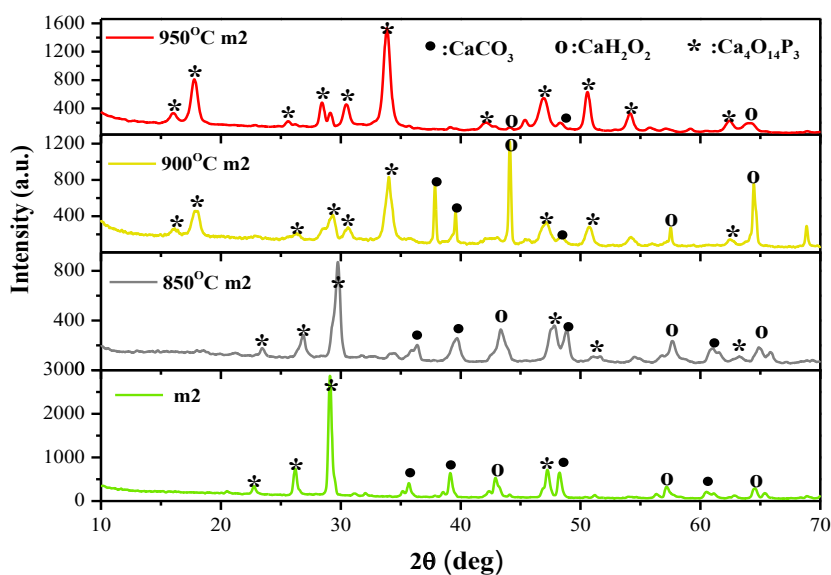


Fig. 1. Diffractogram patterns of the duck eggshells at mesh 200 (M2) with temperature variations of 850 °C, 900 °C, and 950 °C

The composition of compounds in a mesh sample of 200 duck eggshells variation temperature detail based on the XRD test results can be seen in the following **Table 2**.

Table 2
XRD mesh 200 (M2) test results on duck eggshells

Code	Hydroxylapatite ($\text{Ca}_4\text{O}_{14}\text{P}_3$) %	Calcium Hydroxide Portlandite (CaH_2O_2) %	Calcium Carbonate (CaCO_3) %
M2	64.89	11.61	23.50
M2 850	56.51	19.84	23.65
M2 900	69.31	11.24	19.44
M2 950	91.87	5.88	2.25

The Relationship of XRD Diffractogram Results with duck egg shells Mesh 200 Temperature Variation.

Fig. 2 shows the relationship between the results of the XRD diffractography test of mesh 200 duck egg shells with variation in temperature.

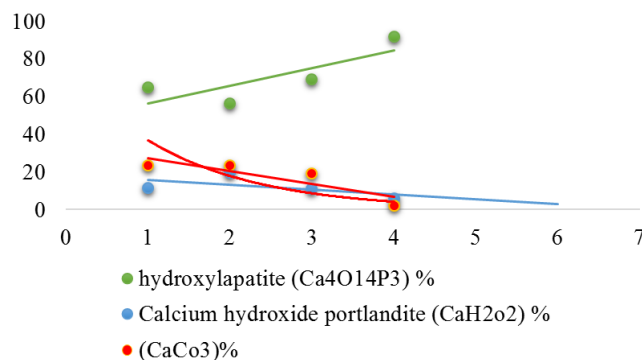


Fig. 2. XRD Test of Duck Eggshell Powder on Mesh 200

The higher the heat treatment temperature on the 200 mesh duck egg shells, the formation of the hydroxylapatite (Ca₄O₁₄P₃) phase is shown to be more significant compared to the phases of Calcium Portlandite (CaH₂O₂) and Calcium Carbonate (CaCO₃) compounds as shown in **Fig. 2**.

3. 3. Test Results Using X-Ray Fluorescence (XRF) Spectroscopy

Table 3 shows the chemical composition of raw duck egg shells and duck egg shells that are heat treated with temperature variations.

The chemical composition of raw duck egg shells and has been heat treated with temperature variation is shown in **Table 4**.

XRF test results are shown in **Table 4**. This showed the highest content of calcium oxide (CaO) in duck egg shells at 99.50 %.

Table 3

Test Results Using X-Ray Fluorescence (XRF) Spectroscopy Mesh 200

Temperature (°C)	Time (Minute)	Composition Chemical Compound (%)									
		Ca	Si	Mg	Px	Cr	Nb	Mo	Zn	Hf	W
Non Heat Treatment	–	99.71	–	–	0.246	0.0286	0.0063	–	–	–	–
850	60	93.16	1.23	5.24	0.338	–	0.338	0.0095	–	–	–
900	60	69.48	–	29.71	0.56	0.068	0.0055	–	0.0608	0.048	0.043
950	60	90.96	–	8.81	0.209	–	0.0061	–	–	–	–

Table 4

Test Results Using (XRF) Chemical Oxide Compounds on Mesh 200

Temperature (°C)	Time (Minute)	Composition Chemical Oxidation Compound (%)												
		CaO	P ₂ O ₅	Cr ₂ O ₃	MgO	SiO ₂	MoO ₃	Nb ₂ O ₅	ZnO	HfO ₂	WO ₃	SnO ₂	In ₂ O ₃	TeO ₂
Non Heat Treatment	–	99.50	0.451	0.02	–	–	–	–	–	–	–	–	–	
850	60	90.22	0.607	–	90.2	2.12	0.009	0.0090	–	–	–	–	–	
900	60	60.31	0.90	0.05	38.6	–	–	–	0.039	0.03	0.03	0.009	0.008	0.0070
950	60	89.63	0.37	–	9.99	–	–	0.0052	–	–	–	–	–	

3. 4. Chemical bond test results using Fourier Transform Infra-Red (FTIR) spectroscopy

The FTIR (Fourier Transform Infra-Red) characterization aimed to determine the chemical bonds present in the sample. This analysis was based on the analysis of the wavelength and

characteristic peaks of the sample. The wavelength indicated specific functional groups in the sample because each functional group has a distinct characteristic peak.

The FTIR spectrum of raw and heat treated duck egg shell samples with temperature variations is shown in **Fig. 3**.

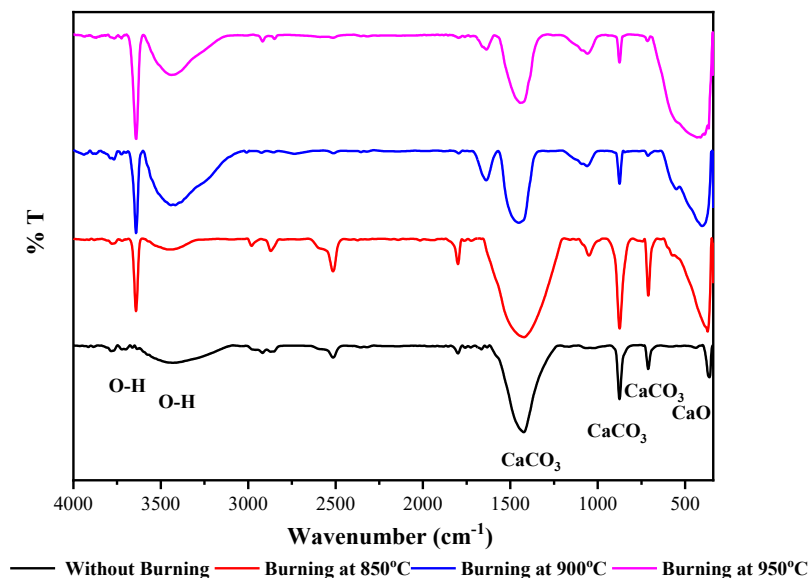


Fig. 3. FTIR results of duck eggshells on mesh 200: *a* – without burning; *b* – burning at 850 °C; *c* – burning at 900 °C; *d* – and burning at 950 °C

Fig. 3 indicates that the higher the heat treatment temperature, the higher the CaCO_3 deforms to CaO , as evidenced by the peak formed from the FTIR spectrum which is getting sharper and higher and there is a change from the amorphous phase to the crystalline phase.

4. Discussion

The results of **Table 1**, showed that the physical characteristic (colour, smell, fineness) of raw duck egg shells and their calcined products from 850 °C to 900 °C to 950 °C for 1 hour. A higher calcination temperature produces the samples which have a whiter [19].

The results of the XRD diffractogram of duck egg shells in **Fig. 1**, showed that the diffraction peak observed at the diffractogram, hydroxyapatite ($\text{Ca}_4\text{O}_{14}\text{P}_3$), was at the diffraction peak at an angle of 25.98° with a hkl plane (201), an angle of 26.5° to the hkl plane (002), an angle of 27.7° to the hkl plane (102), an angle of 29.3° to the hkl plane (210), an angle of 34.24° to the hkl plane (202), an angle of 44° to the hkl plane (113), an angle of 47.24° to the hkl plane (222), an angle of 50.1° to the hkl plane (321), an angle of 54.2° to the hkl plane (104), and an angle of 62.4° to the hkl plane (502), which corresponded to JCPDS Card No. 00-009-0432. Meanwhile, calcium carbonate (CaCO_3) was observed at a diffraction peak at an angle of 39.5° to the hkl plane (113), an angle of 48.4° to the hkl plane (016), and an angle of 59.8° to the hkl plane (122), which corresponded to JCPDS Card No.00-047-1743. Based on the comparison of the diffractogram patterns, it was concluded that the hydroxyapatite ($\text{Ca}_4\text{O}_{14}\text{P}_3$) phase dominated the sample composition and showed that the majority of the peaks formed were hydroxyapatite, according to the JCPDS data.

Previous studies have described the decomposition of CaCO_3 to CaO at low calcination temperatures for an hour, between 830 °C and 850 °C for an hour, 900 °C for an hour. The product was synthesized from powder C and DCPD heat treatment at different temperatures 800 °C for an hour [5]. Chemical composition (CaO) of chicken eggshells whose calcination temperatures were 700 °C, 800 °C, 850 °C, 900 °C and 1000 °C for an hour each, reaching the most substantial peak of CaO (HA), increasing at a calcination temperature of 850 °C to 900 °C, and decreasing at

a calcination temperature of 1000 °C so that the egg calcination temperature was set at 900 °C [5]. The product synthesized from powder C and heat-treated DCPD at temperatures (800 °C, 900 °C, 100 °C, 1100 °C) for three hours showed the highest peaks of CaO with HA at 1100 °C [2].

In this research, the resulting XRD diffraction pattern is shown in **Fig. 1**. Uncalcined and calcined raw duck eggshell powder at temperatures of 850 °C, 900 °C, and 950 °C for an hour each shows the formation of a crystalline phase from CaCO₃ to Ca₄O₁₄P₃ was significant and different. The small peak of HA from uncalcined duck eggshell powder was 64.89 %, with CaCO₃ of 23.50 %. The peak of CaO increased with increasing calcination temperature from 900 °C resulting in HA of 69.31 % reduced CaCO₃ of 19.44 %, and the highest peak with increasing HA formation at a calcination temperature of 950 °C was 91.87 %, with a very significant decrease in CaCO₃ of 2.25 %.

The phase transformation from calcium carbonate (CaCO₃) to calcium oxide (CaO) occurred at 930.4 °C [20]. The composition of compounds in a mesh sample of 200 duck eggshells in detail based on the XRD test results can be seen in the **Table 2**.

The results of **Fig. 2**, showed that the formation of the binding phase of the hydroxyapatite compound along with the increase in temperature from the phase change from calcium carbonate (CaCO₃) to hydroxyapatite (Ca₄O₁₄P₃) was higher, although at 850 °C the temperature was slightly different, indicating that the value of hydroxyapatite decreased due to air humidity (high content of H₂O) during the sample combustion process so that the Calcium (Ca) and Carbon (C) elements (catenuation elements) ranged and efficiently bind to H₂O compounds to form high CaH₂O₂ and CaCO₃, and there was still an impurity phase which resulted in the degradation of CaCO₃ bonds to CaO not optimal or perfect [5]. This was following the results of the FTIR test.

The results of **Table 3**, showed the XRF test results of duck eggshell powder on Mesh 200 without burning, temperatures of 850 °C, 900 °C, and 950 °C for an hour of burning as shown in **Table 3** indicated that the elements of Ca, Si, Mg, and Px were abundant and dominant in the duck eggshell powder, which found that the calcium contents: (Ca) M2 = 99.71 %, M2 850 = 93.16 %, silica (Si) = 1.23 %, (Mg) = 5.24, M2 900 = 69.48 %, (Mg) = 29.76 %, (Px) = 0.56, M2950 = 90.96 %, and (Mg) = 8.81 %.

The results of **Table 4**, showed the XRF test results of duck eggshell powder on 200 mesh that received heat treatment and those that did not. P₂O₅ and MgO, the chemical composition of the CaO oxide did not appear to change significantly or not much different for each temperature change. The lowest CaO content was in the sample at 950 °C. This was influenced by the burning temperature, minerals, or compounds. In addition, there were also new compounds with short compositions that also affected changes in the chemical composition of CaO after burning. The results of previous research obtained the chemical composition (CaO) of chicken eggshells at 96.64 % [16]. Another research found the chemical composition (CaO) of chicken eggshells at 98.124 % and duck eggshells at 98.925 % [17]. This research found that the chemical composition (CaO) was higher at 99.50 %, with calcium (Ca) at 99.71 %.

FTIR spectrum of the duck eggshells, before and after heat treatment at 850 °C, 900 °C, and 950 °C, generally showed five significant absorption bands, namely at 3431.36 cm⁻¹, 1423.47 cm⁻¹, 875.68 cm⁻¹, 711.73 cm⁻¹, and 360.69 cm⁻¹. Bands 875.08 cm⁻¹ and 1423.47 cm⁻¹ corresponded to the vibrations of the carbonate group at wavelengths of cm⁻¹ to 4000 cm⁻¹. At a temperature of 850 °C, a high concentration of CaCO₃ (wide broadband) at a wavenumber of 1500 cm⁻¹ causes high water-binding (H₂O) to form CaH₂O₂ with a widening of the absorption band at 3431.36 cm⁻¹. Due to heating, however, a sharp absorption band with strong intensity at 3641.6 cm⁻¹ appeared from O–H water. In accordance with previous researchers that calcination of 700–900 °C for an hour showed that 3643 cm⁻¹ band was strong (OH), 3435 cm⁻¹ (OH), 1630 cm⁻¹ was strong (C = O), and 500–580 cm⁻¹ (Ca–O) [20]. The absorption bands (*d*) at 1423.47 cm⁻¹, 875.68 cm⁻¹, and 711.73 cm⁻¹ originated from CaCO₃ (CO₂₂), while the absorption band at 360.69 cm⁻¹ came from bond stretching vibrations of CaO [21]. This indicated that the formation of CaO at a temperature of 950 °C was much higher than the formation of CaO at lower temperatures. However, not all CaCO₃ in this burning had been converted to CaO because the absorption band at 1438.9 cm⁻¹ from CaCO₃ was still of moderate intensity. Increasing the heating temperature of duck eggshell powder increased the formation of CaO from CaCO₃.

This research is still in the early stages which only looks at the characterization of the hydroxyapatite content of duck egg shells through physical and chemical tests (XRF, XRD, FTIR). The results show that the content of hydroxyapatite (hydroxylapatite) is very high at 91.87 % with elemental calcium (Ca) of 99.71 %.

It is necessary to carry out further research on duck egg shell research using various methods of synthesizing hydroxyapatite-sized nanoparticles that will produce optimal properties for biomedical industrial applications, especially bones and teeth. This is evident from the ratio of Ca/P hydroxylapatite ($\text{Ca}_4\text{O}_{14}\text{P}_3$) obtained is still = 1.34, while the applicable standard is 1.66.

The results of this study are very important to encourage the local industry in the field of ceramic biomaterials related to medical bone and dental implant materials through the nanoparticle-generating (HA) method. This has to be done because so far the materials have been imported from abroad at high prices.

5. Conclusions

Research suggests that duck eggshell powder can be an alternative and a potential hydroxyapatite (HA) source for biomedical applications. XRF test results indicated that duck eggshell powder obtained a chemical composition of CaO of 99.50 % with an element of calcium (Ca) of 99.71 %.

The XRD test results of duck eggshell powder on mesh 200, HA of uncalcined duck eggshell powder was 64.89 % with CaCO_3 at 23.50 %. The peak of CaO increased along with the increase in calcination temperature from 900 °C, resulting in HA at 69.31 %, reduced CaCO_3 at 19.44 %, and the highest peak with the increasing formation of HA at 950 °C, calcination temperature of 91.87 %, with a very significant decrease in CaCO_3 at 2.25 %. The FTIR test results showed the rate of formation of CaO or HA in eggshell powder on mesh 200 was fast because the level of fineness was better, making it easier for the calcination process or heating of the sample so that the higher the heating temperature or calcination temperature, the faster the formation of CaO from CaCO_3 .

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