ANALYSIS OF PHYSICAL PROPERTIES AND COMPRESSIBILITY OF AVIAN EGGSHELL NANOPOWDERS IN SOLID STATE REACTION

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Abstract

Eggshell is bioceramic material that produces by avian that commonly contains of 94 % calcium carbonate, 1 % magnesium carbonate, 1 % calcium phosphate, and 4 % other organic element. This study proposed to investigate the synthesis and characterization of avian eggshell powders. The avian eggshell that used in this study involved chicken, duck, and quail eggshells. The characterization of avian eggshell nanopowder for reducing their grain size from micro to nano involved ball milling process (solid state reaction) with the variation of milling times (3, 5, and 7 hours) and sintering temperature at 1000 °C for 2 hours. X-Ray Diffraction (XRD) test presented the phase characterization of quail eggshell nanopowder which ball-milled for 7 hours, obtained the smallest crystallite size at 19.2 nm. Scanning Electron Microscopy (SEM) test presented the morphological analysis that showed changes in grain size and shape of each variety of the avian eggshell such as spherical, oval, wormlike, cubical, triangular, and some irregular grains. Energy Dispersive X-Ray (EDX) test presented the compound in avian eggshell powders that showed Ca and O level were the highest, while C was the lowest level. Fourier Transform Infrared (FTIR) test presented the possibility of the functional group of the avian eggshell powders that showed Ca-O, Ca=O groups, CaCO₃, asymmetric C-O, -CO₃, amide, C=O, -OH, alkyl CH, and C-H. While compressibility shown the increase along with the decrease of crystallite and particles size in cubical grain. The highest compression ratio is 67.75 % for chicken eggshell nano powder with 5 hours milling time at 2000 kgf of compression loading.

Keywords: Phase identification, compressibility, avian eggshell, solid state reaction, ball milling, milling time, morphology, molecular bonding, bioceramic, grain shape.

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1. Introduction

Avian eggshells are bioceramic materials that produced by avian, functioning to protect the egg from mechanical damage [1]. The eggshells are complex permeable bioceramic materials and very structured with large mixing both organic and non-organic phases that showed simple overlap between non-calcified and calcified eggshell membrane [2]. Eggshell contains about 8.5–11 % total mass of the egg, with thickness about 355 μm [3, 4]. Generally, eggshell arranged by 94 % calcium carbonate (CaCO₃), 1 % magnesium carbonate (MgCO₃), 1 % calcium phosphate (Ca₃(PO₄)₂), and 4 % organic matter, usually protein, collagen, and sulfated polysaccharides [3–6].

Utilizations of eggshell biological wastes are highly recommended in our society because of environmental and economic reasons. It is known that eggshell wastes contain valuable organic and inorganic component which can be used in commercial product with new value in this waste materials [5]. Especially in Indonesia, that has abundant eggshell wastes. This is because the amount of egg consumption by the Indonesian people is quite high, besides that the shell waste is also

produced from the remnants of the hatchery in the chicken nursery industry [7]. Another reason is because the egg production in Indonesia increases each year, for example, the production of quail egg in 2013 was 18,936 tons then increases to 25,272 tons in 2017 [8].

Eggshells itself have been used in various research such as catalyst for biodiesel and used as a binder [9]. The eggshell also used as filler and precursor for cement mortar in making of gypsum, even used as drug delivery in medical sector [10, 11]. For other applications, eggshells also used as reinforcement at cast metal alloy, even used as basic material to make hydroxyapatite which can be used as an implant material for bone and used as tooth filler [7, 12, 13]. In the form of CaCO_3 , many application has been discussed. One of them is the application for alkali-activated aluminosilicate binders [14]. It was revealed that the addition of CaCO_3 intensified the nano-structured formation processes in the direction of formation of Na-Ca zeolite-like phases and shortening the required time for cement stone to gain water resistance in normal conditions [15].

Based on these data, the objective of this research is to utilize eggshell waste into CaCO_3 powder in an easy and inexpensive way that can later be used in various applications. The synthesis that was carried out to obtain CaCO_3 from eggshells was by ball-milling process, therefore to get the best results, this study used variations in milling time and carried out characterization in the form of phase identification, morphology, functional groups, and compressibility values. The results of this characterization will be useful for the application of CaCO_3 as a tooth filler in dental implants.

2. Materials and methods

In this research the specimen that used are variation of dried avian eggshells which includes chicken, duck, and quail eggshell. The synthesis of avian eggshells done with ball mill method using planetary ball mill (MTI QM-3SP2), which intend to produce particles in nano-sized by mixing 300 g micro-sized of eggshell with 100 ml of acetone, to obtain the optimum result of final particle size [16]. The mixing was carried out with the variation of milling time, i. e. 3, 5, and 7 hours for each variety of the eggshell. After ball mill process, drying was done at temperature 110 °C for 1 hour [15]. The next process is crushing for 1 hour [8]. After crushing, sintering process has been done at temperature 1000 °C for 2 hours [7]. After sintering, do crushing for 1 hour [8]. The characterization process aims to identify phase, morphology, elemental composition, and functional groups using XRD (PanAnalytical E'xpert Pro), SEM-EDX (FEI Inspect-S50), and FTIR (Shimadzu IRPrestige21). For compressibility test, it used cold pressing by using universal testing machine (ILE IL-904) with load of 1000 kgf and 2000 kgf for 90 seconds [17].

Fig. 1 shows the samples after ball milling process for 7 hours that consist of sintering and non-sintering powder.

Fig. 1. Avian eggshells after ball mill process: *a* – chicken eggshell non-sintering; *b* – duck eggshell non-sintering; *c* – quail eggshell non-sintering; *d* – chicken eggshell after sintering; *e* – duck eggshell after sintering; *f* – quail eggshell after sintering

Fig. 1, a, b, and c are the chicken, duck, and quail eggshell without sintering process and crushed for 1 hours, respectively. **Fig. 1, d, e, f** are the chicken, duck, and quail eggshell with sintering process at 1000 °C for 2 hours in environmental condition and crushed for 1 hours.

3. Results

Naming of samples in this discussion using (CES) for chicken eggshell, (DES) for duck eggshell, and (QES) for quail eggshell with (M3H) for 3 hours of milling time, (M5H) for 5 hours of milling time, and (M7H) for 7 hours of milling time.

3. 1. Phase Identification

The results of XRD test were shown in **Fig. 2**. Intensity, FWHM, d-spacing, and crystallite size are shown in **Table 1**. The crystallite size was calculated using Scherrer (1) as [8, 15]:

$$d = \frac{k \cdot \lambda}{\beta \cos \theta} \quad (1)$$

Where d is the diameter of crystallite, K is the constant (0.89–0.9), λ is the wavelength (1.5406 Å), and β is Full-Width Half Maximum (FWHM).

Fig. 2. Diffractogram graph of avian eggshell nanopowders

XRD test results were analyzed using Match! software (v3.8.0.137) with crystallography reference database used (COD-Inorg REV218120 2019.09.10). The results of XRD test shown that all samples have several peaks, namely [102], [104], [006], [110], [108], [211], and [212] which appropriate with trigonal phase. The highest peak is positioned at 34,1 °2Th. The existence of peak indicated the presence of CaCO_3 , with the form of $\text{Ca}(\text{CO}_3)$ which has crystal system of monoclinic, calcium carbonate calcite which has crystal system of rhombohedral, and calcium carbonate and calcite which has crystal system of trigonal (hexagonal axes).

Based on **Fig. 2**, it can be seen visually that the XRD results have similar pattern. However, **Table 1** shows that there is a difference in intensity and FWHM. Also, based on **Table 1**, it can be seen that the intensity has a correlation with the crystallite size. The higher the intensity, the larger crystallite size originating from the increase of FWHM [2]. The smallest crystallite

size which counted is 19.2 nm at quail eggshell nanopowder with 7 hours of milling time, and the largest crystallite size which counted is 30.2 nm at duck eggshell nanopowder with 3 hours of milling time and quail eggshell nanopowder with 5 hours of milling time.

Table 1

Intensity, FWHM, d-spacing, and crystallite size of avian eggshell nanopowders

Samples	X-Ray Diffraction (correspond to peak [110])			
	Intensity (cts)	FWHM (rad)	d-spacing (Å)	Crystallite size (nm)
CES M3H	260.02	0.006181956	2.62657	23.5
CES M5H	238.64	0.006869616	2.62356	21.1
CES M7H	255.92	0.006181956	2.62236	23.5
DES M3H	285.00	0.004808382	2.62849	30.2
DES M5H	274.76	0.005496042	2.62877	26.4
DES M7H	260.26	0.005496042	2.62810	26.4
QES M3H	275.07	0.006181956	2.62807	23.5
QES M5H	277.43	0.004808382	2.62466	30.2
QES M7H	262.90	0.007557276	2.63164	19.2

The correlation between milling time and crystallite size was shown in **Fig. 3**, while the correlation between type of avian eggshell powder and crystallite size was shown in **Fig. 4**.

Fig. 3. Correlation between milling time and crystallite size **Fig. 4.** Correlation between type of avian eggshell powder and crystallite size

3. 2. Morphological Characterization

The morphology of avian eggshell was observed by using SEM [2–4]. The SEM results of avian eggshell powders with variation of milling times, shown in **Fig. 5**.

Fig. 5. Morphology of avian eggshell nanopowders:

a – CES M3H; *b* – CES M5H; *c* – CES M7H; *d* – DES M3H; *e* – DES M5H; *f* – DES M7H;
g – QES M3H; *h* – QES M5H; *i* – QES M7H

Fig. 5 shows the morphology of avian eggshell nanopowders with magnification of 100000 times. **Fig. 5, a** shows the morphology of chicken eggshell nanopowder with 3 hours of milling time was spherical with few of oval with the smallest size was 52.78 nm and the largest size was 68.59 nm. **Fig. 5, b** shows the morphology of chicken eggshell nanopowder with 5 hours of milling time was have the form of wormlike with the smallest size was 37.32 nm and the largest size was 76.49 nm. **Fig. 5, c** shows the morphology of chicken eggshell nanopowder with 7 hours of milling time was roundness and few of oval with the smallest size was 32.05 nm and the largest size was 96.87 nm.

Fig. 5, d shows the morphology of duck eggshell nanopowder with 3 hours of milling time was spherical with few of oval with the smallest size was 32.05 nm and the largest size was 107.8 nm. **Fig. 5, e** shows the morphology of duck eggshell nanopowder with 5 hours of milling time was cubical with the smallest size was 37.99 nm and the largest size was 81.80 nm. **Fig. 5, f** shows the morphology of duck eggshell nanopowder with 7 hours of milling time was spherical and few of irregular with the smallest size was 47.44 nm and the largest size was 88.77 nm.

Fig. 5, g shows the morphology of quail eggshell nanopowder with 3 hours of milling time was spherical, with few of cubical and triangular with the smallest size was 49.54 nm and the largest size was 94.38 nm. **Fig. 5, h** shows the morphology of quail eggshell nanopowder with 5 hours of milling time was spherical and few of oval with the smallest size was 56.05 nm and the largest size was 75.77 nm. **Fig. 5, i** shows the morphology of quail eggshell nanopowder with

7 hours of milling time was cubical, spherical, and few of triangular with the smallest size was 88.20 nm and the largest size was 157.5 nm.

In Fig. 5, *a–d*, the uniformity in the structure of grains on the surface showed that agglomeration was the sediment particles on the membrane of eggshell nanopowders grains [2]. While in Figure Fig. 5, *e–i*, the uniformity in the structure of grains on the surface showed that agglomeration was occurred as particles deposited on the membrane of eggshell nanopowders grains [2]. There is no significant change in the particles size which cause by kinetic energy used in milling process that causing the reduction of particle size can be achieved by longer milling time [5].

3. 3. Functional Groups Characterization

The functional group of avian eggshell nanopowders has been observed by using FTIR test. The results of FTIR test were showed in Fig. 6.

Fig. 6. FTIR spectra of avian eggshell nanopowders

Fig. 6 shows that the strong peaks were observed at the bands 3644 and 1520 cm^{-1} . Bands at 3644 and 2517 cm^{-1} possibly indicate to the presence of hydroxyl alcohol group (-OH) stretching during adsorption of water by CaO and acidic hydrogen group (-OH) stretching, respectively [8, 18]. Band at 1520 cm^{-1} can be related to the presence of carbonate mineral (CO_3) in the matrix of eggshell powders [18, 19]. Bands at 854 and 874 cm^{-1} possibly related to in-plane and out-plane deformations, which indicate to the presence of calcium carbonate (CaCO_3) [8, 18, 20]. Bands at 874 and 1520 cm^{-1} also related with asymmetric C-O with carbonate group vibration [8]. Bands at 2909 and 2872 cm^{-1} represent C-H vibration and assign to alkyl CH, which indicating the existence of organic layers, built from amino acids, in the eggshells [8, 18]. Bands at 1788 and 1643 cm^{-1} correspond with C=O and carbonyl group stretching (amide), respectively [18]. Bands at 500–580 cm^{-1} and 586 cm^{-1} shows the presence of Ca-O bond and Ca=O group, respectively [8, 20]. The differences of FTIR spectra of avian eggshell powders can be occur because of chemical treatment like oxidation, addition, and substitution that would effect the chemical nature of membrane top layer [21]. It also known that the final stoichiometry depends on the control and monitoring of the milling conditions [6].

3. 4. Analysis of compressibility

Compressibility and compression ratio has been calculated using (2), (3) [22, 23], respectively. The results have been sorted by the grain shapes that found on each sample and has been shown in Table 3. Fig. 7–10 has showed the correlations between physical properties and compressibility of the samples.

$$\rho_p = \frac{m}{V}, \quad (2)$$

$$CR = \frac{h_0 - h_p}{h_0} \cdot 100 \%, \quad (3)$$

where ρ_p is the compressibility (g/cm^3); m is compact mass (g); V is compact volume (cm^3); CR is compression ratio (%); h_0 is height at zero pressure (cm), h_p is compact height (cm).

Table 3

Crystallite sizes, particle sizes, compression ratio, and compressibility of eggshell nanopowders

Grain shapes	Samples	Crystallite size (nm)	Particle sizes (nm)	Compression ratio (%)		Compressibility (g/cm^3)	
				1000 kgf	2000 kgf	1000 kgf	2000 kgf
Spherical	QES M5H	30.2	56.05	59.50	61.00	1.56	1.60
Oval	DES M3H	30.2	32.05	63.25	63.00	1.64	1.67
	DES M7H	26.4	47.44	61.25	67.00	1.64	1.76
	CES M3H	23.5	52.78	64.00	67.50	1.56	1.70
Cubical	DES M5H	26.4	37.99	58.00	64.00	1.63	1.76
Triangular	QES M3H	23.5	49.54	60.00	61.50	1.57	1.65
Spherical	CES M7H	23.5	32.05	60.75	61.25	1.56	1.66
Oval	QES M7H	19.2	88.2	53.75	60.75	1.56	1.61
Wormlike	CES M5H	21.1	37.32	63.50	67.75	1.63	1.71
Irregular							

Fig. 7, 8 show that the compressibility and compression ratio of samples with spherical and oval grain shapes have increase on both 1000 and 2000 kgf of load. It is because of the increasing of compressibility and compression ratio along with the decreasing of crystallite and particle sizes [23, 24]. While Fig. 9, 10 show that the compressibility and compression ratio in samples with cubical, triangular, spherical and oval grain shapes slightly decrease on both 1000 and 2000 kgf of load, due to morphology such as particle sizes and grain shapes take effect on compressibility [25]. It is because the irregular grain shapes of grain are more difficult to compact than spherical grain shapes, due to it can make it easier to shear deformations to happen [26]. In this reasearch, the milling times does not have significant effect to the compressibility.

Fig. 7. Compressibility in spherical and oval grain shapes

Fig. 8. Compression ratio in spherical and oval grain shapes **Fig. 9.** Compressibility in cubical, triangular, spherical, and oval grain shapes **Fig. 10.** Compression ratio in cubical, triangular, spherical, and oval grain shapes

4. Discussion of experimental results

The results of data analysis showed that the crystallite size with a longer milling time had a smaller size. Meanwhile, larger crystallite sizes can be due to agglomeration and also the sintering process which can cause larger grain sizes [7]. This is because at the same time, the grain

size increases as the temperature increases, it approaching $1\text{ }\mu\text{m}$ at $1000\text{ }^{\circ}\text{C}$ (1273 K) [27]. The phases found in this study are calcium carbonate and calcite which have a trigonal crystal system (hexagonal axes), $\text{Ca}(\text{CO}_3)$ which has a monoclinic crystal system, and calcium carbonate calcite which has a rhombohedral crystal system. The crystalline phase in this study did not change significantly, it was indicated by all the phases found that were still included in the carbonate mineral. This means that the milling process cannot change the structure of the test specimen. Theoretically, the energy supplied from this process cannot overcome the breakdown of the structure [15].

Based on SEM result, it is also known that the dominant grain diameter size of the powdered poultry egg shell with the variation of the milling time is on average below 100 nm which makes it classified as a nanopowder. In the test specimens of eggshell powder, it can be seen that the particle size is not much different, it could be due to the milling time which is not too different. This is because the kinetic energy used in the milling process which causes a reduction in particle size can be obtained with a longer milling time [28].

Differences in the FTIR spectra of eggshell powder can occur due to chemical treatments such as oxidation, addition, and substitution which can result in chemical properties of the top layer of the membrane [21]. It is also known that the final stoichiometry depends on controlling and monitoring the milling conditions [28].

This research offers a synthetic method for processing large amounts of eggshell waste using a solid state reaction. When compared to other synthesis methods, such as sol-gel and coprecipitation, the solid state reaction method is a method that can produce a large number of products. The drawback of this method is that the results obtained have a morphology that is not uniform and tends to agglomerate, therefore, to achieve the uniform size of nanoparticles it is necessary to carry out further synthesis or by using high energy ball milling process. The correlation between physical properties and compressibility is influenced by the grain shape and the size of the crystal or particle. Meanwhile, in this research, it was shown that the milling time did not have a significant effect on grain shape, crystal size, and particle size. This indicates that the milling time used in this study has small effect on the correlation between physical properties and compressibility of poultry eggshell nano powder (chicken, duck, and quail). From this results, CaCO_3 derived from eggshell is a good candidate for tooth filler because it has good compressibility for almost all the type of eggshells. From all characterization results, the three types of eggshells have similarities with each other. But if we look more closely, duck eggshells have a more stable character in terms of crystallite size, compressibility, and uniform morphology not to mention about the availability compare to the quail eggshell. This study focused on the use of eggshells as implant material and from this study, the best eggshells were duck eggshells with a milling time variation of 5 hours. From the simulation results in the previous research, CaCO_3 from eggshell powder was increase the strength of mastication 40 times and the most important parameter is the crystallite size of CaCO_3 [7, 8]. Other application that related to the implant material is hydroxyapatite (HA) that can be obtain from CaCO_3 and natrium phosphate. The biocompatibility and bioactivity properties of HA from eggshell powder has similarities with inorganic components of hard tissue in natural tissue of bones and teeth [29, 30].

5. Conclusion

Nanopowders of the avian eggshell wastes had been successfully produced by the ball mill process. The smallest crystallite size which counted is 19.2 nm at quail eggshell nanopowder with 7 hours of milling time, and the largest crystallite size which counted is 30.2 nm at duck eggshell nanopowder with 3 hours of milling time and quail eggshell nanopowder with 5 hours of milling time. The morphology shows that the major shape of the grains is spherical, but there is no significant change to the grains of avian eggshell nanopowders. The elemental composition represents the dominant element that can be found in avian eggshell nanopowders are calcium (Ca), oxygen (O), and carbon (C). The functional group shows the existence of CaCO_3 , CaO , and carbonate mineral contained in avian eggshell nanopowders. While compressibility of avian eggshell nanopowders will increase along with decreasing of crystallite and particle sizes. The grain shapes will take effect of the compressibility because it can effect

deformations. Based on the results, by knowing the physical properties of the avian eggshell nanopowders, it can be used for multipurpose applications such as material to make hydroxyapatite, tooth filler, catalyst, reinforce, and also become proper material for mass production of CaCO_3 and CaO .

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