

## Identification of Calcium Carbonate ( $\text{CaCO}_3$ ) Characteristics from Different Kinds of Poultry Eggshells Using X-Ray Diffraction (XRD) and Fourier Transformation Infra-Red (FTIR)

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### Abstract

Initial studies have been conducted to study the properties of calcium carbonate ( $\text{CaCO}_3$ ) from a wide range of poultry such as domestic chicken eggshell, Chicken Broiler egg shell, duck egg shell and quail egg shell. This initial study was conducted to determine whether the initial identification of a wide variety of egg shells of various kinds of birds contains Calcium Carbonate, knowing the nature of the initially formed crystals covering the structure, the study of crystal system, lattice parameters, space group and diffraction field. Proving the existence of Calcium Carbonate by FTIR is to understand the formation of calcium carbonate absorption band. Preliminary results from the nature of Calcium Carbonate wide variety of birds by using x-ray diffraction and analysis on chi 1.2 GSAS program, is for every eggshell which are Domestic Chicken, Chicken Broiler, duck and quail have almost the same initial properties of the crystal structure of  $\text{CaCO}_3$  with a system formed rhombohedra crystals with lattice constant values  $a = b = 3.516 \text{ \AA}$ ,  $3.500 \text{ \AA}$ ,  $3.522 \text{ \AA}$ ,  $3.519 \text{ \AA}$  and  $3.505 \text{ \AA}$  and  $c = 12.180 \text{ \AA}$ ,  $12.125 \text{ \AA}$ ,  $12.127 \text{ \AA}$ ,  $12.199 \text{ \AA}$ ,  $12.190 \text{ \AA}$  and  $12.142 \text{ \AA}$ ,  $R_3C$  space group and orientation of the diffraction planes [hkl] in ( $2\theta$ )  $34,325^\circ$ ,  $34,110^\circ$ ,  $34,135^\circ$  and  $54,275^\circ$ . Further evidence was obtained by using FTIR absorption band that calcium carbonate ( $\text{CaCO}_3$ ) in the sample eggshell, chicken eggshell, duck eggs, quail eggs and much more is formed on the Ca-CO group at wave number  $1405 \text{ cm}^{-1}$ ,  $873 \text{ cm}^{-1}$  and  $712 \text{ cm}^{-1}$ .

*Keywords:*  $\text{CaCO}_3$ , X-Ray Diffraction, and FTIR

### 1. Introduction

Calcium carbonate ( $\text{CaCO}_3$ ) is the calcium salt found in chalk, limestone, marble. Calcium carbonate in the form of powder, white, odorless, tasteless, stable in water. Practically insoluble in water, solubility in water increases with little or ammonium salts and carbon dioxide dissolved in nitric acid to form a gas bubble. Given Calcium carbonate ( $\text{CaCO}_3$ ) itself can be found in large quantities in nature, with applications mostly as raw material for the ceramic calcium carbonate development in rapidly growing technology and research, such as hydroxyl apatite material synthesis as an alternative to the teeth and bones of human<sup>1</sup>. Rapid technological developments have forced research in all fields of science and technology is no exception to

continue to innovate in the technology of waste utilization and waste<sup>2</sup>. It is particularly interesting to study the waste bin of various kinds of poultry egg shell, with an assumption that a lot of egg shells contain calcium carbonate. Utilization of waste eggshell as a material for synthesizing other compounds those are able to combine to produce a new compound.

Potential shell waste or egg shell waste in Indonesia is quite large, which produces 178,566.33 tons a year. Eggshell may cause environmental pollution because it is difficult to be degraded by soil microbes<sup>3</sup>. Alternative methods to process and use eggshell in a way that is beneficial to the environment, e.g the process of shell material into a substrate that can be used as material manufacturers Hydroxiapatite synthesis (HAP) that

serves as the replacement of bone or teeth in humans by using a combustion method (solution combustion).

Many studies using egg shells, e.g made of and hidroxyapatit characterization of a snailshell<sup>4</sup>. Snailshell produced from the boiling process, cleaning, and drying, calcined at a temperature of 800°C for 4 hours to be used as a powder. The powder was identified phases of calcium and calcium levels contained by X-Ray diffraction and atomic spectroscopy absorbtion. The results of the drying temperature 110°C, only slightly formed hydroxyapatite and many impurities. Synthesis results are analyzed with X-ray diffractometer and Fourier transform infrared analysis showed that hydroxyapatit formed on the sample. The use of eggshell as ingredients hydroxy apatite has been done by purwasasmita<sup>5</sup> characterization using XRD (X-Ray Difracton), FTIR (Fourier Transform Infrared) and SEM (Scanning Electron Microscopy ).

This study is only a preliminary assesment of various kinds of eggshells which are domestic chicken eggs, chicken broiler, duck and quail. This is done because it has been much research done on the direct synthesis of hydroxyapatite materials, without knowing in advance the initial identification of the basic material. Characterization of the starting material has very significance, because it involves the following of properties synthesized material or would be processed further

## 2. Methodology

The tools used in this study are: (a) mortar, (b) sieve, (c) glass preparations, (d) a spatula, (e) Phillips Diffractometer PW 3710 / 40kV, (f) FTIR, (g) ICDD, PPE and BELLA software. This study used a Domestic Chicken eggshell, Chicken Broiler, duck and quail. This material is in the form of shells obtained from the residual waste or disposal form.

The initial phase is the management of eggshell material by cleaning with alcohol. Followed by the second stage is to make the eggshell into a powder with a mortar, and then passes 150 mesh sieve. The third stage is done by heating at 100°C to vaporize H<sub>2</sub>O. The fourth stage is the testing of the samples by XRF to determine the elements that are deposited on powder samples. Samples were prepared in a manner to four

eggshell powder was placed on top of the glass preparations, then measuring the diffraction pattern by means of conditions on the source CoK $\alpha$ , 1.7902Å wavelength, voltage 40 kV, current 30 mA, mince speed of 0.5/sec, kind of continuous and chopped 2 $\theta$  angle 15°-90°. Diffractogram results were analyzed using the PPE, BELLA and ICDD software, to determine the crystal structure and phases that may be formed in the sample.

Fourier Transformation Infrared (FTIR) is a tool used to determine bond in a molecule vibrates due to the frequency. FTIR principle is given in terms of energy absorption of infrared radiation by molecules that result in vibration at the molecular bond. Spectrum characterization is out of the wave number as independent variables and the transmittance (%) as the dependent variable.

## 3. Result and Discussion

Based on observations of the XRF results for all of the domestic chicken eggshell, Chicken Broiler, duck and quail, in Table 1 shows the results of quantitative XRF characterization, where the elemental composition of Ca has a greater value than other elements contained in the shell material, include Si, P, S, Ti, Fe, Zn and Sr. This can be explained from the values of weight% and the molecular weight obtained showed the highest values of the other elements.

Table 1. XRF Result of domestic eggshell, Chicken broiler, duck and quail

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

While qualitatively, can be seen in Fig. 1 in the form of a graph of peak formation for each element of the XRF characterization results.

XRF characterization chart patterns can qualitatively explain the change of the peak varies. Where elements of Ca showed peak with the highest intensity among other elements. Further proof that every egg shells contain calcium carbonate ( $\text{CaCO}_3$ ) i.e the diffraction test. Based on observations footage to four eggshell powder (domestic chicken, broiler chicken, duck and quail) is at  $\theta = 34,165^\circ, 34,325^\circ, 34,32^\circ, 34,11^\circ, 34,135^\circ$  and  $34,275^\circ$ . From the location of the peaks can be determined also large lattice parameters. Lattice parameters can be determined by matching with ICDD data software that meet the rules of a layout pattern of diffraction peaks have a tendency to each other the same pattern, according to  $h^2 + k^2 + l^2$  is [104], [200], [220], [311] and [222].

In Fig. 2 shows the diffraction pattern of four eggshell (domestic chicken, chicken broiler, duck and quail). PCDFwin is a data bank containing all the research data on the crystal structure of the material and the data are only contained 53 data banks in accordance with the crystal structure of  $\text{CaCO}_3$ . From the results it can be seen PCDFwin value of the distance between the field orientation (d), lattice constants, space group and the name of a crystalline mineral that is most important as well as the shape of the crystal structure of the material.

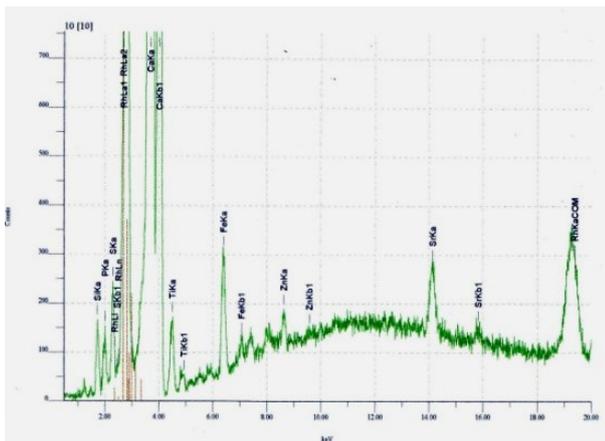


Fig. 1. XRF pattern of domestic chicken, chicken broiler, duck and quail eggshell.

The shape of the crystal structure of shell eggs, chicken, duck and quail, with a compound obtained  $\text{CaCO}_3$  rhombohedra crystal system with space group for each sample generated at the  $R_3C$ , which has a value for each sample of field orientation [104], whereas for the lattice parameter values different for each sample. For samples with chicken and chicken have the same lattice constant values ( $a = b = 4.995 \text{ \AA}$  and  $c = 17.06 \text{ \AA}$ ), whereas for sample ducks also have the same value of the lattice constant ( $a = b = 4.990 \text{ \AA}$  and  $c = 17.00 \text{ \AA}$ ) and to sample quail has a value of lattice constant ( $a = b = 4.989 \text{ \AA}$  and  $c = 17.06 \text{ \AA}$ ).

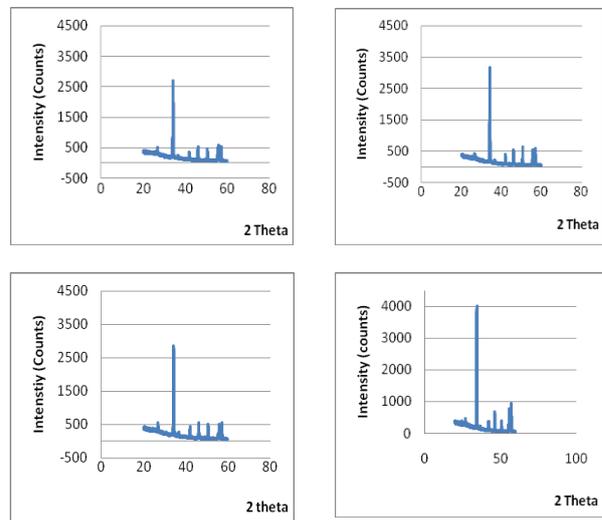


Fig. 2 Diffraction pattern of eggshell: (a) Domestic chicken, (b) chicken broiler, (c) duck and (d) Quail.

Proving that the presence of calcium carbonate ( $\text{CaCO}_3$ ) with the formation of clusters of Ca-CO bond stretching of carbonate also occurs in the sample on the eggshell samples are in the area around 1405, 873, and  $712\text{ cm}^{-1}$  with which the wave numbers  $1405\text{ cm}^{-1}$  is the In-Plane Bending, wave number of  $873\text{ cm}^{-1}$  is the Out-

Out-Plane Bending, and wave number of  $712\text{ cm}^{-1}$  is the asymmetric stretching region.

These results are consistent with the results of the study reported by (Shan, 2007) indicated that the infrared spectra of the Ca-CO group stretching of carbonate that appears on the  $1418, 873, 707\text{ cm}^{-1}$ . From

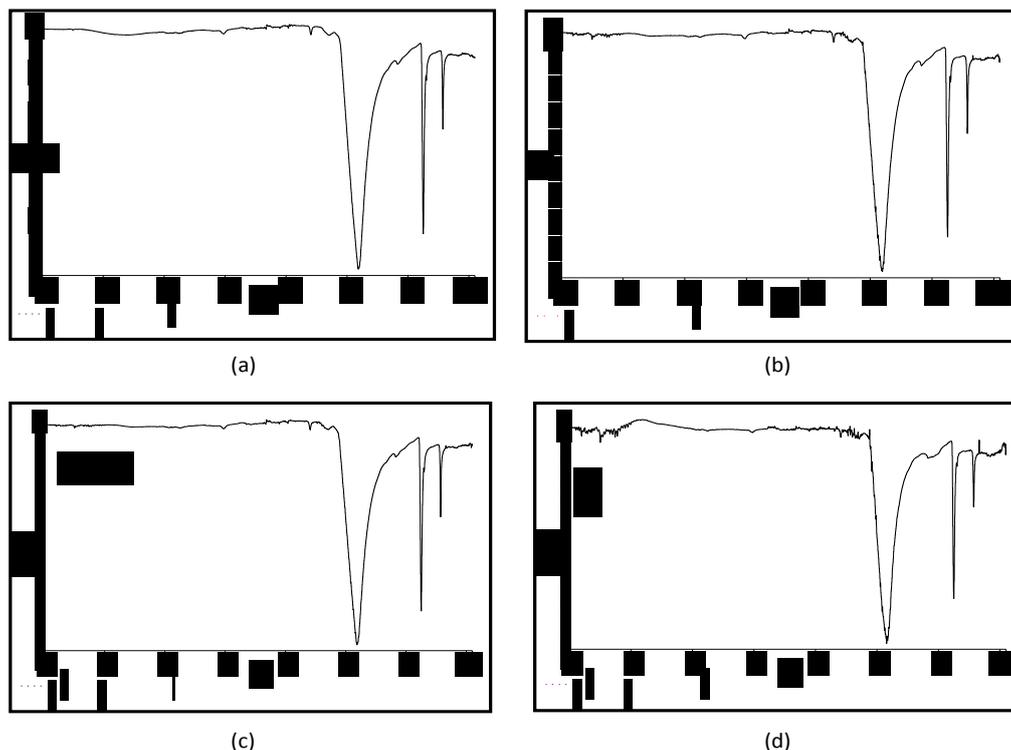


Fig. 3 FTIR pattern of eggshell: (a) Domestic chicken, (b) chicken broiler, (c) duck and (d) Quail.

Plane Bending, and the wave number of  $712\text{ cm}^{-1}$  is the asymmetric stretching region. In the chicken egg shell formed Ca-CO group in the area around 1405, 873, and  $712\text{ cm}^{-1}$  with which the wave number  $1405\text{ cm}^{-1}$  is the In-Plane Bending, wave number of  $873\text{ cm}^{-1}$  is the Out-Plane Bending, and the wave number of  $712\text{ cm}^{-1}$  is an asymmetric stretching region. In the duck eggshell Ca-CO clusters formed in the area around 1405, 873, and  $712\text{ cm}^{-1}$  with which the wave number  $1405\text{ cm}^{-1}$  is the In-Plane Bending, wave number of  $873\text{ cm}^{-1}$  is the Out-Plane Bending, and wave number of  $712\text{ cm}^{-1}$  is the asymmetric stretching region. While quail eggs are formed in Ca-CO group in the area around 1421, 874, and  $712\text{ cm}^{-1}$  with which the wave number  $1421\text{ cm}^{-1}$  is the In-Plane Bending, wave number of  $874\text{ cm}^{-1}$  is the

these results also showed that the calcium carbonate ( $\text{CaCO}_3$ ) in the sample shell eggs, chicken eggs, duck eggs, quail eggs and much more form the Ca-CO group at wave number  $1405\text{ cm}^{-1}$ ,  $873\text{ cm}^{-1}$ ,  $712\text{ cm}^{-1}$

#### 4. Conclusion

Results of X-Ray Fluorescence characterization (XRF) of the material eggshell highest weight% Ca elements shown in the percentage of 95.603%. The crystal structure formed has been showed rhombohedral crystal system with lattice constant values for samples with eggshell-range chickens and chicken have the same value of the lattice constant ( $a = b = 4.995\text{ \AA}$  and  $c = 17.06\text{ \AA}$ ), The results of the characterization of the Fourier Transformation Infra-Red (FTIR) of the samples of each material eggshell formation bonding OH group,

CH, C = O, CO, and Ca-CO bond is a group of compounds forming CaCO<sub>3</sub> and Ca (OH)<sub>2</sub>. Fourier Transformation Infra-Red (FTIR) were obtained that the bond formed at the OH group absorption region 3866 cm<sup>-1</sup>, CH formed in the region 2511 cm<sup>-1</sup>, C = O group formed in the 1795 and 1647 cm<sup>-1</sup>, whereas the CO group formed in the region 1191 cm<sup>-1</sup>, and Ca-CO group formed in the 1405 cm<sup>-1</sup>, 873 cm<sup>-1</sup>, 712 cm<sup>-1</sup>. The results also showed that the calcium carbonate (CaCO<sub>3</sub>) in the sample shell eggs, chicken eggs, duck eggs, quail eggs and more formed in Ca-CO group at wave number 1405 cm<sup>-1</sup>, 873 cm<sup>-1</sup>, 712 cm<sup>-1</sup>.

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