CHAPTER III
MATERIALS AND METHOD

3.1 Materials and Equipments

The materials which were used in this research were hen’s eggshell and pro-analyze Ca(OH)₂ as calcium source, pro-analyze (NH₄)₂HPO₄ and pro-analyze H₃PO₄ as phosphate source. Sample preparation was performed in-house hydrothermal reactor at Biophysics Laboratory, Physics Department IPB. Sample was characterized by using X-Ray Diffractometer (XRD), Fourier Transform Infra Red (FTIR) spectrometer, and Scanning Electron Microscope (SEM).

3.2 Experimental Method

Washing eggshell to remove inner membrane of eggshell was done by using aquade. Calcination of these eggshell was held at temperature 1000°C with temperature rate 5°C/minute within 5 hours. The result of this process was calcium oxide (CaO) which is used as calcium precursor, with the reaction as follows:

\[ \text{CaCO}_3 \rightarrow \text{CaO} + \text{CO}_2 \]

![Washed eggshell](image1.png)  ![Calcination at T=1000°C for 5 hours](image2.png)  ![Result of calcination](image3.png)

Figure 3. Calcination of eggshell process.

There were two types of sample, named sample A and B. Sample A was prepared by using 1 M CaO from hen’s eggshell and 0.6 M (NH₄)₂HPO₄. While sample B was prepared by using 1 M Ca(OH)₂ and 0.6 M H₃PO₄. Each solution was mixed in a hydrothermal reactor. CaO was added by (NH₄)₂HPO₄, while Ca(OH)₂ was added by H₃PO₄ by using burette with dropping rate 6.67 ml/minute.
Subsequently, the mixed solution was heated in house hydrothermal reactor at temperature 300°C and at 20 psi pressure condition for 8 hours. To control homogeneity of solution, stirring was held during the heating process. After heating process, sample was decanted for about 12 hours, and then it was dried using furnace at temperature 110°C for 5 hours. Result of sample was sintered at temperature 1000°C for 2, 4, and 6 hours. The results sample from A types was called A1, A2, and A3 relating with 2, 4, and 6 hours of sintering time, respectively. The same code was given to sample B as B1, B2, and B3.

![Diagram showing hydrothermal process followed by sintering process.](image)

Figure 3.2 Hydrothermal process followed by sintering process.

These samples were characterized by using X-Ray diffractometer (XRD), Fourier transform infrared (FTIR) spectroscopy, and scanning electron microscope (SEM). XRD characterization was held by using Shimadzu XRD610 which use Kα x-ray characteristics of Cu (wavelength 1.54060 x 10^-10 m) with the range of diffraction angle 10° - 80°.

FTIR characterization was held for getting information of functional group in the sample. FTIR characterization used BRUKER model TENSOR 37 FTIR...
Spectroscopy. Two milligram of sample compacted into a pellet with a hundred of KBr to be irradiated by infra red with wavenumber in a range of 4000-400 cm$^{-1}$.

SEM characterization was held by using JEOL JCM-35C scanning electron microscope. Before characterization, the sample was coated by gold-palladium (80% of Au and 20% of Pd).