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PRODUCTION AND CHARACTERIZATION OF NANO-$CACO_3$ FROM CLAMSHELL (GELOINA SP.) BY TOP-DOWN METHOD

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ABSTRACT

The utilization of nano-$CaCO_3$ is currently growing very fast in various fields. The research aims to produce and analyze the properties of nano-$CaCO_3$ from clamshells (Geloina sp.). The production of nan-$CaCO_3$ was done by the top-down process using high-energy milling. The clamshell (Geloina sp.) is the potential resource of nano-$CaCO_3$. The nano-$CaCO_3$ can be produced by the milling process. The main factor that affected the yield is the number of steel balls, while the speed of rotation and number of cycles have a negative effect. The EDX analysis shows that the nano-$CaCO_3$ has high purity. The nano-$CaCO_3$ from clamshell (Geloine sp.) can be applicated as a drug delivery system and catalyst.

Keywords: Nano-$CaCO_3$, Geloina sp., Top-Down Method

1. INTRODUCTION

The utilization of nano-$CaCO_3$ is currently growing very fast in various fields. In the drug field, nano-$CaCO_3$ is one of the intelligent carriers because of its biocompatibility and biodegradability, especially its sensitivity to pH Xing et al. (2020). The toothpaste that contains nano-$CaCO_3$ can be used as an anticaries drug on the teeth Adnyani et al. (2020). In the industrial field, nano- $CaCO_3$ can be used as a catalyst Mosaddegh et al. (2013). Nano-$CaCO_3$ also can be used as a reliable, durable, and environment-friendly alternative to diminishing fly ash Poudyal et al. (2021). The nano- $CaCO_3$ can also as an additive in the epoxy resin adhesive to increase the shear strength Kaybal et al. (2017). Nano- $CaCO_3$ can also decrease the flowability and the setting time of fresh cement paste Liu et al. (2012).

Nano-$CaCO_3$ can be made from chemicals and natural resources by several processes. From chemicals, nano- $CaCO_3$ can be produced from CaO Adnyani et al. (2020) and CaCl2 Xing et al. (2020). From natural resources, nano- $CaCO_3$ can be produced from materials that contain $CaCO_3$ such as eggshells Mosaddegh et al. (2013), clamshells Widyastuti & Intan Ayu Kusuma (2017), pearl shells Wahyuningsih et al. (2019), and rocks Akhwady and Bayuaji (2017). Clamshell (Geloina sp.) is one abundance of natural resources of calcium carbonate ($CaCO_3$). The production of Nano- $CaCO_3$ from clamshells can be conducted by a top-down method and a bottom-up method. In the top-down method, the materials are milled until the nano-size is achieved Kamboj et al. (2020) Mosaddegh et al. (2013). While the bottom-up method, nano- $CaCO_3$ can be produced by the precipitation process Ismail et al. (2022). The top-bottom method is simpler compared to the bottom-up method.

This study aims to produce nano- $CaCO_3$ from clamshell by top-bottom method and characterize the nano- $CaCO_3$ produced. Three main variables such as the number of balls, speed rotation, and the number of cycles is studied. The characteristics of nano- $CaCO_3$ are studied using Fourier Transform Infra-Red (FTIR) and Scanning Electron Microscope (SEM).

2. MATERIALS AND METHODS

2.1. MATERIALS

The Clamshell was obtained from the local market in the Cilacap Regency. The sodium hydroxide (NaOH) was obtained from Merck.

2.2. PREPARATION

The clamshell was washed and crushed until the particle size of 50 – 80 mesh. The powder of the clamshell was then dried at the temperature of 105oC. The clamshell powder was then deproteinized using NaOH solution Eke-Ejiofor and Moses (2019).

2.3. MILLING PROCESS

The milling process was using high-energy milling. High-energy milling was carried out with a number of steel balls of 20 with a diameter of 3 mm. The high-energy milling process uses a speed of rotation of 300 rpm and a number of cycles of 500,000 cycles. After the milling process, the milled $CaCO_3$ was then sieved using a 500 mesh. The yield of the milling process was calculated using Eq. 1.

$$\text{yield, \%} = \frac{w1}{w0} x100\%$$

Equation 1

where w0 is the weight of feed $CaCO_3$ and w1 is the weight of nano-$CaCO_3$. The design experiment is shown in Table 1.

Table 1

Table 1 Design Experiment		
	Variables	Value of Variables
	-1	1
Number of balls (X1)	10	20
Speed of rotation (X2)	300	400
Number of cycles (X3)	5,00,000	10,00,000

Characterizations

The functional groups of nano- $CaCO_3$ were studied using Fourier Transform Infra-Red (FTIR). The morphology of nano- $CaCO_3$ was analyzed using Scanning Electron Microscope – Energy Dispersive X-Ray (SEM-EDX).

3. RESULTS AND DISCUSSIONS

3.1. YIELD OF NANO-$CACO_3$

Table 2 shows the yield of nano- $CaCO_3$ obtained. The yields obtained are a range of 13.67 - 43.29%. Table 3 shows the effect of the main and interaction variables. Factorial design analysis shows that the main effect that effluence the yield is the number of balls (X1). While the most influential variable interaction is the interaction between the number of balls (X1) and the number of cycles (X3).

Table 2

Table 2 The yield of nano-$CaCO_3$ obtained				
Run	X1	X2	X3	Yield
1	-	-	-	40.95
2	+	-	-	34.85
3	+	+	-	43.29
4	-	+	-	35.54
5	-	-	+	13.67
6	+	-	+	40.84
7	+	+	+	15.42

Table 3

Table 3 Effect of variables	
Effects	Values
X1	8.85
X2	-0.67
X3	-49.31
X12	-64.13
X13	5.55
X23	-6.73
X123	-60.99

Figure 1 shows the main effects plot for yield. The number of steel balls has a positive effect (8.85), at a higher of a number of steel balls the yield of nano- $CaCO_3$

obtained will be increased. The speed of rotation has a slightly negative effect (0.67), with the addition of the speed of rotation, the yield will decrease slightly. While the number of cycles has a negative effect (-49.31). The addition of the number of cycles will decrease of yield of nano- $CaCO_3$. From the experimental data, it can be recommended that the best condition of nano- $CaCO_3$ using high-energy milling is at the number of steel balls of 20, the speed of rotation of 300 rpm, and the number of cycles of 500,000 cycles.

Figure 1

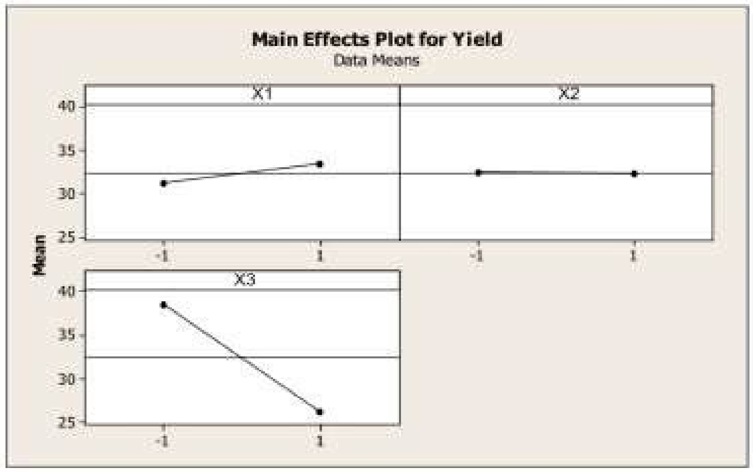

Figure 1 Main Effects Plot of Process Variables

3.2. 3FTIR SPECTRUM OF NANO- $CACO_3$

Figure 2 shows the FTIR spectrum of nano- $CaCO_3$. There are three main peaks, respectively 1465.9, 1450.47, and 856.39 cm-1. The three peaks show the vibration of CO32- Ramasamy et al. (2017). Figure 3 shows the EDX analysis of nano- $CaCO_3$. EDX analysis shows the chemical composition of nano- $CaCO_3$. The mass percentage of Ca, C, and O are 28.15%, 17.28%, and 53.85% respectively. From the EDX analysis can be seen that nano-$CaCO_3$ produced has high purity.

Figure 2

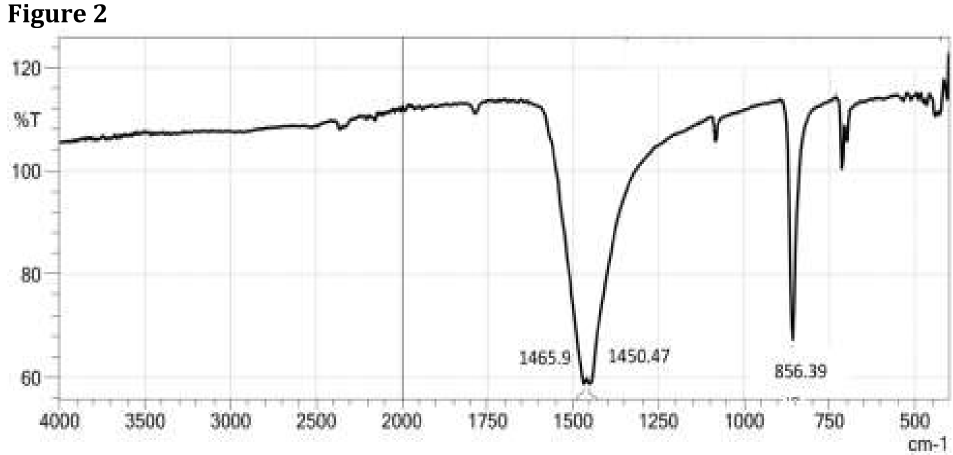

Figure 2 FTIR Spectrum of $CaCO_3$

Figure 3

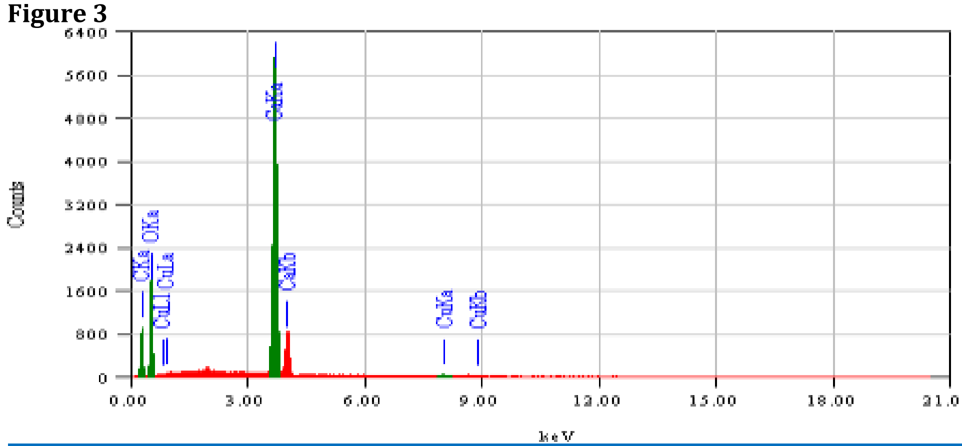

Figure 3 EDX Analysis of Nano-$CaCO_3$

3.3. MORPHOLOGY OF NANO-$CACO_3$

Figure 4 Show the morphology of nano- $CaCO_3$ obtained from Geloina sp. Figure 3 shows that the nano- size was obtained during the milling process. The nanoparticle or ultrafine particle is usually defined as a particle of matter that is between 1 and 100 nanometres (nm) in diameter. The bigger particle size occurred during the milling process because of the agglomeration of nano- $CaCO_3$. This is following the results of the factorial analysis which shows that the number of cycles factor has a negative effect. The longer the cycle, the more particles agglomerate.

Figure 4

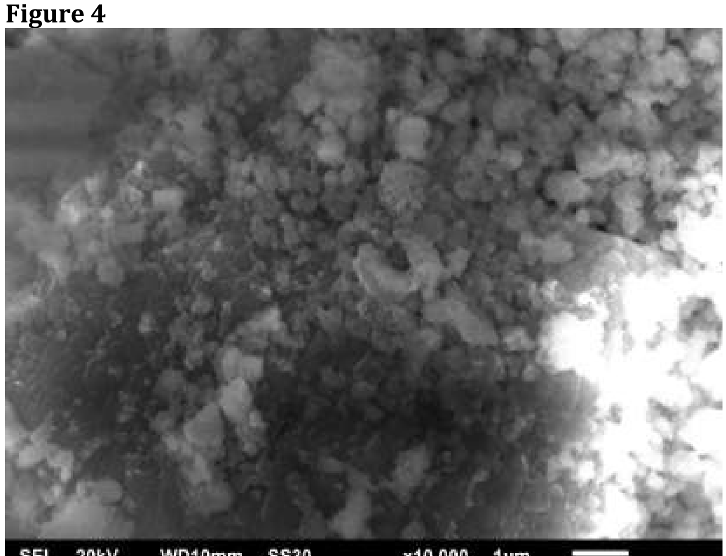

Figure 4 Morphology of Nano-$CaCO_3$

4. CONCLUSIONS AND RECOMMENDATIONS

The clamshell (Geloina sp,) is the potential resource of nano- $CaCO_3$. The nano-$CaCO_3$ can be produced by the milling process. The main factor that affected the yield is the number of balls, while the speed of rotation and number of cycles have a negative effect. The EDX analysis shows that the nano- $CaCO_3$ has high purity. The nano- $CaCO_3$ from clamshell (Geloine sp.) can be applicated as a drug delivery system and catalyst.

CONFLICT OF INTEREST

None.

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