Microstructure and Physical of Al_2O_3 $2SiO_4$ $2H_2O$ Kaolinite Particle Analysis by Shacking Time and Powder Metallurgy

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Abstract: The shacking time effect on the kaolinite material morphology, physical and grain size have been analyzed. The aim of this study was to synthesize kaolinite material from Indonesian kaolin to gain nanoscale particle reduction by shacking time and powder metallurgy cycle. Kaolinite particle powder was successfully synthesized from thick kaolin solution. The increase in the shacking time resulted in the homogenous dispersion of kaolinite particles, the reduction of grain size particle clustering, and the reduction of distances between its particles. The significant grain refining during shacking was revealed which showed reduction of particle size resulting from longer milling time. SEM analysis of the nanoparticle explained that designated ball shacking presents to the crystalline refining development. It can be discussed that these morphological and microstructural variations of $Al2O_3$ $2SiO_4$ $2H_2O$ particle powders developed by designated ball shacking time were found to present to an improvement in the density, grain refinery, grain size of kaolinite material particle. The modification of particle grain size is possible into an initial nanoscale by employing shacking and milling powder metallurgy process. A significant reduction of grain size reduction was acquired in all cases.

1 INTRODUCTION

Kaolin's group minerals and its derivative metaform is characterized by a rather simple chemical composition of Si, Al, O, and H (Dill, 2016). Kaolin is relatively pure clay and it has been widely used in ceramic industries for years (Chen, Lan and Tuan, 2000). The main chemical elements of kaolin, is a hydrous aluminum silicate of the approximate composition $2H_20$-$A1_2O_3$-$2SiO_2$. Structurally, kaolinite material consists of alumina octahedral sheets and silica tetrahedral sheets stacked alternately and has the theoretical composition 46.54% Si, 39.5°/0 $A12O_3$, 13.96% H2O (Prasad, Reid and Murray, 1991).

The main mineral component of kaolin is kaolinite, which consists of layers held together through hydrogen bonds. Each layer consists of a two-dimensional arrangement of Al-centred octahedral (O) and a two-dimensional arrangement of Si-centred tetrahedral (T) (Zsirka *et al.*, 2015). The layer is formulated of tetrahedral (Si-O) and octahedral (Al-O) films bonded through common oxygen. Moreover, kaolinite occupies a unique asymmetric interlayer arrangement with two chemically different surfaces: oxygen of the tetrahedral film and inner surface hydroxyls of the octahedral film (Matusik and Matykowska, 2014). This enables for the synthesis of new nanomaterial products with precisely defined properties (Matusik and Bajda, 2013).

Kaolinite is a refractory material since it is a non-metallic material capable of enduring high temperatures and suitable as construction materials for industrial furnaces. Their primary purpose is derived from their resistance to high temperatures (Aramide and Seidu, 2013). One of its application is for refractory metal furnace material. However, to develop reliable metal furnace it requires optimum material alloy and optimum grain size.

The particular grain size of the material generates different atomic bonding (Taylor, Meyersm and Ashworth, 2007). In the term of material selection that process would affect micro-

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structure and which determines the properties of the material (Callister Jr and Rethwisch, 2009). Therefore the aim of this work is to prepare AL_2O_3 $2SIO_4$ $2H_2O$ particle using mechanical shacking milling method in order to reduce the existing grain size to nanoscale particle reduction, dispersion, and morphology of particles. Through milling process, it is possible to acquire solid materials with wider surface area and different particle sizes (Leonel, 2014).

2 EXPERIMENTAL

2.1 Material

For the preparation of the microstructures, four samples of Indonesian kaolin were processed. The untreated kaolin was measure in the ground is 2.24µm. The solvent used: ethanol 70%. The chemical compositions of the raw kaolin are given in Table 1, 2, 3 and 4. This chemical composition acquired by SEM EDX testing from the untreated sample. The test given in four different spots to get the average chemical composition of the sample.

2.2 Kaolinite Particle Synthesis

The kaolin powder was purchased from the local chemical store in Semarang and placed in a plastic bag prior to the measurement of its weight. 9 grams of kaolin sample was prepared in a separated container prior to the treatment. The kaolin powder, ethanol and ball mill were placed in the stainless chamber of the shacking mill machine. It was then shacked and milled in eight axes to optimize its kaolinite particle fabrication in 30, 60 and 90 minutes individually with ethanol solution maintaining a liquid ratio of kaolin and ball mill 1:10 by mass. The thick solution was collected for the synthesis of kaolinite particle. The sample was handled for drying purpose and moved from the chamber into a separated container prior characterization sample preparation. A standardize coating was done prior to the sample characterization process to investigate its microstructure.

2.3 Structural Characterization Method

Scanning Electron Microscopy (SEM) - Energy Dispersive X-Ray (EDX) analyzer from PhenomProx was employed for structural characterization of the samples to determine the chemical elements, microstructure, dispersion, morphology, and its grain size. Each sample was placed on a carbon type and coated by Aurum in 18mA for optimal result. Untreated sample as the first sample was analyzed by Scanning Electron Microscopy (Choi *et al.*, 2016) and Energy Dispersive X-Ray (EDX) analyzer (Bhattacharyya and Behera, 2017).

However, the other three sample which has been treated were analyzed by Scanning Electron Microscopy (Takeda *et al.*, 2013) since there is no additional chemical element process during the particle synthesis process. The result of each characterization shown in Figure 2 to 9.

3 RESULT AND DISCUSSION

It is acknowledged that milling processes of crystalline material enable a significant change in the morphology of powders as a result of great plastic deformation of the particles within the milling process (Hossein-zadeh and Razavi, 2013).

3.1 Particle Chemical Composition

The kaolinite particle chemical composition analysis was taken from four different spots as shown in Figure 1.

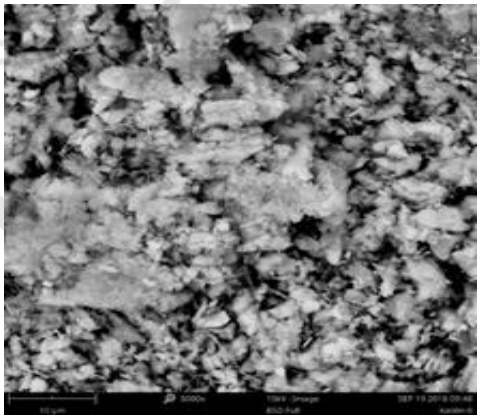

Figure 1: Scanning Electron Microscopy of untreated kaolinite material morphology.

Table 1: Kaolin particle chemical composition in spot 1.

Element	Weight Percentage
O	59.3%
Al	16.1%
Si	13.7%
Sr	10.0%
Ba	0.9%
K	0%

Table 2: Kaolin particle chemical composition in spot 2.

Element	Weight Percentage
O	61.0%
Al	14.8%
Si	15.9%
Sr	8.3%
K	0%

Table 3: Kaolin particle chemical composition in spot 3.

Element	Weight Percentage
O	57.4%
Al	16.0%
Si	13.9%
Sr	9.6%
K	3.1%

Table 4: Kaolin particle chemical composition in spot 4.

Element	Weight Percentage
O	58.4%
Al	16.3%
Si	14.0%
Sr	10.7%
Ba	0.6%

Figure 1 shows Scanning Electron Microscopy of untreated kaolinite material morphology, the chemical composition testing was done in four different spots. It is possible to examine the detail chemical composition as shown in table 1, 2, 3 and 4. Major chemical compositions of the sample are Oxygen, Aluminium and Silica are visible in a significant amount. However, there is a phenomenon that the sample has Strontium, Barium, and Kalium at spot number 3. It can be discussed as the uniqueness of the Indonesian kaolin chemical composition influenced by the natural development in the soil.

3.2 Particle Microstructure

The micro kaolinite particles were synthesized and its morphology and dispersion are shown in Figures below. Figure 2 describes the microstructure, morphology and kaolinite particle dispersion of the untreated sample in 5,000x magnitude and 10µm length. While Figure 3 describes the microstructure, morphology and kaolinite particle dispersion of the untreated sample in 20,000x magnitude and 5µm length. The grain size was investigated that it is still possible to perceive the presence of crystalline materials with a relatively big size throughout the surface of the sample.

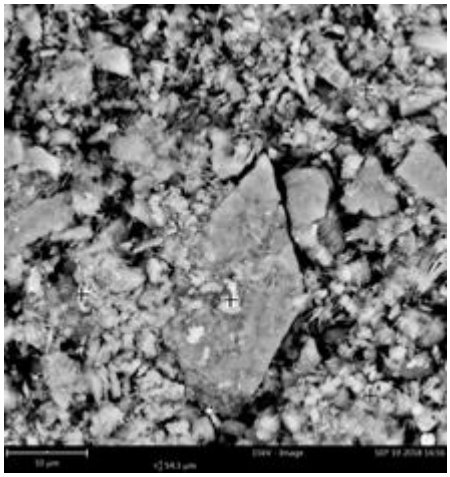

Figure 2: Scanning Electron Microscopy of untreated kaolinite material.

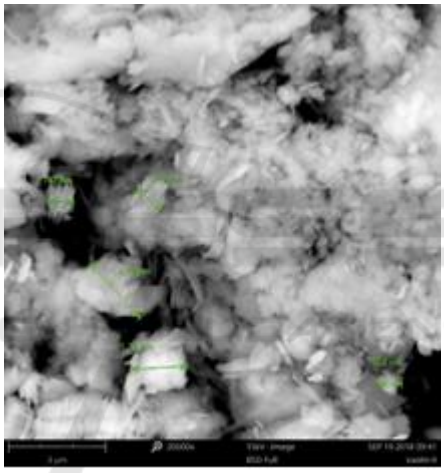

Figure 3: microstructure, morphology and kaolinite particle .

The grain size investigation was done in five different spots as shown in Figure 2 above. The initial diameter of grain size was investigated, it is 2.24µm. The results examined in Figure 4 and 5 describe the existence of different morphological transformation phenomena which occur simultaneously along with the shacking process (Leonel *et al.*, 2014). It can be explained that the particular particles initiated to crack due to the shacking mechanism. As a matter of fact that kaolinite material is a brittle material, the breakage of it is particle occurs in a wide dispersion throughout the particle surface as shown in Figure 4 and 5.

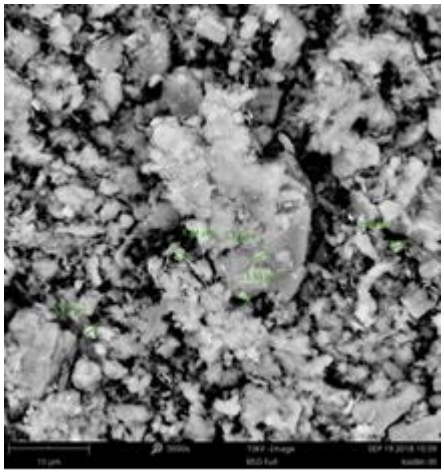

Figure 4: Scanning Electron Microscopy of kaolinite material morphology after 30 minutes of treatment.

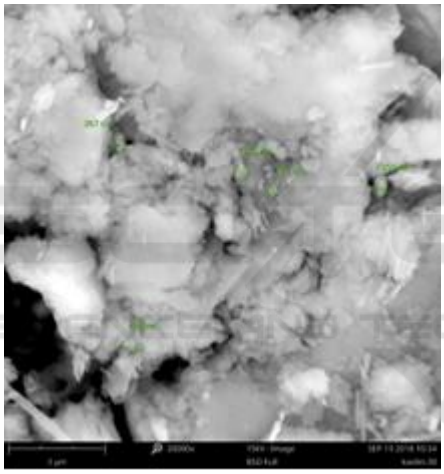

Figure 5: Scanning Electron Microscopy of kaolinite material grain size after 30 minutes of treatment.

It is explained that the dispersion of its morphology of the particle is wider, the pores visibility of the particles are relatively reduced. This means there is a reduction of the particles into a smaller grain size. As the result, there is an escalation of the surface contact area of each particle. It shows on Figure 6 that the grain size particles range is between 990nm - 1.07µm. More detail result can be obtained from Figure 7 which explains that the grain size range is between 312 – 297nm. Through both Figure 8 and 9 it can be examined that there is a shrinkage in the width of the peak demonstrating the formation of a slightly more homogeneous particle in comparison with the material before the treatment process (Hubadillah *et al.*, 2016). There is a significant grain size decrement subject to each particle of the kaolinite material. It shows that the morphology of the material is more homogenous and the pores number are decreased tremendously. The diameter of grain size obtained in the range of 211 – 190nm.

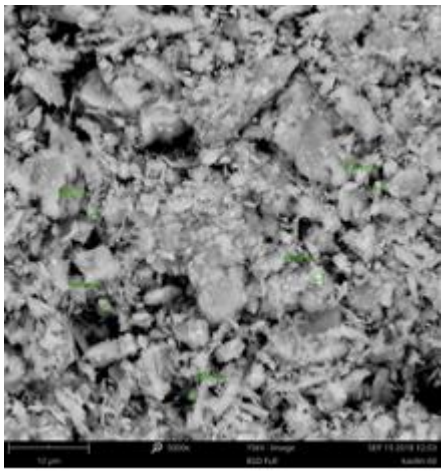

Figure 6: Scanning Electron Microscopy of kaolinite material morphology after 60 minutes of of treatment.

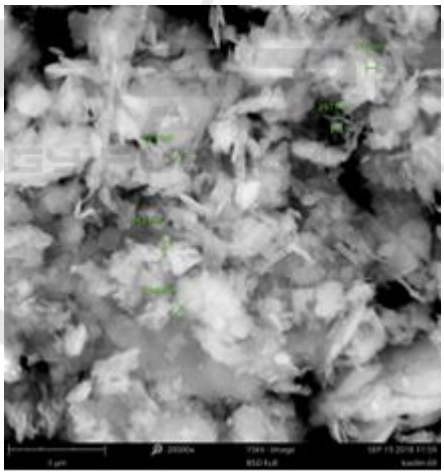

Figure 7: Scanning Electron Microscopy of kaolinite material grain size after 60 minutes of of treatment.

Furthermore, it can be investigated further from Figure 9 that the surface area of contact is larger than the previous ones. Besides, $AL2O_3$ $2SiO_4$ $2H_2O$ nanoparticles materialize in large arrays initiate to disperse within kaolinite particles with a better homogeneity and smaller area by escalating the shacking life (Toozandehjani *et al.*, 2017). The significant disparity in the crystalline size and in the lattice strain of kaolinite particle is associated to great plastic deformation and grain size refinement

occurring in particles in the presence of $AL2O_3$ $2SiO_4$ $2H_2O$ initial nanoparticles as the main subscription to the shacking cycle (Mobasherpour, To and Ebrahimi, 2013).

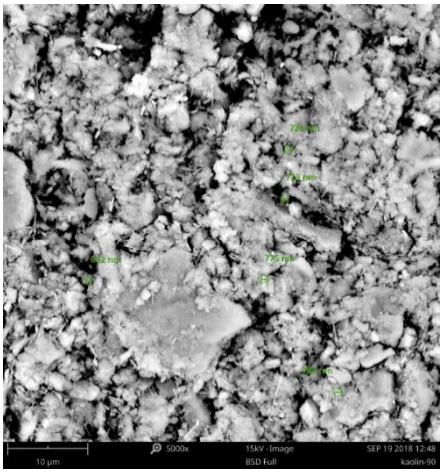

Figure 8: Scanning Electron Microscopy of kaolinite material morphology after 90 minutes of treatmen.

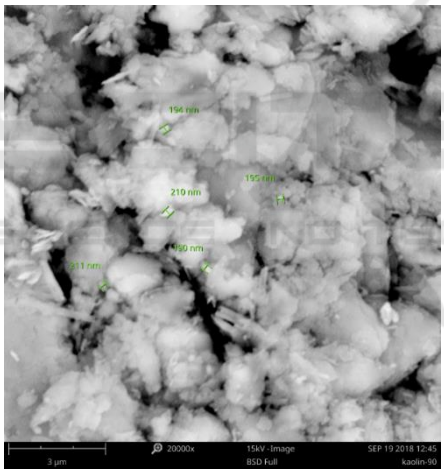

Figure 9: Scanning Electron Microscopy of kaolinite material grain size after 60 minutes of treatment.

4 CONCLUSIONS

Kaolinite particle powder was successfully synthesized from thick kaolin solution by shacking and milling of power metallurgy process. The shacking-milling mechanism was a crucial step for generating the quality product of particle. The initial grain size of the untreated sample range is 2.24µm to 660nm. Furthermore, nanoparticles appear in large clusters start to disperse within kaolinite particles with a better homogeneity after 90 minutes of treatment. As the result, the grain size diameter has been reduced to 190nm. The by-product initial nanoparticle hydrous alumina-silica of material formed in this process is a non-hazardous material.

Thus, we can convey the process followed in this paper is an accessible, affordable and environment-friendly method for kaolinite particle synthesis from Indonesia kaolin. The modification of particle grain size is possible into a nanoscale by employing shacking and milling powder metallurgy process. A significant of grain size reduction was acquired in all cases.

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