Influence of high energy milling time on nano-quartz structure from West Sumatera

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Abstract. Mineral silica is widely available in nature. Silica in nanometer size has a high economic value in the world steel industry. Methods that can be implemented in making nanomaterial one of them is High Energy Milling 3D Motion. The purpose of this study was to determine the effect of grinding time on nanostructures in the form of quartz crystal structure and particle size. The results showed that for grinding time 6, 12, 24 hours and 30 hours decreased the size of quartz particles. The size of the quartz particles was consecutively equal to 464 nm, 380 nm, 345 nm, and 293 nm with hexagonal structures.

1. Introduction
Silica is a mineral found in quartz sand mining. Quartz sand mining is commonly found in West Sumatra, Indonesia [1]. Silica is a type IV metal oxide which has good abrasion resistance, high insulators and high thermal stability. Stability. Silica has two structures: amorphous silica and crystals. The structure of quartz atoms is tetrahedral, a silicon atom surrounded by 4 oxygen atoms [2]. The content of silica minerals such as quartz, cristobalite, and trydrite, has many applications in technology especially if it is nanometer-sized (smaller than 100nm). The nanometer-sized materials have a number of chemical and physical properties that are superior to bulk materials. Nanomaterials have the advantage of being readily absorbed, larger touch surface areas [3], and anti-corrosion [4].

Many methods can be used to create nano-sized materials. Each method has its advantages and disadvantages [5][6][7][8]. One method for producing materials in nano sizes is the high energy milling method. High energy milling is a unique technique using the collision energy between the crushing ball and the wall of space that is rotated and driven in a certain way. The qualified nanoparticles depend on, among others, the time of milling [9][10][11]. In other words, knowledge of optimum milling time will provide information about the structure, size and characteristics of the surface and bulk of nanoquartz particles which impact on its application in the industry. In this paper, we focus on the effect of high energy milling time on the nanoquartz structure.

2. Research Method

2.1. Preparation of manganese Powder
Silica powders were prepared by high energy milling method using natural quartz sand from daerah Tanah Datar, Sumatera Barat, Indonesia (88.9% SiO) as starting material. The tool used for the preparation of silica is the Ball Mill High Energy Milling Ellipse 3D Motion (HEM-E 3D). Variations of milling time used are 6, 12, 24 and 30 hours.
2.2. Characterization

The Silica oxide samples were characterized by an X-ray powder diffractometer (XRD, CubiX3Cement) using Cu-Kα radiation (λ = 1.5406 Å) at 45 kV and 40 mA and were used to determine the identity of any phase present and their crystallite size. The crystalline size was calculated using the Scherrer formula, \( D = \frac{0.9 \lambda}{\beta \cos \theta} \), where \( \lambda \) is the wavelength of X-ray radiation, \( \beta \) is the full width at half maximum (FWHM) of the peaks at the diffracting angle \( \theta \) \([12][13]\). The surface morphology of the quartz was obtained using scanning electron microscopy (SEM). The stages of the research are summarized in figure 1.

![Diagram](image)

**Figure 1.** The stages of the research

3. Results and discussion

3.1. Quartz Structure

Figure 2 shows the XRD pattern of samples prepared with grinding time at 6 hours and room temperature (26°C). The results of the diffraction pattern showed silica samples were dimilled with a time of 6 hours mineral quartz periclase minerals, cristobalite, and brucite. The peak on the Quartz XRD pattern is compared with the standard \([11]\). The XRD pattern of quartz formed at 20.89, 26.66, 36.56, 39.48, 40.30, 42.46, 45.81, 50.15, 54.89 and 59.97°. Quartz Structure for hexagonal.

![Graph](image)

**Figure 2.** Spectra XRD of silica with milling time at 6 hour
Figure 3 shows an XRD pattern of samples prepared with milling time at 12 hours. The results of the diffraction patterns showed in the sample of silica, mineral periclase, cristobalite, and brucite, increasingly disappearing from the sample. Quartz Structure for hexagonal.

![XRD Pattern of Silica with milling time at 12 hours]

**Figure 3.** Spectra XRD of Silica with milling time at 12 hour

Figure 4 is a plot of intensity against the 2 angles of theta (2θ) of the X-ray diffraction pattern of the silica samples being dimilled for 24 hours. The results of diffraction patterns show that the highest peak is dominated by quartz minerals. While the other peak appears mineral periclase the longer milling time then the peak will be lost. Quartz Structure for hexagonal.

![XRD Pattern of Silica with milling time at 24 hours]

**Figure 4.** Spectra XRD of Silica with milling time at 24 hour

Figure 5 is a plot of intensity against the 2 angles of theta (2θ) of the quartz X-ray diffraction pattern of 30-milling results seen that the highest peak is dominated by quartz minerals in which no other mineral appears. Quartz Structure for hexagonal.

![XRD Pattern of Silica with milling time at 30 hours]

**Figure 5.** Spectra XRD of silica with milling time at 30 hour
Based on X-ray diffraction pattern on silica powder with milling time variation it can be seen that at milling time 30 hours obtained pure mineral quartz. Uncontaminated with other minerals. In addition, the increase in milling time will decrease the intensity of mineral quartz. The decrease in intensity due to the smaller particle size, but not affect the structure of mineral quartz, for hexagonal. In addition to the decrease in intensity, the widening of the curve also occurs at any time the milling is done. The decrease in intensity due to changes in crystal size, this change not results in the crystal structure of the shaping element changed [9]

3.2. Morphological characteristics

The surface morphologies of quartz with milling at different time were examined by SEM technique. Figure 6 shows a typical SEM image of quartz at milling time from 6, 12, 14, 30 hour with 33,000x magnifications. The SEM images indicated nearly spherical quartz

![SEM images of nanoquartz at milling time(a). 6 hour; (b).12 hour; (c).24 hour; (d).30 hour; with 33,000x magnification](image)

**Figure 6.** SEM images of nanoquartz at milling time(a). 6 hour; (b).12 hour; (c).24 hour; (d).30 hour; with 33,000x magnification

The SEM results also show that the particle size is getting smoother as the milling time increases (Table 1). Grinding time is 6 hours, the average particle size is 464 nm. At 12 hours of grinding, the average particle size was reduced to 380 nm. Furthermore, for a 24-hour grinding period, the particle size becomes 345 nm. At milling time 30 hours the average particle size is 293 nm. Based on the quartz mineral morphology form, for a 6-hour grinding time, the shape is round but not homogeneous. At the time of the 12-hour grinding, the shape begins to be slightly homogeneous. At the time of
grinding 24 hours, there is little merging between particles. At 30 hours of milling, the quartz mineral is in the form of a homogenous round.

**Table 1.** SEM Data Analysis of Milling Results Against Particle Size

| Samples | Milling Time (hours) | Avg. Particle Size (nm) |
|---------|----------------------|-------------------------|
| 1       | 6                    | 464                     |
| 2       | 12                   | 380                     |
| 3       | 24                   | 345                     |
| 4       | 30                   | 293                     |

4. Conclusion

In short, silica powder from natural minerals is synthesized by using high energy milling. The quartz mineral crystal structure does not depend on milling time. The milling time variation produces the same hexagonal quartz crystal structure. Increased milling time causes morphological changes and quartz particle size to become smaller. Because the particle size of the quartz is still in the order of hundreds of nanometers, it is advisable to continue milling by increasing the milling time, to obtain a quartz particle size below 100 nm.

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