

Characterization of Silica Sand Due to The Influence of Calcination Temperature

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Abstract. The characterization of natural silica from *Tanah Datar* region has been done due to variation of calcination temperature. Characterization is done using X-Ray Fluorescence (XRF) and X-Ray Diffraction (XRD). The results of the XRF test show that the content of silica content is 88.957%. Based on XRD test it is found that temperature increase causes structural changes in natural silica. At a temperature of 26 ° C the silica has a quartz phase and in the temperature range of 800 ° C to 1000 ° C there is a new phase, cristobalite. As for the temperature of 1100 °C appears a tridymite phase. Furthermore, the higher the given temperature causes of the crystal size of the crystal be small. In addition to phase and crystal size, there is also a change of lattice parameters.

1. Introduction

Silica (SiO₂) has wide application in various technology applications. Utilization of silica is most widely used for raw materials manufacture glass and glass industry, cement mixture, detergent manufacture, welding coating, cleaning and electrolyte. The last few years the utilization of silica and calcium is made in nano size into bioactive materials for repair of bone tissue. Other applications in the industry are for the production of pigments, pharmaceuticals, ceramics and catalysts, waterglass manufacture, as welding coatings, cleaning, electrolysis, hard cement manufacture, heat-resistant cement, building cement and acid-resistant cement [1,2,3].

Each country has a different silica concentration. The difference is in accordance with the state of the natural structure of each country. Quartz sand known as white sand is one of the weathered rocks that contain the main mineral of silica. Thus, the natural state and weathering processes that occur is one cause why the concentration and silica structure is different.

Materials that are heated to a certain temperature will affect the structure of the material. When a material is heated at a fixed rate of warming there will be a physical change such as phase change [4]. Silica has various crystalline forms such as α -quartz, β - quartz, α -cristobalite, β -cristobalit and tridymite [5]. Other forms are stishovite and fluorite, but rarely appear. With the many structures that have made silica widely used in the industrial world.

Temperature plays an important role in the structural change of silica. At room temperature ($\pm 28^\circ$ C) the silica will be amorphous crystals. At 200-300°C temperature, the cristobalite phase is seen. At a temperature of 163°C, the tridymite phase is observed. Silica glass has an amorphous crystalline structure at a temperature of 500-600°C [3]. At temperatures of 800-900° C, it is quartz and when the temperature is increased to 1000 ° C it will change to tridymite and critobalite. This structure difference is due to the presence of a silica-forming material that depends on the occupied region.



Temperatures in addition to causing a change in phase also cause changes that occur in crystal size and lattice parameters.

This article discusses the structural changes of natural silica due to variation of calcination temperature. This information is important to know the emergence of new phases in the valuable silica economically at a certain temperature.

2. Methods

2.1. Sample Preparation:

The sample used is silica sand material obtained from *Tanah Datar* District, Indonesia. The resulting silica sand is separated from the impurities of the garbage, before being smoothed the quartz sand first, the quartz sand smoothing is done using a bowl mill. After smoothing the sieving is done with an automatic sieve. The sample is heated using the furnace. The crushed quartz sand is heated using 800°C, 900°C, 1000°C, 1100°C temperatures with a duration of 2 hours.

2.2. Characterization

The refined silica sand is characterized using XRF with type Epsilon 3 to determine the content and the element content of the quartz sand. Then the heated sample was characterized using XRD Type CubiX3 Cement (CuK α 1,5606 Å, 40 mA, 45 kV) to determine the characteristics of the silica oxide in terms of the phase and crystal structure that emerged. The process of identifying phase and structure is obtained from search match using high score plus program. The crystal size was calculated using the Scherrer formula, $D = 0.9 \lambda / \beta \cos \theta$, where λ was the wavelength of X-ray radiation, β was the full width at half maximum (FWHM) of the peaks at the diffracting angle θ [6].

3. Results and Discussion

3.1 Silica Sand Concentration

The result of the measurement obtained from X-Ray Fluorescence (XRF) on silica sand from *Tanah Datar* District, Indonesia is in the form of peak intensity of element with potential energy. The XRF test results show that the silica concentration is 88.957%. The silica content of this area is different from other regions due to different natural conditions and weathering processes and the amount of impurities in the soil and rock layers of the area [7].

3.2 Effect of temperature on silica structure

3.2.1 Phase

Based on XRD data analysis for each sample it can be seen that the variation of the calcination temperature influences the natural silica phase that is formed. The silica phase formed for each temperature is shown in Figure 1.

Figure 1 illustrates the effect of temperature on changes in the natural silica phase. At temperature 26 °C silica is quartz. At a temperature of 800 °C there appears a new phase of cristobalite beta at an angle of 50.40 with a hkl [331] and an angle of 64.10 with a hkl plane [034]. At a temperature of 900 °C there appears to be a change in the silica phase in which the crystal quartz is missing or does not appear. Temperature 1000°C visible cristobalite beta phase appears at an angle of 43.13 with the hkl plane [222]. When the temperature of 1100 °C occurs the addition of a new phase of tridymite alpha with hkl [311] and re-emerges the quartz crystal field [022] in the diffraction pattern. To see the phase formed can be seen in Table 1.

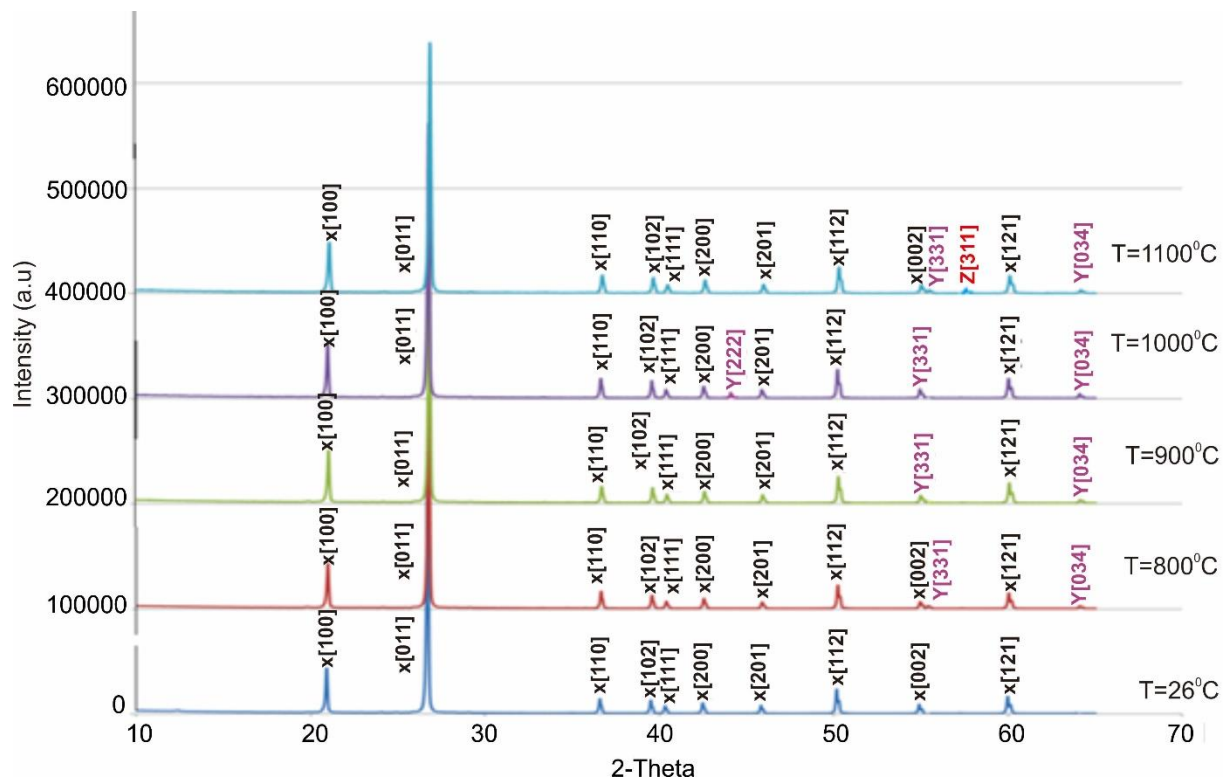


Figure 1. Silica phase with temperature variation

Table 1. The silica phase formed on each sample

Temperature	Quartz phase	Cristobalite phase	Tridymite phase
26 °C	√	-	-
800 °C	√	√	-
900 °C	√	√	-
1000 °C	√	√	-
1100 °C	√	√	√

In Table 1, the quartz phase appears at each temperature. For the cristobalite phase begins at Temperature 800 °C, the tridymite phase appears only at 1100 °C. In accordance with the silica phase diagram, the beta tridymite will appear at elevated temperatures. Temperature can affect the growth of silica crystals, such as the emergence of new phases. The effect of temperature on phase change on silica can be seen from figure 2. At room temperature of 26°C phase that appears only quartz phase. This phase arises because at room temperature the silica is in an amorphous state [5]. At normal temperatures quartz will be in quartz trigonal structure (α -quartz) and then turn into hexagonal β -quartz at temperature 573 °C. In this case, the emergence of new phase began to be seen at 800°C temperature in the presence of cristobalite phase at the angle of 55.4° and 64.1° as well as the temperature 900°C. At a temperature of 1000°C arise tridymite. Based on the phase diagram of silica, at temperature 1000°C will appear tridymite. In certain circumstances tridymite does not appear at low temperatures because tridymite is not an original form of β -quartz. So that tridymite can be replaced by β -quartz [1]. Silica under atmospheric pressure has three crystalline forms: quartz, tridymite and cristobalite. Tridymite appears at temperatures of 870°C to 1470°C [5].

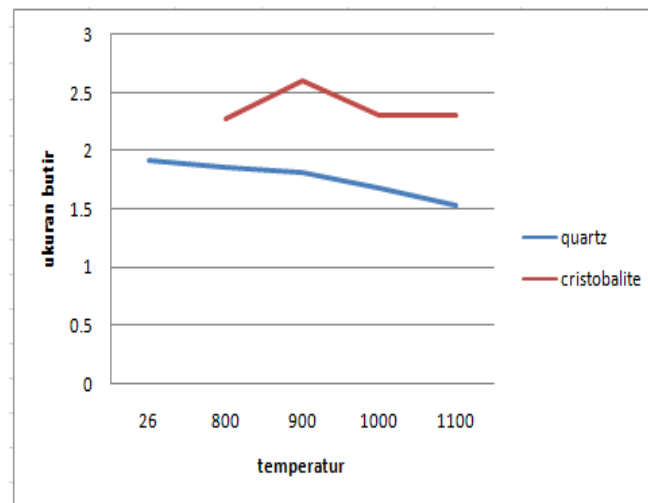


Figure 2. Relation temperature and crystal size

Figure 2 shows only two silica minerals quartz and cristobalite, whereas tridymite is not clearly influenced by temperature because tridymite only appears at 1100°C only. Quartz crystal size can be seen that its value decreases with increasing temperature. For cristobalite the size of the crystal is raised at 900°C and then decreases as the temperature is raised.

Seen in Figure 2 the size of the quartz crystals decreases as the temperature increases, the higher the given temperature the greater the FWHM value the smaller the crystal size becomes. The higher the intensity formed the smaller the crystal size of the crystal. This is reinforced by the X-ray diffraction pattern where as the temperature of the diffraction peak increases, it is higher and sharper. The cause of temperature affect the crystal size is due to rising temperatures so the diffusion of atoms is also increasing, thus increasing the formation of the same new phase nuclei. Between these nuclei there is a crystal boundary that separates it and is separated by pores. Temperature is continuously erected, causing the reduction / removal of the pores tersebut. When the pores are so small they can no longer lock the crystal boundaries against the effect of the crystal growth forces [4]. Increasing heating temperatures result in smaller crystal size.

3.2.2 Lattice parameters

In the XRD measurement data that has been matched with JCPDF data, the composite phase of quartz sand is obtained. It also obtained a crystal structure which includes lattice parameters (α , β , γ , a , b , and c), group space, crystal system contained in silica minerals from quartz sand. Changes in crystal structure due to temperature variation can be seen in Table 2 for quartz silica minerals.

Based on Table 2 it is seen that the lattice parameters of silica with temperature variations. Where at temperature 26°C the lattice parameter of quartz $a = b = 4,9124 \text{ \AA}$, $c = 5,4039 \text{ \AA}$ with hexagonal crystal form, for lattice parameter of cristobalite $a = b = c = 7,4170 \text{ \AA}$ with cubic crystal form. At 800 °C the lattice parameters of quartz $a = b = 4,9019 \text{ \AA}$, $c = 5,3988 \text{ \AA}$, with hexagonal crystal form, for lattice parameters of cristobalite $a = b = c = 7,2679 \text{ \AA}$ with cubic crystal form. At 900°C the lattice parameters of quartz $a = b = 4,8915 \text{ \AA}$, $c = 5,3885 \text{ \AA}$, with hexagonal crystal form, for lattice parameters of cristobalite $a = b = c = 7,2679 \text{ \AA}$ with cubic crystal form. At temperature 1000°C, the lattice parameters of quartz $a = b = 4,900 \text{ \AA}$, $c = 5,400 \text{ \AA}$. At the temperature of 1100°C the lattice parameters of quartz $a = b = 4,8715 \text{ \AA}$, $c = 5,3885 \text{ \AA}$, lattice parameters of cristobalite $a = b = 5,1190 \text{ \AA}$, $c = 7,1683 \text{ \AA}$ with tetragonal crystal form whereas for tridymite the lattice parameter is $a = 18,4940 \text{ \AA}$, $b = 4,9910 \text{ \AA}$ and $c = 25,832 \text{ \AA}$ with its monoclinic crystal form. Increased temperatures cause the phase change of a material, it also causes the structure of the material also to change. The structural changes that occur are hexagonal structured quartz phases with lattice parameters $a = b = 4.9124 \text{ \AA}$, $c = 5.4039 \text{ \AA}$ which dominantly shrinks as temperature increases. Likewise with cristobalite lattice parameters that begin $a = b = c = 7.4170 \text{ \AA}$ tends to decrease with increasing temperature.

Table 2. The lattice parameters of silica to temperature

T (°C)	Phase	Lattice parameter						Space group	Crystal system
		a (Å)	a (Å)	a (Å)	α	β	γ		
26	Quartz	4.9124	4.9124	5.4039	90	90	120	P3221	Hexagonal
	Cristobalite	-	-	-	-	-	-	-	-
	Tridymite	-	-	-	-	-	-	-	-
800	Quartz	4.9124	4.9019	5.3988	90	90	120	P3121	Hexagonal
	Cristobalite	7.2679	7.2679	7.2679	90	90	90	P213	Cubic
	Tridymite	-	-	-	-	-	-	-	-
900	Quartz	4.8915	4.8915	5.3885	90	90	120	P3221	Hexagonal
	Cristobalite	7.2679	7.2679	7.2679	90	90	90	P213	Cubic
	Tridymite	-	-	-	-	-	-	-	-
1000	Quartz	4.9000	4.9000	5.4000	90	90	120	P3221	Hexagonal
	Cristobalite	7.2679	7.2679	7.2679	90	90	90	P213	Cubic
	Tridymite	-	-	-	-	-	-	-	-
1100	Quartz	4.8715	4.8715	5.3885	90	90	120	P3221	Hexagonal
	Cristobalite	5.1190	5.1190	7.1683	90	90	90	P41212	Tetragonal
	Tridymite	18.494	4.9910	25.832	90	117	90	Cc	Monoclinic

4. Summary

In Summary, the concentration of silica from *Tanah Datar*, Indonesia is 88.957%. Increased calcination temperature affects the structure of natural and emerging phases of silica. At a temperature of 26 °C the silica has a quartz phase and in the temperature range of 800°C to 1000°C there is a new phase, cristobalite. As for the temperature of 1100°C appears tridymite phase. In addition, the increase in calcination temperature affects the size of the crystalline silica. The higher the given temperature the smaller the crystal size of the crystal. In addition, there is also a change of lattice parameters of quartz phase hexagonal structures with lattice parameters $a = b = 4.9124 \text{ \AA}$ $c = 5.4039 \text{ \AA}$ which dominantly becomes smaller as temperature increases. Likewise with cristobalite lattice parameters that begin $a = b = c = 7.4170 \text{ \AA}$ tends to decrease with increasing temperature.

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