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Zeolite-Based Silica Synthesis With Calcination Temperature Variations 100°C, 150°C and 200°C

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Abstracts

This research on Zeolite-Based Silica Synthesis with Calcination Temperature Variations 100 °C, 150 °C, and 200°C. The purpose of this study was to determine the effect of calcination temperature on the silica structure contained in zeolites. Synthesis is made from zeolite, NaOH and HCl by sol-gel method and temperature calcination treatment 100 °C, 150 °C, and 200°C. Zeolite samples were characterized by XRD *(X- Ray Diffraction).* The results showed that sample A with temperature 100°C contains Quartz silica, moganite silica (SiO_2), and Aluminum Oxide (Al_2O_3). Sample B with temperature 150 °C contains Quartz silica (SiO_2), and Anorthite ($AlCaO_8Si$). Sample C with 200°C contains Quartz silica and Aluminium Oxide (Al_2O_3). As for the crystallite size in sample A measuring 678.51 nm, sample B measuring 488.16 nm and sample C measuring 488.13 nm.

Keywords: Zeolit, Silica and XRD *(X-Ray Diffraction)*

1. Introduction

Zeolites were first discovered by Freiherr Axel Cronstedt, a mineralogist from Sweden in 1756. Zeolites have been known for more than 200 years, where the European community at that time used them for jewelry and were collected in various museums. Zeolite is a term that comes from the Greek word Zeo which means foam and lythe (stone) which means stone.

Zeolite is an aluminasilicate in the form of a three-dimensional crystal structure with a uniform pore size. Zeolite has unique properties, which is porous and can act as a cation exchanger. Zeolites contain Na, K, Mg, Ca, and Fe ions as well as water molecules. In addition there is also a sillica (Si) content. Previously zeolites were incorporated into clay rock types or silicate minerals. However, starting in 1984, zeolites were classified as a separate mineral type. Clay rock is a type of alumino silicate mineral, but this rock has a layered structure and its ion exchange is caused by hydroxyl groups, with H ions being replaced by other ions. Meanwhile, zeolite ion exchange is caused by the inclusion of Al isomorphs in tertrahedra Si. All Al atoms in zeolites are tetrahedral in shape, however, in clay rocks it is octahedra.

Zeolites can be divided into two, namely natural zeolites and synthetic zeolites. Natural zeolites are found around volcanic areas (sedimentary rock areas). Besides that, it can also be found in the area around hot springs. Zeolite that comes from one area, may have the same texture and color as another area, but it still has a different chemical composition. This is due to different environmental hydrothermal conditions.

Synthetic zeolites can be found in chemical stores. This zeolite is more widely used than natural zeolite because it has a high level of purity and uniform pore size. The material often used to make synthetic zeolite is kaolin. Kaolin is a type of clay which contains silica (Si) and alumina (Al).

Silica is a chemical compound with the molecular formula SiO2 (silicon dioxides) which can be obtained from mineral silica, vegetable and crystal synthesis. Mineral silica is a compound that is commonly found in mining or mining materials in the form of minerals such as quartz sand, granite, and phosphar containing silica crystals (SiO2). Silica is formed through strong covalent bonds and has a structure where four oxygen atoms are bonded in a tetrahedral angle position around a central atom, namely the silicon atom. In general, silica is in the form of amorfterhydrous, however, if combustion continues at temperatures above 650 °C, the crystallinity level will tend to increase with the formation of quartz, crystobalith, and triymite phases.

Silica synthesis can be carried out chemically using zeolite minerals. The results of silica synthesis are influenced by variations in materials, catalyst activity, method, temperature used and so on. So that silica has a small and uniform pore structure.

One method that can be used for silica synthesis is sol gel. The sol gel process is a process which can be described as the formation of an oxide network through a progressive polycondensation reaction of precursor molecules in a liquid medium or a process to form a material through a sol, gel from the sol and finally the removal of the solvent. Sol is a colloid suspension whose dispersed phase is solid (solid) and the dispersing phase is liquid (liquid).

One of the characteristics of the silica structure can be done using XRD (X-Ray Dirfraction). Characterization was performed using XRD (X-Ray Diffraction). XRD is used to determine the structure of a material, in this research it is silica. The material can be distinguished whether it has crystalline or amorphous properties.

The principle of the XRD (X-ray powder diffraction) device is that the X rays produced from a certain metal have a certain wavelength, so that by varying the angle of reflection, an elastic reflection can be detected. So according to Bragg's Law the distance between atomic planes can be calculated by the resulting diffraction data at certain angles.

Based on this background, the study was conducted to determine the effect of calcination temperature on the silica structure contained in zeolites using the sol-gel method. With variations in calcination temperature 100 °C, 150 °C, and 200°C.

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2. Experiments Procedure

This research was conducted from November 2017 to January 2018 at the Material Physics Laboratory of the Walisongo State Islamic University Semarang and the UNNES Physics Laboratory.

The material used in this research is zeolite obtained from the main sari shop at Jalan KH Agus Salim Blok B3, shop Jurnatan, Purwodinatan, Central Semarang. 4M NaOH, 1M HCL demineralized water. The equipment used in this research is a hot magnetic stirrer, oven, furnace, beaker glass, sieve, digital balance, ceramic plates, pH paper, filter paper, drop pipette, mortar, 200 mesh sieve.

The sample was mashed using a mortar and sieved using a 200 mesh sieve. Then synthesized using the sol gel method. The steps of the sol gel method are 40 grams of zeolite added 320 ml of NaOH 4M and stirred until homogeneous so that it becomes a solid sodium silicate. The sodium silicate solid is refurned at 500˚C for 30 minutes until the solid is whitish brown. The residue that has been obtained is dissolved in 400 ml of demineralized water for 3 nights to form a sodium silicate solution. The solution that has been formed is then filtered using filter paper to separate the insoluble brown precipitate. The resulting sodium silicate solution is brownish yellow.

The sodium silkat solution was then titrated using 1M HCL drop by drop using a magnetic stirrer until the pH was neutral. The neutral PH solution was left to rest for 72 hours. The formed gel was washed with demineralized water and the residue was calcined in an oven with temperature variations of temperature 100 ℃, 150 ℃, and 200℃ for 2 hours. The silica powder formed is smoothed. Then the silica powder was characterized using an XRD (X-Ray Diffractometer) to determine the type of silica structure.

3. Result and Discussion

The samples in this study were characterized using XRD (X-Ray Difraction). XRD is a tool used to characterize the crystal structure, the crystal size of a solid material. When analyzed using XRD, all materials containing certain crystals will reveal specific peaks. In this study, 3 samples were used with variations in calcination temperature, namely 100 ℃, 150 ℃, and 200 ℃. Following are the results of sample characterization using XRD.

Based on XRD results, sample A with a temperature of 100 ℃ has several contents, the first is Quartz silica (SiO_2). When shot with Cu-Ka of 1.54 A at an angle of 2 Tetha (2 θ) of 26.49 ° , d of 3.3644, I / Ic of 4.76 resulting in an FWHM value of 0.002041 rad. Second, moganite silica (SiO_2). When shot with a Cu-Ka of 1.54 A at an angle of 2 Tetha (2θ) of 26.47 °, d of 3.3670, I / Ic of 2.17 produces an FWHM value of 0.003501 rad. The third is Aluminum Oxide (Al2O3). When shot with Cu-Ka of 1.54 A at an angle of 2 Tetha (2θ) of 34.71 °C, d of 2.5831, I / Ic of 0.86 results in an FWHM value of 0.009338 rad.

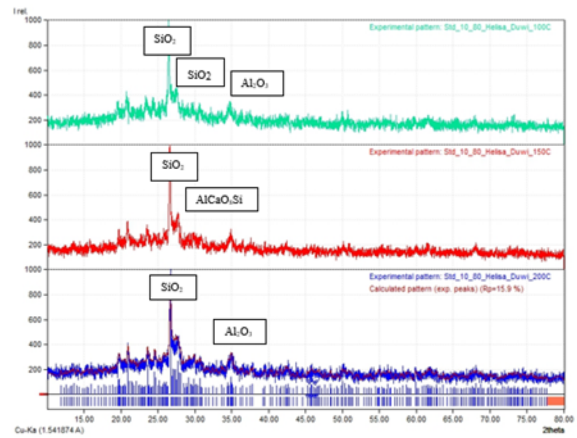

Figure 1. XRD's result

In sample B with a temperature of 150 ℃ has several contents, the first is silica Quartz (SiO_2). When shot with a Cu-Ka of 1.54 A at an angle of 2 Tetha (2 θ) of 26.68 ° , d of 3.3417, I / Ic of 0.9950 results in an FWHM value of 0.002918 rad. Second, Anorthite ($AlCaO_8Si$). When shot with Cu-Kas of 1.54 A at an angle of 2 Tetha (2θ) of 28.11 °, d of 3.17, I / Ic of 0.63 produces an FWHM value of 0.001751 rad.

In sample C with a temperature of 200 ℃ has several contents, the first is Quartz silica (SiO_2). When shot with a Cu-Ka of 1.54 A at an angle of 2 Tetha (2 θ) of 26.65 ° , d of 3.3435, I / Ic of 4.73 results in an FWHM value of 0.002918 rad. Second, Aluminum Oxide (Al_2O_3). when shot with Cu-Ka of 1.54 A at an angle of 2 Tetha (2θ) of 34.91 °, d of 2.5716, I / Ic of 0.86 results in an FWHM value of 0.004668 rad.

The data above shows that the three samples contain Quartz silica (SiO_2). Even in sample A with a temperature of 100, it contains silica moganite. However, in the three samples there were still contents other than silica, among others Al_2O_3 and $AlCaO_8Si$.

From the XRD graph with the highest peak of SiO2, the crystallitesize can be measured using the following equation

$$t = \frac{0.9\lambda}{\beta_{hkl}\cos\theta_{hkl}}$$

The following is a table for calculating the crystal grain size of the three samples

Table 1. Calculation of the crystallite size value

No	Suhu (°C)	2θ	λ	$FWHM(ra$	t (nm)
A	100	26.49	1.54	0.002041	678.51
B	150	26.68	1.54	0.002918	488.16
C	200	26.65	1.54	0.002918	488.13

The table above explains that an increase in temperature affects the crystal grain size. The greater the temperature, the smaller the crystal grain size.

4. Conclusion

Based on the research that has been done, it can be concluded that the three samples contain quartz silica with different crystal grain sizes. In sample A measuring 678.51 nm, sample B measuring 488.16 nm and sample C measuring 488.13. The greater the temperature, the smaller the crystal grain size.

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