

Alumina-silica Synthesis Uses Glass Waste as Silica Source: Hydrothermal Time Variations

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Abstract: Synthesized a silica-alumina material where glass waste as the silica source and Tetra-ethylorthosilicate (TEOS) as the supporting silica source has done. The Al source as aluminum hydroxide $Al(OH)_3$ and cetyltrimethylammonium bromide (CTAB) as a structure directing agent. The synthesis of silica-alumina material was carried out by mixing sodium silicate, TEOS, sodium aluminate, CTAB surfactant, and distilled water, then stirring using a magnetic stirrer for ± 30 minutes to produce a gel. The gel obtained was then heated at a temperature of $100^\circ C$ with varying heating times of 48, 96 and 144 hours, then aged for 2 days. The solid washed using distilled water and dried, then the solid was calcined at a temperature of $550^\circ C$ for ± 5.5 hours. The final products were characterized by FTIR, XRD and Nitrogen gas adsorption methods using the BET equation. Based on data analysis, it can be concluded that increasing the hydrothermal time actually reduces the product crystallinity and porosity.

Keywords—glass waste; hydrothermal time; silica-alumina; crystallinity

1. INTRODUCTION

As the use of glass equipment increases, more glass waste is produced. Due to the glass is brittle, it can easily break and can no longer be used. Glass is a material composed of components that are not easily degraded by microorganisms, thereby triggering environmental damage. Reduction the impact on environmental damage, glass waste can be processed into new materials that have better usability. One of them is used as a source of silica in the synthesis of silica-alumina materials. In previous research, the use of glass waste to synthesize zeolites was developed [1] [2]. Apart from that, glass waste has also been used as raw material to synthesize geopolymers [3].

The use of surfactants in the synthesis of porous materials is important because they are responsible for the formation of pores in the material. Cetyltrimethylammonium Bromide (CTAB) is a cationic surfactant which acts as a structure-directing agent [4][5][6]. Synthesis parameters that are generally studied are hydrothermal time and heating temperature, because these factors influenced the stability and crystallinity of the product.

Various studies have been carried out regarding the synthesis of silica alumina. Bao et.al [7] have been successfully synthesized mesoporous silica-alumina with enhanced specific surface. Mesoporous silica-alumina with enhanced Brønsted acidity have been successfully synthesized via a novel acetic acid-based sol-gel process [8][9]. Nurlela et al. [10] have synthesized zeolite material from TEOS as the silica source and CTAB surfactant as the pore forming agent. Synthesized mesoporous silica (MPS) material based on sodium silicate as a silica source and CTAB surfactant as a pore forming agent with variations in

hydrothermal time (6 hours, 24 hours and 72 hours) by Ertan [11]. In this research, the formation of mesoporous silica (MPS) material with a stable structure occurred with a hydrothermal time of 24 hours. Synthesis Rho zeolite by Mousavi [12] showed that the crystal size increases from 1.45 to 1.90 μm by increasing synthesis time from 4 to 10 days. Synthesis time is also effective on the crystal morphology. Khan et al. [13] have synthesized zeolite-A with varying heating times of 1 hour, 2 hours, 4 hours, 6 hours, 12 hours and 24 hours. The optimum heating time for the formation of zeolite-A was 24 hours. Sriatun and Ananto [14] have synthesized sodalite with various hydrothermal time. Utchariyajit et al. [15] have synthesized the mesoporous material SAPO-5 with a silica source derived from silica powder, an Al source derived from aluminum hydroxide, and Triethylamine (TEA) surfactant as a directing molecule. In this research, variations in heating time were carried out at a temperature of $190^\circ C$, namely 0.5 hours, 1 hour, 1.5 hours and 2 hours. The optimum heating time for the formation of SAPO-5 mesoporous material is 2 hours. Lee et al [16] have also synthesized zeolite A, where the Silicon and aluminum were respectively extracted from SB (sandblasting waste) and LCDWG (liquid crystal display waste glass) particles using sodium hydroxide and distilled water. One of the synthesis parameters studied is the crystallization time were carried out for 24 hours, 36 hours, and 48 hours. The crystal growth of zeolite A was more sensitive to changes induced by crystallization duration and temperature under high alkalinity conditions.

In this research, silica-alumina material was synthesized using used glass waste as a silica source, $Al(OH)_3$ as an Al source, CTAB surfactant as a pore forming agent. To determine the effect of heating time on the synthesis of silica-alumina material, variations in heating time were carried out, namely 48 hours, 96 hours and 144 hours.

2. METHODOLOGY

2.1 Materials and equipment

The materials were used glass waste, distilled water, Na_2CO_3 , NaOH , $\text{Al}(\text{OH})_3$, TEOS (tetra-orthosilicate) and CTAB (cetyltrimethylammonium bromide). Meanwhile, the equipment and instruments used were glassware, nickel crucibles, analytical balances, magnetic stirrers, hot plates, pH indicators, Teflon vessels, Whatman® 42 filter paper, ARISTON ovens, VULCAN 3-130 furnaces, AAS Perkin-Elmer 26 spectrophotometer, Thermo-Nicolet Avatar 360 FTIR Spectrophotometer, Shimadzu 6000 X-RD Diffractometer.

2.2 Preparation of Sodium Silica and Sodium Aluminate

Sodium silicate

This method refers to Sriatun et al. The glass waste is destructed to be fine glass powder is formed and sifted to 100 mesh size. Amount of 10 grams of glass powder was added 17.6 g of Na_2CO_3 , mixed thoroughly, then heated at 900°C for 5.5 hours. The solid was dissolved in 200 mL of distilled water. In the next step, the mixture was filtered to obtain sodium silicate filtrate. The dissolved silica content was determined by AAS.

Sodium aluminate

A total of 30 grams of NaOH dissolved in 100 mL of distilled water, then stirring with a magnetic stirrer at 100°C . Twenty grams of $\text{Al}(\text{OH})_3$ were added to the sodium hydroxide solution while stirring until dissolved. The mixture was diluted to 250 mL and then allowed to stand at room temperature to obtain sodium aluminate.

2.3 Synthesis of silica-alumina material

A total of 9.1 grams of CTAB surfactant was dissolved in 34.8 ml of distilled water. The mixture was stirred and heated at 60°C for 30 minutes. The resulting solution was placed in glass beaker I and cooled to room temperature. At the same time, 20.8 ml of TEOS solution was mixed with 7.2 ml of sodium silicate solution (from glass waste). They were stirred for 30 minutes. The mixture obtained was then added to the resulting glass beaker I and 12.2 ml of sodium aluminate was added. The entire mixture was stirred using a magnetic stirrer until a homogeneous mixture. When stirring, the pH is also checked at ± 10 using a pH paper indicator. If the pH obtained is too alkaline, it needed dilute to acetic acid at a concentration of 25% is added drop by drop while stirring. The stirring continues until the mixture turns into a gel phase. Next, the mixture was placed in a closed Teflon container and heated in various heating times of 48 hours, 96 hours and 144 hours at a temperature of 100°C . Then an aging process was carried out for 48 hours for each sample. The resulting solid is washed with distilled water to remove impurities. The residue/solid phasa was dried in an oven for 2 hours at 70°C and weighed

until a constant weight. Next, the calcination process was carried out in a furnace for ± 5.5 hours at a temperature of 550°C .

2.4 Material Characterizations

The final products were characterized using an FTIR spectrophotometer to determine functional groups, meanwhile the XRD to determine the crystallinity and the BET method to determine the pore size, surface area and pore volume of the material.

3. RESULTS AND DISCUSSIONS

3.1 SILICA-ALUMINA MATERIAL AS A PRODUCT OF SYNTHESIS

Synthesis of silica-alumina based on silica from glass waste using the direct method, at various heating times, namely 48 hours, 96 hours and 144 hours at a temperature of 100°C . The purpose of varying the heating time is to determine the effect of heating time on the crystallinity and pore size of each sample. The addition of CTAB surfactant aims to act as a pore forming agent in the material and is expected to produce a resulting material that has a larger and more uniform pore size.

The silica source used not only come from glass waste but also other materials are added to support the silica source, namely TEOS solution. The TEOS solution was used because the silica content from sodium silicate extraction was relatively small, it was 10324.48 mg/L or 20.648%. The small amount of silica extracted from glass can be caused by the fact that the silica content in the glass source used is small, while the content of other compounds in the glass source is thought to be quite high, such as CaO , MgO , K_2O , Na_2O . The Tetraethyl-orthosilicate (TEOS) has a high level of purity so that no purification process is required before use.

Sodium silicate is polar while TEOS is non-polar. The addition of TEOS aims to increase the silicate content in the mixture. Dissolving the surfactant Cetyltrimethylammonium bromide (CTAB) in distilled water is accompanied by stirring and heating at 60°C for 30 minutes, so that the dissolving process of the CTAB surfactant is faster without damaging the structure of the CTAB surfactant. At concentrations equal to or greater than CMC, CTAB surfactant forms micelles where the hydrophobic groups will merge with each other, while the hydrophilic groups (surfactant heads) will move away from the hydrophobic groups, thus forming a cylindrical circle. The small micelles formed will be surrounded by a silica-alumina polymer framework.

In the initial stage, the CTAB surfactant dissolved in distilled water will undergo ionization to become CTA^+ ions and Br^- ions. The hydrophilic group (surfactant head) is a positively charged ammonium group, while the hydrophobic group (surfactant tail) consists of hydrocarbon chains. The hydrophilic end and the hydrophobic end are far from each

other to form cylindrical micelles. The positively charged surfactant head will bond with the negatively charged TO₄ unit (T= Si/Al) due to electrostatic interactions. One oxygen atom from the TO₄ group bonds with the ammonium group from the CTA⁺ ion while the other oxygen atom will bond with another Si-O-Si or Si-O-Al group.

When all the ingredients are mixed and stirred continuously and the pH is adjusted, a process of homogenization of the mixture will occur, the solution becomes thick and then turns into a gel solution. The formation of the gel phase indicates a condensation polymerization process between silica and alumina to form long chains of silica-alumina. The polymerization-condensation process takes place continuously along with the interaction between CTA⁺ micelles and silica alumina and subsequently forms a silica-alumina framework. The polymerization-condensation process that occurs is the initial process of nucleation and crystal growth. At the crystal formation stage, the amorphous gel will experience a rearrangement of its structure due to heating so that an embryonic crystal nucleus can form. In this situation, equilibrium occurs between the crystal nucleus embryo, the remaining amorphous gel, and the supersaturated solution, this condition is called the metastable phase. Continuous heating process will cause the metastable phase to change into a stable phase. The stable phase is the phase where crystals form in a stable state [17]

The heating process can grow crystal embryos that have been formed in the gelation process so that they can form crystal units that have a better crystal structure. After the heating process is carried out, the next stage is the aging process for 48 hours. This process aims to organize and stabilize the structure of the silica-alumina material.

The final process in the synthesis of alumina silica is calcination which aims to decompose the CTA⁺ surfactant in the pores of the material into CO₂, O₂ and H₂O. The calcination process was carried out at a temperature of 550°C for ± 5.5 hours.

3.2 EFFECT OF HYDROTHERMAL TIME ON FUNCTIONAL GROUPS IN SILICA-MATERIAL MATERIAL

Determination of functional groups of calcined materials using FTIR at wave numbers 400-4000 cm⁻¹. The presence of bending and stretching vibration absorption areas of Si-O or Al-O indicates fingerprint areas of the silica-alumina material. FTIR spectra from each sample with variations in heating time of 48 hours, 96 hours and 144 hours can be seen in Fig. 1.

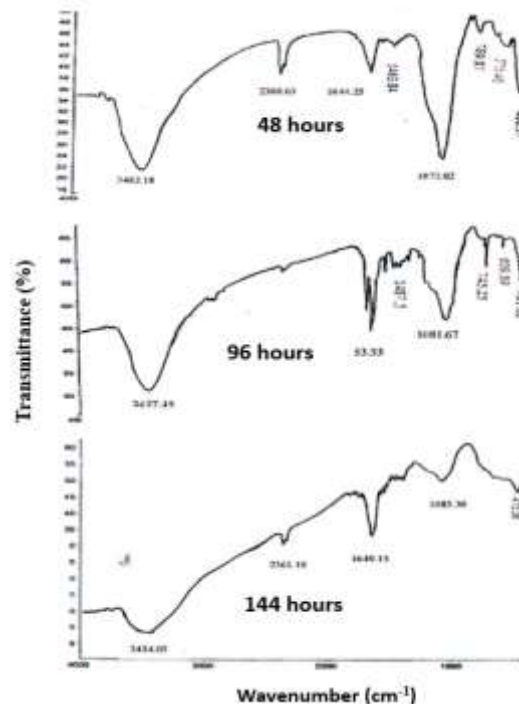


Fig. 1. FTIR spectra of silica-alumina materials at various hydrothermal time

Table 1. Intrepretation of FTIR data

Silica alumina standart	Wavenumber (cm ⁻¹)			Interpretation
	48 h	96 h	144 h	
300 - 420	466.37	481.00	472.36	Bending vibration of Si-O-Si
650 - 500	575.40	609.89	-	External linkage (double ring)
800 - 700	789.87	745.25	-	Symmetrical stretching vibration Si-O (Si-O-Si)
1250 - 900	1072.02	1081.67	1083.30	Asymmetrical stretching vibration of O-T-O
	1463.84	1487.55	-	
1645-1655	1644.25	1653.33	1649.15	Bending vibration -OH (Si-OH)
	2360.63	-	-	Stretching vibration C=N
3600 - 2500	3462.18	3457.45	345.03	Symmetrical stretching vibration -OH (Si-OH)

Fig. 1 shows the FTIR spectra of the synthesized sample. The 144 hours sample did not show the presence of a double ring at wave numbers 650-500 cm⁻¹. The double ring is an external connection between one layer and another, so that a three-dimensional structure has not been formed in the silica-alumina framework. In addition, the 144 hours sample did not show any stretching or bending vibrations from Si-O and Al-O, meaning that a silica-alumina framework had not yet been formed. Asymmetric stretching vibrations of O-T-O (T=Si/Al) in the 1250-900 cm⁻¹ region have been demonstrated by samples of 48 hours, 96 hours and 144 hours, however in this study the 48 hours sample had the lowest

transmittance indicating its absorbance was the strongest which is also supported by the presence of Si-O-Si bending vibrations, which indicates that the 48 hours sample has a better silica-alumina framework than the 96 hours and 144 hours samples. This is in accordance with research conducted by Ma et.al. [18] which states that the absorption at 1090 cm^{-1} , 790 cm^{-1} and 450 cm^{-1} indicates the existence of internal bonds in the TO_4 tetrahedral where the peak is not sensitive to changes structure.

According to Sadrara et al. [19], the presence of -OH bending vibrations and -OH stretching vibrations indicates the presence of silanol groups (Si-OH) on the defect side in the silica-alumina framework. In this study, the three samples 48 hours, 96 hours, and 144 hours contain -OH bending vibrations and -OH stretching vibrations, however the 144 hours sample has a wide absorption band so it is possible that the defect side contained in the silica-alumina framework of the 144 hours sample is greater. much more than the 96 hours and 48-hours samples. The existence of this side defect is suspected to be damage and irregularity in the silica-alumina framework. In this research, the presence of C-H groups was also found in the 48 hour and 96 hours samples in the 1463.84 cm^{-1} and 1487.55 cm^{-1} areas. The presence of C-H group vibrations indicates that the CTA^+ template molecule still remains. This is supported by the presence of a vibration wave number of 2360.63 cm^{-1} in the 48 hours sample which indicates the stretching vibration of $\text{C}\equiv\text{N}$ which is thought to originate from fragmentation of the CTA^+ compound which was not completely decomposed during the calcination process. This situation will result in the pores in the silica alumina framework remaining closed and possibly reducing the surface area and pore volume in the silica-alumina material. The calcination process which is not carried out simultaneously is thought to have an effect on the process of releasing surfactant molecules because the humidity and air pressure conditions are different at each time. Based on the FTIR analysis previously described, the 48 hours sample has a better structure than the 96 hours and 144 hours samples.

3.3 SILICA-ALUMINA CRYSTALLINITY

The diffractogram patterns of the 48 hours, 96 hours and 144 hours samples varied greatly. The diffractogram pattern of the 48 hours sample has sharper diffraction peaks compared to the 96 hour and 144 hours samples. The diffractogram pattern of the 48 hours, 96 hours and 144 hours samples can be seen in Fig. 2.

Hydrothermal time is very influential in the formation of crystals of silica-alumina material. In accordance with Figure 2, it can be seen that as the hydrothermal time increases, the crystallinity decreases. A decrease in crystallinity is possible because when a certain hydrothermal time is reached, the structure tends to become unstable.

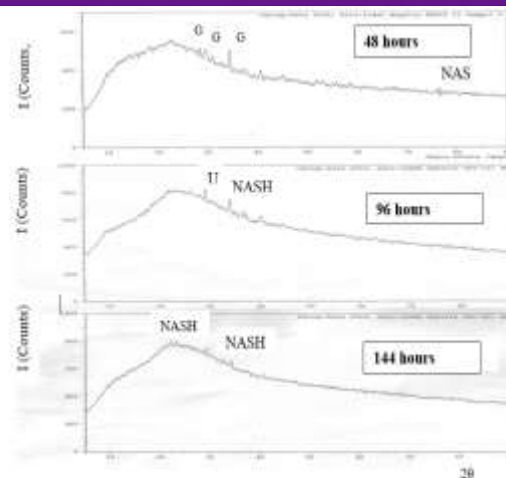


Fig. 2. The XRD pattern of silica-alumina materials at various hydrothermal time

This is in accordance with West, (1984) who states that the structure of a solid is a function of time and temperature, where at a certain time and temperature a solid will be more stable and when it passes through a certain phase its stability can change. Providing a relatively constant hydrothermal temperature of 100°C in the silica-alumina synthesis does not have much influence on the crystallinity of the resulting silica-alumina structure as the hydrothermal time increases. This is in accordance with Patwardhan (2001), who states that a temperature of 100°C still produces a silica-alumina framework that has low crystallinity. According to Warsito (2009), the use of CTAB surfactant is able to provide a uniform pore size of the silica-alumina material, resulting in good crystallinity. This was not proven in this study, the use of CTAB surfactant only had a very small effect on the formation of the silica-alumina framework. This is possible because the interaction between the CTAB surfactant and the silica-alumina polymer framework during the gelation process is not optimal, causing the crystal formation process in the silica-alumina material to not be optimal. Apart from that, using an oven as a heating medium does not have a good crystallinity effect when compared to using an autoclave. In an autoclave, the system pressure is considered, because saturated water vapor at a pressure above atmospheric pressure is used to heat the entire contents of the autoclave [20] whereas in an oven there is no continuous supply of water vapor from outside the system, so this is thought to influence the crystallinity of the material results.

3.4 IDENTIFICATION COMPOUNDS

Table 2. The value 2 theta product

Standard (JCPDS)			Samples		
Name	2 Theta (°)	Intensity (counts)	Name	2 Theta (°)	Intensity (counts)
Gmelinite (PDF No. 12-0229)	34.56	100	48 h	34.48	100
	22.44	40		22.46	41
	31.62	40		31.08	37
Sodium aluminate silicate	20.98	76	96 h	20.0	24
	49.78	13		49.61	18
	62.07	31		62.81	13
	73.06	5		73.10	9
	83.36	6		83.35	5
Ussingite (PDF No. 28-1037)	29.82	100	144 h	29.18	100
	23.13	52		22.58	52
	23.82	43		23.7	44
Sodium Aluminate Silicate Hydrate (PDF No. 38-0247)	26.60	50		26.36	51
	33.82	20		33.64	18
	30.39	50		30.18	27
Sodium Aluminate Silicate Hydrate (PDF No. 38-0247)	22.98	100		22.55	100
	29.78	11		29.37	80
	34.22	5		34.16	70

Based on the JCPDS database, Table 2 shows that the 48 hours sample has a 2θ value that is almost the same as the JCPDS standard and is identified as a mixture of Gmelinite and Sodium Aluminate Silicate (NAS) even though the diffraction pattern has a lower sharpness than the JCPDS standard. The crystal properties of Gmelinite (12-0229) are as follows: crystal composition formula $\text{Na}_4(\text{Si}_8\text{Al}_4)\text{O}_{24} \cdot 11\text{H}_2\text{O}$, Mohs scale hardness: 4.5, hexagonal & dihexagonal dipyrimal crystal system, has specific gravity: 2.04-2.17. The 96 hours sample was identified as a mixture of Ussingite and Sodium Aluminate Silicate Hydrate (NASH) with a peak sharpness pattern lower than the JCPDS standard. The crystal properties of Ussingite (28-1037) are as follows: crystal composition formula $\text{Na}_2\text{AlSi}_3\text{O}_8(\text{OH})$, zeolite group 2, pinacoidal triclinic, density 2.48 g/cc, hardness 6-7, refractive index 1.543-1.545 while the 144 hours sample was identified as the Sodium Aluminate Silicate Hydrate compound.

3.3 THE PORE STRUCTURE OF THE SYNTHESIS PRODUCT

The pore size distribution is an important aspect of adsorption and is necessary for the evaluation of porous media as well as membranes. Pore size distribution influences adsorption capacity, adsorption kinetics and adsorption selectivity, and is the basis for adsorbent synthesis. The pore

size classification is micropore ($d < 20 \text{ \AA}$), mesoporous ($20 < d < 500 \text{ \AA}$), and macropore ($d > 500 \text{ \AA}$) [21]. This classification was based on the adsorption of nitrogen gas at a normal boiling point by porous solids with a large range of pore sizes.

Data regarding the pore structure of the synthesized alumina silica solids are presented in Table 3.

Table 3. Comparison of the average pore radius, specific surface area and total pore volume of silica-alumina samples

Samples	Average of pore radius (Angstrom)	Specific surface area (m^2/g)	Total pore Volume (cm^3/g)
48 h	13.51	21.34	9.33
96 h	13.79	9.94	4.44
144 h	13.42	6.84	2.97

Table 3 shows that the pore size in each sample is not as expected, because the majority fall into the micropore category (0-2 nm or 0-20 \AA). The formation of microporous material is thought to be caused by the pore cavities not being opened evenly. Influencing factors include the calcination process not being optimal, causing the surfactant to not be completely decomposed, which results in a decrease in the surface area of the resulting material because the pores formed will be filled with compounds from the remaining surfactant. The use of a furnace system without the flow of inert gas (nitrogen) is thought to make it difficult to release surfactant molecules in the silica-alumina material so that the process of removing surfactant molecules does not take place optimally.

The three samples have similar pore sizes, but the 48 hour sample has the largest surface area and pore volume, it is possible for more pores to form in the 48 hours sample than in the 96 hours and 144 hours samples. The more pores formed, it is possible to increase the surface area and total volume of pores.

In this case, as the hydrothermal time increases, the surface area and pore volume decrease, which is not as expected. Pore formation is not only influenced by heating time, but there are other factors that also influence it, namely temperature, the higher the hydrothermal temperature, the larger the size of the pores formed, besides that, according to Ishizaki et al. [22] if the Si/surfactant composition ratio is excessive and not balanced with directing molecules will reduce the pore radius. In the analysis using the BET method, the best results were obtained for the 48 hours sample because it had a larger surface area and pore volume than the 96 hours and 144 hours samples.

4. CONCLUSIONS

The synthesis of silica-alumina material has been successfully carried out using glass as a base material. Increasing hydrothermal time causes the tendency for the crystallinity and porosity of the resulting material to decrease. The product with the best crystallinity was obtained for a hydrothermal time of 48 hours.

5. ACKNOWLEDGMENT

We thank the funding providers who have supported the implementation of this research.

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