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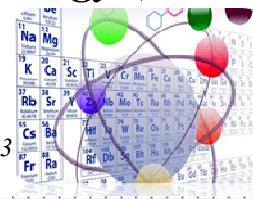
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Characterization of Silica Nanoparticles from Pumice as an Aerogel Adsorbent

Rahma Hidayah*, Desy Kurniawati, Alizar

Department of Chemistry, Faculty of Mathematics and Natural Sciences, Universitas Negeri Padang, Padang 25132, Indonesia.

*rahma.hidayah2000@gmail.com

ABSTRACT

Pumice is often found around the banks of rivers, this stone is a type of igneous rock formed from volcanic eruptions. One of the compounds contained in pumice is silica. Therefore, the synthesis of silica nanoparticles from pumice using the sol gel method was used because it is simpler and more efficient in terms of cost and processing time. The initial step was by reacting pumice powder and NaOH at 70°C - 80°C then synthesized by adding 2M HCl to form a gel or white precipitate, soaking in ethanol, toluene, hexane solutions was then synthesized to become an aerogel. Silica synthesis results into silica aerogels were characterized by FTIR and XRF. From FTIR silanol and siloxane functional groups were found, and the SiO₂ composition increased to 93,299% after synthesis.

Keywords: pumice, silica, silica aerogel, adsorbent, sol gel method

1. INTRODUCTION

Pumices is often found in Indonesia, the distribution of pumice is found in riverside areas. This stone is a type of igneous rock that is formed from the results of volcanic eruptions, this stone can also be called volcanic glass rock which comes from gas bubbles¹. Pumice contains several compounds, namely oxides, such as K₂O, MgO, CaO, Fe₂O₃ and SiO₂. which combine then form a natural composite with the highest content in silica (SiO₂) of 70.21%².

One of the compounds in this stone is silica. Silica is used by some people, namely it is used as a moisture-proof product to store goods so that they last longer. It is also developed in various fields of industry, health and so on. This silica compound will later become silica aerogel. Silica aerogel is a highly porous, hydrophobic and inert ceramic material. This stone has unique characteristics, namely high porosity

(80% to 99%) low thermal conductivity (< 0.005 W/mK) low density (0.003 g/cm³ to 0.35 g/cm³) low refractive index and high surface area (500 - 1600 m²/g) so that the silica aerogel in this stone can be applied in various industrial sectors, especially as a thermal insulator, catalyst and adsorbent³.

Silica aerogel found from pumice can be used to reduce metal content in water. Water is the main natural resource and has a very important function for human life and other living things. If the quality of the water decreases, there will be influence (influence) from the surrounding water which has been polluted, both from impurities and residual heavy metal waste. The types of metals such as cadmium (Cd), chromium (Cr), iron (Fe), copper (Cu), lead (Pb), nickel (Ni) and manganese (Mn) exceed the quality standards, from household waste. as well as industry⁴.

Many other researchers have made silica aerogels, starting from different samples, namely, silica from Lapindo mud, silica from bagasse ash, silica from pond water and silica from old newspapers⁵. Making silica aerogel also uses various variations, namely, stirring time, solution concentration and so on. The results obtained are also of good value with optimum conditions that are efficient and of economic value⁶.

In this study, it encouraged the authors to try to utilize silica compounds to become airgel but with a different sample, namely pumice originating in the Pariaman City area. It is made using the Sol Gel method, this method is used because it is simpler and more efficient in financing and processing time. In this research, several variations of NaOH concentration and optimum variation of silica aerogel were carried out. The goal is to obtain silica aerogel with an optimal shape so that it can reduce the metal content in water.

2. EXPERIMENTAL

2.1. Chemicals, Equipment and Instrumentation

The tools used Chemical glass, Erlenmeyer, Dropper pipette, Stir bar, Funnel, Mortar and pestle, Filter paper, Furnace, Sieve 150 μ m, Oven, pH meter, Crucible cup, Magnetic stirrer. The instruments used for its characterization are XRF, FTIR. The materials used are pumice, 4 M NaOH, 2 M HCl, TEOS, ethanol, hexane, aquadest.

2.2. Research Procedure

2.2.1. Pumice Preparation

The pumice is pounded and washed using distilled water, then dried in a furnace at 700°C for 4 hours. The dried pumice is then crushed, the results of the grinding are filtered through a 150 μ m sieve to obtain finer pumice powder. The powder was then washed with distilled water 4 times and dried (calcined) in a furnace at 700°C for 1 hour. This calcination aims to activate the silica from the fine powder of pumice to make it easier to extract.

2.2.2. Extraction of fine powder of pumice

Pumice fine powder was taken 10 grams, then added 100 ml of solution with various concentrations of NaOH 4M . Then it was heated with a magnetic stirrer for 1 hour with a temperature range of 70°C - 80°C . The results of heating will become mush, then filtered using filter paper and the filtrate is taken. Then the filtrate is dripped with 2 M HCl with a pH of 8 which will form a white precipitate then filtered and the filter results are washed with distilled water to obtain silica hydrogel. The silica hydrogel is then put into the

Syringe, this is used to print the silica, then in the oven with a temperature of 50°C until it reduces slightly. Remove the hydrogel from the oven, then leave it in the desiccator overnight to solidify.

2.2.3. Preparation of Silica Aerogel

Silica gel formed at the best/optimum conditions, washed with ethanol for 24 hours at room temperature to obtain alcogel results. Then the alcogel was put into a mixed solution of 5 ml of ethanol, TEOS, hexane for 24 hours. After that, it is aged for 18 hours at room temperature or in a desiccator until it solidifies. Then it is characterized using the FTIR instrument.

3. RESULTS AND DISCUSSION

3.1. Characterization of FTIR and XRF on the Synthesis of Silica Nanoparticles from Pumice

The FTIR analysis of pumice with 4M silica, it shows that the shape of the pumice curve with black color and the silica synthesis curve from 4M pumice with purple color are almost similar. This FTIR analysis was carried out in order to find out the functional groups of silica present in pumice⁷. Based on the spectrum results on silica, characterization of silica was carried out by identifying functional groups based on infrared absorption spectra data. The silanol groups (Si-OH) and siloxane groups (Si-O-Si) are active sites on the silica surface which can be used for adsorption purposes. Characterization using infrared spectroscopy aims to determine the presence of silanol groups (Si-OH), siloxane (Si-O-Si), and other groups. The results obtained on the characterization using FTIR showed good results⁸.

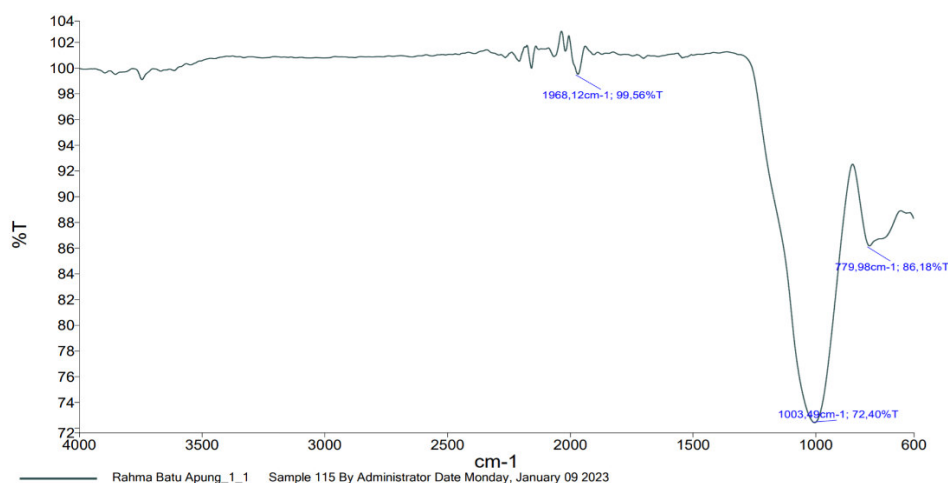


Figure 1. FTIR shows that the shape of the pumice curve with black color

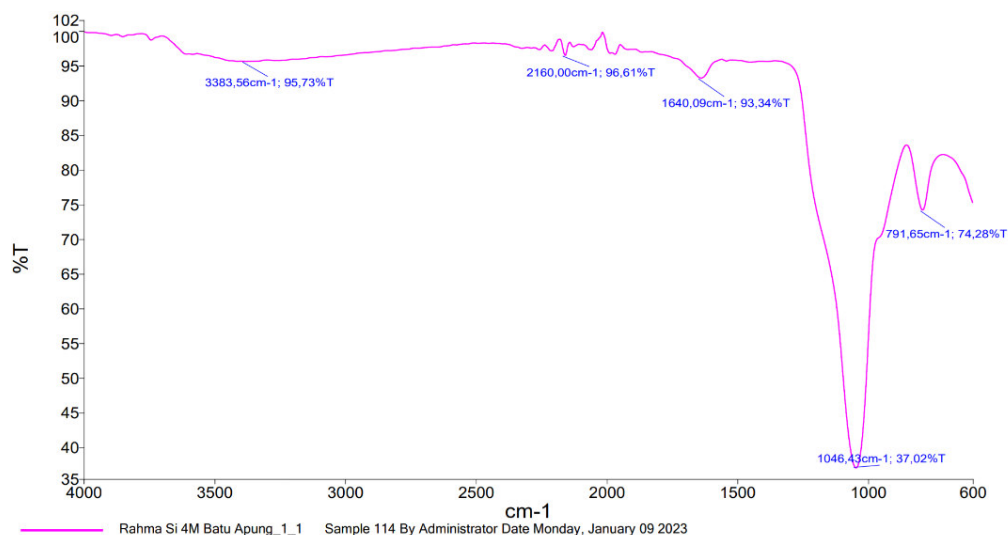


Figure 2. FTIR the silica synthesis curve from 4M pumice with purple color are almost similar

The figure you can see the FTIR spectrum data of SiO₂ synthesized from pumice and pumice, experienced a not so significant shift.⁸ There are peaks of pumice in the wave number range 4000-600, namely there are 3 peaks with a wavelength of 1968.12 cm⁻¹; 1003.49cm⁻¹; 779.98 cm⁻¹ while the peak obtained by SiO₂ from the synthesis of pumice is that there are 5 peaks with a wavelength of 3383.56 cm⁻¹ this number is a new peak indicating the vibration range of the OH group from Si-OH, at wave number 2160, 00cm⁻¹; 1640.09cm⁻¹; 1046.43cm⁻¹; 791.65 cm⁻¹ is the absorption of O-H bending in strengthening the OH group of Si-OH. It means that SiO₂ synthesized from pumice found the desired C-H or O-H chemical groups, namely silanol groups (Si-OH), and siloxane groups (Si-O-Si)⁹.

Table 1. Percentage Composition of Chemical Elements in Pumice with XRF

Chemical Element	Percentage
SiO ₂	64,411%
K ₂ O	12,038%
Al ₂ O ₃	9,081%
CaO	4,29%
P ₂ O ₅	4,224%
Fe ₂ O ₃	3,627%

Table 2. Percentage Composition of Silica Chemical Elements from Pumice with XRF

Chemical Element	Percentage
SiO ₂	93,299%
K ₂ O	0,101%
Al ₂ O ₃	1,375%
CaO	0,881%
P ₂ O ₅	3,732%
Fe ₂ O ₃	0,146%

It is known that in the XRF analysis the chemical element of silica in pumice is 64.411% and the silica obtained in pumice purification is 93.299%, indicating that the analysis carried out has achieved very good results.

3.2. FTIR Characterization on the Synthesis of Silica Nanoparticle Aerogel from Pumice

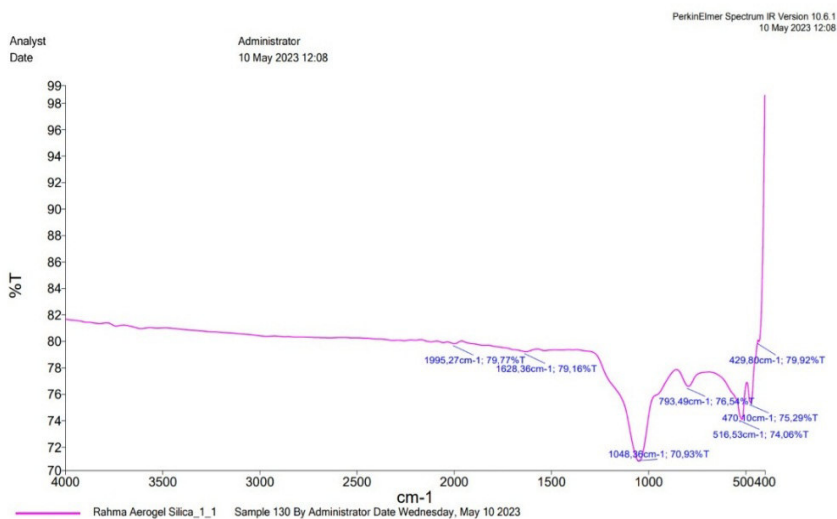


Figure3. FTIR Characterization on the Synthesis of Silica Nanoparticle Aerogel from Pumice

The graph shows the curve of silica aerogel made from synthetic silica with a wavenumber range of 4000-400 in which 7 significant peaks are formed. At a wavelength of 1995.27 cm⁻¹; 1628.36 cm⁻¹ indicates -OH bending vibration of silanol ($\equiv\text{Si-OH}$), the wavelength of 1048.36 cm⁻¹ indicates Si-O asymmetric stretching vibration of siloxane ($\equiv\text{Si-O-Si}\equiv$), at number wave 793.49 cm⁻¹ shows symmetrical stretching vibration of Si-O on siloxane ($\equiv\text{Si-O-Si}\equiv$), then the wave number is between 516.53 cm⁻¹; 470.10cm⁻¹; 429.80 cm⁻¹ the emergence of a new peak indicating that the bending vibration of the siloxane group ($\equiv\text{Si-O-Si}\equiv$)₁₀.

4. CONCLUSION

The synthesis of silica from pumice, quite a lot of silica is produced, this is evidenced from testing using XRF with a composition of 93.299% and FTIR testing of silica synthesis with a wave number range of 4000-600 at a wave number of 2160.00 cm⁻¹; 1640.09cm⁻¹; 1046.43 cm⁻¹ and 791.65 cm⁻¹ found silanol groups (Si-OH), and siloxane groups (Si-O-Si).

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