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Synthesis of silica gel from glass waste for adsorption of Mg^{2+} , Cu^{2+} , and Ag^+ metal ions

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Abstract. Glass waste is broken glass which is generally no longer used but on the other hand the content of silica in glass is quite high at around 72.4% so that the glass has the potential to be extracted silica for further use for making silica gel. In this study, silica extraction was carried out by reacting glass and 1.5 M sodium hydroxide solution and then heating it in a furnace at 400°C. Silica gel synthesis was carried out by the sol-gel method with citric acid catalyst at pH 5. From the FTIR results, there were two main functional groups of silica gel, which were silanol and siloxane groups at wave number of 920 and 1100 cm^{-1} respectively. Silica gel obtained has acidity, water content, and water capacity 1,625; 60; and 112 mmol/g adsorbent. While the ability of silica gel adsorption on metal ions Mg^{2+} , Ag^+ and Cu^{2+} was 17.68; 12.75; and 9.48 mmol/g adsorbent respectively

Keywords: glass, silica gel, adsorption

1. Introduction

Silica gel is one of the inorganic materials that has been widely known to have stable properties against mechanical and temperature influences. Silica gel has many uses, among others, as a chromatographic stationary phase, rubber tire filling material, components in the ceramic, adhesive, detergent, and pharmaceutical products industry [1]. Generally, silica gel is synthesized from the basic material of quartz sand which is a source of silica [2]. Quartz has a large silica content of almost 100% and is crystalline, therefore it has a high extraction temperature of 1300°C. Glass as a raw material for making glass has a silica content of 72.4% [3] and has the properties as a true amorphous solid so that the melting point of glass is lower than quartz. This lower melting point allows the extraction of silica to be carried out at a lower temperature. Several studies that have been carried out for the synthesis of silica gel are the basic ingredients of rice husk ash [4-7]. But the use of glass to extract silica and use it for the synthesis of silica gel has not been done much [8].

According to Orgaz-Orgaz [9], synthesis silica gel can be carried out through a process of hydrolysis and condensation. It is explained in detail that the extraction of sodium silicate from SiO_2 can be carried out by reacting SiO_2 with NaOH. Sodium silicate that is formed is then hydrolysed to form silicic acid. Silica gel is formed from the condensation of silicic acid [10, 11].

In this study, synthesis of silica gel was carried out with glass base material and NaOH with citric acid catalyst at pH 5 and calcination temperature 400°C. The silica gel obtained was characterized by FTIR to determine the presence of functional groups and determined the level of acidity, water content, water capacity, and adsorption ability of metal ions Mg^{2+} , Cu^{2+} , and Ag^+ .



2. Materials and Methods

2.1. Materials

The chemicals used for this research study were as follows: clear glass, sodium hydroxide (Merck), citric acid (Merck), distilled water, sulphuric acid (Merck), phenolphthalein indicator (Merck), hydrochloric acid (Merck), $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ (Merck), $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$ (Merck) and AgNO_3 (Merck).

2.2. Extraction of silica from clear glass waste

The clear glass was washed with distilled water, ground, sifted, and washed with 200 mL of 5% H_2SO_4 for 3 hours, then filtered and washed with distilled water until the filtrate is neutral and dried. 10 grams of glass powder were then reacted with NaOH 1.5 M 180 mL and heated with a furnace at a temperature of 400°C . The results obtained were dissolved in distilled water and analysed with AAS to determine the dissolved Si content in the filtrate.

2.3. Synthesis of silica gel

A filtrate of 50 mL was reacted with 4 M citric acid dropwise to pH 5, then allowed to stand for 24 hours to produce a hydrogel. Silica hydrogel is washed, dried, and ground, so that a dry gel (xerogel) was formed. Silica gel powder was analysed by FT-IR spectrophotometer to determine the presence of silanol and siloxane groups, determination of acidity level, water content, water adsorption capacity, and silica gel adsorption test for metal ions Mg^{2+} , Cu^{2+} , and Ag^+ .

3. Result and discussion

3.1. Silica gel from clear glass waste.

Based on the results of AAS analysis, the concentration of silica extracted from 10 grams of glass was 0.2143 mol/L, hence the silica content extracted was 30%. This value was still quite low compared to the silica content in glass expressed by Scholes and Greene [12] which was 72.4%. This low value is certainly due to many factors, including different types of glass and varying extraction efficiency

The reaction of the formation of silica gel was conducted by adding citric acid to sodium silicate to pH 5. The addition of citric acid produces free silicic acid which formed silicic acid polymers. The polymer aggregate joined into a polymer ball forming an aqua gel. According to Kalapathy *et al.* [13], at this stage condensation between polymer balls continued and there was shrinkage in the volume of aqua gel followed by the releasing of sodium salts hence a rigid gel was produced called hydrogel. In this study the time needed for hydrogel formation was 24 hours. In the initial condition of sodium silicate was very alkaline (pH 12-13), sodium silicate was in the form of Si-O^- (silanol deprotonated). Then after the addition of citric acid gel formation began to occur as in reaction (1) and (2).



Gel formation was stopped at pH 5 when a soft hydrogel was obtained. Addition of acid caused an increase in the number of silanol groups and a reduction in the amount of deprotonated silanol, however, the number of deprotonated silanol groups was still dominant. In conditions that were close to neutral, the concentration of the silanol and deprotonated silanol groups was balanced in relatively large amounts, hence the condensation reaction and gel formation were faster. The weight of the synthesized silica gel was 1.2660 g.

3.2. Functional groups of silica gel.

The spectrogram pattern of FT-IR silica gel is presented in Fig. 1 while the spectrogram interpretation is shown in Table 1.

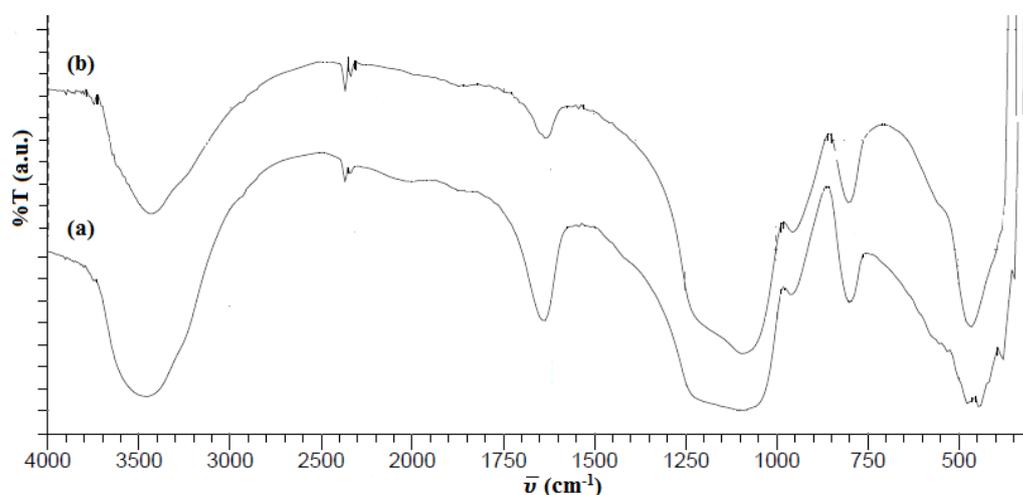


Figure 1. FTIR spectra of (a) Kiesel G-60 silica gel and (b) synthesis silica gel

Table 1. Interpretation of G-60 Kiesel silica gel spectrum and synthesis of silica gel [14, 15]

Wave number (cm-1)		Interpretation
Kiesel G-60 Silica Gel	Synthesised Silica Gel	
3415.7	3433.1	-OH stretching vibration of H ₂ O
1629.7	1639.4	-OH bending vibration of H ₂ O
1097.4	1087.8	Vibration Si-O asymmetry stretching from Si-O-Si
956.69	956.69	-OH stretching vibration of Si-OH
800.4	794.6	Vibration of Si-O symmetry stretching from Si-O-Si
451.3	470.6	Si-O-Si bending vibration

It was seen that the Kiesel 60 silica and synthesized silica gel showed similar absorption patterns but with different intensities. In the area between 3300-3750 cm⁻¹ which shows the presence of -OH group caused by -OH stretch vibration of H₂O. In the absorption area around 1600 cm⁻¹ indicates the presence of an -OH group which is characterized by a -OH bending vibration of H₂O. The area around 1100-1000 cm⁻¹ indicates the presence of siloxane groups caused by vibration of Si-O asymmetry stretching from Si-O-Si and in an area of about 800 cm⁻¹ by Si-O symmetry stretching vibration from Si-O-Si. These two vibrations appear in different wave number regions because they have different energies. In asymmetry stretch vibration has a greater energy so that it appears in the larger wave numbers as well. The buckling vibration of Si-O-Si is seen in an area of about 450 cm⁻¹. At an area between 956 cm⁻¹ which shows the presence of an -OH group due to the -OH stretch vibration of Si-OH. Both samples showed uptake in the same area but different intensities. The spectra shift towards higher wavelengths compared to the G-60 silica gel silica spectra which proves that silica gel synthesis has greater Si-O-Si bond strength compared to standard silica gel.

3.3. Determination of the acidity level of silica gel

Determination of the level of acidity was done by volumetric methods. According to Bronsted Lowry, acid is a compound capable of donating protons. According to Lewis, acid is a species that can act as an electron pair acceptor. Silica gel which has a silanol group acts as Bronsted acid which can donate protons while NaOH which has OH⁻ is a Bronsted base that can accept protons. Acidity level of synthesized silica gel obtained was 1.625 mmol/g. Therefore, the acidity level was determined by the release of H⁺ from the -OH group hence the acidity level was directly correlated with the amount of silanol groups that are owned by the silica gel.

3.4. Determination of silica gel moisture content

Determination of moisture content was done to determine the moisture content of silica so that the molecular formula of silica gel ($\text{SiO}_2 \cdot n\text{H}_2\text{O}$) was known. Initially the synthesized silica gel was heated at 100°C to dehydrate silica gel. Heating at 100°C produced dehydrated silica gel in which free water molecules will escape and leave only water bound to the framework [16]. Then the silica gel was incubated at 600°C to vaporize all the water contained in silica gel so that the amount of water bound to silica gel was known. The water content of the synthesized silica gel is 0.064 mol, so the resulting chemical formula for silica gel is $\text{SiO}_2 \cdot 0.064\text{H}_2\text{O}$.

3.5. Determination of silica gel water adsorption capacity

Adsorption is the event of absorption of a substance on the surface of another substance. Water adsorption capacity by silica gel was carried out by heating the synthesized silica gel at 100°C for 24 hours to dehydrate silica gel from free water. Then the dried silica gel was put into a desiccator inside which a beaker glass containing water was placed. Silica gel was left in the desiccator for 24 hours for optimal water absorption. The water adsorption capacity by silica gel was 0.112 mol / g.

3.6. Silica gel adsorption on Mg^{2+} , Cu^{2+} , and Ag^+ metals ions.

Silica gel which has silanol and siloxane groups is known to absorb hard metal ions such as Na^+ , Mg^{2+} , Ca^{2+} , and Fe^{3+} [17]. Silica gel has an active group $-\text{OH}$ which can release H^+ to form O^- which can bind hard metals because O^- is a hard base according to the Pearson hard-acid base theory. Hard bases interact more easily with hard acids and soft bases are easier to react with soft acids. In this study, the adsorption was carried out on three types of metals, which are Mg^{2+} as a hard acid, Cu^{2+} as a boundary line and Ag^+ as a soft acid. It is expected that the synthesized silica gel absorbed Mg^{2+} metal more than the other two metals because Mg^{2+} was a strong acid [18]. Metal adsorption ability is shown in Table 2. Table 2 shows that the synthesized silica gel tends to bind Mg^{2+} metal ions compared to the other two metals because of the silica gel synthesis properties as hard acids which prefer to bind to hard bases.

Table 2. Synthesis of silica gel adsorption capacity for metal ions Mg^{2+} , Cu^{2+} , and Ag^+ .

Metal ion	Adsorption capacity (mmol/g)	
	Silica gel synthesis	Kiesel G-60 Silica Gel
Mg^{2+}	17.68	14.20
Cu^{2+}	9.48	12.89
Ag^+	12.75	14.53

4. Conclusion

Silica has been extracted from a glass with a silica content of 0.214 mol / L for every 10 grams of glass. Silica gel was successfully synthesized from glass as indicated by the appearance of Si-OH and Si-O-Si functional groups with a pattern similar to Kiesel silica G-60 gel. The silica gel obtained was 1266 g which having acidity, water content, and water capacity of 1.625; 60 and 112 mmol/gram of adsorbent respectively. While the ability of silica gel adsorption for metal ions Mg^{2+} was greater than Ag^+ and Cu^{2+} with adsorption capacity was 17.68; 12.75; and 9.48 mmol/gram of adsorbent respectively. In general, the silica gel adsorption ability synthesized into Mg^{2+} metal ion was greater than G-60 kiesel silica gel.

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