The influence of polyethelene glycol concentration on silica xerogel synthesis from geothermal sludge to Pb(II) ions adsorption

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The influence of polyethelene glycol concentration on silica xerogel synthesis from geothermal sludge to Pb(II) ions adsorption

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Abstract. Geothermal solid waste contains around 80-95% of silica, which can be used for getting valuable materials. Synthetic silica can be produced from sodium silicate with polyethylene glycol (PEG) as a capping agent. The purpose of this research is to obtain silica xerogel from geothermal sludge, determine the effect of concentration variations of PEG to particle size and Si-OH/Si-O-Si groups ratio of silica xerogel on the adsorption ability to Pb (II) ions. The sol-gel method was applied to synthesize the product. The xerogel successfully resulted in deriving from geothermal solid waste with the addition of PEG. FTIR spectra indicated the presence of Si-OH stretching vibration at 954 cm^{-1} and Si-O-Si asymmetry stretching vibrations at 1089 cm^{-1}. PSA data showed that silica gel with 0%, 2%, 4%, and 5% were 46.63; 197.01; 468.35; and 569.07 μm, respectively, and increase in particles size and the group' ratio of Si-OH to Si-O-Si. The addition gel with a variation of PEG 2% slightly increased the Pb(II) ions adsorption capacity at various concentrations of 80 mg/L.

1. Introduction

Geothermal sludge is waste from the geothermal industry, especially geothermal power plants, generally obtained in abundance. This mud has not been utilized optimally even though it contains a large silica source up to 95%, especially in Central Java's Dieng geothermal power plant Indonesia. Due to its large silica content, this waste can be used as a base material for making silica gel and other materials with higher economic value [1]. Furthermore, silica xerogel from this waste can be utilized as some useful materials such as a nanofiller material manufacturing [2], additional material in cement [3], composite materials [4], drug delivery [5], and including as an adsorbent for radioactive waste and heavy metals [6-7].

Lead is a heavy metal that is dangerous either when it enters the body or in the environment [8-9]. The maximum lead content in water recommended by WHO is less than 0.01 ppm [10]. If its presence exceeds the threshold value, it will impact human health, causing brain damage, seizures, and death. Its toxicity in small doses and continuously in children can cause neurotoxicity (nerve poisoning) and behavioral disorders [11]. Lead is a non-essential heavy metal, meaning a metal whose benefits are unknown to the body and toxic [12]. Because of the danger that some materials are applied to remove Pb (II) ions with adsorption process [13-15].

In the synthesis of silica gel, to obtain the gel with good quality in pore size, a structure-directing agent such as polyethylene glycol (PEG) is generally added [16-19]. On the other hand, there is often an aggregation or agglomeration process in its synthesis that will affect the final reaction product [20-

21]. To overcome this, it is highly recommended to add PEG as a capping agent to prevent agglomeration for obtaining silica gel with relatively small particle size. However, in previous studies, PEG addition does not easily make the particle size smaller [20]. Therefore, in this study, the addition of PEG will be carried out and its effect on the particle size distribution and the number of active sites of silanol (Si-OH) to siloxane (Si-O-Si) groups on silica xerogel in affecting adsorption of Pb (II) ions. It is necessary to vary the addition of PEG concentrations in the synthesis of silica xerogel from geothermal waste. Furthermore, silica gel synthesis will be carried out using the sol-gel method known as the simple method and can be carried out at room temperature [22-23]. Several previous studies using different molecular weight variations of PEG obtained different xerogel properties and used synthetic pure materials in their synthesis [20,24,25,26]. In this study, PEG-6000 and natural materials were specifically used as xerogel synthesis materials.

2. Material and methods

2.1 Tool and *materials.*

2.1.1 Tools. The tool used in this study were beaker glasses, watch glasses, stirrers, dropper pipettes, magnetic stirrers, vial bottles, measuring flask, Erlenmeyer, glass funnel, pH indicator (Merck), filter paper, thermometer, reflux column, analytical balance, XRF (PANalytical axios), FTIR(Perkin Elmer spectrum 100), PSA(Horiba LA-960), Oven(Binder), AAS(Perkin Elmer AAnalyst 200).

2.1.2 Materials. The materials used in this study were geothermal waste (geothermal sludge), Merck 37% HCl, Merck NaOH, Merck PEG 6000, distilled water, Merck Pb(NO$_3$)$_2$

2.2 Experimental

2.2.1 Geothermal waste preparation. Geothermal sludge was obtained from the Dieng Geothermal Power Plant (PLTPB), Central Java, Indonesia. The sludge was first washed using distilled water, followed by a heating process using an oven at 100°C until completely dried. Furthermore, crushed and sieved with a 150-mesh (147-µm) sieve [1]. The result was fine powder geothermal waste.

2.2.2 Production of sodium silicate. Fine powder geothermal waste 10g was refluxed using 100 mL of 3M NaOH for 1 hour with constant stirring using a magnetic stirrer for dissolving silica and producing a sodium silicate solution. The solution then cooled at room temperature and filtered using Whatman-41 filter paper. The resulting filtrate was a sodium silicate solution, which was used as a precursor for the synthesis of silica gel.

2.2.3 Silica xerogel synthesis. Sodium silicate that has been made was added to PEG 6000, which concentration variation of 0%, 2%, 4%, and 5%. Since PEG was completely dissolved, then 1M hydrochloric acid solution was added gradually to start the hydrolysis-condensation reaction. Changes in pH values were monitored using a pH meter. White gel started to form when the pH value was decreasing to less than 11. The gel was adjusted to a pH value of 4. Silica gel formed then aged for 8 hours and heated in an oven at 80°C for 12 hours. Then washed it with distilled water to neutral pH. Finally, the result heated using an oven again at 80°C for 24 hours for obtaining silica xerogel.

2.2.4 Pb(II) ions adsorption on silica xerogel. A total of 15 mL of Pb (II) ions solution with a variety of concentrations of 20 ppm was added with 0.03 grams of silica gel and shaken with a 100 rpm shaker for 30 minutes, allowed to stand for 2 hours, and filtered. The resulting filtrate was analyzed with AAS. The same treatment was carried out at various 40, 60, 80, and 100 ppm concentrations.

2.2.5 Characterization. The level of mineral content in fine powder geothermal waste was analyzed with XRF while Si-OH and Si-O-Si functional groups in synthesized silica gel indicated with FTIR. PSA measures the particle size of the silica xerogel. AAS to determine the remaining Pb (II) ions present in a solution after the adsorption process.

3. Results and discussion

3.1 Geothermal waste

Geothermal waste material is from the Geothermal Power Plant (PLTPB) in Dieng. Before using the waste as a primary precursor to produce silica gel, at first, it was tested with X-Ray fluorescence (XRF) to determine the components present in the geothermal sludge. The results of the analysis are showed in table 1.

Table 1. Geothermal Waste Composition

Component	Concentration (%)
SiO_2	95.016
P_2O_5	1.904
Fe_2O_3	1.763
CaO	0.530
Sb_2O_3	0.213

The data indicates that the waste content is dominated by silicon dioxide (SiO_2) up to 95.016%. Other components including P_2O_5, Fe_2O_3, CaO, and Sb_2O_3 with consecutive concentrations of 1.904%; 1.763%; 0.530%; and 0.213%. The large concentration of silicon dioxide is expected to produce large amounts of sodium silicate to obtain large amounts of silica gel.

3.2 Sodium silicate *from geothermal waste extraction*

Sodium silicate is obtained by reacting to geothermal waste with an alkaline solution, which is sodium hydroxide. Because of some impurities in it, before being reacted with sodium hydroxide, the geothermal waste was cleaned. The fine powder, which is specific in grain size, refluxed using sodium hydroxide. The reflux process was carried out at a temperature lower than the water boiling point for an hour to extract the silica content containing in the fine powder geothermal waste. Then the result of reflux was filtered for getting a filtrate. This process can produce around 93 mL of the filtrate of sodium silicate, which was slightly thick and brownish in shape, then after filtering, the color becomes slightly clear yellow. That color is presumed as preliminary evidence that the silica in the waste has reacted with sodium hydroxide. The product of sodium silicate is a precursor for getting silica gel.

3.3 Silica *gel*

Silica gel synthesis was carried out by adding a 1M hydrochloric acid solution to the sodium silicate solution drop wisely. This acid acts as a catalyst in the sol-gel reaction. Before adding the acid, the sodium silicate solution was mixed with PEG 6000 solution, and the pH was measured as the initial pH.

The white gel was formed (hydrogels), was then treated with aging at 60°C for 8 hours. As a result, the gel volume was reduced, and the structure solidified. In this process, a condensation reaction occurs in the hydroxy groups to form new bonds, which cause the network to become stiffer and more robust, and there is a shrinkage of the gel volume [23].

The silica hydrogel was repeatedly rinsed to remove the remaining NaCl until the pH was neutral, heated in an oven for 10 hours at 80°C to form a dry gel. The dry gel was rinsed using distilled water to remove PEG, which adhered to the gel surface and dried at 80°C for 15 hours. The result of the process is a dry silica gel solid called xerogel. Xerogel is a dry silica gel produced by drying the pores'water phase through an evaporation process [27]. The xerogel obtained was analyzed using FTIR and PSA.

3.3.1 Function groups on silica gel. FTIR analysis was carried out on the synthesized silica gel with variations in the addition of concentrations of PEG. The results of the FTIR analysis on silica xerogel are presented in figure 1.

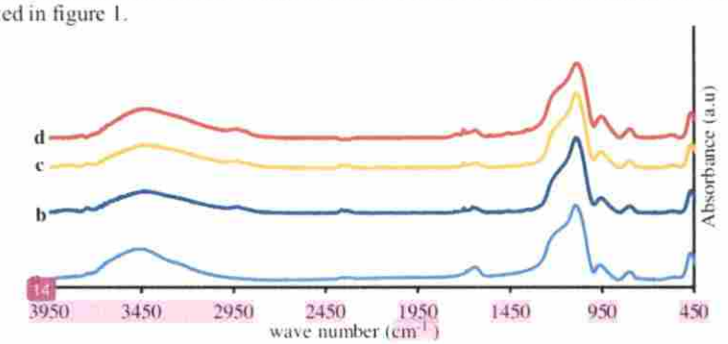

Figure 1. FTIR spectrum of silica xerogel with variations in PEG 6000 concentration of (a) 0% (b) 2% (c) 4% and (d) 5%

Figure 1 is an FTIR spectrum of silica xerogel with a variation of polyethylene glycol concentration (PEG) 6000 at 0%, 2%, 4%, and 5%. The peaks in the wavenumber 1300-450 cm^{-1} are a combination of vibrations found in silica, where the peak 460-450 cm^{-1} is the rocking vibration of Si-O-Si [28]. The next three peaks below 1300 cm^{-1} are at a wavenumber of about 800 cm^{-1} indicates the symmetry stretching vibration of Si-O-Si, 954 cm^{-1} denotes the Si-OH stretching vibration, and the wide peak at about 1089 cm^{-1} represents the asymmetric stretching vibration of Si-O-Si[29]. The peaks at waves 2800-1350 cm^{-1} refer to overtone and a combination of vibrations of water molecules and SiO_2 network. The peaks at the waves of 2860 cm^{-1} and 2924 cm^{-1} signify the stretching vibrations of symmetry and asymmetry from -CH_2- originating from the PEG chain [30]. There is the appearance of a vibration peak -CH2- which is quite intense for the concentration variation of PEG 2%, 4%, and 5%, otherwise, there was no such peak at 0%. The peaks at the numbers 4000-3000 cm^{-1} show the overtone and the combination of stretching vibrations of H_2O [28]. From this spectrum data, it can be seen that the silica xerogel from geothermal waste has been formed, and there are still PEG molecules in it.

Specific information on the region of wave number 1250-950 cm^{-1} is needed to determine xerogel character due to the content of the ratio of Si-OH and Si-O-Si groups. Therefore, the quantization of the peaks in the 1250-950 cm^{-1} region was carried out by deconvolution of the FTIR spectrum. Figure 2 is a deconvolution of the FTIR spectrum of silica xerogel with concentration variations of PEG 0%, 2%, 4%, and 5% using the "fityk" application with the same HWHM for all individual peak components. This physical analysis is used to determine the difference in composition by measuring the absorbance intensity associated with the type of functional group in each sample, then calculating the area ratio of Si-OH groups to Si-O-Si groups so that the area of Si-OH/ Si-O-Si is obtained.

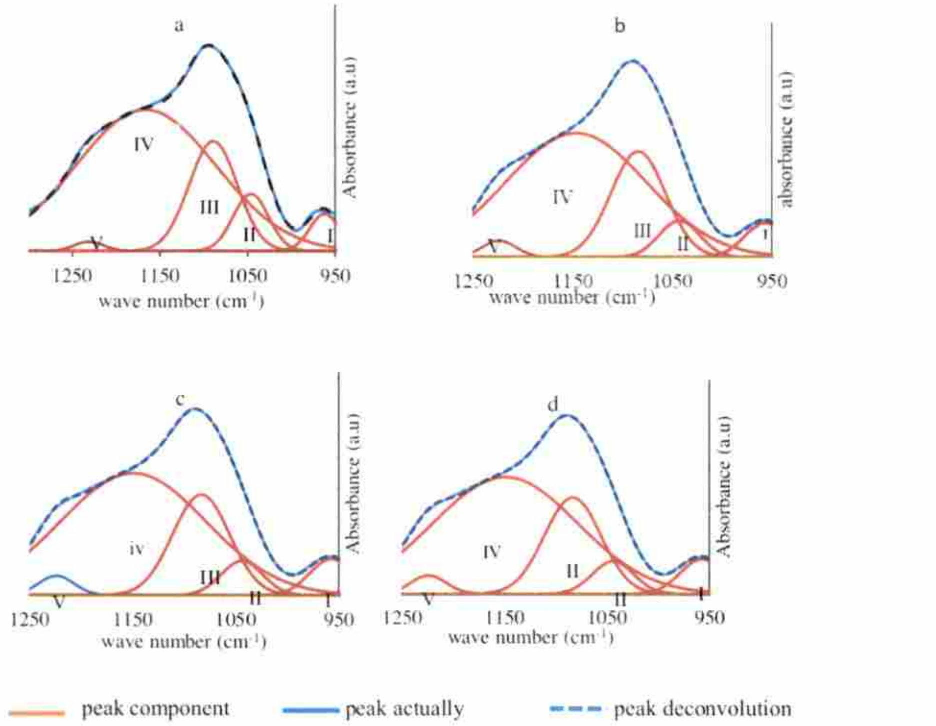

Figure 2. The deconvolution of FTIR silica xerogel spectrum with variations in PEG concentration of (a) 0% (b) 2% (c) 4% and (d) 5%

Figure 2 indicates five peak components that form the peak of the asymmetric stretching asymmetry Si-O-Si. We can find out the two structures of Si-O-Si present in the synthesized silica xerogels from this deconvolution. At first, wave numbers around 1200 cm^{-1}(IV) and 1090 cm^{-1}(III) are TO Transversal Optical) and LO (Longitudinal Optical) stretching vibrations of Si-O-Si, which have a cyclic structure.In contrast, the second, Si-O-Si stretching vibrations with a linear structure, is shown by the peak component at the wavenumber around 1040 cm^{-1}(II)[32]. Meanwhile, the peak component around 960-940 cm^{-1}(I) is a stretching vibration of Si-OH, and the peak component around 1250-1210 cm^{-1}(V) is presumed a twist vibration of -CH2-.

The Si-OH/Si-O-Si ratio can be determined from the deconvolution that has been done. The ratio results are presented in Figure 3. From the ratio value, it is known that silica gel with increasing PEG addition will accrue silanol groups. The groups'silanol increases due to the addition of sodium silica dropwise into the PEG solution, The silica particles will adsorb the PEG hydrophilic portion through hydrogen bonds to form a steric effect, which can prevent further growth of silica particles causing agglomeration [20-21].

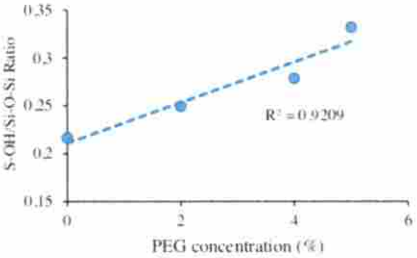

Figure 3. Graph of Si-OH to Si-O-Si ratio in silica xerogel

3.3.2 Particle size distribution of silica xerogel. PSA analysis was carried out to determine the effect of PEG concentration variations on the particles size's diameter and distribution of the obtained silica gel. The results of the measurement can be seen in table 2.

Table 2. Average particles size of silica xerogel by variation in PEG concentrations

Concentration PEG 6000	Size (μm)
0%	46.63
2%	197.33
4%	455.35
5%	569.07

Table 2 reveals that the variation of PEG 0%, 2%, 4%, and 5% obtained an average particle size of 46.63 μm, 197.33 μm, 455.35 μm, and 569.07 μm, respectively. The Concentration of PEG 5% has the largest average particle size. Besides that, with the increase in the concentration of PEG, the average particle size become wider. Polyethylene glycol makes silica perforated so that after the silica is evaporated or in the oven, it makes silica has a broad particle size. These results reinforce previous research that at high PEG numbers, secondary aggregates of the PEG chain can be formed, which results in sodium silicate molecules being trapped in the PEG chain. This causes silica particles to form in the PEG matrix and form silica groups, resulting in silica gel, which has a larger particle size and broader particle size distribution [20].

Figure 4 shows a shift in the particle size distribution of the silica xerogel. In addition to the PEG concentration, the graph gets to the right, which indicates the larger the particle size and the wider the distribution of particle size. The smallest silica size on the left graph belongs to the sample with the addition of 0%, and then to the right with the addition of 2%, then 4%, and the rightmost shift is the silica xerogels sample with the addition of 5%. The larger particle size distribution with PEG addition is thought to be due to PEG absence from the silica surface and the need to optimize time and temperature during the calcination process.

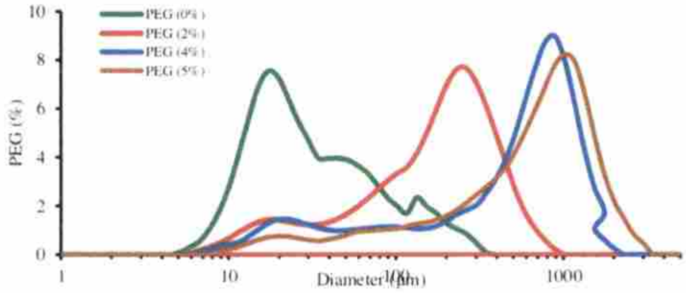

Figure 4. Graph of the particles size distribution of xerogel with PEG concentration addition

3.4 Adsorption *of Pb (II) ions on silica xerogel*

Silica gel has many benefits, one of which is as an adsorbent. In this research, the application of synthesized silica gel was used to adsorb Pb (II) metal ions in water. Adsorption is done at various concentrations, namely concentrations of 20, 40, 60, 80, and 100 mg/L. A total of 0.03 grams of silica gel was added to the $Pb(NO_3)_2$ solution, then shaker for 30 minutes, after which it was filtered, and the filtered solution was then analyzed using Atomic Absorption Spectrophotometry (AAS).

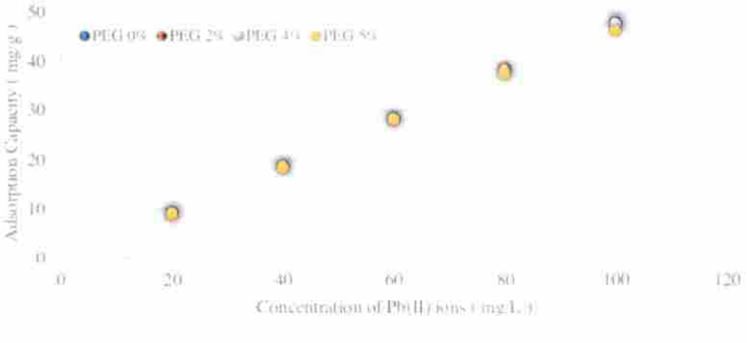

Figure 5. Graph of Pb (II) ions adsorption on silica xerogels.

Figure 5 is a graph of the ability of Pb (II) ions adsorption by silica with variations in the addition of PEG. The concentration of Pb (II) ions can be adsorbed by silica with each variation of the addition of PEG 0%; 2%; 4%; and 5%. In all variations of PEG concentration, the adsorption ability of xerogel increased significantly with the increase in the concentration of Pb (II) ion from 20 to 100mg/L. At the addition of 0%, the adsorption ability increases from a concentration of 20 mg/L(9.214mg/g) to 100 mg/L(47.94mg/g). Whereas the of addition of 2%, 4% and 5% ere 9.084mg/g-47.695mg/g, 9.042mg/g-47.639mg/g, 8.904mg/g-46.228mg/g, respectively. At each concentration of Pb (II) ion, the addition of PEG actually decreased the adsorption ability although not significantly, only at a concentration of 80mg/L the addition of 2% slightly increased the adsorption ability from 38.01mg/g to 38.50mg/g. Of all the variations in the addition of PEG, which has the least adsorbed Pb (II) ion, is the addition of 5% PEG.

The ratio of Si-OH to Si-O-Si groups tends to improve (Figures 2 and 3). With PEG concentration, increasing this ratio is expected to increase the silanol groups on the xerogel frame. It is hoped that Pb (II) ions adsorption tends to increase, presumably by the ion exchange mechanism, but this is not the case. Only the addition of 2% PEG occurs the adsorption went up slightly at 80mg/L. The addition of further concentrations does not follow it. It is suspected that other factors were involved in this adsorption. Based on the average particle size measurement, the addition of the PEG concentration increases its size. Besides that, the distribution of particle size becomes wider. The larger the particle size is thought to result in a smaller surface area. In the adsorption process, the surface area is an essential factor influencing its success. The larger the surface area, the better the adsorption and vice versa. It is estimated that the larger the particle size makes the best adsorption in the addition of 4% and 5% PEG concentrations but instead in the addition of 2%.

4. Conclusion

Silica xerogel can be synthesized from geothermal sludge waste. The addition of PEG concentration increased the Si-OH to Si-O-Si ratio nevertheless, the particle size diameter and distribution also increased respectively from 46.63; 197.01; 468[illegible]; and 569.07 μm for variations of 0%, 2%, 4%, and 5%. The addition of PEG did not significantly affect the adsorption of Pb (II) ions except the addition of 2% at a concentration of 80mg/L of 30g xerogel within 30 minutes.

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