Kinetics Adsorption of Strontium(II) by Silica Xerogel from Fly Ash

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Abstract A study of liquid waste adsorption containing strontium was carried out using the adsorption method using silica xerogel adsorbent. Silica xerogel synthesized from coal fly ash using the sol-gel method, produces a specific surface area of 84.8884 m²/g. This study was intended to determine the appropriate kinetics law. Data were obtained by following changes in Sr concentration to the time of adsorption at temperature of 25 °C and 35 °C. The data obtained was matched with 5 kinetic models, namely zeroth order kinetics, first order, second order, pseudo first order, and pseudo second order. Based on data analysis by means of linear regression and the number of minimum sum squared of errors, the adsorption kinetics model that is suitable for the adsorption of strontium by silica xerogel is a pseudo second order. The reaction rate constant at 298 K and 308 K were 0.1023 g /mg/min and 0.0716 g/mg/min respectively. The value of the activation energy is negative so the higher the temperature, the lower the adsorption rate. It is indicate that the adsorption is reversible.

1. Introduction
Strontium-90 is a radioactive waste nuclide produced from nuclear reactors. These radionuclides emit beta particles with the highest decay energy of 0.546 MeV and a half-life of 28.6 years [1,2,3]. For safety reasons, radioactive waste has never been carelessly discharged into the environment because it has been regulated by national legislation and does not conflict with internationally applicable regulations. The level of danger of radioactive waste from other hazardous wastes can be distinguished by the causes and mechanisms of interaction with the target. A hazard characteristic of radioactive waste is emitting radiation that can ionize or damage a target so that it becomes unstable or dysfunctional [3]. Strontium-90 produced from nuclear reactors can enter the human body in two ways, namely ingestion then enter the body through the human food chain and inhalation, ie radioactive dust particles through breathing, then settles inside body as a source of internal radiation, so that it can cause negative effects on human health [4].

To reduce the negative impact of water contaminated by strontium carried out by various treatments such as precipitation, extraction, ion exchange, reverse osmosis, and adsorption. Adsorption is a relatively simple, easy to do, and economical way, effective for low solute concentrations. Adsorption requires absorbent material. Several journals report materials used to adsorb Sr including Florisil impregnated with Cyphos IL-101 (Negrea et.al, 2013), synthetic Hydroxyapatite (HAP) (Nishiyama, 2015), kaolinite (Zigong, 2017), and Nut shell activated carbon (Duca et al, 2018) [5, 6, 7, 8, 9].
This research uses silica xerogel from fly ash to adsorb Sr(II). Silica xerogel can be made by utilizing high content Si of wastes that are not economically utilized such as rice husk ash and coal fly ash. Burning coal as a source of electrical energy produces solid waste in the form of fly ash. This ash has not been utilized properly and is only stacked in landfills, causing environmental problems. Fly ash is composed of several porous oxides (mainly silica) and unburned carbon which have good potential as adsorbents. However, the high level of crystallinity of silica and the content of unburned carbon can cause a decrease in adsorption capacity [10]. Fly ash is a fine particle material which is dominantly shaped in a solid or hollow sphere shape. This material is a ferrous aluminous silicate compound with the main elements Si, Al, Fe, Ca, K, and Na. The mineralogy is influenced by the coal origin. Fly ash is classified as a dangerous and toxic material [10].

This research utilized coal fly ash as a source of silica as the basis for making silica xerogel. The silica xerogel was used to reduce Sr(II) levels in simulated waste. Based on the research of Affandi, et al. (2009), the silica xerogel which synthesized from bagasse ash by the sol-gel method, using silicate sources from sodium silicate has a surface area of 69-152 m²/g, a pore volume of 0.059-0.137 cm³/g, and a pore diameter of 3.2-3.4 nm which indicates it is mesoporous [12-14]. In a study conducted by Sarand, et al. (2015) showed that silica xerogel was successfully synthesized increased the decrease in Pb²⁺ and Cd²⁺ in aqueous solutions [15].

This study was intended to determine the kinetics model that is suitable for the adsorption of strontium with xilika xerogel made from coal fly ash. The kinetic model will be approached with the reaction rate law of zeroth order, first order, second order, pseudo first order, and pseudo second order. By determining the adsorption kinetics model, it will be obtained important parameters that can be used as initial data for its application in industry [16]. These are necessary with the wide use of adsorption in the industry, especially in waste treatment.

2. Methods
2.1. Materials
The main material used is fly ash obtained from waste fuel from PT Madukismo. Before being used, the ash was processed according to research conducted by Anggia (2016) [17]. The fly ash was dried at 100 °C for 1 hour, then sieved to pass 120 mesh, part of it was analyzed by using XRF. The XRF analysis results showed that contents of silica 82.184%, alumina 5.042%, iron 3.038%, and calcium 1.184%. Based on the results of this analysis, it indicates that fly ash has the potential to be used as a source of silica as a raw material for making silica xerogel. Recovery of the silica is done by weighing 50 grams of fly ash, dissolved in 300 mL of 3 M NaOH solution, then refluxed for ± 5 hours while stirring at 300 speeds rpm. The mixture then filtered and the filtrate formed is sodium silicate, taken as a base material for making silica xerogel. The making of silica xerogel refers to the method in the research of Affandi, et al. (2009) [12].

The filtrate containing sodium silicate add with HCl solution in concentration of 3M so the pH become 7 and thus forming gelatin, filtered with filter paper, then washed with distilled water for three times every four hours. The mixture then heated to it boiling point for 5 hours under reflux with constant stirring. After that, the mixture is filtered to get sodium silicate solution then neutralized with 3M HCl solution to the gelatin form. At the aging stage silica gel is washed with a mixture of isopropanol and n-hexane (1:1) 3 times for 1 day to form a gel. The formed gel is dried at 50 °C for 24 hours. This gel is silica xerogel which is used as Sr(II) adsorbent material and analyzed by X-Ray Fluorescence (XRF), surface area by BET method, and functional groups by XRD method.

Based on the results of the XRF analysis of silica xerogel, the contain of silica 94.599%, alumina 4.937%, iron 0.072%, and calcium 0.038%. After being analyzed using the BET method, the silica xerogel produced had a specific surface area 84.8884 m²/g. This material is used as a research material for adsorbent in Sr adsorption kinetics.

Strontium as a simulated waste adsorbate was prepared by dissolving Sr(NO₃)₂ made by Merck with an initial concentration of 50 ppm.

Supporting materials used in this experiments include: sodium hydroxide (NaOH), hydrochloric acid (HCl), nitric acid (HNO₃), isopropanol, n-hexane, aquadest (H₂O), murexide, ethylene glycol, and 70% ethanol.
2.2. Procedures
The simulation solution containing strontium waste was made to varying concentrations of 50 and 100 mg /L. The Sr(NO3)2 solution was pipetted 10 mL, put into a 100 mL erlenmeyer, silica xerogel was added as much as 0.1 g. Stirring using a shaker with a speed of 120 rpm during time variation. After stirring is complete, the solution is filtered, and the filtrate. The filtrate was analyzed by using a UV-Vis spectrophotometer with a murexide complexing solution to determine the remaining of Strontium concentration in the solution (Ct). The number of Sr adsorbed per weight of silica xerogel at any time is calculated by the equation:

\[ q_t = \left( \frac{C_0 - C_t}{W} \right) V \]  

(1)

where \( C_t \) (mgL\(^{-1}\)) is the liquid phase concentrations of Sr at any time, \( C_0 \) (mgL\(^{-1}\)) is the initial concentration of the Sr(II) in solution, \( V \) is the volume of the solution (L), and \( W \) is the mass of dry adsorbent (g).

To obtain the value of the activation energy an experiment was experimented on for different temperatures of 25 and 35 °C or 298 and 303 K.

3. Results and Discussion

3.1. Kinetics models
The experimental data is processed to be matched with the reaction kinetics model of zeroth order, first order, second order, pseudo first order, and pseudo second order.

A reaction is called zero order if the magnitude of the reaction rate is not affected by the concentration of the reactant. Equations of zero order reaction rates are expressed in Equation (2).

\[ -\frac{d[C]}{dt} = k[C]^0 \]  

(2)

The Equation (2) can be written as equation follows:

\[ C_t = C_0 - kt \]  

(3)

The first order kinetics model shows that the reaction rate is directly proportional to the concentration of reactant with first power that can be expressed by Equation (4).

\[ -\frac{d[C]}{dt} = k_1[C] \]  

(4)

Equation (4) can be transformed into a linear equation as follows:

\[ \ln C_t = -k_1 t + \ln C_0 \]  

(5)

where \( C_t \) is concentration any time, \( k_1 \) is first order rate constant, and \( C_0 \) is initial concentration. The plot between ln Ct/lnC0 with t is a linear kinetics curve of order 1 [19].

The 2nd order kinetics model shows that the reaction speed is directly proportional to the square of the reactant concentration. The kinetics of second order reaction can be written with Equation (6):

\[ -\frac{d[C]}{dt} = k_2[C]^2 \]  

(6)

Equation (6) can be linearized into Equation (7).
\[ \frac{1}{C_t} = \frac{1}{C_0} + k_2 t \]  

(7)

where \( k_2 \) is the second order of reaction rate constant. When the plot between \( 1/C_t \) and \( t \) is a linear line the kinetics model is the second order [20-21].

The pseudo-order first order model is based on the surface complexation reaction between the adsorbent and adsorbate. The pseudo first order equation is as follows:

\[ \frac{dq_t}{dt} = k_1 s (q_e - q_t)^1 \]  

(8)

The kinetic equations of pseudo first order reactions can be expressed by Equation (9) [22].

\[ \ln (q_e - q_t) = \ln q_e - k_1 s t. \]  

(9)

The pseudo-order second order model is based on the surface complexation reaction between the adsorbent and adsorbate. The pseudo-order 2 kinetics equation is as follows:

\[ \frac{dq_t}{dt} = k_2 s (q_e - q_t)^2 \]  

(10)

The linear form of the pseudo second order reaction equation kinetics can be written with the following equation.

\[ \frac{t}{q_t} = \frac{1}{k_2 s q_e^2} + \frac{t}{q_e} \]  

(11)

Plotting \( t/q_t \) with respect to \( t \) is a linear curve with slope of \( 1/q_e \) and the intercept is \( 1/k_2 s q_e^2 \) (Liu, 2018). The pseudo second system equation (Ho and McKay's model), can be used to estimate the mechanism that occurs [22].

Analysis data of the initial concentration, concentration at some time, and concentration at equilibrium, are processed to find out the appropriate kinetics model. The results of the experiments were carried out at 25 °C (298 K), initial Sr concentration of 50 ppm, volume of solution 10 mL, and weight of silica xerogel 0.1 g are presented in Table 1.

**Table 1.** Experimental results at 25 °C (298 K), initial Sr concentration of 50 ppm

| Time, min | (Ct) (ppm) | Qt, mg/g | ln Ct | 1/Ct | ln(qeqt) | t/qt |
|----------|------------|----------|-------|------|----------|------|
| 0        | 50.0000    | 0        | 3.9120| 0.02 | 1.4816   | 0    |
| 2        | 26.0286    | 2.3857   | 3.2592| 0.0384| 0.7003   | 0.8383|
| 4        | 17.3143    | 3.2571   | 2.8515| 0.0578| 0.1336   | 1.2281|
| 6        | 11.6000    | 3.8286   | 2.4510| 0.0862| -0.5597  | 1.5572|
| 10       | 7.8900     | 4.2114   | 2.0651| 0.1268| -1.6683  | 2.3745|
| 15       | 5.6000     | 4.4400   | 1.7228| 0.1786| 3.3784   |      |
| 20       | 5.6000     | 4.4400   | 1.7228| 0.1786|          |      |

Based on the data in Table 1, were fitted with the reaction kinetics model of zeroth order, first order, second order, pseudo first order, and pseudo second order. From the analysis of experiments data the equation will be obtained and the value of \( R^2 \) and sum of square of errors (SSE) of each model can be calculated. The results of the data fitting with the kinetics model are presented in Table 2.
Table 2. Determination of the Adsorption Kinetics Model (T=298 K)

| Model, order | Equation | $R^2$ | SSE   |
|-------------|----------|-------|-------|
| Zeroth      | $C_t = -4.55t + 41.486$ | 0.68  | 0.95249 |
| First       | $\ln C_t = -0.1863t + 3.7296$ | 0.91  | 0.12573 |
| Second      | $1/C_t = 0.010t + 0.018$ | 0.99  | 0.06668 |
| Pseudo first| $\ln(qe - qt) = 0.312t + \ln qe$ | 0.91  | 3.08520 |
| Pseudo second| $t/qt = 0.2454t + 0.163$ | 0.99  | 0.00500 |

The values of $R^2$ and SSE values in Table 2 it is concluded that the suitable kinetic model for the adsorption process of strontium by silica xerogel is pseudo-second order. This model was chosen because give $R^2$ values close to 1 and the sum of squared of errors is the smallest. Taking this into account, the pseudo-second order kinetics constant of strontium adsorption by silica xerogel is $k_{2s}$ is 0.1023 g/mg/min at 298K. Strontium adsorption with initial concentration of 50 ppm by silica xerogel following the pseudo-second order kinetics model can be interpreted as the dominant adsorption that takes place by intra particle diffusion. In this study it can be explained that kinetically, strontium will predominantly diffuse into the pores of silica xerogel and stick to the pore walls. Strontium diffusion process into the pores of silica xerogel will take place until the surface of the pore is saturated or cannot absorb the strontium anymore. In this kind of adsorption, the chemical reaction seems significant in the rate-controlling step and the pseudo-second order chemical reaction kinetics provide the best correlation of the experimental data and the adsorption's mechanism is chemically rate controlling and because of this it is called chemisorption. In this mechanism, the kinetics of sorption should correspond to a reversible second order.

To determine the activation energy, the Arrhenius equation is used, based on two different temperatures, namely at 25 ºC (298 K) at 35 ºC (308 K). Comparison of experimental results at temperatures 298 and 308 is presented in Figure 1.

![Figure 1. Plots of pseudo second-order reactions at temperatures 298 and 308 K](image)

Based on data of the two experimental temperatures, related to the Arrhenius equation the relation of the reaction rate constants to temperature can be written with the equation:

$$ k = A e^{-E/RT} $$

Equation (12) can be linearized as follows:
\[
\ln k = \ln A - \frac{E}{RT} \tag{13}
\]

The rate constant, \(k\), values, at temperatures 298 and 308 K are 0.1023 and 0.0716 g/mg/minutes, respectively, and the activation energy is -9.6113 J/K/mol. The negative value means that the higher the temperature, the slower the adsorption rate that can occur because the reaction kinetics are pseudo second order and reversible. This is similar to the results of Marzewski's research on the adsorption of phenoxyacid pesticides as the speed and capacity of adsorption decrease with increasing temperature [23].

3.2. Silicaxerogel character

To ensure that the material obtained was silica xerogel, characterization was carried out. Characterization includes determining functional groups, crystallinity testing, and determining specific surface area.

3.2.1. Functional groups. A functional group is a collection of atoms that bind together and give characteristics to the physicochemical properties of compounds such as solubility, acidity, and chemical reactivity in a molecule. Characterization of silica xerogel functional groups was carried out by Fourier Transform Infrared (FTIR) instruments. The principle of FTIR is to use infrared waves to produce vibrations in silica xerogel molecules. Characterization using FTIR aims to determine the presence of silica xerogel functional groups which are marked by the presence of silanol and siloxane groups [24]. The results of determining xerogene silica functional groups using FTIR are presented in Figure 2.

![FTIR Spectra of silica xerogel from fly ash](image)

Based on the Figure 2, it is known that there is a small absorption that appears at wave number 3433.42 cm\(^{-1}\) which is a bond of Si-OH group (silanol), then sharp absorption appears at wave number 1095.57 cm\(^{-1}\) which is asymmetric stretching vibrations of Si-group O on Si-O-Si (Siloxane) group bonds. The buckling vibration of the siloxane group is shown in the absorption band of wave number 794.67 cm\(^{-1}\) indicating the presence of Si-C groups, and the absorption band at 470.63 cm\(^{-1}\) indicated the buckling vibration of the hydroxyl group bonding of Si-O-Si (silanol) groups. The absorption band at a wavelength of 1635.64 cm\(^{-1}\) shows the vibrational buckling of the hydroxyl group from the silanol group. In general, the absorption bands that appear in silica xerogel wave numbers from coal fly ash
indicate that the functional groups are contained in silica xerogel are silanol (Si-OH) and siloxane (Si-O-Si) groups. Based on this description, it is certain that the product obtained is silica xerogel. [25].

3.2.2. Crystallinity Test. The next characterization process is to determine the structure of the synthesized silica xerogel material. Characterization uses X-ray diffraction (XRD) instruments to obtain information about the crystallinity of the silica xerogel from coal fly ash. The working principle of XRD is to utilize the results of diffraction arising from X-ray scattering on silica xerogel material. The spectrum results can be used to infer whether the silica xerogel made is amorphous or crystalline. The results of the sloping position spectrum indicate that the silica xerogel structure is amorphous, whereas if the XRD spectrum results are getting sharper due to repeated X-ray scattering at the same angle then the silica xerogel structure is crystalline. In general, crystalline silica tends to have atoms bonded regularly. Silica accumulates in living things, whether animals or plants have an amorphous form, in contrast to silica that is not derived from living things such as rocks and dust that has crystalline silica structure. Amorphous silica is considered to be more reactive than crystalline silica and has an irregular spherical structure. Irregular structure can cause high surface area. The results of the silica xerogel crystallinity test using the X-Ray Diffraction tool are presented in Figure 3.

![Figure 3. Silica xerogel from coal fly ash XRD diffractogram](image)

Figure 3 shows the results of the diffractogram of silica xerogel with the peak widened at an angle of 2θ is 22.696. According to Kalaphaty (2000) a wide peak shape with a peak center around 2 peaks of 21-22 shows that silica xerogel is amorphous. This amorphous nature causes silica xerogel to be reactive and can be used as an adsorbent. The arrangement of atoms in amorphous silica occurs randomly or with a low degree of order. The results of this spectrum are similar to studies conducted by Affandi et al (2009) who made silica xerogel each from the basic material of bagasse ash [12]. This widening peak is caused by x-rays being unable to be diffracted by the amorphous silica xerogel structure so that the x-ray diffraction angle read by the tool becomes irregular due to scattering.

3.2.3. Test Surface Area Specific. Determination of the specific surface area in silica xerogel is done by BET testing using a Surface Area Analyzer. The specific surface area obtained using BET analysis is 84.8884 m$^2$/g. These results are the same as those conducted by Affandi et al (2009) who made silica xerogel from bagasse ash with a specific surface area of 69-152 m$^2$/g [12]. In addition to knowing the specific surface area of the silica xerogel produced, by BET testing using the Surface Area Analyzer it can also be seen the pore volume and pore diameter of the silica xerogel. The pore volume and pore diameter of silica xerogel produced were 0.17213 cc/g and 4.05545 nm, respectively. With a specific surface area, pore diameter and large pore volume, silica xerogel is able to adsorb strontium. From the results of the surface area test, according to the IUPAC (International
Union of Pure and Applied Chemistry) classification, silica xerogel made can be classified as mesoporous material, whose pore size ranges from 2-50 nm.

4. Conclusions

The kinetics of adsorption by silica xerogel from fly ash follow pseudo 2nd order models. The reaction rate constant at 298 K was 0.1023 g/mg/min and at 308 K was 0.0715 g/mg/min. The value of the activation energy is negative so the higher the temperature, the lower the adsorption rate. The product of silica silica xerogel is in the form of amorphous and has a specific area 84.88884 m²/g.

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