Adsorption of Mercury Using Different Types of Activated Bentonite: A Study of Sorption, Kinetics, and Isotherm Models

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Abstract 17784

Mercury is a hazardous element because of its toxicity and harmful effects on human health. Various traditional and low-cost methods have been developed to remove mercury from wastewater. This study used local raw material as an alternative adsorbent to treat mercury-contaminated wastewater. Activated bentonite was prepared using different chemical activators (H3PO4, HCl, and ZnCl2) in various concentrations. Then, it was dried at 200°C for an hour. The materials were characterized by SEM-EDS. Its percent removal and isotherm models were analyzed. In this study, the most effective activator was H3PO4 and the experimental data matched the Freundlich model.

Keywords: activated bentonite, chemical activators, mercury, wastewater treatment

1. Introduction

Water contamination by toxic metals, such as Hg^{2+} has become a critical issue for the environment. Water contaminated by these metal ions is harmful not only to humans but also to other species and the environment (Smith, 2019). Mercury contamination in water and the environment is increasing because of its wide use in mining (including small-scale gold mining), paper industry, battery production, ombrotrophic peatlands, and combustion of coal (Coggins et al., 2006; Li et al., 2018; Smith, 2019). To address this issue, researchers have formulated various methods and seek to find new materials capable of reducing the amount of mercury to less than 5 ppb.

Methods developed in the past decades to remove or at least to reduce mercury in wastewater include electrochemical methods, atomic sorption, and electrophoresis (Garg and Prasad, 2016; Mansilla et al., 2018; Naswir et al., 2019). However, most of the methods have limitations, such as complicated processing, high-cost instrument, more challenging resource sourcing, and time-consuming operations. Therefore, simpler and low-cost method and material for mercury removal is needed. One of such low-cost material is bentonite. Being abundant, bentonite has appealed to scientists because of its unique properties and low-cost production.

This material has good porosity and large surface area (Didi et al., 2009; Hebbar et al., 2018; Javed et al., 2018; Sanz-pérez et al., 2019). Various bentonite applications have been developed in various fields including adsorbent (Lee, 2015), isotherm modelling (Zheng et al., 2009), and removal of paraquat from water (Sidhoum et al., 2013), with metal ion sorption as one of the essential applications. This material has good sorption of metal ions, especially mercury, and no concerns about its negative impacts has been reported. In other words, bentonite is safe for humans and the environment.

Bentonite is suitable for industrial applications. It is extremely hydrophobic (Mambrini et al., 2013) and has been used to remove metal ions in solution. As raw material like activated carbon, bentonite can convert into activated bentonite to generate a larger surface area and well-developed pores. Recently, bentonite has been developed because this material exhibits vibrant intercalation chemistry. Meanwhile, being an economical and eco-friendly method, adsorption has been widely used to remove mercury from water. However, there are few researches focusing on bentonite’s sorption mechanism. Although several studies reported that bentonite was activated by using HCl (Bendou and Amrani, 2014), the best activator to produce active bentonite has not been found yet.
This paper describes and explains the best activators to produce activated bentonite. The prepared bentonite was modified by acid, base, and salt activators (HCl, H₃PO₄, NaOH, KOH, NaCl, and ZnCl₂). Bentonite microstructure was characterized by Scanning Electron Microscope with Energy Dispersive X-ray Spectroscopy (SEM-EDS), while bentonite elements were characterized using X-ray Fluorescence. In this work, sorption capacity, removal, kinetic study, and isotherm models will also be described. The study showed that low-cost modified adsorbent (bentonite) is a great significance to the mercury sorption and is safe for both environment and human health in different activators.

2. Methods

2.1 Materials

Bentonite was collected from Sungai Rengas, Jambi Province, Indonesia. Mercury solution was prepared using mercury nitrate (Hg(NO₃)₂). The activators used were phosphoric acid (H₃PO₄), zinc chloride (ZnCl₂), hydrochloric acid, or muriatic acid (HCl). While the equipment used included shaving shaker 100 mesh, furnace carbolite AAF 11 7 PID 301, magnetic stirrer, beaker, pipette, filter, Erlenmeyer glass, atomic absorption spectrophotometry (AAS) Perkin Elmer 900F, Memmert Oven, and SEM-EDS JEOL JSM 6510 LA.

2.2 Preparation of Bentonite

Bentonites were prepared as the support materials. Bentonites were dried at 105°C using Memmert Oven for 24 h. This treatment is to reduce the water content in bentonites. It was then pyrolyzed using furnace carbolite AAF 11 7 PID 301 at 200°C for an hour before being sieved on shaving shaker at 100 mesh and was put in the Erlenmeyer glass.

2.3 Characterization and Measurement

Bentonites were analyzed using SEM-EDS JEOL JSM 6510 LA to determine bentonites pore structure. Sorption of mercury was measured by AAS Perkin Elmer 900F. The sorption measurements of mercury were performed at room temperature (±28°C). Activators were added (200 mL) into 50 g of bentonite with various concentrations (0.5, 1, and 1.5 M). The treatments were carried out to enlarge bentonite pore size and surface area. The resulting bentonites were then filtered. Finally, samples were mixed using a stirrer at 200 rpm for 0, 5, 10, 15, 20, 25, and 30 minutes.

2.4 Experiment Design

The preliminary studies informed that activators give different abilities for mercury sorption. However, only some of them presumed that contact time and concentration of activators could be taken as the key to efficiency parameters. This work used a static model in a laboratory scale. In this case, adsorption capacity, percent removal and kinetic studies were analyzed using pseudo kinetic order, while isotherm model was analyzed by using Langmuir and Freundlich models. The best of activated bentonite was also characterized.

3. Results and Discussion

3.1 Surface Morphology of Activated Bentonites

Surface morphology of the activated bentonites was analyzed using SEM (Figure 1). A previous study informed that the surface morphology would be related to surface area and these properties started to decrease at 100°C (Toor et al., 2015). In this study, thermal activation under high temperature (200°C) could remove the water content and other molecules. Pyrolysis process increased the surface area because of the removal of the adsorbed, hydrate volatile organic, water molecules, and organic compounds attached to the natural bentonite surface. Calcination above 100°C simultaneously can alter the physical and chemical properties of bentonite (Wampler, 2006). However, the different composition and structure of natural and activated bentonites upon heating can vary depending on bentonite’s chemical properties.

The different results of surface morphologies of the different chemical activators from activated bentonite were presented. Thermal activation at 200°C did not significantly influence its surface morphology, especially for HCl and ZnCl₂. This process was because of the use of the industrial Memmert oven instead of the atmospheric with nitrogen or carbon monoxide. However, the acid-activated bentonite showed different surface morphology. This porous structure indicated a process of leaching has taken place, making the bentonite surface more porous. As shown in Figure 1, the best porous surface in this study was H₃PO₄-activated bentonites.
Bentonite adsorption is caused by the interlayer aluminosilicate structure at a ratio of 2:1. This material consists of one octahedral layer and flanked by two tetrahedral lines. \( \text{SiO}_2 \) and \( \text{Al}_2\text{O}_3 \) are the main components in developing montmorillonite tetrahedral layer. It is in line with the EDS analysis results in which \( \text{SiO}_2 \) and \( \text{Al}_2\text{O}_3 \) oxides are the main components of bentonite. Negative ions in octahedral bentonite layer will be balanced with other positive ions, such as \( \text{Ca}^{2+} \), \( \text{Mg}^{2+} \), \( \text{Na}^{+} \), and \( \text{K}^{+} \). The ability of bentonite to attack will affect type of activator used. The use of activator with higher \( \text{H}^+ \) equivalent value will result better deterrence capabilities.

### Table 1. Composition of activated bentonites

| Elements (% of Mass) | \( \text{H}_3\text{PO}_4 \) | HCl | ZnCl\(_2\) |
|----------------------|-----------------|-----|-----------|
| C                    | 4.77            | 14.24 |
| O                    | 64.61           | 48.13 | 41.00   |
| Al                   | 12.25           | 13.30 | 17.79   |
| Si                   | 20.29           | 23.92 | 20.62   |
| Cl                   | -               | -    | 0.75     |
| K                    | 0.68            | 1.08  | 0.40     |
| Ti                   | 0.25            | -    | 0.40     |
| Fe                   | 1.91            | 2.53  | 1.73     |
| P                    | -               | 6.27  |          |

### 3.3. Sorption Studies

Mercury has been selected to study and evaluate the performance of activated bentonite. Sorption capacity and bentonite sorption were calculated using Equation 1.

\[
\text{qe} = \frac{V (\text{Co} - \text{Ce})}{M}
\]  

Where \( V \) is the volume of mercury solution (mL or L), \( \text{Co} \) is initial mercury concentration (mg/L), \( \text{Ce} \) is the final mercury concentration for the ability of bentonite sorption and equilibrium concentration for sorption capacity, \( M \) is bentonite mass (g), and \( \text{qe} \) is sorption capacity/bentonite sorption ability. Percent removal of mercury was calculated using Equation 2.

\[
\text{Percent removal} = \frac{100 (\text{Co} - \text{Ce})}{\text{Co}}
\]  

Where \( \text{Co} \) is the initial concentration, and \( \text{Ce} \) is the final concentration of mercury.

The first concentration of mercury solution was 5, where 50 g of bentonite was added into 200 mL mercury solution in 1, 5, 10, 15, 20, 25, and 30 minutes at 200 rpm using magnetic stirrer at neutral pH 6 at room temperature. Then, the concentration of mercury in the solution was measured uses AAS Perkin Elmer 900F.
Activated at the various concentration of 0.5, 1, and 1.5 M, bentonite’s sorption ability is presented in Figure 2. It shows that the best chemical activator for Hg sorption is $\text{H}_3\text{PO}_4$ at 1.5 M concentration. This study showed that higher concentration of chemical activators would have an impact on sorption ability. $\text{H}_3\text{PO}_4$ activators are able to hydrate organic molecules from bentonite better, limit tar formation, and decompose organic compounds.

However, bentonite material is a unique material and is slightly different from other materials commonly used to prepare activated carbon (i.e., coconut shell). Acid activation is also reported to change montmorillonite on bentonite. Montmorillonite decompose during the pyrolysis and the activation using chemical activators. This treatment also showed a change in the crystal structure (Önal et al., 2002).

**Figure 2.** Effect of contact time on the mercury concentration change in solution

**Figure 3.** Effect of contact time on the sorption of mercury in solution
As seen in Figures of 2 and 3, the best activators are H₂PO₄ with concentration 1.5 M. This research showed that the higher the concentration of activators, the larger the impact of sorption capacity will be. This study informs that the significant concentration will have an impact on the cleaning of the surface area. Chemical activation has an effect on degrading or hydrating bentonite during the carbonization process, limiting tar formation, assisting in decomposition of bentonite compounds for subsequent activation, dehydrating water trapped in bentonite cavity, removing hydrocarbon deposits generated during the carbonization process and protecting the surface of the bentonite, to reduce the possibility of oxidation. These activators affected to suppress hydrocarbon impurities in their pores.

### 3.5 Kinetic Studies

In kinetic studies, pseudo-first and second-orders were used to find the reaction models. Pseudo-first and second-orders were calculated using the bentonites interface's experimental kinetic data. The models of bentonite sorption by pseudo-first and second-order were calculated by Equation 3.

\[
\frac{dq_t}{dt} = K (qe - qt)
\]  
(3)

Where \( qe \) is equilibrium sorbent amount in \( \mu g/kg \), \( qt \) is the amount of sorbent in \( t \) time on \( \mu g/g \), and \( K \) is the pseudo-first-order reaction per minute. The condition of \( qt = 0 \) when \( t = 0 \) or \( qt = qt \) at \( t = t \), using Equation 4.

\[
qt - qe (1 - e^{-kt})
\]  
(4)

Maximum exponential rises an equation with two parameters, nonlinear regression, single, as in Equation 4.

\[
Y = a(1 - e^{bx})
\]

For isotherm study, the experiment used several concentration variation of mercury in solutions (30, 50, 70, 90, 100, 150, 200, 300, 400 and 500) ppm (mg/L). These mercury solutions were added into 1 g activated bentonite with 1.5 M H₂PO₄, HCl, and ZnCl₂. This concentration was chosen because it has the highest sorption ability. Isotherm models will be compared with ARE and chi-square value to check the isotherm's best model.

The models of the isotherm are presented in Figure 4 to show that activated bentonite (H₂PO₄) followed the Langmuir model. However, as seen in \( R^2 \) of this regression, they have the same value at 0.98, so the next challenge is to find the best model of these isotherm models.

Equations of 10 and 11 can be used to find the best model of bentonite sorption, where \( C_0 \) is the X-axis, \( qe \) on this works is \( Y1 \), \( qe \) Langmuir model is \( Y2 \) and \( qe \) Freundlich model is \( Y3 \) (Table 4). This research was carried out at room temperature (±28°C).
Table 2. Freundlich check use ARE and chi-square

| Ce  | qe (Freundlich Model) | qe (This research) | ARE  | Chi-square |
|-----|----------------------|--------------------|-------|------------|
| 1.295 | 7.743               | 5.741              | 3.487 | 0.518      |
| 4.740 | 11.666              | 9.052              | 2.887 | 0.586      |
| 9.924 | 14.732              | 12.015             | 2.261 | 0.501      |
| 15.712 | 17.032             | 14.858             | 1.464 | 0.278      |
| 20.580 | 18.548              | 15.884             | 1.677 | 0.382      |
| 49.566 | 24.462              | 20.087             | 2.188 | 0.799      |
| 84.775 | 29.003              | 23.045             | 2.585 | 1.224      |
| 161.148 | 35.526             | 27.770             | 2.793 | 1.693      |
| 236.012 | 40.075             | 32.798             | 2.219 | 1.322      |
| 335.516 | 44.784             | 32.897             | 3.613 | 3.155      |
| **Total** | **25.174**         | **10.447**         |       |            |

Table 3. Langmuir check use ARE and chi-square

| Ce  | qe (Langmuir Model) | qe (This research) | ARE  | Chi-square |
|-----|---------------------|--------------------|-------|------------|
| 1.295 | 2.316               | 7.176              | 6.773 | 10.201     |
| 4.740 | 7.419               | 11.315             | 3.443 | 2.046      |
| 9.924 | 13.072              | 15.019             | 1.297 | 0.290      |
| 15.712 | 17.585              | 18.572             | 0.531 | 0.055      |
| 20.580 | 20.449              | 19.855             | 0.299 | 0.017      |
| 49.566 | 29.523              | 25.108             | 1.758 | 0.660      |
| 84.775 | 33.968              | 28.806             | 1.792 | 0.784      |
| 161.148 | 37.761              | 34.713             | 0.878 | 0.246      |
| 236.012 | 40.361              | 39.997             | 0.412 | 0.073      |
| 335.516 | 40.121              | 39.541             | 0.185 | 0.014      |
| **Total** | **17.369**         | **14.388**         |       |            |

Table 4. Comparison of Langmuir and Freundlich Model for activated bentonite using H_3PO_4

| C (x) | Y1  | Y2  | Y3  |
|-------|-----|-----|-----|
| 1.295 | 7.743 | 2.316 | 11.666 |
| 4.740 | 11.315 | 7.418 | 14.732 |
| 9.924 | 15.019 | 13.072 | 17.032 |
| 15.712 | 18.572 | 17.585 | 18.548 |
| 20.580 | 19.855 | 18.572 | 20.580 |
| 49.566 | 25.108 | 25.108 | 49.566 |
| 84.775 | 33.968 | 33.968 | 84.775 |
| 161.148 | 37.761 | 37.761 | 161.148 |
| 236.012 | 40.361 | 40.361 | 236.012 |
| 335.516 | 41.121 | 41.121 | 335.516 |
| **Total** | **44.783** | **44.783** | **44.783** |

As seen in Figure 4, Freundlich model is closer than Langmuir. This result showed that bentonite sorption following the Langmuir model, although R^2 of Freundlich is larger than that of Langmuir. However, the value of R^2 equals to 0.98. This research showed that activated bentonite has heterogeneous layers. Every layer has different sorption abilities. This isotherm gives an expression which defines the surface heterogeneity and the exponential distribution of active sites and their energies (Ayawei et al., 2017).

Figure 4. Compilation of isotherm models from activated bentonites using H_3PO_4

Figure 5. Compilation of isotherm models from activated bentonite using HCl
Ce | qe (Freundlich Model) | qe (This research) | ARE | Chi-square
---|---|---|---|---
2.295 | 9.277 | 5.541 | 6.742 | 1.504
5.740 | 12.393 | 8.852 | 4.000 | 1.012
9.940 | 14.739 | 12.012 | 2.270 | 0.505
13.712 | 16.315 | 15.258 | 0.693 | 0.069
19.580 | 18.258 | 16.084 | 1.352 | 0.259
40.566 | 22.980 | 21.887 | 2.226 | 0.693
83.775 | 28.895 | 28.770 | 2.226 | 1.166
156.148 | 35.174 | 35.998 | 4.000 | 2.270
220.012 | 39.196 | 39.097 | 0.889 | 0.261
304.516 | 43.433 | 39.097 | 1.109 | 0.433

Total | 22.211 | 6.365

Table 6. Langmuir check use ARE and chi-square

Ce | qe (Langmuir Model) | qe (This research) | ARE | Chi-square
---|---|---|---|---
2.295 | 2.316 | 7.176 | 2.316 | 7.743
5.740 | 7.419 | 11.315 | 3.443 | 2.046
9.940 | 13.072 | 15.019 | 1.297 | 0.290
13.712 | 17.585 | 18.572 | 0.531 | 0.055
19.580 | 20.449 | 19.855 | 0.299 | 0.017
40.566 | 29.523 | 25.108 | 1.758 | 0.660
83.775 | 33.968 | 28.806 | 1.792 | 0.784
156.148 | 37.761 | 34.713 | 0.878 | 0.246
220.012 | 39.306 | 40.997 | 0.412 | 0.073
304.516 | 40.361 | 41.121 | 0.185 | 0.014

Total | 17.369 | 14.388

Table 7. Comparision of Langmuir and Freundlich model for activated bentonite using HCl

Ce (X) | Y1 | Y2 | Y3
---|---|---|---
2.295 | 7.176 | 2.316 | 7.743
5.740 | 11.315 | 7.418 | 11.666
9.940 | 15.019 | 13.071 | 14.732
13.712 | 18.572 | 17.585 | 17.032
19.580 | 19.855 | 20.449 | 18.548
40.566 | 25.108 | 29.522 | 24.482
83.775 | 28.806 | 33.966 | 29.003
156.148 | 34.713 | 37.757 | 35.525
220.012 | 39.306 | 40.997 | 40.075
304.516 | 41.121 | 40.359 | 44.783

R² from Figure 6 showed the same value at 0.96. These models would be compared using ARE and chi-square to reach the sorption model, where K = 0.04386 and qmax = 43.1034 for Langmuir and K = 7.13674 and 1/n = 0.3158 for Freundlich. Tables of 5 and 6 are used as references to find the best isotherm model, where Ce is the X-axis, qe on this works is Y1, qe Langmuir model is Y2, and qe Freundlich model is Y3. This experiment were carried out at room temperature. Therefore, activated bentonite using ZnCl₂ corresponds to Freundlich model (0.99). This result was also corrected with ARE and chi-square to find the best model of the isotherm.

Based on the analytical statistic (Table 7), the chi-square is less than ARE. It means that this result is correct for both models. The last is activated bentonite using ZnCl₂. This isotherm model is in accordance with Freundlich model. Thus, all of the activated bentonite showed Freundlich model.

This model describes reversible and unideal adsorption process. This model is not limited to monolayer formation as its application to the multilayer adsorption is possible. This model informed that affinities and adsorption heat do not need to be evenly distributed with the heterogeneous surface.
Actually, this model can be developed into another isotherm model, such as Sips and Jovanich Isotherm because these models followed homeopathic patch approximation proposed by the theory of Oliver and Ross (Ayawei et al., 2017). Freundlich’s model is also similar to the Halsey isotherm model. This model fits multilayer adsorption as well as the heterogeneous surface (Ayawei et al., 2017). Freundlich model is a hybrid with Redlich-Peterson isotherm model (Al-Ghouti and Da’aana, 2020). These models informed that researchers throughout the world could predict various membrane pore sizes and thicknesses (Ge et al., 2006). Ge et al. (2006) found that when the variation is more considerable, it will impact on delayed total saturation (Bruggen, 2003).

4. Conclusion
Bentonite is a potential material for an effective adsorbent. This study proved that bentonite has good adsorption ability of mercury. The bentonite activated using a variety of acid, base and salt activators showed that each activator has different ability to clean the bentonite pores. The isotherm model suitable for mercury adsorption by bentonite is the Freundlich model.

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