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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 (H_3PO_4, HCl, and $ZnCl_2$) 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 H_3PO_4 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, H_3PO_4, NaOH, KOH, NaCl, and $ZnCl_2$). 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 ($Hg(NO_3)$). The activators used were phosphoric acid (H_3PO_4), zinc chloride ($ZnCl_2$), 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 $ZnCl_2$. 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 H_3PO_4-activated bentonites.

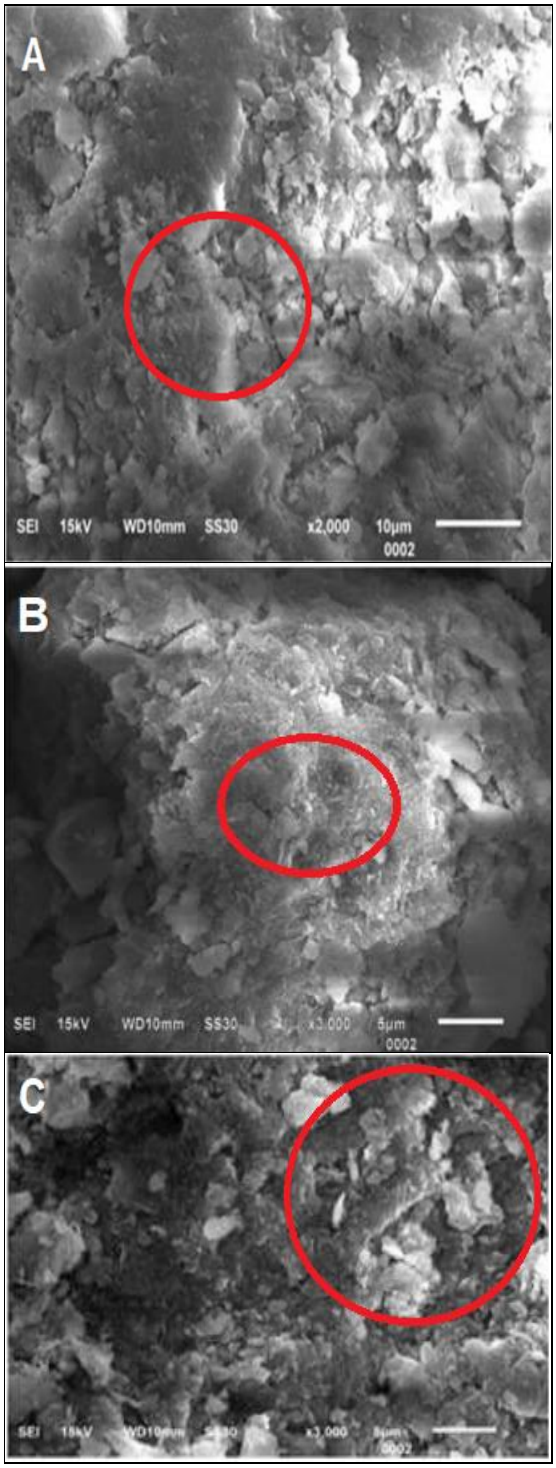

Figure 1. Surface morphology of activated bentonite using (a) HCl, (b) $ZnCl_2$ and H_3PO_4

3.2 Chemical Composition of Bentonites

EDS (combined with SEM) was used to identify the chemical composition of bentonite. This instrument showed that different activator had different impact on the materials' element and oxide. The majority of oxide in bentonite is SiO_2.

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. SiO_2 and Al_2O_3 are the main components in developing montmorillonite tetrahedral layer. It is in line with the EDS analysis results in which SiO_2 and Al_2O_3 oxides are the main components of bentonite. Negative ions in octahedral bentonite layer will be balanced with other positive ions, such as Ca^{2+}, Mg^{2+}, Na^{+}, and K^{+}. The ability of bentonite to attack will affect type of activator used. The use of activator with higher H^{+} equivalent value will result better deterrence capabilities.

Table 1. Composition of activated bentonites

Elements (%)	H_3PO_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	0.04
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.

$$qe = \frac{V\,(Co - Ce)}{M} \qquad (1)$$

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

$$\text{Percent removal} = \frac{100\ (Co - Ce)}{Co} \qquad (2)$$

Where Co is the initial concentration, and 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 H_3PO_4 at 1.5 M concentration. This study showed that higher concentration of chemical activators would have an impact on sorption ability. H_3PO_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).

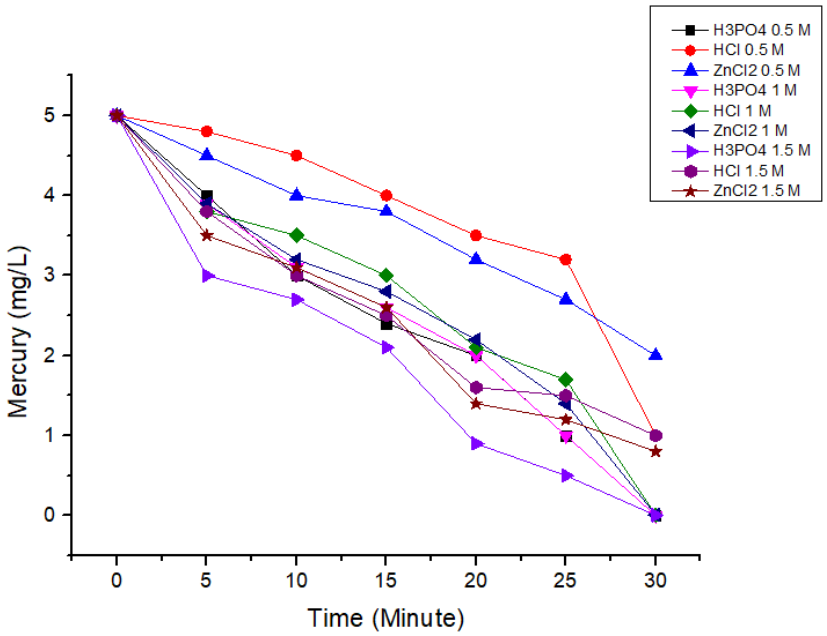

Figure 2. Effect of contact time on the mercury concentration change in solution

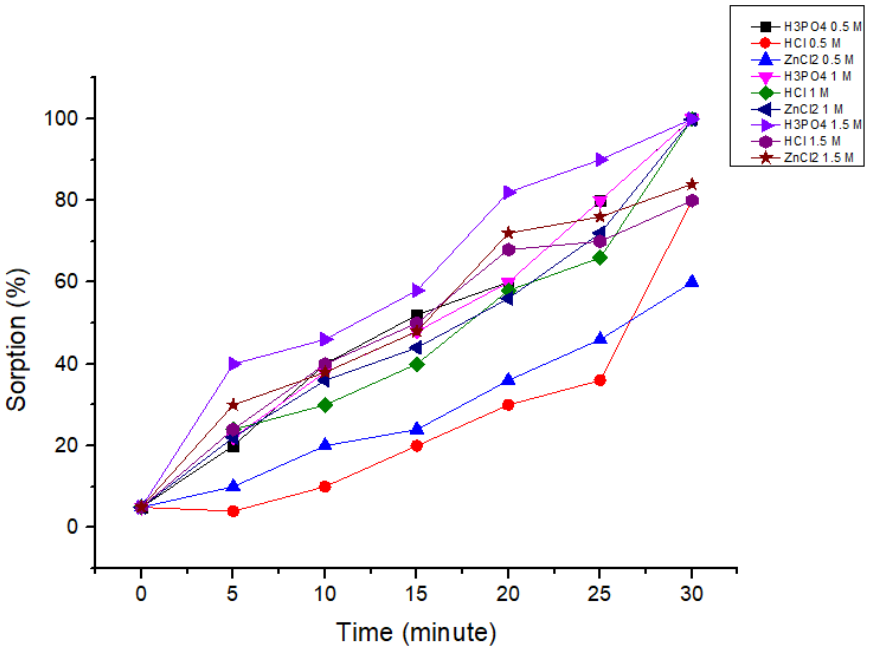

Figure 3. Effect of contact time on the sorption of mercury in solution

As seen in Figures of 2 and 3, the best activators are H_3PO_4 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.

$$\frac{\partial qt}{\partial q} = K\,(qe - qt) \qquad (3)$$

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

$$qt - qe\,(1 - e^{(-k\cdot t)}) \qquad (4)$$

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

$Y = a^{(1 - e^{-bx})}$ was used in Equation 3 to fit the experimental data. The model of pseudo-second-order is used according to Equation 5.

$$\frac{\partial qt}{\partial t} = K2\,(qe - qt)^2 \qquad (5)$$

K2 is pseudo second-order rate constant (μg/(kg·min)). This equation can provide integration of Equation 3 to prove Equation 6.

$$qt = \frac{qe^2\cdot K2\cdot t}{1 + K2\cdot qe\cdot t} \qquad (6)$$

Regression linear of the hyperbola equation with one and two parameters from Equation 6 results in Equation 7.

$$y = \frac{ax}{b + x} \qquad (7)$$

Therefore, Equation 5 can be reformulated as Equation 8.

$$qt = \frac{qe\cdot t}{\frac{1}{K2\cdot qe} + t} \qquad (8)$$

The sorption data was in accordance with the Freundlich reaction using Equation 9.

$$S = Kf\cdot C^{1/n} \qquad (9)$$

Where S is the sorption (mg/kg), C is the concentration at equilibrium (mg/L), and Kf is dimensionless of the sorption parameter. The last n is linearity parameter. Then, isotherm models were analyzed using the Average Relative Error (ARE) equation as follows.

$$ARE = \frac{100}{n}\sum \frac{|qe - qe'm|}{qe} \qquad (10)$$

Where qe'm is the value of qe from the isotherm model. The model compared with chi-square value into Equation 11.

$$x^2 = \sum \frac{(qe - qe'm)^2}{qe'm} \qquad (11)$$

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 bentonites with 1.5 M H_3PO_4, HCl, and $ZnCl_2$. 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 (H_3PO_4) 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 K = 7.13674 and 1/n = 0.3158 for Freundlich and K = 0.04386 and qmax = 43.1034 for Langmuir. Tables of 2 and 3 are used as a reference to find the best model of isotherm, where Ce 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	24.482	20.087	2.188	0.789
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	39.306	40.997	0.412	0.073
335.516	40.361	41.121	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	7.176	2.316	7.743
4.740	11.315	7.418	11.666
9.924	15.019	13.071	14.732
15.712	18.572	17.585	17.032
20.580	19.855	20.448	18.548
49.566	25.108	29.522	24.482
84.775	28.806	33.966	29.003
161.148	34.713	37.759	35.525
236.012	40.997	39.304	40.075
335.516	41.121	40.359	44.783

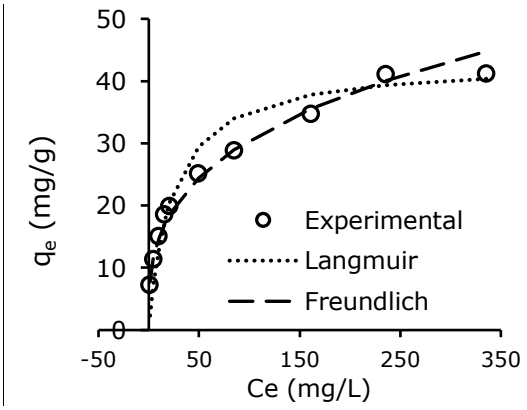

Figure 4. Compilation of isotherm models from activated bentonites using H_3PO_4

As seen in Figure 5, 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).

Table 5. Freundlich check use ARE and chi-square

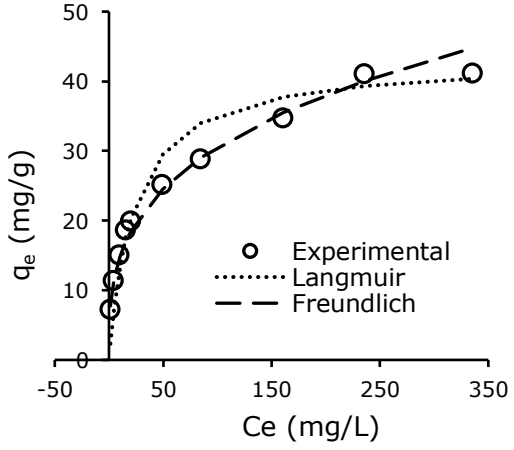

Figure 5. Compilation of isotherm models from activated bentonite using HCl

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	0.500	0.052
83.775	28.895	23.245	2.430	1.105
156.148	35.174	28.770	2.226	1.166
220.012	39.196	35.998	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	6.773	10.201
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. Comparission 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	20.448	18.548
40.566	25.108	29.522	24.482
83.775	28.806	33.966	29.003
156.148	34.713	37.759	35.525
220.012	40.997	39.304	40.075
304.516	41.121	40.359	44.783

R^2 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 $ZnCl_2$ 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 $ZnCl_2$. This isotherm model is in accordance with Freundlich model. Thus, all of the activated bentonite showed Freundlich model.

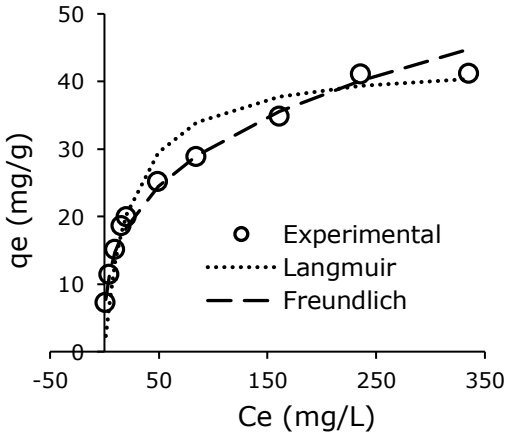

Figure 6. Compilation of isotherm models from activated bentonites using $ZnCl_2$

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'ana, 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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