Enhancing Adsorption Efficiency of Cd Heavy Metal Ions using Aceh's Natural Bentonite: A Design Expert 13 Optimization Study

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Received: July 22, 2023 Approved: July 29, 2023

Abstract

Wastewater containing heavy metal ions presents a formidable challenge to environmental sustainability because heavy metals are nonbiodegradable and toxic. Cadmium has been identified by scientific consensus as one of the most perilous and deleterious chemicals. These metal ions have a significant effect on many illnesses in living organisms, including humans. Therefore, an effective absorption technique is required to mitigate the issues caused by this waste. Adsorption is frequently used to remediate heavy metal waste and improve the processing process because of its versatility and suitability as a technology. The principal aim of this investigation was to augment the adsorptive potency of cadmium (Cd) ions by optimizing the utilization of natural Aceh bentonite. The optimization process utilizes Design Expert 13, specifically Box Behnken Design with Response Surface Methodology. The results of the optimization revealed the significance of bentonite mass (1-2 g), stirring time (30-200 minutes), and initial Cd adsorbate concentration (100-500 mg/l) on the absorption of Cd (II) ions. Optimal conditions for Cd (II) ion adsorption by Aceh natural bentonite were achieved with an agitation time of 2 mass, 126.8 minutes of agitation time, and Cd (II) ion concentration of 500 mg/l. Under these conditions, the adsorption of Cd (II) ions by Aceh natural bentonite was 220.5 mg/l, and the desirability of this outcome was 0.84.

Keywords: heavy metal, adsorption, optimization, efficient, desirability

Abstrak

Air limbah yang mengandung ion logam berat menjadi tantangan serius bagi keberlanjutan lingkungan karena logam berat bersifat tidak terdegradasi dan beracun. Kadmium telah diidentifikasi oleh konsensus ilmiah sebagai salah satu zat kimia paling berbahaya dan merusak. Ion logam ini memiliki pengaruh signifikan pada berbagai penyakit yang mempengaruhi makhluk hidup, termasuk manusia. Teknik penyerapan yang efektif diperlukan untuk mengatasi masalah yang disebabkan oleh limbah ini. Adsorpsi sering digunakan untuk mengatasi limbah logam berat dan meningkatkan proses pengolahannya karena sifatnya yang serbaguna dan cocok sebagai teknologi. Tujuan utama dari penelitian ini adalah untuk meningkatkan daya serap ion Kadmium (Cd) melalui optimasi penggunaan bentonit alam Aceh. Proses optimasi menggunakan Design Expert 13, khususnya dengan Box Behnken Design dan Response Surface Methodology. Hasil optimasi menunjukkan bahwa massa bentonit (1-2 g), waktu pengadukan (30-200 menit), dan konsentrasi adsorbat Cd awal (100-500 mg/l) secara signifikan mempengaruhi penyerapan ion Cd (II). Kondisi optimal untuk penyerapan ion Cd (II) oleh bentonit alam Aceh dicapai dengan 2 gram massa bentonit, waktu pengadukan selama 126,8 menit, dan konsentrasi ion Cd (II) sebesar 500 mg/l. Pada kondisi-kondisi ini, penyerapan ion Cd (II) oleh bentonit alam Aceh mencapai 220,5 mg/l, dan tingkat keinginan dari hasil ini adalah 0,84.

Kata kunci: logam berat, adsorpsi, optimasi, efisien, desirabilitas

1. Introduction

The industrial growth of a country, ranging from small to large industries, reflects its level of development. While the sector exerts a positive influence by engendering novel commodities and employment opportunities, it also exerts a deleterious effect by generating pernicious waste products and noxious heavy metals. Improper management of heavy metals in waste can lead to environmental contamination, damaging the environmental balance system [1], [2]. The adsorbent used for adsorption is natural bentonite [3], [4]. Indonesia possesses natural mineral resources found in several provinces, including clay. The soil's natural mineral content comprises montmorillonite, chlorite, illite, halloysite, and kaolinite. Montmorillonite, a mineral prevalent in the soil, is distinguished by its expansive characteristics and high cation exchange capacity. Bentonite, a variety of clay, is prominently composed of

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Bentonite is a type of mineral found in large quantities in Indonesia. The utilization of adsorbents is widespread in numerous applications owing to their distinct features, namely an interlayer structure that can be tailored to increase their absorption capability [9], [10]. Numerous varieties of materials possess the capability to serve as adsorbents in the adsorption process of heavy metals, encompassing but not restricted to bentonite, activated charcoal, zeolite, and magnetite, ascertained by Monika et al. [11]. Among these options, bentonite stands out as a relatively cheap and abundant natural adsorbent. The fundamental constituents of the material comprise alumina and silica. However, the effectiveness of bentonite during the adsorption process may not reach its maximum potential in the event of lacking prior modification. Untreated bentonite can absorb water content, leading to instability in its application [12].

Natural bentonite exhibits a low level of absorption primarily because of the presence of mineral impurities. However, this absorption capacity can be significantly enhanced through a purification process called activation. As a result of activation, bentonite acquires a high absorption capacity. Moreover, bentonite possesses the unique ability to absorb and bind water within its structural layers. Its characteristics include low resistivity, expansive properties, and remarkable cost-effectiveness. Additionally, it is significant to mention that bentonite displays properties of non-corrosiveness and, due to its composition based on clay, it showcases exceptional durability, thereby rendering it remarkably resistant to facile deterioration [13], [14].

The principal dissimilarity between the present investigation and antecedent inquiries is situated in the refinement procedure of the primary source constituent. In this research, the raw material is purified from impurities through the intercalation process of Na ions. Additionally, bentonite adsorbents are applied to enhance the absorption capacity of Cd²⁺ metal. The chosen bentonite used as an adsorbent is sourced from the Trieng Gadeng sub-district, Pidie Jaya. The primary objectives of this study are to investigate the individual effects and interactions, increase the uptake of Cd (II) ions, and optimize the process using Design Expert 13, Design Box Behnken, and Response Surface Methodology (RSM).

2. Material and Methods

Materials used
In the current study, several materials were utilized to investigate a particular subject. The primary component examined was Bentonite, which was sourced from Trieng Gadeng sub-district, situated in the Pidie Jaya district of Aceh Province, Indonesia. Bentonite is a clay mineral known for its wide-ranging industrial applications, and its inclusion in the research aimed to explore specific properties or behaviors. Furthermore, the investigation utilized several distinct compounds, including cadmium sulfate, formally known as CdSO₄, sodium chloride, and an acetate buffer procured from the reputable pharmaceutical company, Merck located in Germany. These chemicals might have been selected to either facilitate reactions or create specific test conditions. The inclusion of distilled water is common in various experimental setups as a control and a solvent.

Adsorbent preparation
In this study, the process of preparing Bentonite for experimentation involved several steps. Initially, the Bentonite was subjected to grinding using a ball mill and then sieved to achieve a particle size of 200 mesh, ensuring uniformity in the sample. To further enhance its properties and remove any moisture, the sieved Bentonite was heated at a temperature of 105°C for two hours. After heating, it was transferred to a desiccator, a specialized container used for drying substances, to cool down and achieve complete dryness. Once the prepared Bentonite was dry and ready, it was combined with 250 ml of acetate buffer, carefully maintaining a pH level of 4.8. The mixture was thoroughly stirred for five hours, allowing the Bentonite to interact with the buffer and facilitating the reaction. During this period, any CO₂ gas formed as a result of the reaction was monitored until no further gas evolution occurred. After a five-hour duration of stirring, the mixture underwent a centrifugal force application for a period of 10 minutes, ultimately yielding its separation. This step aimed to separate the Bentonite from the liquid components of the mixture, allowing researchers to analyze and investigate the properties and characteristics of the treated Bentonite [15].

Adsorption Experiment
The adsorption of Cd (II) ions was carried out using natural Aceh bentonite, and the intercalation of Na from NaCl was performed in batch Erlenmeyer flasks. Aceh natural bentonite was purified up to 25 grams in a measuring flask and then added to 500 ml of distilled water. The mixture was stirred with a magnetic stirrer for 24 hours at 70°C. Subsequent to this, the solution underwent separation via decantation,
following which the precipitate was subjected to drying at a temperature of 105°C. Next, 100 ml of Cd (II) solution with varying concentrations and doses of bentonite were placed in the flask and stirred at 200 rpm. Subsequently, the mixture was filtered for the analysis of Cd (II) residue. The adsorption of Cd (II) ions utilizing natural bentonite and intercalation was assessed by means of the subsequent equation.

\[
Cd(II) \text{ ion uptake} = (C_0 - C_t)V
\]  

(1)

Capacity (absorptive power) \( q_t = \frac{(C_0 - C_t)V}{m} \)  

(2)

Where \( C_0 \) represents the initial concentration of Cd\(^{2+} \) (mg/l), \( C_t \) represents the remaining concentration of Cd\(^{2+} \) at time \( t \) (minutes), \( V \) is the volume of the Cd solution (ml), and \( m \) is the mass of bentonite (g). Cd\(^{2+} \) ion absorption is measured in mg/l, while Cd\(^{2+} \) ion absorption capacity is measured in mg/g. \( Y_1 \) represents the response variable for Cd\(^{2+} \) uptake by natural bentonite, while \( Y_2 \) represents Cd\(^{2+} \) uptake by intercalated bentonite.

**Experiment Design and Analysis**

The influence of the variables \( X_1, X_2, \) and \( X_3 \) individually and the interaction with the uptake of natural bentonite (\( Y_1 \)) can be written in equation (3).

\[
Y_1 = f (X_1, X_2, X_3)
\]  

(3)

Description:

\( Y_1 = \) Absorption of Cd\(^{2+} \) ions (mg/l) by natural bentonite  
\( X_1 = \) Mass of natural bentonite (g)  
\( X_2 = \) Mixing time (minutes)  
\( X_3 = \) Concentration of ion Cd\(^{2+} \) in beginning (mg/l)

The influence of the variables \( X_1, X_2, \) and \( X_3 \) individually and the interaction with the uptake of intercalated bentonite (\( Y_2 \)) can be written in equation (4).

\[
Y_2 = f (X_1, X_2, X_3)
\]  

(4)

Description:

\( Y_2 = \) Absorption of Cd\(^{2+} \) ions (mg/l) by intercalated bentonite  
\( X_1 = \) Mass of intercalated bentonite (g)  
\( X_2 = \) Mixing time (minutes)  
\( X_3 = \) Initial Cd\(^{2+} \) ion concentration (mg/l)

The experimental design uses Design Expert 13, Design Box Behnken, Response Surface Methodology (RSM). Experimental design data as in Table 1.

**Table 1.** Design and Experimental Analysis of the Effect of Bentonite Mass (\( X_1 \)), Agitation Time, and Initial Concentration of Cd (II) on Natural Aceh Bentonite Uptake and Intercalated Na Ion Bentonite (Na-Bentonite)

<table>
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<tr>
<th>Run</th>
<th>( X_1 ) (g)</th>
<th>( X_2 ) (minute)</th>
<th>( X_3 ) (mg/l)</th>
<th>( Y_1 ) (mg/l)</th>
<th>( Y_2 ) (mg/l)</th>
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<tr>
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<td>2</td>
<td>115</td>
<td>500</td>
<td>211.2621</td>
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3. Results and Discussion

Table 1 presents the experimental design and analysis of the effect of bentonite mass (X₁), stirring time, and initial concentration of Cd (II) on the adsorption of natural Aceh Bentonite and intercalated Na ion bentonite (Na-Bentonite). The study’s objective is to evaluate the potential use of natural Aceh bentonite and Na-bentonite as adsorbents to reduce the concentration of Cd (II) in a solution. The parameters chosen for the study were bentonite mass (X₁), stirring time, and initial concentration of Cd (II). The current investigation utilized an experimental approach that was consistent with the principles of a completely randomized design (CRD), which included the incorporation of three factors and three levels. The assessment of the concentration of Cd (II) in the solution, following its adsorption by bentonite, was executed utilizing an atomic absorption spectrophotometer.

The experimental results demonstrate that both types of bentonites exhibit high adsorption efficiency in removing Cd (II) from the solution. Na-Bentonite displayed a higher absorption capacity compared to natural Aceh bentonite. Moreover, the augmentation of bentonite mass and duration of stirring resulted in an elevation of the Cd (II) uptake. Nevertheless, the initial concentration of Cd (II) failed to exhibit a noteworthy impact on the adsorption potential of the two types of bentonites. This investigation provides essential perspectives on the possible application of bentonite as an adsorbent for remedying heavy metal contamination in water, with Na-Bentonite exhibiting exceptional efficacy. Further investigation is necessary to fully grasp the interaction mechanism that occurs between heavy metals and bentonite, while also striving to optimize experimental conditions.

The findings from Table 1 indicate that the experimental factors, including the mass of Aceh Natural bentonite, agitation time, and initial concentration of Cd (II), had a significant impact on the adsorption capabilities of both Aceh natural bentonite and Na-intercalated bentonite. Notably, the highest uptake results were observed during run 16, where the optimal conditions were achieved: a bentonite mass of 2 grams, stirring time of 20 minutes, and an initial Cd concentration of 300 mg/l. Under these conditions, the maximum removal of Cd (II) reached 230 mg/l for Aceh natural bentonite and 235.8 mg/l for Na-intercalated bentonite. To further comprehend the effects of each experimental factor and their interactions, Table 2 presents detailed results. This information provides valuable insights into the adsorption efficiency and effectiveness of the tested bentonite types in removing Cd (II) from the solution. The study’s significance lies in its potential contribution to tackling heavy metal pollution in water, as both types of bentonites exhibited high adsorption efficiency. Na-intercalated bentonite displayed superior performance compared to Aceh natural bentonite, indicating its promise as an effective adsorbent for Cd (II) removal.

Table 2. Analysis of variance (ANAVA) Effect of Natural Bentonite Mass and Intercalated Bentonite (X₁), Agitation Time (X₂), and Initial Cd (II) Concentration on BA and BI Absorption (JK = Sum of Squares, DK = Degrees of Freedom, KT = Middle Square, F-v = F value, p-v = p value)

<table>
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<tr>
<th>Source</th>
<th>JK</th>
<th>DK</th>
<th>KT</th>
<th>F-v</th>
<th>p-v</th>
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<th>JK</th>
<th>DK</th>
<th>KT</th>
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<td>9194.90</td>
<td>16.98</td>
<td>0.0043</td>
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<td>670.24</td>
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intercalation, optimization, and desirability so that the manipulation of certain variables will impact the overall effect can be seen based on the optimization of the RSM (response surface methodology), the Box Behnken Design (BBD) method, the graph shows the effect of these three variables, their interactions in influencing the absorption process of Cd (II) by the tested bentonite materials. The outcomes of the ANOVA assist in comprehending the contribution of each variable and facilitate the identification of the most influential factors in the adsorption process.

Table 2 presents the outcomes of the analysis of variance (ANOVA) that examine the effects of Natural Bentonite Mass and Intercalated Bentonite (X₁), Agitation Time (X₂), and Initial Cd (II) Concentration on the absorption capacity (BA and BI) of the examined bentonite types. The ANOVA comprises various parameters such as JK (Sum of Squares), DK (Degrees of Freedom), KT (Mean Square), F-v (F value), and p-v (p value). These statistical measures are instrumental in evaluating the importance of each factor and their interactions in influencing the absorption process of Cd (II) by the tested bentonite materials. The outcomes of the ANOVA assist in comprehending the contribution of each variable and facilitate the identification of the most influential factors in the adsorption process.

The final equation in the code factor for Aceh’s natural bentonite:

\[ Y_1 = 141.15 + 36.8 X_1 + 33.9 X_2 + 39.95 X_3 + 20.9 X_1 X_2 + 26.8 X_1 X_3 + 19.4 X_2 X_3 - 5.8 X_1^2 - 10.9 X_2^2 - 27.8 X_3^2 \]  

The final equation in code factors for Na-bentonite (intercalated bentonite):

\[ Y_2 = 166.9 + 36 X_1 + 34.6 X_2 + 41.8 X_3 + 18.5 X_1 X_2 + 26.4 X_1 X_3 + 12.9 X_2 X_3 - 18.3 X_1^2 - 18X_2^2 - 35.8 X_3^2 \]

Equation (5) describes the influence of natural Aceh bentonite mass (X₁), agitation time (X₂), and the initial concentration of Cd (II) (X₃), and the interaction is very significant with a correlation R² = 0.92. Equation (6) describes the influence of the mass of Na-bentonite (X₁), agitation time (X₂), and the initial concentration of Cd (II) (X₃), and the interaction is very significant with a correlation R² = 0.97. Absorption of Na-bentonite > Aceh bentonite without the intercalation of the bentonite mathematical model, equation 6, is more accurate than equation (5). From the data in Table 1, Table 2 and equations (5) and (6), a 3-D graph shows the effect of these three variables, their interaction, optimization, and desirability so that the overall effect can be seen based on the optimization of the RSM (response surface methodology), the BBD method (Box Behnken Design), the level of accuracy, and also being able to compare the two bentonite qualities. Equation (5) represents the impact of natural Aceh bentonite mass (X₃), agitation time (X₂), and initial Cd (II) concentration (X₁), indicating a highly significant interaction with a correlation coefficient (R²) of 0.92. Similarly, equation (6) demonstrates the influence of Na-bentonite mass (X₁), agitation time (X₂), and initial Cd (II) concentration (X₃), with a highly significant interaction and a higher correlation coefficient of 0.97.

Notably, the absorption capacity of Na-bentonite was found to surpass Aceh bentonite without intercalation, with equation (6) providing a more accurate model than equation (5). In order to conduct an all-encompassing assessment of the information featured in Tables 1, and 2, in conjunction with equations (5) and (6), a tridimensional graph was generated to provide a comprehensive representation of the collective impact of these variables, their interconnectedness, optimization, and desirability. By utilizing response surface methodology (RSM) and the Box Behnken Design (BBD) method, the graph showcases the overall impact and allows for comparison between the two bentonite qualities. This approach facilitates identifying the optimal conditions for maximum Cd (II) adsorption and enables a comprehensive assessment of accuracy and performance. Through this analysis, researchers can gain insights into the adsorption potential of both bentonite types and make informed decisions regarding their application in Cd (II) removal, thus contributing to addressing heavy metal pollution in water and environmental remediation efforts.
Fig. 1. (a) Plot of experimental and predicted response surface for natural bentonite (b) Plot of experimental and predicted response surface for Na-bentonite (BI)

Fig. 2. (a) 3-D response surface plot of bentonite mass ($X_1$, g) and stirring time ($X_2$, minutes) at Cd (II) removal concentration ($Y_1$, mg/l) for natural bentonite (b) for intercalated bentonite (Na bentonite)
From Figure 1, Figure 2, Figure 3 and Figure 4 3-D the influence of bentonite mass \( (X_1) \), stirring time \( (X_2) \), and initial \( \text{Cd}^{2+} \) ion concentration on the removal of \( \text{Cd}^{2+} \) ions individually and the interaction was very significant. One noteworthy finding is the effect of intercalation of Na ions, transforming Ca-bentonite into Na-bentonite, which leads to increased absorption capacity. This process involves replacing Ca atoms with Na atoms, resulting in enhanced adsorption performance. The 3-dimensional plots provide a comprehensive visual representation of how varying the bentonite mass, stirring time, and \( \text{Cd}^{2+} \) ion concentration affects the overall adsorption efficiency. The current discoveries hold significant value in the enhancement of the adsorption procedure and in the comprehension of the correlation among the factors implicated. The research’s significance is rooted in its potential applications in dealing with heavy metal pollution in water. Through the comprehension of the singular and interactive influences of bentonite mass, stirring duration, and initial \( \text{Cd}^{2+} \) concentration, enhanced adsorption systems that exploit bentonite as an efficacious adsorbent for \( \text{Cd}^{2+} \) eradication can be formulated. Ultimately, this exploration furnishes significant perceptions towards the advancement of sustainable resolutions for environmental reclamation and aqueous refinement.
Figures 5 (a) and (b) show the overall optimization results for bentonite with a mass of 2 grams, a stirring time of 126.78 minutes, a Cd$^{2+}$ ion concentration of 500 mg/l. The quantification of Cd$^{2+}$ (removal) was documented at 220.468 mg/l for natural bentonite and 225.768 mg/l for intercalated bentonite. Figure 5 (a) and (b) show that the comprehensive optimization results adsorption capability of Cd$^{2+}$ ions escalate from natural bentonite to intercalated bentonite. This means that intercalated bentonite is more effective in removing Cd$^{2+}$ ions from solution. This shows that the intercalation process has succeeded in increasing the absorption properties of bentonite, so that it becomes more efficient in binding Cd$^{2+}$ ions.

The intercalation of bentonite is endowed with the potential for utilization in waste treatment or the mitigation of heavy metal ion concentration, notably Cd$^{2+}$, across an array of industries. The use of intercalated bentonite can be a better alternative in improving water quality and the environment as a whole. This demonstrates that the intercalation procedure has triumphed in augmenting the adsorption characteristics of bentonite, thereby rendering it more proficient in entwining Cd$^{2+}$ ions. This overall optimization can have potential applications in waste treatment or in reducing the concentration of heavy metal ions, such as Cd$^{2+}$, in various industries, including mining, agriculture, and others. Thus, the use of intercalated bentonite can be a better alternative to improve water quality and the environment in general.

4. Conclusion

In conclusion, this study evidences the remarkable influence of bentonite mass, stirring time, and initial concentration of Cd$^{2+}$ ions on the uptake or removal of Cd$^{2+}$ ions, with both individual and interaction effects being of utmost significance. The employed mathematical model for prediction demonstrated a high level of accuracy, as exemplified by the negligible disparity between predicted and actual data. Furthermore, it can be observed that the R$^2$ coefficients of 0.92 and 0.97 for natural and intercalated bentonite, respectively, provide additional evidence for the effectiveness of the model. Optimal conditions for Cd$^{2+}$ ion removal was achieved at a bentonite mass of 2 grams, a stirring time of 126.78 minutes, and an initial Cd$^{2+}$ concentration of 55 mg/l. The removal of Cd$^{2+}$ ions reached 220.468 mg/l for natural Aceh bentonite and 225.768 mg/l for intercalated Na bentonite.
The study highlights the potential for improving the quality of Aceh's natural bentonite through the intercalation of Na ions obtained from NaCl. The process employed herein considerably amplifies the adsorption proficiency of bentonite, lending it an efficacious potential as an adsorbent for curtailing heavy metal contamination, most notably Cd2+ ions in aqueous systems. The implications of these findings are invaluable in comprehending the adsorptive capability of bentonite and its practical utilization for environmental remediation endeavors. Intercalated Na bentonite exhibits superior performance, offering prospects for its practical use as an efficient adsorbent in water purification systems to combat heavy metal contamination. Further research is recommended to delve deeper into the mechanism of interaction between bentonite and heavy metals, allowing for the optimization of experimental conditions and broader applications in addressing water pollution challenges.

5. References