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Adsorption of Thorium(IV) from Aqueous Solution Using Natural Bentonite from Pacitan, Indonesia

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ABSTRACT

Separating and utilizing thorium from industrial by-products is essential for economic, energy, and environmental reasons. Natural bentonite can be an effective adsorbent for removing various contaminants, including thorium. This study aims to determine the effectiveness of natural bentonite from Pacitan as an adsorbent for thorium removal. Batch adsorption experiments were conducted using a surrogate thorium solution to evaluate the effects of adsorbent dosage, contact time, and adsorption kinetics and isotherms. The study shows that the adsorbent's pHpzc is 7. The adsorbent also shows a cation-exchange capacity of 90.69 meq/100g. The adsorption experiments revealed that using 0.1 g of adsorbent in a 50 mL solution containing 100 ppm thorium yielded an adsorption efficiency of 99.34%. Experimental observations determined an optimum contact time of 120 minutes with 99.80% efficiency. The adsorption data were well-fitted by the pseudo-second-order kinetic model and the Langmuir isotherm, suggesting chemisorption-dominated behavior consistent with monolayer coverage on homogeneous active surface sites. These findings indicate that natural bentonite from Pacitan is a promising, cost-effective material for removing thorium from wastewater.

Keywords: Pacitan bentonite, adsorption, thorium, cation-exchange capacity, wastewater treatment

INTRODUCTION

Monazite is a radioactive mineral in the Naturally Occurring Radioactive Materials (NORM) category, a by-product of tin mining. Monazite contains radioactive elements such as uranium and Thorium, as well as rare-earth elements (REEs) [1], [2]. Monazite can be strategically and economically optimized as a source of REEs and nuclear fuel. Separation of uranium and Thorium from tin slag has been carried out using several methods to recover and utilize these high-value elements [3]. Thorium, in the form of Technology-Enhanced Naturally Occurring Radioactive Materials (TENORM), is abundant in Bangka Belitung, Riau, and Kalimantan and has potential as a

source of electrical energy and nuclear fuel, attracting interest from companies developing green atomic energy. Therefore, separating and utilizing Thorium from monazite is essential for economic, energy, and environmental purposes [4], [5].

However, every processing technology will produce hazardous by-products and waste if not properly managed. Waste produced by radioactive mineral processing can be solid or liquid. This waste is classified as TENORM because of the increased potential for exposure compared to its initial state, resulting from human activities or technological processes. Management of NORM/TENORM waste containing Thorium requires a deep understanding of the potential for radiation exposure and the need to implement radiation protection measures. In addition, NORM/TENORM waste management also involves appropriate regulations and management techniques to minimize environmental and human health risks. TENORM can contain Thorium compounds such as Thorium dioxide, nitrate, and oxalate, which can cause contamination and corrosion. TENORM can contain radioactive elements and can be managed to reduce radiation risk. TENORM must be considered and treated in a manner appropriate to minimize radiation risks [6]–[8].

TENORM control strategies can be implemented by separating Thorium, precipitating it with phosphate ions, and using adsorbents such as bentonite. Adsorption is widely regarded as a superior method compared to ion exchange and solvent extraction. Its widespread application is attributed to its high capacity, low operational costs, and sustainability; the process allows straightforward regeneration without the drawback of sediment formation [8], [9].

One adsorbent that can absorb or remove pollutants such as Thorium in wastewater is natural bentonite [10], [11]. In the adsorption process, bentonite adsorbs contaminants from its surface or in its pores, thereby reducing their concentration. Bentonite has high porosity and ion-exchange capacity, allowing it to perform better in adsorption than other adsorbents. The tiny colloidal particle size of bentonite enables it to exhibit faster adsorption performance than granular adsorbents [12]. Bentonite also has higher selectivity than activated carbon in removing Th^{4+} from the solution. Other materials, such as polyhydroxyethylmethylacrylate-pumice composites, have shown good regenerability over five cycles; however, their synthesis complexity and cost may limit practical application.

Despite the favorable properties of bentonite as an adsorbent, studies specifically examining natural, unmodified bentonite from Indonesian sources, particularly Pacitan, for thorium removal remain limited. Most existing work has focused on modified or synthetic adsorbents, leaving the performance of locally available natural bentonite poorly characterized. This study, therefore, evaluates the thorium removal efficiency of natural bentonite from Pacitan using a surrogate thorium solution in a batch adsorption setup. Critical variables, including adsorbent dosage, contact time, and adsorption kinetics and isotherms, were systematically investigated. The point of zero charge and cation-exchange capacity of the adsorbent were also determined to support the development of a cost-effective, locally sourced material for thorium management in radioactive wastewater.

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METHODOLOGY

Materials and Chemicals

In this study, the natural bentonite used was collected from Pacitan, East Java, Indonesia. It is classified as a Ca-type montmorillonite, with an initial specific surface area of 77.17 m2/g, as determined by Brunauer-Emmett-Teller (BET) analysis in a previous study [13].

All chemicals used in this study were of analytical grade. NaOH (≥97%, MERCK), NaCl (≥99%, MERCK), HCl (37%, MERCK), HNO_3 (65%, MERCK), ethylenediamine (99.5%, MERCK), $CuSO_4 \cdot 5H_2O$ (≥99%, MERCK), $Th(NO_3)_4 \cdot 5H_2O$ (≥99%, MERCK), ascorbic acid (≥99%, Sigma-aldrich), ethanol (≥99.5%, MERCK), thorin indicator (MERCK), and deionized water were used in this study. A surrogate thorium solution of 100 ppm was prepared by dissolving 12.28 mg of $Th(NO_3)_4 \cdot 5H_2O$ in 50 mL of deionized water, with 3 drops of HNO_3 added to prevent thorium hydrolysis.

Characterization of the Point Zero Charge

The determination of pHpzc aims to characterize the specific application of a material based on the particle surface charge. The pH drift method described by F. Wei et al. [14] was adopted. A total of 0.3 g of adsorbent was mixed with 20 mL of 0.01 N NaCl solution at initial pH values of 2, 4, 6, 7, 8, 10, and 12, adjusted using 0.1 M HCl or 0.1 M NaOH. The suspensions were stirred for 2 hours at room temperature (±25°C), after which the equilibrium pH was recorded. All measurements were conducted in triplicate and reported as the mean value. The pHpzc was identified at the point where the initial pH equals the equilibrium pH (ΔpH = 0).

Determination of Cation Exchange Capacity (CEC)

The cation-exchange capacity (CEC) was determined using the $[Cu(en)_2]^{2+}$ complex method following the procedure described by [15]. The $[Cu(en)_2]^{2+}$ 0.01 M complex was prepared by mixing 20.5 mL of ethylenediamine solution (1.5025 g in 25 mL of distilled water) with 10 mL of $CuSO_4$ solution (3.99 g of $CuSO_4$ in 25 mL of distilled water), then diluted in a 1L volumetric flask with distilled water. A series of five standard variations was generated; 0.01 M $[Cu(en)_2]^{2+}$ was measured into 50 mL volumetric flasks in volumes of 7.5, 10, 12.5, and 16 mL, then diluted to the graduation mark. To evaluate the cation exchange capacity (CEC), a 0.1 g sample of the adsorbent was combined with 8 mL of 0.01 M $[Cu(en)_2]^{2+}$ complex, diluted with 17 mL of distilled water, and stirred for 30 minutes. After filtering the mixture, the filtrate was analyzed using a UV-Vis spectrophotometer (Shimadzu U-2600, λ = 546 nm) to determine the concentration. All measurements were conducted in triplicate and CEC values are reported as the mean value.

Adsorption Experiment

The adsorption experiment was performed in a laboratory using batch mode with 50 mL of a Th solution variation, shaken in an orbital shaker at 100 rpm at room temperature (±25°C). The Effect of adsorbent mass was examined in the 0.01-0.5 g range, while the impact of contact time was varied from 5 to 120 minutes. The initial thorium concentration was fixed at 100 ppm for all experiments unless otherwise stated. Following each experiment, the adsorbent was separated by filtration.

The thorium concentration in the filtrate was determined spectrophotometrically using thorin as the complexing indicator. A 1 mL aliquot of the sample was transferred into a 25 mL volumetric flask. Subsequently, 5 mL of 5% (w/v) ascorbic acid solution and 5 mL of 0.1% (w/v) thorin solution were added. The flask was filled to the graduation mark with HCl solution (pH 0.8), and the absorbance was measured at λ = 545 nm using a UV-Vis spectrophotometer (Shimadzu U-2600). All experiments were conducted in triplicate, and results are expressed as the mean value.

Using the mathematical expressions in Equations (1) and (2), the study analyzed adsorption kinetics using both pseudo-first-order and pseudo-second-order models to determine the best fit for the data.

$$ln(q_e - q_t) = ln\, q_e - k_1 t \quad (1)$$

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{t}{q_e} \quad (2)$$

The amount of adsorbate taken up at equilibrium conditions is defined as q_e, the adsorption capacity at time t is defined by q_t, and the adsorption rate constants are k_1 and k_2. Moreover, the experimental data were correlated with the Langmuir and the Freundlich equations to determine the adsorption mechanism, as represented by Equations (3) and (4), respectively.

$$\frac{c_e}{q_e} = \frac{c_e}{q_m} + \frac{1}{q_m b} \quad (3)$$

$$log\, q_e = log\, k_f + \frac{1}{n} log\, c_e \quad (4)$$

The parameters for both isotherms are defined as follows: q_m is the saturation capacity (mg/g), and b (L/mg) is a constant reflecting the adsorption intensity. Additionally, the distribution coefficient k_f (mg/g) characterizes the adsorption capacity, with the material's performance proportional to k_f.

RESULTS AND DISCUSSION

Determination of pHpzc and CEC

A pH condition known as the Point of Zero Charge (pHpzc) occurs when the overall charge on the particle surface is zero. The pHpzc must be determined to establish the ideal pH for absorption, as pH levels significantly impact the absorption of various chemical compounds. According to Figure 1, natural bentonite from Pacitan has a pHpzc of 7, indicating that the adsorbent will develop a positive surface charge when conditioned at pH < 7, thereby facilitating the effective adsorption of anions under these conditions. However, the adsorbent surface becomes negatively charged when adsorption is conducted at pH > 7, thereby allowing cations to be adsorbed effectively under these conditions. Because pH influences the adsorbent's surface charge and the cation or anion's capacity for adsorption, pH has a significant role in adsorption [16].

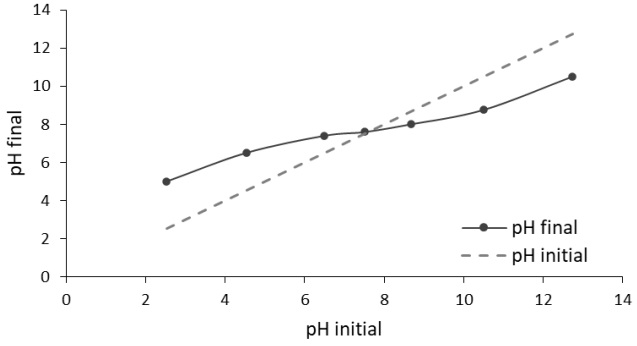

Figure 1. pHpzc natural bentonite from Pacitan

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The determination of cation exchange capacity in natural bentonite from Pacitan aims to ascertain the number of cations that can occupy the interlayer spaces of bentonite. Adding $[Cu(en)_2]^{2+}$ complex solution to bentonite replaces Na^+ ions in Na-Bentonite within the interlayer spaces of bentonite.

From the data in Table 1, natural bentonite from Pacitan has a CEC of 90.69 meq/100g. Thus, it may be said that the cation exchange capacity of the natural bentonite is high. This is because the natural bentonite has a pH of 8.98, which is higher than the pHpzc, and thus permits efficient cation adsorption. There is a strong correlation between cation exchange capacity and bentonite properties. According to other publications on bentonite modification, the addition of acid can alter the surface structure and pore volume [13]. Therefore, further investigation is required to understand the relationship between CEC and the large surface area and pore volume of bentonite.

Adsorption Studies

Effect of Adsorbent Mass

Figure 2 illustrates the impact of varying bentonite dosage (0.01–0.5 g) on the adsorption process. The obtained results show that the adsorption rate of Thorium concentration increases with increasing bentonite mass. This improvement may be driven by increased active-site density, resulting from a larger surface area and a higher concentration of functional groups on the adsorbent [17]. However, in this study, the maximum adsorption of Thorium onto the adsorbent was observed at a mass of 0.1 g, with an adsorption efficiency of 99.34% in a 50 mL solution containing 100 ppm Thorium. Also, further increases in adsorbent dosage result in a decline in removal efficiency. This may be the consequence of the material reaching its theoretical maximum capacity, as evidenced by the formation of a monolayer at the surface [9], [18].

Table 1. CEC value of Raw Bentonite

pH	8.98
Absorbance	0.0510
Concentration (M)	0.0014
$[Cu(en)_2]^{2+}$ Not adsorbed (mmol/g)	0.6931
Total $[Cu(en)_2]^{2+}$ (mmol/g)	1.6000
CEC (mmol/g)	0.9069
CEC (meq/100g)	90.69

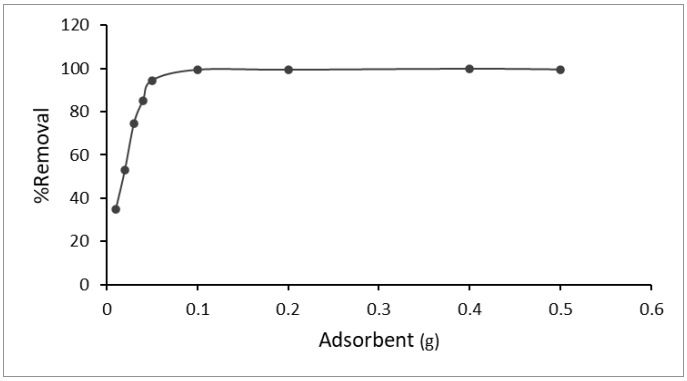

Figure 2. Effect of natural bentonite from Pacitan mass as an adsorbent on Thorium removal from adsorbate solution

Furthermore, this behavior is attributed to the presence of a finite number of discrete binding sites, each energetically equivalent and capable of accommodating only a single adsorbate molecule [19]. In addition, increasing the adsorbent dosage while keeping the aqueous volume fixed can reduce site accessibility. This occurs because the particles may aggregate, thereby reducing the total effective surface area available for Thorium uptake. Another factor that takes effect is the aggregation of adsorbent particles due to collisions within a high mass of adsorbent, which reduces the adsorption surface area and lengthens the diffusion path, thereby slowing the adsorption process [18].

Effect of Contact Time

The kinetic behavior of Thorium uptake by bentonite was evaluated by varying the contact duration from 5 to 120 minutes. As illustrated in Figure 3, thorium removal efficiency increased progressively with contact time, reaching a maximum of 99.98% at 120 minutes, which was identified as the optimum contact time. During the initial stage (5–15 min), rapid uptake occurred due to the abundant availability of unoccupied binding sites on the bentonite surface. A slight decrease in efficiency was observed at 30 minutes, which may be attributed to a transient redistribution of loosely bound thorium ions at the adsorbent surface before more stable bonding configurations are established [18]. Beyond 30 minutes, efficiency resumed its upward trend, increasing steadily through 60, 90, and 120 minutes. The continued rise in removal efficiency over time, alongside the diminishing rate of increase between 90 and 120 minutes, suggests that the system was approaching equilibrium near the end of the study period. Based on these findings, 120 minutes was selected as the optimum contact time for subsequent experiments.[18], [20].

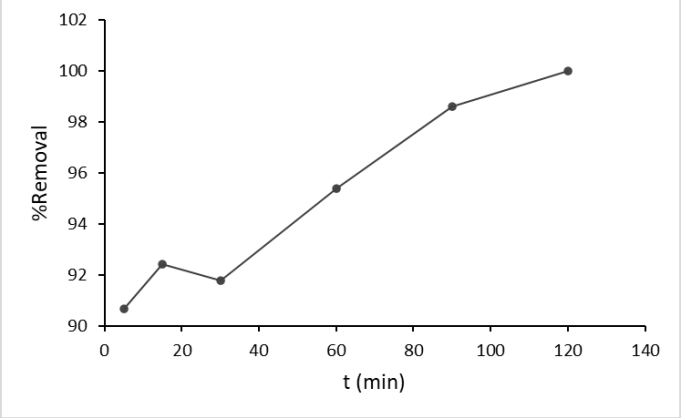

Figure 3. Effect of adsorption time of natural bentonite from Pacitan as an adsorbent on Thorium removal from adsorbate solution

Adsorption Isotherm Models

To illustrate the adsorbent's surface characteristics, equilibrium data were analyzed using adsorption isotherm models. These frameworks are instrumental in estimating removal outcomes and determining the necessary adsorbent dosage across diverse experimental setups. In this work, the Langmuir and the Freundlich isotherms models were employed. The former assumes a uniform surface with monolayer coverage, whereas the latter accounts for multilayer adsorption on heterogeneous sites [21]. In this research, the equilibrium sorption behavior of

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Pacitan-sourced natural bentonite toward Th was evaluated using several isotherm frameworks.

As evidenced by the parameters in Table 2 and the graphical representations in Figures 4 and 5, the Langmuir isotherm provided a better fit to the Th adsorption data. This preference, as indicated by the higher linear correlation coefficient (R^2), suggests that thorium ions are adsorbed onto specific, homogeneous surface sites in a monolayer arrangement. The maximum adsorption capacity was calculated to be 126.58 mg/g.

Table 2. Isotherm parameters for Th adsorption using natural bentonite from Pacitan

Isotherm model	**Parameter**	**Parameter values**
Langmuir	q_m (mg/g)	126.5823
	K_L(L/mg)	0.0009
	R^2	0.9944
Freundlich	K_f	26.1637
	n	3.4734
	R^2	0.7860

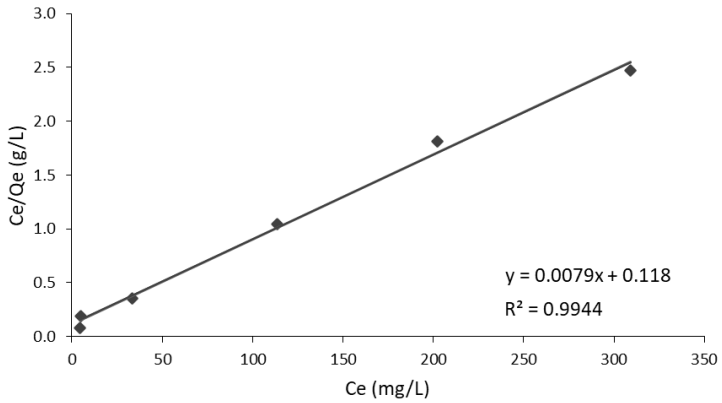

Figure 4. Adsorbent isotherm Langmuir model of Thorium on natural bentonite from Pacitan

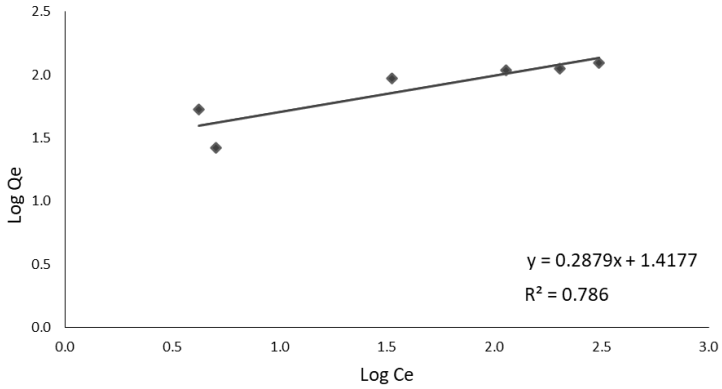

Figure 5. Adsorbent isotherm, the Freundlich model of Thorium on natural bentonite from Pacitan

Adsorption Kinetics Models

The study of chemical kinetics determines reaction rates and reaction dynamics. A component of the reaction rate equation is the reaction order. The reaction order of an element is the power of its concentration in the reaction rate equation. Adsorption kinetics describe the rate of solute uptake by an adsorbent as a function of time. To evaluate the adsorption of Th by Pacitan natural bentonite, the experimental data were analyzed using foundational pseudo-first-order and pseudo-second-order empirical models [21].

The kinetic parameters and associated plots are detailed in Figures 6 and 7 and in Table 3. Evaluation of the linear correlation coefficients (R^2) indicates that the pseudo-second-order model more closely aligns with the observed data than the pseudo-first-order alternative. Such strong statistical agreement confirms that chemisorption is the rate-controlling mechanism. This implies that the total uptake potential depends on the concentration of active functional sites at the clay interface [22].

Table 3. Kinetic model for Th adsorption using natural bentonite from Pacitan

Kinetic model	Parameter	Parameter values
Pseudo-first-order	q_e (mg/g)	13.2765
	K_1(min^{-1})	-0.0327
	R^2	0.6747
Pseudo-second-order	K_2(g/mg.min)	53.1915
	q_e (mg/g)	0.0104
	R^2	0.9994

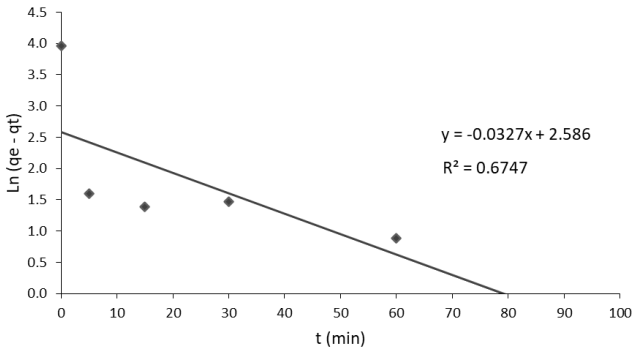

Figure 6. Pseudo-first-order kinetic model for Thorium adsorption into natural bentonite from Pacitan

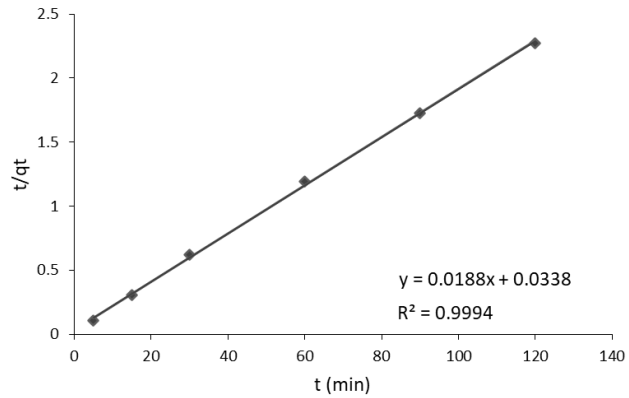

Figure 7. Pseudo-second-order kinetic model for Thorium adsorption into natural bentonite from Pacitan

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CONCLUSION

Natural bentonite from Pacitan was characterized by a point of zero charge (pHpzc) of 7 and a cation-exchange capacity of 90.69 meq/100 g. Batch adsorption experiments demonstrated that an adsorbent mass of 0.1 g in 50 mL of 100 ppm surrogate thorium solution achieved the maximum removal efficiency of 99.34%. The optimum contact time was 120 minutes, yielding a thorium removal efficiency of 99.98%. The adsorption equilibrium data were best described by the Langmuir isotherm model (R^2 = 0.9944), with a maximum adsorption capacity of 126.58 mg/g. The adsorption kinetics were described by the pseudo-second-order model (R^2 = 0.9994). These results demonstrate that natural bentonite from Pacitan is a promising and cost-effective material for thorium removal from wastewater.

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