

Urania

Jurnal Ilmiah Daur Bahan Bakar Nuklir

Beranda jurnal: <https://ejournal.brin.go.id/urania>



LIGAND INTERACTION IN THORIUM ADSORPTION BY PACITAN BENTONITE

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(Submitted: 30–01–2025, Revised: 15–03–2025, Accepted: 17–03–2025)

ABSTRACT

LIGAND INTERACTION IN THORIUM ADSORPTION BY PACITAN BENTONITE. Many studies have investigated the use of bentonite as an adsorbent; however, questions remain regarding the effects of sulfate and phosphate on thorium (Th) adsorption. Sulfate may reduce adsorption through the formation of Th-sulfate complexes and competition for adsorption sites between SO_4^{2-} and UO_2^{2+} , whereas phosphate is an important component of subsurface systems and plays a key role in controlling Th mobility. This study investigated Th adsorption by natural and acid-activated Pacitan bentonite as adsorbents for radioactive waste treatment. Because of its large surface area and porosity, bentonite has a high adsorption capacity, which can be further enhanced by acid activation. The adsorbents were characterized by FTIR and XRD to evaluate structural changes after activation. Batch experiments were conducted to examine the effects of pH, contact time, temperature, and complexing ligands, including sulfate, carbonate, and phosphate, on Th adsorption. The results showed that natural bentonite exhibited high Th adsorption efficiency from the beginning of the experiment, whereas acid-activated bentonite showed a gradual increase in adsorption efficiency over time. Th complexation with sulfate decreased adsorption efficiency, while phosphate improved Th stability in activated bentonite. These findings indicate that Pacitan bentonite, in both natural and acid-activated forms, is a promising adsorbent for Th-containing radioactive waste, provided that the adsorption conditions are properly optimized.

Keywords: Thorium adsorption, acid activation, ligands, radioactive waste, bentonite, Pacitan.

INTRODUCTION

Bentonite is a clay mineral with a high adsorption capacity and is therefore widely used in various industrial applications, including paint production, water treatment, and as a raw material in toothpaste and cosmetic products. In the environmental sector, bentonite has been widely applied in waste treatment technologies, particularly for adsorbing hazardous chemicals and preventing soil pollution [1].

However, the application of bentonite in radioactive waste treatment remains limited. Most previous studies have focused on its use in water filtration and liquid-waste treatment. The use of bentonite as an adsorbent for radioactive waste has not been extensively explored, even though its adsorptive properties suggest strong potential for handling hazardous and difficult-to-manage radioactive contaminants. Therefore, this study contributes to the development of alternative methods for radioactive waste treatment and provides a broader understanding of the potential of bentonite in environmental applications [2].

Predicting and understanding thorium (Th) migration at contaminated sites requires knowledge of the factors that govern Th uptake by minerals. Several parameters can influence Th sorption, including pH, contact time, and the presence or absence of complexing ligands such as sulfate, carbonate, and phosphate. These inorganic ligands are among the most important factors affecting Th uptake and transport [3]. This study addresses the use of bentonite as an adsorbent because important questions remain regarding the effects of sulfate, carbonate, and phosphate on Th adsorption [4]. Sulfate may slightly reduce adsorption due to the formation of uranyl-sulfate complexes and the competition between SO_4^{2-} and UO_2^{2+} . Meanwhile, phosphate is a major component in subsurface systems and plays an important role in regulating mobility. Previous studies have shown that phosphate can compete for surface sites in systems containing Th and iron oxides, thereby decreasing Th adsorption, while Th-phosphate precipitation may also reduce the dissolved Th concentration in water [5].

The uptake and transport of Th can also be kinetically controlled. Its sorption behavior is often described as an initial fast stage, occurring within a very short time,

followed by a slower uptake stage over hours or days. Accordingly, this study examined Th sorption onto bentonite as a function of pH and contact time in the presence and absence of sulfate, carbonate, and phosphate. The results are expected to clarify the sorption behavior of bentonite and support its application in radioactive waste treatment technology to minimize radiation hazards [6][7].

METHODOLOGY

Instruments and materials

The instruments used in this study included a Thermo Scientific Nicolet iS10 Fourier-transform infrared (FTIR) spectrometer to identify functional groups in the bentonite structure, a Rigaku SmartLab X-ray diffractometer (Cu source) to analyze the crystal structure of bentonite, and a Shimadzu UV-2600 ultraviolet-visible (UV-Vis) spectrophotometer to determine the Th concentration in solution, along with standard laboratory equipment. The materials used included natural Pacitan bentonite as an adsorbent in both its natural and acid-activated forms; a Th solution (Th^{4+}) prepared by diluting Th nitrate with distilled water to obtain a Th-nitrate concentration of approximately 200 ppm, as verified by UV-Vis; H_2SO_4 for bentonite activation; NaOH and HCl (0.1 M) for pH adjustment; and complexing ligands consisting of NaCl (0.01 M), Na_2SO_4 (0.005 M), and Na_2HPO_4 (0.003 M) to investigate the effect of ligands on Th adsorption.

Experimental Procedure

The experiment was carried out at laboratory scale using a batch method to evaluate the effects of several parameters on Th adsorption by bentonite. Pacitan bentonite was washed with deionized water and dried at 105°C. For the acid-activated bentonite, the material was soaked in 0.5 N H_2SO_4 for 24 h, washed to neutral pH, and dried again. The dried bentonite was then ground with a mortar and pestle and sieved to 200 mesh. In the adsorption experiments, 0.5 g of bentonite was mixed with 20 mL of Th solution in a 250 mL Erlenmeyer flask, and the pH was adjusted to 3-8 using 0.1 M NaOH or 0.1 M HCl. The suspension was stirred with a magnetic stirrer for 5-1440 min to evaluate the effect of contact time. For the temperature study, experiments were conducted at 25°C,

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45°C, and 60°C. To evaluate the effect of complexing ligands, bentonite was tested in three solution conditions: A2 (0.01 M NaCl), A3 (0.01 M NaCl + 0.005 M Na₂SO₄), and A5 (0.01 M NaCl + 0.003 M Na₂HPO₄). After adsorption, the liquid and solid phases were separated by centrifugation. The Th concentration in the final solution was measured by UV-Vis spectrophotometry at 547 nm using the TOPO method. Adsorption efficiency was then calculated using the corresponding equation:

$$\text{Adsorption (\%)} = \frac{C_1 - C_2}{C_1} \times 100 \quad (1)$$

Here, C_1 is the initial Th concentration and C_2 is the Th concentration after adsorption. The experimental data were analyzed to evaluate the relationships among pH, contact time, temperature, and the presence of ligands on Th adsorption efficiency. A comparison between natural and acid-activated bentonite was used to assess the effect of activation on adsorption performance.

RESULTS AND DISCUSSION

Characterization

The FTIR spectra in Figure 1 shows clear differences between natural bentonite and activated bentonite, indicating changes in surface functional groups. Natural bentonite exhibited typical clay-mineral bands, including a broad peak in the 3400-3600 cm⁻¹ region that was associated with O-H stretching vibrations in the clay structure. The band at approximately 1000-1100 cm⁻¹ corresponded to Si-O-Si

and/or Si-O-Al vibrations characteristic of the silica tetrahedral framework. The band near 1600 cm⁻¹ was attributed to the bending vibration of adsorbed water in the bentonite structure [8].

Activated bentonite showed changes in both the intensity and position of several bands. In the 3400-3600 cm⁻¹ region, the stronger O-H stretching band indicated an increase in hydroxyl groups after activation (e.g., with acid or base). The Si-O-Si band near 1000-1100 cm⁻¹ also became more pronounced, suggesting changes in structural order after treatment. This suggested that the activation process had improved or strengthened the mineral structure, allowing bentonite to have more stable or better-organized physical and chemical properties. Meanwhile, the decreased band intensity around 1600 cm⁻¹ indicated a reduction in adsorbed water content. A lower water content may have exposed more active sites on the mineral surface, thereby improving adsorption capacity. Activated bentonite showed higher intensity in some bands, especially around 3400 cm⁻¹ and 1000 to 1100 cm⁻¹, indicating structural changes due to activation. This changes may have included the growth of active groups on the surface (such as O-H), the removal of impurities or specific ions in the structure through the activation process [9]. Activation modified the bentonite structure by altering surface functional groups and removing impurities, which may have enhanced the availability of active sites for adsorption.

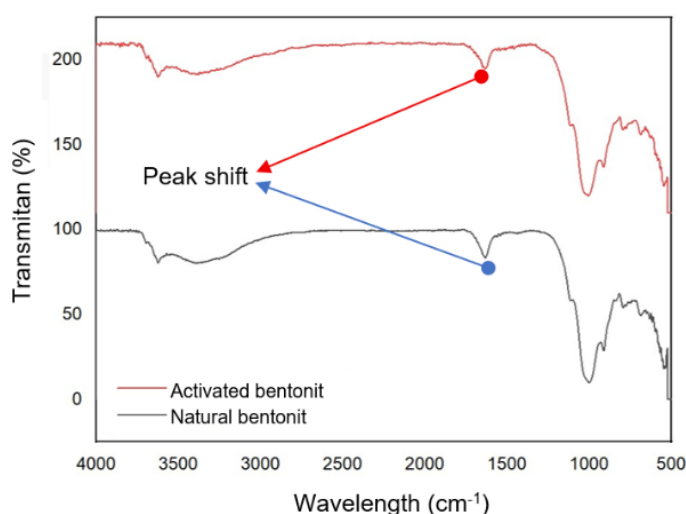


Figure 1. FTIR spectra of natural and activated bentonite.

Natural bentonite exhibited sharper diffraction peaks and higher intensity, particularly at 2θ values of approximately $20-30^\circ$, as shown in Figure 2. The main peak around $26-27^\circ$ indicated the presence of quartz (SiO_2), which is one of the natural mineral components of bentonite. Additional peaks at lower angles, around $5-10^\circ$, might be attributed to montmorillonite layers, the main phase in bentonite, although other minerals such as illite or kaolinite might also contributed at specific 2θ positions [10]. Acid-activated bentonite showed changes in peak intensity and peak position, suggesting increased structural regularity and changes in interlayer spacing after acid treatment. The appearance of several peaks in the $20-30^\circ$ region also indicated mineralogical changes after activation [11][12].

The increase in crystallinity after activation might be related to the removal of impurities and improved structural regularity. Acid attack could dissolve metal ions such as Al^{3+} and Fe^{3+} from the montmorillonite structure, generating new pores and increasing surface area, which might enhance adsorption capacity [13]. Peak shifts might reflect the partial removal of

exchangeable ions and modification of the layer structure, whereas the reduction of some peaks might indicate partial degradation of montmorillonite and the removal of amorphous phases. Activated bentonite showed an increase in surface area and number of active sites, which could increase the Th adsorption capacity. Activated bentonite had more anionic Lewis acid sites, which could enhance the electrostatic interaction with cationic ions such as Th^{4+} . However, excessive activation could damage the montmorillonite layer structure, decreasing adsorption capacity. If excessive activation occurred, the montmorillonite structure might have been damaged, which could have reduced the number of active sites and decreased the adsorption capacity [14]. From the XRD results, activated bentonite had a higher diffraction intensity, indicating increased crystallinity. Higher crystallinity could reduce the amorphous nature of bentonite, thereby reducing the flexibility in adsorbing Th^{4+} ions [15]. The more flexible layered structure of natural bentonite might allow Th to penetrate more deeply into the pore system.

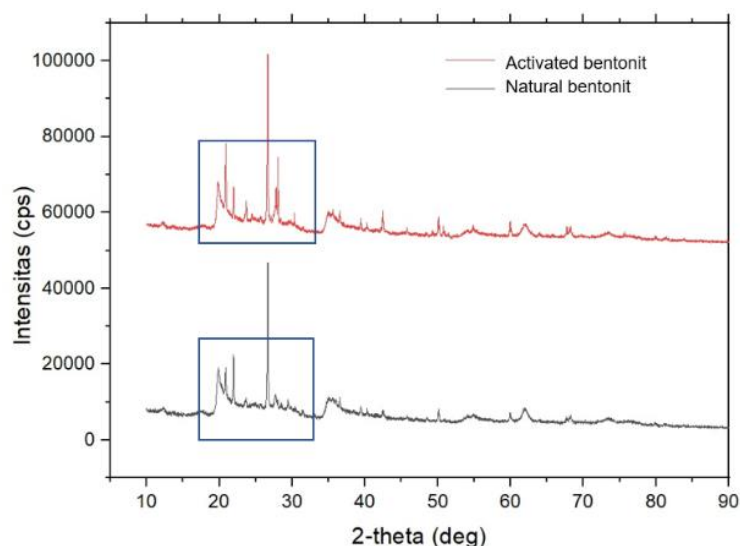


Figure 2. XRD patterns of natural and activated bentonite.

Effect of contact time on Th adsorption by bentonite

Th adsorption by natural bentonite showed a stable trend over the contact-time range of 5-1440 min. The recovery values ranged from 99.80% to 100%, indicating the high efficiency of natural bentonite for Th

adsorption (Figure 3). No significant difference was observed among the three complexing-ligand systems (NaCl ; $\text{NaCl} + \text{Na}_2\text{SO}_4$; and $\text{NaCl} + \text{NaHPO}_4$), indicating that these solutions did not substantially affect adsorption by natural bentonite.

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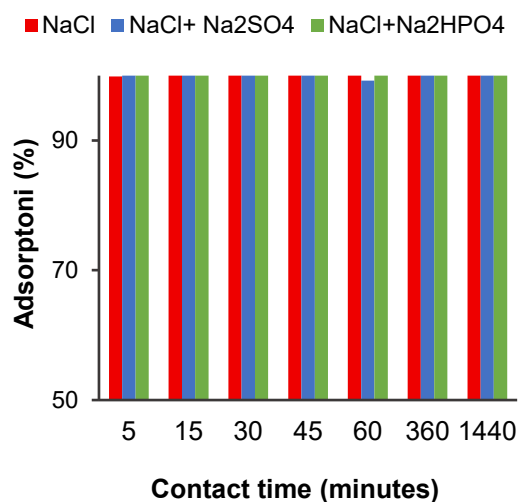


Figure 3. Adsorption behavior of natural bentonite as a function of contact time.

The rapid adsorption of Th by natural bentonite might be attributed to its porous structure and large specific surface area. These characteristics allowed Th ions to interact quickly with the bentonite surface, where active sites such as hydroxyl groups (-OH) and exchangeable ions could bind the metal ions directly. The pore structure also facilitated Th diffusion into the bentonite particles with relatively low resistance [12]. The complexing ligands did not appear to significantly affect adsorption by natural bentonite, likely because the concentrations of Cl⁻, SO₄²⁻, and PO₄³⁻ species in the supporting solutions were relatively low and did not strongly compete with Th for active sites. In addition, the affinity of natural bentonite for multivalent ions such as Th was much greater than for monovalent ions such as Na⁺, so Th adsorption remained very high. The temporary decrease in Th recovery at 60 min in the NaCl + Na₂SO₄ system, as shown in Figure 3, might be caused by a combination of ion competition, Th-sulfate complexation, changes in bentonite surface properties, and shifts in the adsorption-desorption equilibrium [16]. Because bentonite particles contained -OH groups and negatively charged surface sites, they could attract and retain positively charged ions such as Th. However, competition might arise between Th and other ions present in solution, including hydrogen ions and sulfate or chloride species, as illustrated in Figure 4.

This competition could influence the amount of Th adsorbed onto bentonite.

However, the sorption efficiency increased again at longer contact times because a new equilibrium was established and Th adsorption became dominant once more. This behavior might be related to Th diffusion into the bentonite pores, progressive equilibration of the system, changes in the bentonite structure, and the reverse reaction of Th complexes in solution. The process of achieving a new equilibrium can be done by changing the concentration or pH in the solution. Montmorillonite had a negatively charged surface due to isomorphous substitution within its lattice, allowing it to interact with cationic species. At low pH, hydrogen ions bound to surface -OH groups and reduced the number of sites available for Th adsorption.

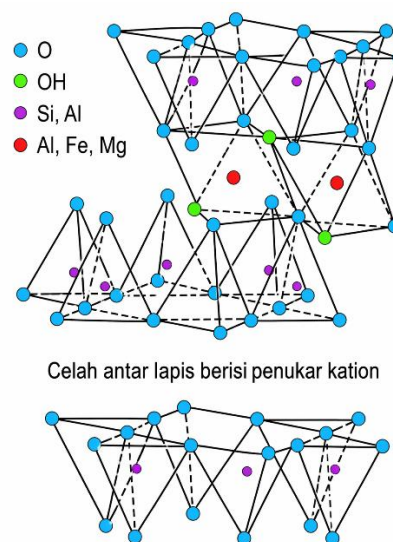


Figure 4. Structure of Montmorillonite, the main mineral constituent of bentonite

At higher pH, OH⁻ ions replaced some of the H⁺ ions on the surface, increasing the availability of -OH groups that could interact with Th ions. Th could also form complexes with OH⁻ ions, forming compounds such as Th(OH)₃ that were not easily adsorbed on the bentonite surface. On the other hand, at low pH, Th was more likely to remain in the form of free ions (Th³⁺) that were more easily interacted with the bentonite surface. At very low pH (strong acid), Th could dissolve into smaller ions or complexes not readily adsorbed by bentonite. At neutral or slightly basic pH, Th³⁺ ions remained stable and were more likely to be adsorbed. Changes in pH therefore affected the availability of active

sites on the bentonite surface, which affected the amount of Th ions that can be adsorbed. At low pH, H^+ ions could reduce the number of available active sites, while at high pH, OH^- ions could increase the availability of active sites. At higher Th ion concentrations, adsorption equilibrium would be reached more quickly. Still, at very high concentrations, the adsorption capacity of bentonite could be reached, which caused a further decrease in adsorption efficiency. Changes in pH would change the form of Th ions in the solution. Th^{3+} ions were more stable at low pH and tended to bind to the bentonite surface. In contrast, at higher pH, the $Th(OH)_3$ complex form was more dominant, which could affect the adsorption process and allowed Th ions to diffuse more quickly into the pores of bentonite.

In addition, the competitive effects of SO_4^{2-} and PO_4^{3-} species might decrease over time, providing a greater opportunity for Th to dominate adsorption on bentonite again [17]. In addition, the presence of Na_2SO_4 compounds could affect the pH in the solution. Suppose the pH of the solution changed during contact time (more acidic or basic). In this case, it could affect the ability of Th to be adsorbed on the bentonite surface, which would change the complex bonds of Th with ions in the solution, as well as the adsorption ability of Th by bentonite surface. This is because, at low pH (acidic), the number of H^+ ions in the solution increased, which could reduce the bentonite charge on the bentonite surface, thereby reducing the attraction of the bentonite surface to Th^{4+} ions. A decreased negative charge on the bentonite surface reduced its adsorption capacity for metal ions such as Th. Conversely, at high pH (alkaline), OH^- ions would increase in the solution, which could increase the negative charge on the bentonite surface, thereby increasing its attraction to Th^{4+} ions. At a certain pH, the surface charge of bentonite could change so that the ionic interaction with Th weakened. This is because the negative charge on bentonite decreased at low pH, while at high pH, it increased. The sorption behavior of Th by acid-activated bentonite showed a different trend from that of natural bentonite, with the sorption grew gradually over time as shown in Figure 5. At the initial contact time (5–45 min), the sorption efficiency was relatively low, with recovery values ranging

from 60% to 80% for NaCl and NaCl + Na_2SO_4 solutions and lower for NaCl + $NaHPO_4$. The sorption recovery increased significantly after 60 min, reaching about 100% at 1440 min for all complexing ligands. This is because acid activation of bentonite increased the interaction of specific ions in the solution with the bentonite surface. Still, it took longer to reach the maximum sorption capacity compared to natural bentonite. The bentonite surface would be more able to interact with metal ions such as Th^{4+} , which could bind to the hydroxyl groups through hydrogen bonding interactions or electrostatic forces [18].

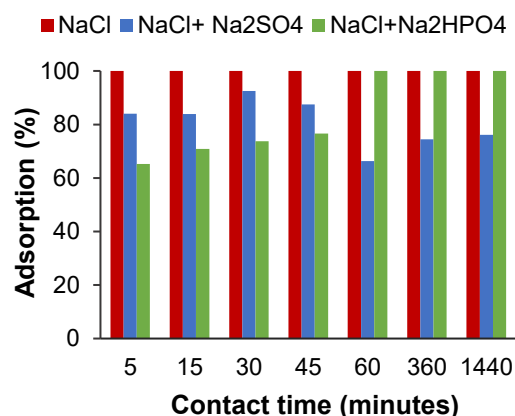


Figure 5. Adsorption behavior of acid-activated bentonite as a function of contact time.

The lowest Th^{4+} adsorption observed in the Na_2SO_4 containing system compared with NaCl and NaCl + $NaHPO_4$ might be explained by the tendency of SO_4^{2-} ions in solution to form stable complexes with Th, such as $Th(SO_4)_4^{2-}$, thereby reducing the amount of free Th^{4+} in solution for adsorption. Due to the larger molecular size and changes in its charge properties, the $Th(SO_4)_4^{2-}$ complex was more challenging to adsorb on the bentonite surface than free Th^{4+} ions. By contrast, NaCl did not significantly complex Th, so more free Th remained available for adsorption. Accordingly, the adsorption behavior of acid-activated bentonite in the NaCl system was closer to that of natural bentonite. NaCl did not form a complex with Th, so Th remained available as a free Th^{4+} ion. In addition, there was no significant competition between Na^+ or Cl^- ions and Th for active sites on bentonite. There was also no modification of the bentonite surface or interference from complexing ions.

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Effect of pH

Figures 6 and 7 present the results of Th sorption on bentonite in 0.01 M NaCl solution as a function of pH. Overall, the sorption curves were only slightly affected by increasing pH, although the effect was more pronounced for acid-activated bentonite at very low pH.

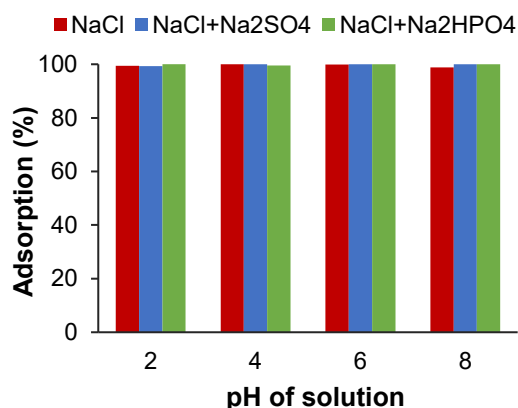


Figure 6. Adsorption behavior of Th on natural bentonite as a function of pH.

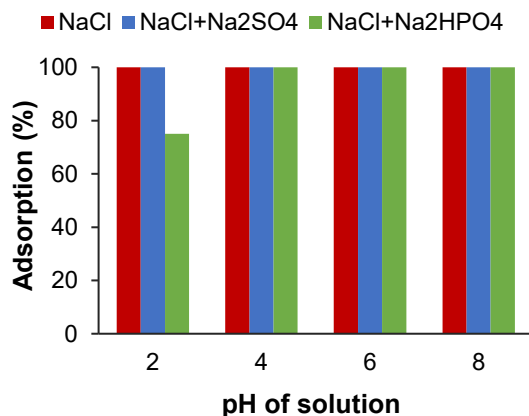


Figure 7. Adsorption behavior of Th on acid-activated bentonite as a function of pH.

Changes in pH had little effect on Th adsorption by natural bentonite (Figure 6). All ligand combinations showed similarly high adsorption, approaching 100% at each pH value. In contrast, acid-activated bentonite showed much lower adsorption at pH 2 than at the other pH values. This behavior might be attributed to the high concentration of H⁺, which competed with Th for the active sites on bentonite [1]. Bentonite's active site (-OH group) tended to be protonated (bonded with H⁺), thus reducing its affinity for Th. In

strongly acidic conditions, the ions formed complexes with other ions in the solution or even remained in dissolved form so that they could not be optimally absorbed by bentonite, which further reduced adsorption efficiency.

The decrease in Th absorption at pH 2 was especially evident in the NaCl + NaHPO₄ system. In contrast, other solutions still showed high absorption because solutions containing HPO₄²⁻ ions could interact with Th⁴⁺ ions to form soluble complexes, such as Th(HPO₄)_x. This complex reduced the amount of free Th⁴⁺ ions available for adsorption by bentonite. The presence of phosphate ions in protonated form (H₂PO₄⁻ or H₃PO₄) could also form a stable complex with Th⁴⁺ ions, thus reducing its adsorption. This effect was less evident in NaCl or NaCl + Na₂SO₄ system which do not appear to interact with Th in the same way under these conditions [19].

Effect of Temperature

Th adsorption by natural bentonite remained high (>95%) at all tested temperatures and in all solution systems as shown in Figure 8. For acid-activated bentonite (Figure 9), the lowest absorption of Th ions at a temperature of 45°C was found in NaCl system compared to other solutions, most likely influenced by ionic effects and ligand-ion interactions. NaCl produces Cl⁻ ions that could compete with Th⁴⁺ ions to bind to the active sites of bentonite. [20]. At 45°C, the increase in the kinetic energy of Cl⁻ ions was significant enough to allow more effective competition for adsorption sites, thereby reducing the amount of Th⁴⁺ ions adsorbed. The decrease in Th⁴⁺ ions adsorption at this temperature occurred because Cl⁻ ions could move faster and competed more efficiently with Th⁴⁺ ions to bind to the bentonite surface. This is because Th³⁺ would be more dominant in interacting with the surrounding environment due to its more potent electrostatic properties and tendency to create more stable bonds than Cl⁻. At higher temperatures, such as 60°C or more, the kinetic energy of Cl⁻ ions and Th⁴⁺ ions would increase further.

However, a further increase in temperature did not constantly improve the competition of Cl⁻ ions against Th⁴⁺ ions significantly. At very high temperatures, excessive kinetic energy could cause desorption of ions from the bentonite surface or even changes in the structure of the

bentonite surface itself. Cl^- ions in NaCl solution could interact with Th^{4+} to form simple complexes such as ThCl_x^{4-x} . This complex could reduce the availability of free Th^{4+} ions in solution to be adsorbed by bentonite. However, this did not affect sulfate and phosphate ions because SO_4^{2-} or HPO_4^{2-} tended not to form stable soluble complexes with Th^{4+} , so Th ions remained available for adsorption. These ions could help increase the affinity of the bentonite surface for Th ions through additional interactions [19].

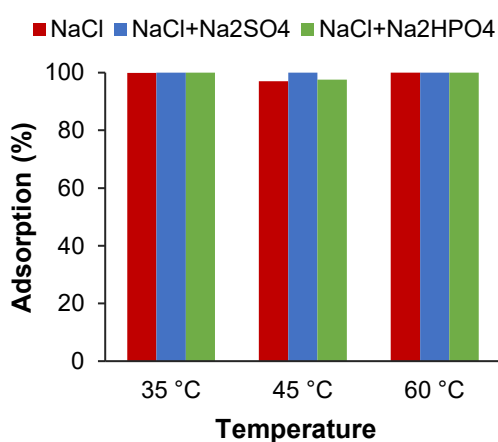


Figure 8. Adsorption behavior of Th with natural bentonite as a function of temperature.

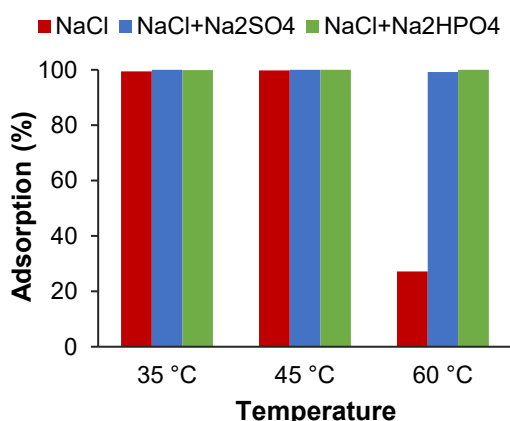


Figure 9. Adsorption behavior of Th with activated bentonite as a function of temperature.

A different trend was observed in acid-activated bentonite. At lower temperatures, Th absorption was more stable, reaching 100% for all complex ligands. However, adsorption decreased at

60 °C by acid-activated bentonite. This decline might be caused by desorption of previously bound Th ions in bentonite surface. The increase in kinetic energy of ions in solution made them easier to release from the active sites of bentonite, especially if the adsorption bonds were weak or partially reversible. This desorption process was more significant in acid-activated bentonite because acid activation produced more active sites with weak bonds (electrostatic interactions) that were more susceptible to ion release at high temperatures. At 60 °C, the structure of activated bentonite could experience microstructural damage or pore collapse, which reduced the adsorption capacity. Solutions containing ions such as Cl^- , SO_4^{2-} , or HPO_4^{2-} at high temperatures could increase the mobility and reactivity of these ions. These ions could compete more strongly with Th ions to bind to the active sites of bentonite [21][22].

CONCLUSIONS

This study demonstrated the potential of Pacitan bentonite, in both natural and acid-activated forms, as an effective adsorbent for thorium ions in radioactive waste treatment. Natural bentonite showed high and rapid thorium adsorption, whereas acid-activated bentonite exhibited a more gradual increase in adsorption over time. Parameters such as pH, contact time, temperature, and the presence of complexing ligands, especially sulfate and phosphate, significantly affected adsorption by acid-activated bentonite but had little effect on natural bentonite. Sulfate reduced adsorption efficiency by promoting thorium complexation, whereas phosphate influenced thorium stability in activated bentonite. Overall, these findings confirm that Pacitan bentonite is a promising material for nuclear waste management, provided that the adsorption conditions are properly optimized. Further studies are recommended to optimize the adsorption properties of bentonite and to expand its application in environmental protection, particularly for the management of radioactive materials.

ACKNOWLEDGMENTS

The authors wish to thank the Head of the Research Center for Nuclear Materials and Radioactive Waste Technology and the Nuclear Raw Material Processing research

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group at the National Research and Innovation Agency (BRIN) for their support in facilitating this research.

AUTHOR CONTRIBUTIONS

The primary contributors to this manuscript are: Roza Indra Laksmana, Mohammad Khotib, Zainal Alim Ma'ud, June Mellawati, Afiq Azfar Pratama, Suci Indryati, and Dwi Luhur Ibnu Saputra.

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