Urania
Jurnal Ilmiah Daur Bahan Bakar Nuklir

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

URANIUM ADSORPTION BEHAVIOR ON PACITAN BENTONITE

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(Submitted: 30–01–2025, Revised: 28–02–2025, Accepted: 05–03–2025)

ABSTRACT

URANIUM ADSORPTION BEHAVIOR ON PACITAN BENTONITE. Uranium adsorption by Pacitan bentonite was investigated to evaluate its capacity for capturing uranium ions under various conditions. Before being used as an adsorbent, Pacitan bentonite was characterized in terms of pore structure, surface area, surface morphology, adsorption capacity, and chemical composition. This study aims to analyze the factors that influencing uranium adsorption on Pacitan bentonite, including contact time, pH, temperature, and the presence of ligands in solution. Both natural and acid-activated Pacitan bentonite were examined to compare their adsorption performance. The results showed that Pacitan bentonite effectively adsorbs uranium, with the highest adsorption occurring under acidic to neutral pH conditions and decreasing at higher pH levels. Acid activation reduced the uranium adsorption capacity due to structural and surface property changes. In addition, the addition of ligands such as NaCl, Na_2SO_4, and Na_2HPO_4 influenced uranium adsorption, with Na_2SO_4 having the most pronounced effect compared to NaCl and Na_2HPO_4. Overall, the results indicate that Pacitan bentonite has strong potential as a uranium adsorbent material, especially under suitable conditions to maintain ion exchange and surface adsorption. These findings provide important insights for developing adsorbent materials for nuclear waste treatment and remediation of uranium-contaminated environments.

Keywords: uranium adsorption, acid activation, bentonite, ion exchange, nuclear waste.

DOI: https://doi.org/10.55981/urania.2025.9213

INTRODUCTION

The world is currently experiencing an increasing demand for electrical energy, particularly in developing countries such as Indonesia. Nuclear Power Plants (NPPs) offer a stable and convenient energy source to support economic growth and improve public health. However, it is important to note that NPPs generate radioactive waste that must be managed carefully in accordance with existing regulations. The management of radioactive waste from NPPs requires investment and advanced technology to ensure human and environmental safety [1]. Radioactive waste from nuclear power plants can exist in solid, liquid, or gaseous forms. This waste must be properly collected, processed, and stored. The management process includes various treatment such as conditioning, incineration, evaporation, and ion exchange to reduce the volume and radiation exposure of the waste, thereby preventing harm to humans and the environment [1][2][3].

The primary source of waste from nuclear reactors are the atomic fuel used, mainly uranium and thorium. Strategies for managing uranium and thorium waste include separation, purification, precipitation, and the use of adsorbent materials. Various radioactive waste treatment methods have been developed, such as solvent extraction, ion exchange, and adsorption. Among these methods, adsorption using suitable adsorbents is considered the most popular, efficient, and practical due to its high capacity, low cost, ease of regeneration, and the absence of sediment formation [1].

One of the adsorbents that can be used to remove radioactive elements such as uranium and thorium from liquid waste is natural bentonite. During the adsorption process, bentonite absorbs pollutants on its surface and within its pores, thereby reducing their concentration of these pollutants in the solution. Bentonite has high porosity and large surface area, which contribute to its superior adsorption performance compared to many other adsorbents [1][4][5]. In addition, its very fine colloidal particle size enables this faster adsorption kinetics compared to granular adsorbents. Bentonite also has higher selectivity than activated carbon in removing uranium and thorium from aqueous solution [6][7].

One of the main producers of bentonite minerals in Indonesia is the Pacitan area. Based on the previous studies, Pacitan bentonite has several advantages compared to bentonite from other regions. It is considered as the highest quality due to its high montmorillonite content (approximately 71%) and its high cation exchange capacity (CEC) of 45.98–62.42 meq/100 g, which makes it more effective in adsorption process [6]. In comparison, the CEC values of bentonite from other regions such as Klaten, Sidoarjo, and Tuban range between 30–50, 25–55, and 30-55 meq/100 g, respectively [8]. In addition, acid activation of Pacitan bentonite has been shown to increase its selectivity for pollutant adsorption, including uranium and thorium. This improvement occurs because acid treatment removes cations from the bentonite surface and increases the number of hydrogen cations (H^+) to exchange with other metal cations, thereby rendering the surface more reactive, according to reaction:

$$Al_2O_3{\cdot}2SiO_2 + 4H^+ \rightarrow 2Al^{3+} + 2SiO_2 + 2H_2O \quad (1)$$

In addition, when bentonite is exposed to acid, some metal cations such as Fe^{2+}, Fe^{3+}, or Al^{3+} that are bound to the bentonite surface can be released and replaced by hydrogen cations (H^+). This substitution increases the bentonite's ability to adsorb specific metal ions or organic molecules, according to reaction (2) [9].

$$Al_2O_3 + 3H_2SO_4 \rightarrow 2Al(SO_4)_3 + 3H_2O \quad (2)$$

Clay minerals are an important consideration in this context, as the sorption of uranium and thorium onto clay minerals has been extensively studied. However, predicting and understanding the migration of uranyl species at contaminated sites requires a thorough understanding of the factors that influence uranium and thorium sorption on mineral surfaces. Several parameters can control the sorption behavior of these elements, including pH, contact time, and the presence or absence of complexing ligands such as sulfate, carbonate, and phosphate. These ligands are the most significant inorganic species in influencing the sorption and transport of uranium and thorium [10].

Extensive research has been conducted on the adsorption properties of bentonite; however, many questions remain regarding the effect of sulfate, carbonate, and phosphate on uranium and thorium adsorption. Sulfate ions can slightly reduce adsorption due to the formation of uranyl-sulfate complexes and competition between SO^{2-} and UO^{2+}. Phosphate, an important component in subsurface system, is vital in regulating mobility. Previous studies have shown that phosphate in systems containing uranium, thorium, and iron oxides has some competitive effects on the iron oxide surface, reducing uranium and thorium adsorption. Competition with surface sites for uranium coordination by forming uranium-aqueous will reduce the adsorption of uranium and thorium. The precipitation of uranium-phosphate will reduce the concentration of uranium in water, where technically, the chemical reaction that occurs is following reaction (3) [11].

$$U^{4+} + 6H_2O \rightleftharpoons [UO_2(H_2O)_6]^{2+} \qquad (3)$$

The sorption and transport of uranium and thorium are kinetically controlled, typically described as fast initial phase occurring on the order of milliseconds, followed by a slower phase occurring on the order of hours or days. In this study, the sorption of uranium and thorium on bentonite was investigated as a function of pH and contact time in the presence and absence of sulfate, carbonate, and phosphate. The results are expected to provide insights into the sorption behavior of bentonite and its potential application in radioactive waste treatment technologies to minimize radiation hazards [12][13].

METHODOLOGY

The instruments used in this study included a Scanning Electron Microscope (SEM) for characterizing the surface morphology of bentonite and a UV-Vis spectrophotometer for analyzing uranium concentration in solution. The specific surface area, pore structure, and physicochemical properties of the bentonite samples were characterized using various techniques, such as Brunauer-Emmett-Teller (BET) analysis and scanning electron microscopy [14][15]. The chemical composition of the bentonite was analyzed using the X-ray Fluorescence (XRF) method, focusing on elements generally found in bentonite.

The materials used in this study included Pacitan natural bentonite, which served as the adsorbent in both its natural and acid-activated forms. The uranium solution (UO_2^{2+}) was prepared from uranyl nitrate diluted in distilled water. Sulfuric acid (H_2SO_4) was used to modify the bentonite structure. A pH buffer system consisting of 0.1 M NaOH and 0.1 M HCl was employed to control the pH during experiment. Complexing ligands—NaCl (0.01 M), Na_2SO_4 (0.005 M), and Na_2HPO_4 (0.003 M)—were used to investigate the effect of ligands on uranium adsorption.

The study was conducted on a laboratory scale using the batch method to evaluate the effect of specific parameters on uranium adsorption by bentonite. Pacitan bentonite was first washed with deionized water and dried at a temperature of 105°C. For acid-activation, the bentonite was soaked in 0.5 N H_2SO_4 solution for 24 hours, then washed to neutral pH and dried again. Adsorption experiments were carried out in a batch system. For pH variation tests, bentonite was mixed with the uranium solution in a 250 mL Erlenmeyer flask, and the pH was adjusted within the range of 3 to 8 using 0.1 M NaOH or 0.1 M HCl. The bentonite and uranium mixture were stirred magnetically for 5 to 1440 minutes to determine the adsorption kinetics under different contact time. Temperature variation studies were performed at 25, 45, and 60°C to observe the effect of temperature on uranium adsorption. To examine the effect of complexing ligands, bentonite was tested in three solution conditions: A1 (0.01 M NaCl), A2 (0.01 M NaCl + 0.005 M Na_2SO_4), and A3 (0.01 M NaCl + 0.003 M Na_2HPO_4). After the adsorption process, the mixture were centrifuged to separate the solution from the residue. The uranium concentration in the final solution was measured using UV-Vis spectrophotometry at a wavelength of 547 nm based on the Trioctylphosphine Oxide (TOPO) method. The adsorption efficiency was calculated using the following equation:

$$\text{Absorption (\%)} = \frac{C_1 - C_2}{C_1} \times 100\% \qquad (4)$$

C_1 and C_2 represent the initial and final uranium concentration, respectively. The experimental data were analyzed to investigate the relationship between pH, contact time, temperature, and the presence of ligands on the efficiency of uranium adsorption. A comparison between natural and acid-activated Pacitan bentonite was conducted to evaluate the influence of acid activation on the adsorption capacity and overall adsorption behavior of the material.

RESULT AND DISCUSSION

Characterization

1. Analysis of specific surface area and pore structure

The results of activation analysis showed that acid treatment significantly increased the adsorption capacity of natural Pacitan bentonite, which can be attributed to the increase in specific surface area and the formation of additional active adsorption sites on the bentonite surface[16].

Table 1 presents surface area, pore volume, and pore size of natural and acid-activated bentonite. Compared with natural bentonite, acid-activated bentonite has a higher surface area (77.7084 m^2/g) and a slightly larger pore volume (0.102012 cm^3/g), while the pore size decreased from 60.993 to 52.510 Å. The increase in surface area and pore volume after acid-activation can be attributed to the removal of impurities and the formation of new pores and active sites during the acid treatment process. Therefore, the increase in surface area of acid-activated bentonite enhances the potential of bentonite to adsorp metal ions such as uranium. However, an increase in surface area does not necessarily increase the adsorption directly. The overall effectiveness of adsorption also depends on the surface properties and pore distribution, which influence ions-surface interaction [18][19]. Natural bentonite has a layered structure that is intact with natural exchangeable cations (such as Na^+ or Ca^{2+}) located between the layers. This structure provides good porosity and an optimal surface area for molecular adsorption. Acid activation can lead to partial dissolution of the mineral layers, particularly the cations within the montmorillonite structure (the main component of bentonite). This process may partially destroy the layered structure, reduce the number of natural pores, and decrease the effective surface area for adsorption [15].

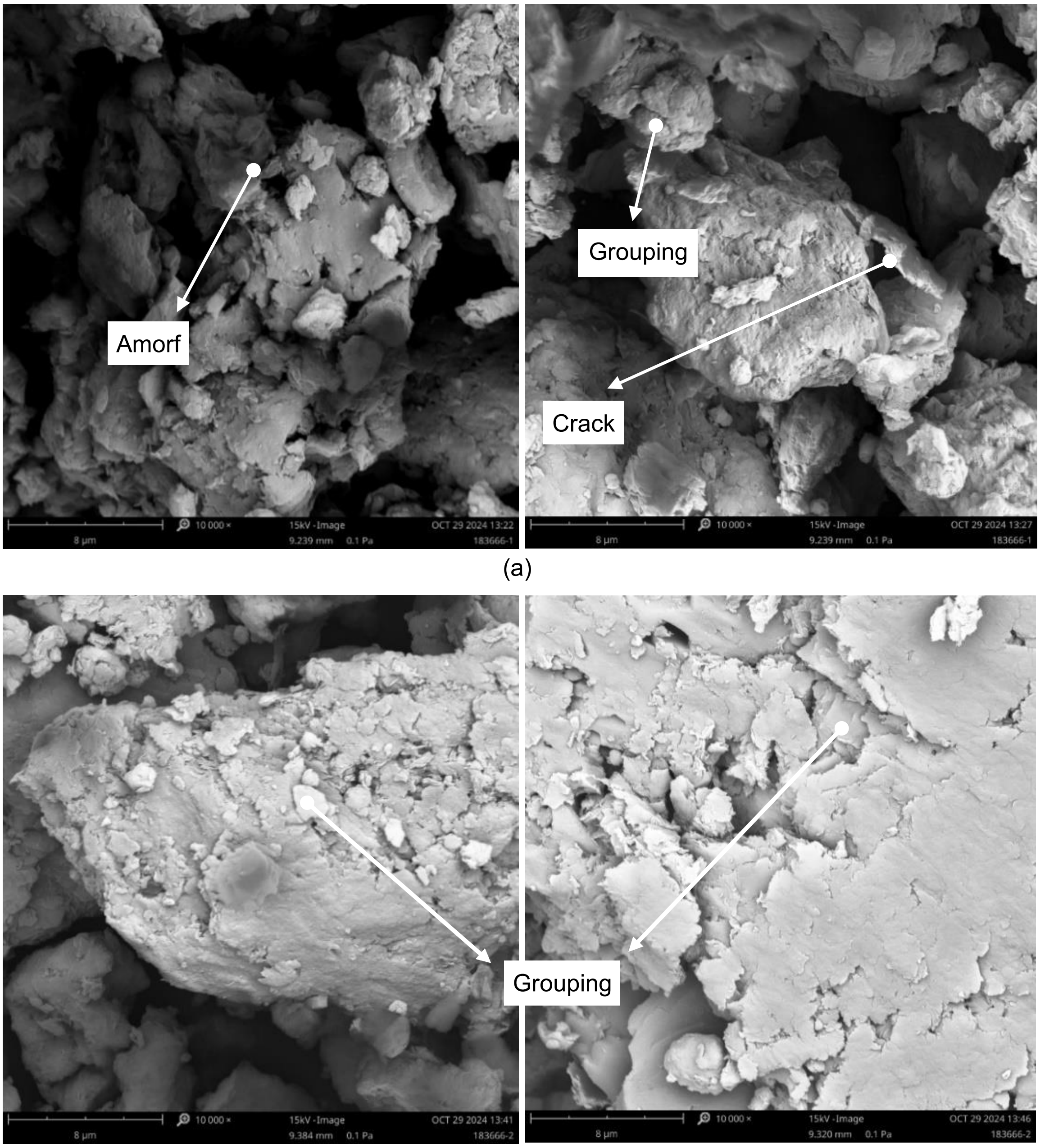
Table 1. Surface area of natural and acid-activated bentonite

Type of bentonites	Surface area (m^2/g)	Pore volume (cm^3/g)	Pore size (Å)
Natural Bentonite	63.409	0.097	60.993
Acid activated bentonite	77.708	0.102	52.510

Pore volume represents the total space or pores within a unit mass of material, which serves as a reservoir for adsorbed molecules or ions. A reduction in pore size is attributed to partial collapse or structural rearrangement of the clay layer [5]. The acid activation process, typically involving strong acid such as HCl or H_2SO_4), removes some of the surface-bound cations and alters the existing crystal structure, thereby increasing the number of active sites and the overall surface area. This process can create new pores or enlarge existing ones, leading to an increase in total pore volume [14]. Larger pores facilitate better diffusion of ions or molecules into the bentonite structure, potentially increasing its absorption capacity [6][15]. Natural bentonite has a larger pore size than acid-activated bentonite. The acid treatment can cause a decrease in pore size due to removal of large surface-bound cations or by changes in the crystal structure that narrow the pore space [7][12][20]. Although acid-activation increase the total pore volume, the smaller pore size may influence the penetration capacity of large molecules or ions, such as uranium, which can be more easily absorbed into materials with smaller pores. The smaller pore size of acid-activated bentonite indicates that this material is more suitable for adsorbing small molecules or ions that can easily diffuse through narrow pores. In contrast, for larger or more complex ions such as uranium, natural bentonite with its larger pores may provide more space for the ions to be adsorbed [13][21].

2. Surface morphology analysis

The SEM analysis results presented in Figures 1a and 1b show the surface morphology of Pacitan natural bentonite before and after acid activation, both shows a heterogeneous surface structure. In Figure 1a, the natural bentonite exhibits amorphous conglomerate groupings and cracks laminated by layered coarse-grained sheets containing many layered zones with cracks. This morphology indicates a high specific surface area, which contributes to a higher adsorption capacity compared to the acid-activated bentonite shown in Figure 1b [7][17].



(a)

(b)

Figure 1. (a). Natural Pacitan bentonite without acid activation; and (b) acid-activated Pacitan bentonite.

3. Chemical composition analysis

The oxide composition of natural and acid-activated bentonite was determined using XRF analysis, and the results are presented in Table 2.

The natural bentonite sample has a lower SiO content (58.609%) compared to acid-activated bentonite (61.347%), indicating a richer silica composition. In contrast, the Al_2O_3 content is slightly more stable between natural and acid-activated bentonite. The increase in SiO_2 content in acid-activated bentonite is caused by hydrogen ions (H^+) from the acid being able to attack the Si-O bonds in the silica structure, causing silica to be released as silicic acid or silica solution, which is more soluble in water. As a result, the SiO_2 content in acid-activated bentonite is slightly higher. While silica is more susceptible to decay in the acid activation process, aluminum (Al_2O_3) is more stable and less affected by acid. Most of the Al_2O_3 in bentonite is contained in clay minerals such as montmorillonite, which has a relatively stable structure under acidic conditions [19].

Table 2. Composition analysis of natural and acid-activated bentonite.

Composition	Natural bentonite (% by weight)	Acid activated bentonite (wt%)
SiO_2	58.609	61.347
Al_2O_3	22.493	22.154
Fe_2O_3	8.160	7.594
MgO	3.809	3.635
CaO	3.391	1.661
K_2O	0.877	0.890
Sc_2O_3	0.009	0
TiO_2	0.808	0.883
P_2O_5	0.958	0.974
SO_3	0.277	0.325
Cl	0.262	0.237
MnO	0.083	0.041
V_2O_5	0.055	0.057
Rb_2O	0.006	0.007
SrO	0.043	0.041
Ta_2O_5	0.018	0.018
Gd_2O_3	0.085	0.082
ZnO	0.022	0.020
Cr_2O_3	0.021	0.021
CuO	0	0.011

Interestingly, the Fe_2O_3 content is higher in natural bentonite, possibly contributing to its color and adsorption properties. The acid activation process appears to have reduced the CaO and MgO content in bentonite, which may be attributed to the leaching of these cations during activation. The reduced CaO and MgO content may change bentonite's cation exchange capacity and swelling properties [12][22]. Previous studies have shown that the adsorption capacity of bentonite towards various pollutants, such as heavy metals and organic compounds, can be influenced by its chemical composition [20][23]. Modified bentonite, with higher silica and lower cation content, can show better adsorption performance for some contaminants. In addition, the acid activation process can also affect the surface and pore structure of bentonite, which can also affect its adsorption and catalytic properties.

Bentonite Adsorption Behavior from the Effect of Contact Time

The adsorption of natural bentonite Pacitan showed a high recovery rate from the beginning (5 minutes) and remained stable at around 100% throughout the 1440 minutes duration. This indicates that bentonite, considering the NaCl complex ligand (A1), has an excellent adsorption capacity and quickly reaches saturation conditions in a short time. Pacitan bentonite activated with acid in uranium adsorption is much lower than without activation, with a gradual increase in time. At the 60th minute, the adsorption showed a significant increase but never approached the value of 100%. This indicates that bentonite activated by acid has a lower adsorption capacity or slower adsorption kinetics than without activation, as seen in Figure 2 and Figure 3. This difference is caused by the physicochemical properties of the modified bentonite (if AA1 is a modified form of A1), such as specific surface area, availability of active sites, or ionic interactions of uranium with the bentonite surface [7][20]. This can be observed in the SEM test in Figure 1a, where in natural bentonite, the hydroxyl groups on the mineral surface remain intact to form hydrogen bonds with adsorbed molecules. Acid activation often removes or damages the hydroxyl groups, thus reducing the ability of bentonite to adsorb polar molecules [13] [18].

Natural bentonite that binds complex ligands with NaCl (A1) tends to replace cationic ions (such as Ca^{2+} or Mg^{2+}) on the bentonite surface with Na^+ ions. This process increases the CEC and expands the interlayer space. NaCl provides a more dispersed and positively charged bentonite surface, thereby the adsorption of U ions is more efficient. Even in a short time, the high adsorption efficiency is due to the U^{2+} ions interacting more easily with the bentonite surface modified with NaCl. Meanwhile, acid activation causes the removal of most of the cations present on the bentonite surface. Furthermore, this process can damage the structure of the bentonite layer, mainly if the acid is used aggressively. This decrease in CEC means that fewer active sites are able to attract and trap U ions, thus reducing the adsorption efficiency [24][25].

Na_2SO_4 increases the stability of the surface charge. Still, sulfate ions compete with uranium ions to attach to the surface, so the adsorption efficiency is more stable but slightly lower than A1. Compared with acid-activated bentonite (AA2), the adsorption capacity of natural bentonite is more efficient because acid activation removes cations such as Al^{3+} and Fe^{3+} that support the bentonite layer structure, causing structural damage. The damaged structure in AA2 reduces the ability of bentonite to adsorb heavy ions, such as uranium, because the inner layer and pores of bentonite become smaller or disappear [19]. In contrast, natural bentonite (A2) maintains its layered structure, which supports the adsorption of uranium ions through cation exchange mechanisms and sulfate ligand interactions [26]. Acid activation produces more silanol (Si-OH) groups than aluminum (Al-OH). Silanol groups are more selective for polar molecules and are less effective in trapping metal ions such as U^{2+}. Furthermore, H^+ ions produced by acid activation can compete with U ions for active sites, reducing adsorption efficiency [27].

HPO_4^{2-} ions can modify the bentonite surface by adding anionic phosphate groups. These groups tend to increase electrostatic interactions with positively charged U ions. However, at a certain pH, phosphate can react with U^{2+} ions, forming precipitates (such as uranium phosphate) not absorbed by bentonite [21][28]. Initially, A3 had a lower adsorption efficiency than A1 and A2 due to the competition of phosphate ions with uranium ions to attach to the bentonite surface. Over time, the adsorption efficiency of A3 increases, but not as good as A1 or A2, due to the precipitation reaction that prevents direct adsorption capacity.

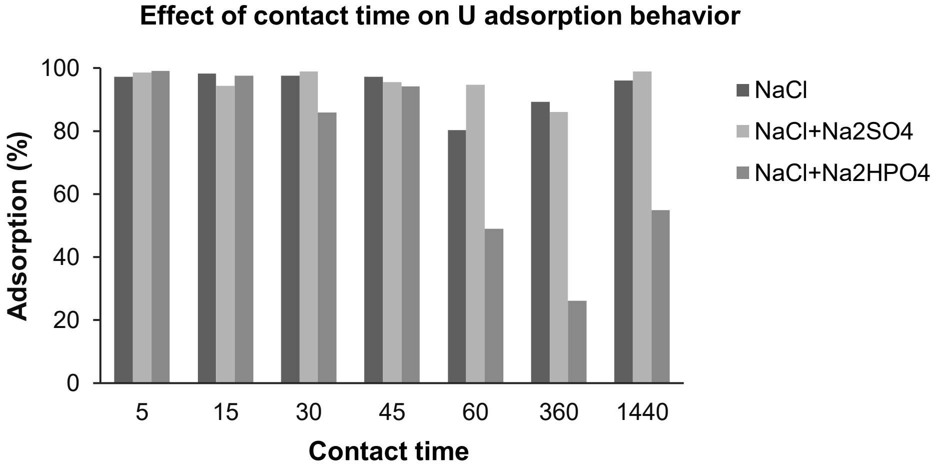


Figure 2. Adsorption behavior of natural bentonite over time.

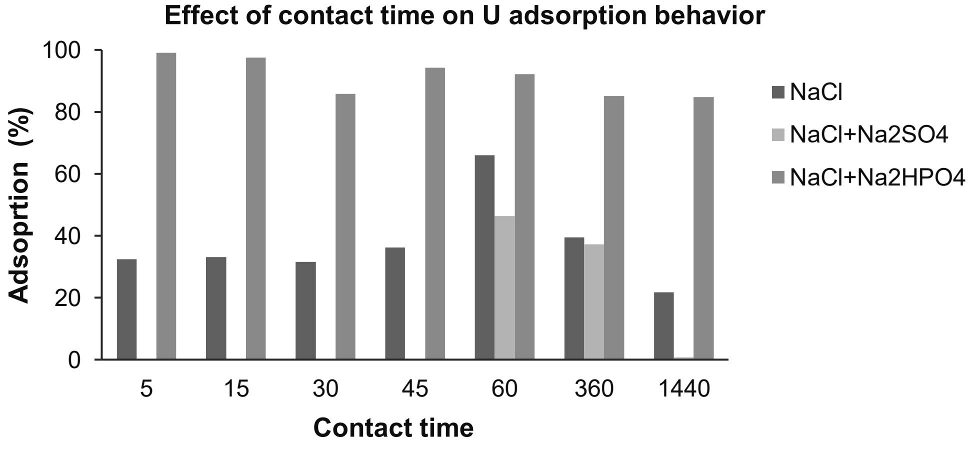


Figure 3. Adsorption behavior of acid-activated bentonite against time.

Effect of Uranium pH

In Figures 4 and 5, the results of uranium sorption on bentonites in 0.01 M NaCl solution is presented as a function of pH. The sorption curve is only slightly influenced by increasing pH.

The effect of pH on uranium speciation shown in Figure 4 shows the increase in absorption from pH 2 to maximum absorption at pH 4 but decreases above pH 4. The amount of uranium adsorbed decreases slightly with increasing pH. The results showed that the pH of the solution significantly affects the efficiency of uranium adsorption by Pacitan bentonite. At low pH, the adsorption capacity of bentonite is higher than at neutral or basic pH. This is due to the protonation of hydroxyl groups on the bentonite surface, which increases the positive charge and the binding capacity of positively charged uranium ions [25].

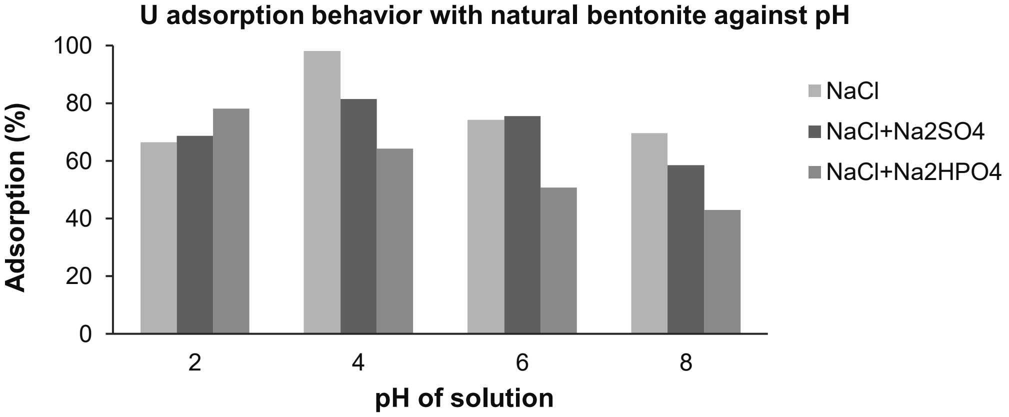


Figure 4. Uranium adsorption behavior with natural bentonite against pH.

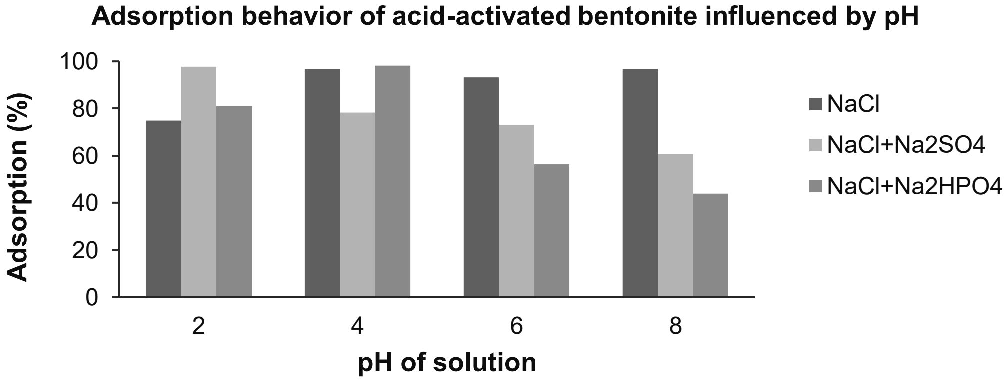


Figure 5. Uranium adsorption behavior with acid-activated bentonite against pH.

At low pH values (approximately 2), uranium is present as uranyl ions (UO_2^{2+}) with a strong affinity for negatively charged surfaces. Acid activation increases the negative charge of the bentonite surface by protonating the surface hydroxyl groups. This increase in negative charge attracts the positively charged uranyl ions, increasing adsorption [27]. The concentration of hydroxide ions (OH^-) increases at higher pH values. These hydroxide ions compete with uranyl ions for adsorption sites on the bentonite surface, reducing uranium adsorption. In addition, other metal ions may be more soluble at higher pH and compete with uranium for adsorption sites. The interaction between uranyl ions and the bentonite surface involves complex reactions with surface hydroxyl groups. The surface area of the complex formed is influenced by pH, and complexes formed at low pH are generally more stable, thus favoring adsorption. Na^+ ions from NaCl can compete with target ions (e.g., U^{6+}) in the adsorption process on the bentonite surface. However, after activation, the active sites of bentonite become more numerous, thus reducing the effect of this competition. SO_4^{2-} ions from Na_2SO_4 can form complexes with metal ions such as uranium, which have the potential to inhibit adsorption depending on the pH conditions and the surface charge of the bentonite [29]. Adding Na_2SO_4 increases the competing ion (SO_4^{2-}) in the solution, which can reduce the efficiency of uranium adsorption, causing lower adsorption compared to A1 at all solution pH. HPO_4^{2-} ions are amphoteric and can affect adsorption through pH changes around the bentonite surface. Na_2HPO_4 can form phosphate complexes with uranium, which are not readily adsorbed by bentonite [29]. This explains the lower performance of A3 compared to A1 and A2.

Activated bentonite is more resistant to this effect due to its more structured surface properties. In natural bentonite, Na^+, Cl^-, SO_4^{2-}, and HPO_4^{2-} ions compete more actively for adsorption sites. As a result, the ability of bentonite to adsorb target uranium ions is reduced. At the same time, activated bentonite has more active sites, which allows it to better overcome this competitive effect [26][27].

Effect of Temperature

In general, temperature significantly influences uranium adsorption in different systems. Increasing temperature tends to increase the solubility of uranium, accelerate the rate of chemical reactions and diffusion, and change the physical properties of the adsorbent [12]. Uranium absorption by natural bentonite compared to acid activated bentonite on temperature variation can be seen in Figure 6 and 7. However, very high temperature can damage the adsorbent and reducing the adsorption efficiency. At high temperatures, inactive natural bentonite has a more stable structure, supporting uranium adsorption. In contrast, acid-activated bentonite can undergo structural changes that make it more sensitive to temperature and degrade, reducing its ability to adsorb uranium [25]. The higher uranium adsorption on natural bentonite compared to acid-activated bentonite can be explained by the changes in physical and chemical properties after acid activation. Natural bentonite has surface and structural properties more favorable for uranium adsorption, as seen in SEM tests (Figure 1). At the same time, acid activation can damage or change these properties, reducing its ability to interact and adsorb uranium efficiently. In natural bentonite, the uranium adsorption efficiency for A1 showed a high adsorption rate at all temperatures (close to 100%), while A2 and A3 showed lower adsorption, especially at higher temperatures. Temperature significantly affected the adsorption of A2 and A3, with a decrease in uranium adsorption at higher temperatures (45 °C and 60 °C). This suggests that natural bentonite is less stable at increasing temperatures when complex ligands are present and are used differently than NaCl. In acid-activated bentonite, the uranium adsorption efficiency tended to be lower for all complex ligand variants than natural bentonite. However, AA3 showed a higher adsorption enhancement than AA1 and AA2 at high temperatures (60°C). Acid activation appears to change the temperature behavior of bentonite, with some ligands becoming more effective at higher temperatures. However, the overall adsorption efficiency remains below 70%.

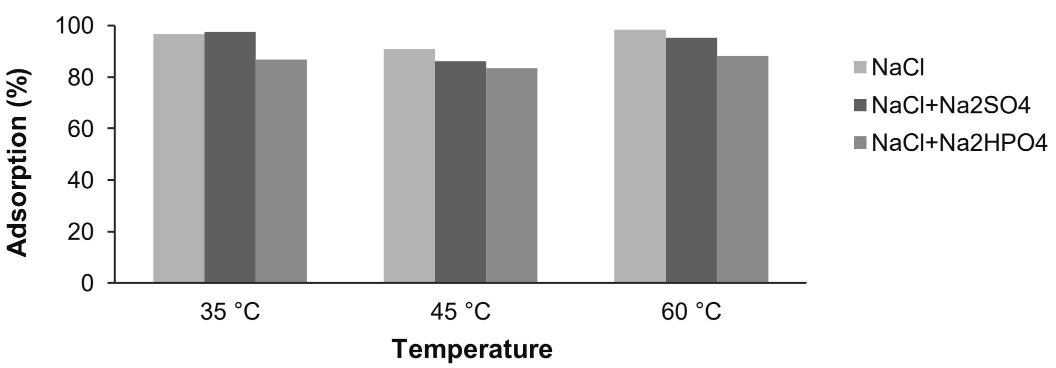


Figure 6. Uranium adsorption behavior with natural bentonite against temperature.

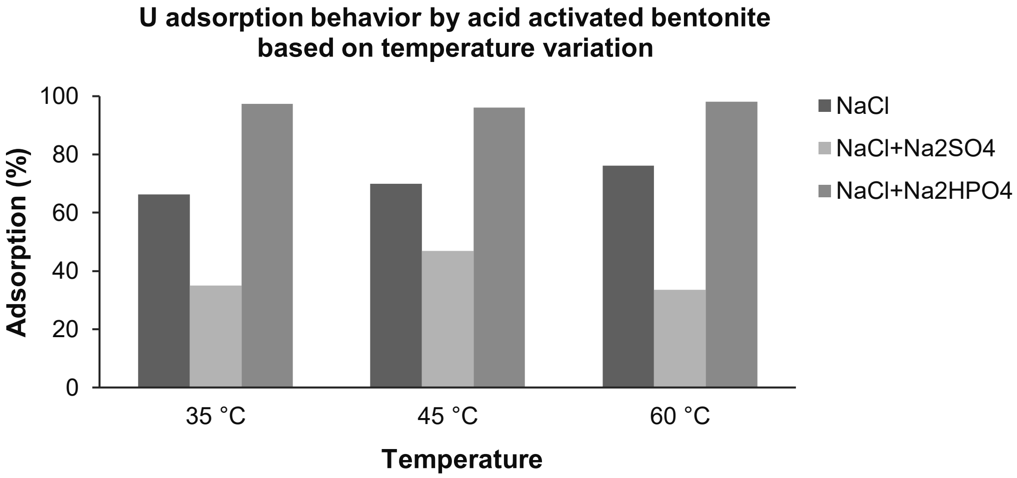


Figure 7. Uranium adsorption behavior with activated bentonite against temperature.

CONCLUSION

The results of this study indicate that Pacitan bentonite has significant potential as a uranium adsorbent, with the best performance occurring at acidic to neutral pH conditions. Acid activation increases bentonite's surface area and pore volume but reduces the overall adsorption capacity due to surface properties and mineral structure changes. NaCl ions positively affect uranium adsorption, while other ions such as SO_4^{2-} and HPO_4^{2-} show competitive effects that reduce adsorption efficiency. In addition, temperature affects the kinetics and capacity of adsorption, with natural bentonite showing better stability than acid-activated bentonite at high temperatures. These results provide important insights into the development of nuclear waste treatment technology using bentonite-based adsorbent materials

ACKNOWLEDGEMENTS

The author is grateful to the Head of PRTBNLR and the Head of the Nuclear Raw Materials Processing Department for their support and assistance with this research.

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