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Environmental Remediation of Contaminated Wastewater with Ammonium Using Clay-Based Adsorbents

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ABSTRACT

Due to a lack of water treatment technology, developing and emerging nations have become significant polluters and water shortage is exacerbated by pollution. Ammonium toxicity is a huge global environmental concern with no clear solution. Population growth and industrialization destroy the ecosystem. Common and industrial products contain ammonium ions. Water pollution damages fish and other aquatic life. An inexpensive and green wastewater treatment method is adsorption. Adsorbent polymers that remove ammonium ions from wastewater have been explored. Ammonium ions are very hazardous when deposited into surface waters. Surfaces of bentonite and montmorillonite clay may attach sodium ammonium ions. They are cheap and abundant, therefore used to treat drain water. Bentonite outperformed montmorillonite in eliminating ammonium ions from water. Bentonite and montmorillonite clays were used to remove residual ammonium ions. These are utilized for bentonitic and montmorillonitic clays. Both clays were absorbed in a neutral pH, and it was free of sulfuric acid, ammonium ions, and phosphorus ions. Montmorillonitic clay boosted TDS by nearly 10% whereas bentonitic clay only raised TDS by 1%. Adsorption may inexpensively filter water and the surface charge of adsorbents affect their adsorption capacity. Ammonium ions may be recycled, and several bioreactors can remove ammonium ions from liquid and solid phases. Iterate over several models and the Freundlich isotherm model outperforms the Langmuir model by 5%. And bentonite clay adsorbs better due to iron oxide content.

INTRODUCTION

Due to a lack of access to water treatment technology, emerging and fast-growing nations have become significant polluters. Growing pollution of aquatic sources endangers aquatic biota and promotes water shortages (Dey et al. 2021, Tse-Lun et al. 2021, Zahra & Mohammad 2013). The toxicity of ammonium ions is a significant global environmental problem, and it is difficult to define acceptable ecosystem boundaries. Population and industrial expansion emit ammonium ions, harming the ecosystem. Ammonium ions are found in numerous home and cleaning products, as well as commercial and industrial fertilizers (Rajoriya & Kaur 2014, Limbachiya et al. 2012, Mithra et al. 2012). Illegal municipal wastewater dumping allows ammonium ions to enter the aquatic environment, affecting fish and aquatic life. Adsorption is one of the most cost-effective and ecologically friendly wastewater treatment methods developed in recent decades. Several adsorbent materials have gained academic attention for their capacity to extract ammonium ions from wastewater efficiently (Chopin et al. 2012, Neori et al. 1998). These contaminants are absorbed from wastewater via oxidation, osmosis, reduction, precipitation, ultra-filtration, electro-dialysis, ion exchange, and electrochemical methods. They create more sludge, are less efficient, and cannot remove as many ammonium ions. Adsorption is the preferred way of obtaining a broad variety of waste materials and natural resources (Duruibe et al. 2007, Young 2005). Clay is a particularly effective adsorbent for ammonium ions because clay is extremely effective, readily accessible, relatively affordable, and potentially reusable and cost-efficient (Buono et al. 2015, Rivett et al. 2008, Gooddy et al. 2014, Reda et al. 2018, 2022). Adsorption is the mass transfer of material from the gas or liquid phase to the surface or interface of a solid phase (i.e., adsorbent). The adsorption process has four stages: Diffusion from the bulk solution to the adsorbent surface through the boundary layer; Pore diffusion or intraparticle diffusion; physical and/or chemical

reaction: adsorption between adsorbate and adsorbent active sites (Aruna et al. 2021, Ibrahim et al. 2022). The first bulk diffusion phase may be avoided by uniformly distributing adsorbent and adsorbate in the solution. Adsorption kinetics is dictated by film diffusion or pore diffusion since the previous step, physical/chemical contact occurs quickly (Mahmoud et al. 2012). Adsorbent liquid volume and surface area affect film diffusion rate i.e., liquid membrane area. Relatively faster liquid-particle relative velocity means a thinner fixed liquid layer on particle surfaces and faster film diffusion (Park & Kim 2005). It is connected to the adsorbents' pore shape and distribution, as well as the adsorbate's molecular size and structure, but is less related to the liquid concentration or adsorbent particle surface area. Adsorption time squared determines pore diffusion rate $t_{0.5}$. Consequently, if the adsorption rate is linear with $t_{0.5}$, the pore diffusion mechanism likely dominates adsorption during these periods (Peez-Corona et al. 1998). Adsorption of ammonium ions from polluted water has great environmental and economic possibilities. Various laboratory studies may produce experimental values for field treatment plant design. Other system variables such as temperature, pH, and other ammonium compounds must be investigated further. Surface modification of adsorbents may improve removal efficacy and should be investigated. Solid waste adsorbents may minimize chemical consumption in water and wastewater treatment (Tian et al. 2010, Saad et al. 1997, El-Shamy et al. 2017a, 2017b, 2017c). Discoveries made in a continuous reactor system may be implemented more efficiently in the field. Research on ammonium ions removal in the presence of other contaminants is vital. Several elements affecting open-air scenarios must be understood (Shehata et al. 2019, Samar et al. 2022, Abdelfattah & El-Shamy 2022). This study is the first to compare two adsorbent media for the construction of community or home-based treatment facilities in industrialized nations. All the variables that occur in open natural environments must be acknowledged (Ashraf et al. 2018, Ibrahim et al. 2022). In batch processing, the adsorbent is combined with ammonium ion solutions for a specified contact time, and the adsorbate is separated by sedimentation or filtering (Farag et al. 2016). In a closed system, the data collected is typically inapplicable to real systems (Namasivayam & Sangeetha 2004). The batch method is used to investigate adsorption processes and compare adsorbents' capacities. Charged contaminants (such as inorganic and organic pollutants) adsorb on adsorbents with oppositely charged surfaces (Frisoni et al. 2001). Because most naturally polluted streams have modest quantities of certain contaminants, effective contaminant removal is critical. Contaminants at ultra-low concentrations should be reduced using adsorbents. Lower-loading adsorbents must be renewed for optimum utilization (Liu et al. 2011). Researchers can improve adsorption rates by optimizing adsorbent design and experimental conditions. Single or more layers of nitrate molecules may be adsorbed on an adsorbent. Monolayer adsorption happens when an adsorbate only covers one molecular layer of the adsorbent surface. Adsorbent surfaces may adsorb more than one molecular layer of adsorbates (Tian et al. 2009, Zohdy et al. 2021). Physical adsorption may be multi-layer, happening on top of a chemically adsorbed monolayer. This study's goal was to prevent ammonium ions from reaching river water sources. Adsorbents were compared in batches using variables such as pH, starting concentration, adsorbent dose, and contact duration (Luo et al. 2010).

MATERIALS AND METHODS

In this study, we will compare the physicochemical absorption of montmorillonitic and bentonitic clays for the treatment of ammonium-contaminated synthetic wastewater as well as drain wastewater from Giza's El-Rahawy drain. Montmorillonitic clay samples from Ayash Clay Mine in the Cairo-Suez area, Egypt, and bentonitic clay samples from Norther Coast West Alexandria. ADWIC, Egypt produces ammonium ions dilutions at various concentrations using ammonium ions solution (pure reagent 33%). The investigation employed analytical-grade chemicals such as NaOH and HCl to modify the pH of the batches. All dilutions were prepared using double distilled water. The authentic drain discharge sample came from Giza's El-Rahawy drain. It was carefully bottled, acidified to pH 3, and sent to the lab for testing (Mehta et al. 2021). This study employed both montmorillonitic and bentonitic clay as active adsorbates. For future usage, they were desiccated after homogenous grinding by crushing into tiny bits and fine mesh-size particles throughout a US Standard Testing Sieve No 100 (150 microns). With the Jasco-FTIR-Spectrometer (Japan), FT-IR montmorillonitic and bentonitic clay powder absorption spectra were measured to identify functional groups that may contribute to the adsorption process. The pH of solutions was adjusted with HCl and NaOH using a Jenway 3510 bench pH meter. The amounts of ammonium ions were determined using the distillation-titration technique, which was utilized to characterize all raw and processed effluents (Wang et al. 2020).

Batch Experiments

A multi-position magnetic stirrer at 200 rpm was used at room temperature (25°C ±2) for mixing beakers containing samples. The supernatant was filtered through the Whatman membrane filters (0.45 μm). The residual ammonium ions concentration is measured according to Standard Methods

for Water and Wastewater Examination. The adsorbed NH_4^+ ions are calculated as:

$$Adsorption\% = (C_0 - C_e)X\frac{100}{C_0} \quad \ldots(1)$$

Where C_0 and C_e are the initial and equilibrium concentrations of ammonium ions in ($mg.L^{-1}$). The number of ammonium ions (qt at time t) adsorbed by adsorbent (M) is estimated utilizing the subsequent equivalence:

$$q_t = (C_0 - C_t)V/M \quad \ldots(2)$$

C_t is the concentration ($mg.L^{-1}$) of ammonium ions after time t min., V is the volume of initial ammonium ions in one liter, and M is the adsorbent mass used in grams. The adsorption capacity (q_e) is defined when equilibrium is reached (Gupta et al. 2011):

$$q_e = (C_0 - C_e)V/M \quad \ldots(3)$$

Adsorption Isotherms

In addition, isotherm models assist in determining the needed dosage of adsorbent and the most appropriate sorbent for the situation. The most common isotherm models for a single solute are Langmuir and Freundlich (El-Kashef et al. 2019).

Langmuir Isotherm

It depends on maximum adsorption corresponding to a saturated monolayer of adsorbate molecules on the surface of the clay.

The Langmuir isotherm is presented in:

$$q_e = \frac{q_{max} * K_l\, C_e}{1 + K_l\, C_e} \quad \ldots(4)$$

The linearized form is:

$$\frac{C_e}{q_e} = \frac{1}{q_m\, K_l} + \frac{1}{q_m} * C_e \quad \ldots(5)$$

$$Slope = \frac{1}{q_m} \quad \ldots(6)$$

$$Intercept = \frac{1}{q_m\, K_l} \quad \ldots(7)$$

Where q_m and K_L are Langmuir constants related respectively to the sorption capacity, and sorption energy, C_e ($mg.L^{-1}$) is the equilibrium concentration and q_e (mg/gm) is the adsorption capacity at equilibrium. R_L is the essential characteristic of the Langmuir dimensionless constant separation factor or equilibrium parameter which is defined by the following equation (El-Shamy et al. 2021).

$$R_L = \frac{1}{1 + K_L * C_0} \quad \ldots(8)$$

R_L calculated values from the above equation show the nature of the adsorption process to be either unfavorable (R_L>1), linear when (R_L=1), favorable when (0<R_L< 1), and irreversible when (R_L=0).

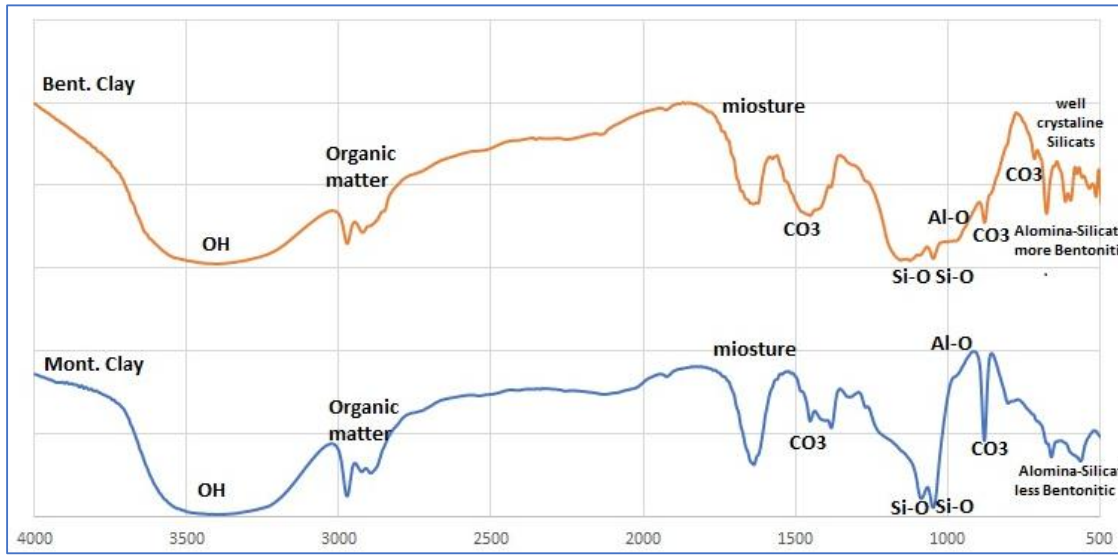

Fig. 1: FTIR Charts for bentonitic and montmorillonitic clay.

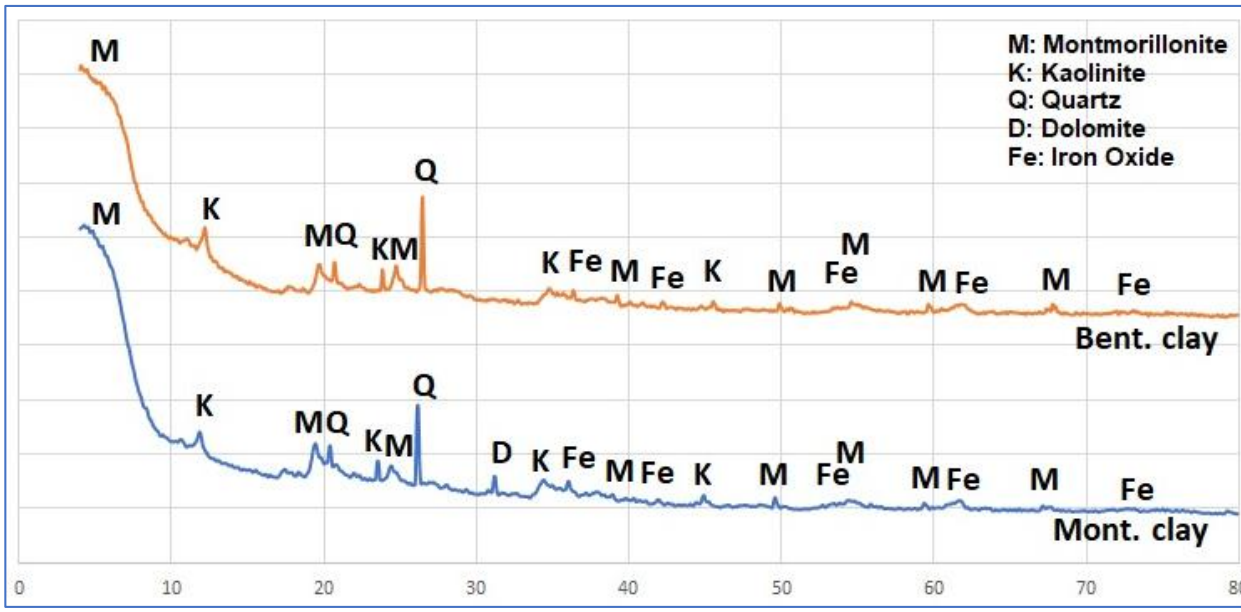

Fig. 2: XRD Charts for bentonitic and montmorillonitic clay.

Freundlich Isotherm

It involves surface heterogeneity and the exponential distribution of active sites and their energies.

The Freundlich isotherm equation is presented as:

$$R_L = \frac{1}{1 + K_L * C_0} \quad \ldots(8)$$

$$q_e = C_e^{\frac{1}{n}} \quad \ldots(9)$$

The linearized form is:

$$\ln q_e = \ln K_F + (\frac{1}{n}) \ln C_e \quad \ldots(10)$$

And the ((n)) calculated from the equation:

$$\text{Slope} = \frac{1}{n} \quad \ldots(11)$$

And the (K_F) is calculated as the anti-ln of the intercept as:

$$\text{Intercept} = \text{lin } K_F \quad \ldots(12)$$

Where q_e (mg.g^{-1}) is the adsorption capacity at equilibrium, C_e (mg.L^{-1}) is the concentration of ammonium ions at equilibrium, K_F is a constant related to the temperature, and n is a characteristic constant (Varshney et al. 1996).

RESULTS AND DISCUSSION

The interaction of loaded clay molecules with ammonium ions was studied using FTIR (Fig. 1), XRD (Fig. 2), and XRF (Fig. 3). The adsorbent's surface area influences the adsorption process's potential effect. Because the clay particles were positively charged, the NH_4^+ ions were likely adsorbed on the surface via complexing with the clay cations and attracting ammonium ions through hydrogen bonding.

Adsorbent Characterization

Adsorption of ammonium ions enhanced the surface clay/ammonium ions ratio. XRD, FTIR, and XRF were used to study NH_4^+-clay interactions. It is stretching at 3049 and 2930 cm^{-1}, as well as two bending modes at 1503 (scissoring) and 695 (wagging) cm^{-1} were identified after integration with –N–H. A rare combination band at 2003 cm^{-1} was also identified. It indicated that clay particles had replaced protons on the –NH_4^+ groups of ammonium ions. Adsorption of NH_4^+ produced a 1403 cm-1 N–H scissoring vibration peak of an ammonium compound. the –NH_2 vibrations shifted blue from 3046 to 3128 cm^{-1}, confirming the clay particles' incorporation of the -NH_4^+ ligand. FTIR detected the chemical environment of the clay/NH_4^+ species. The peak regions show that 86% of –NH_2 groups protonated as –NH_4^+ groups. After clay loading, the –NH_2 groups complexed

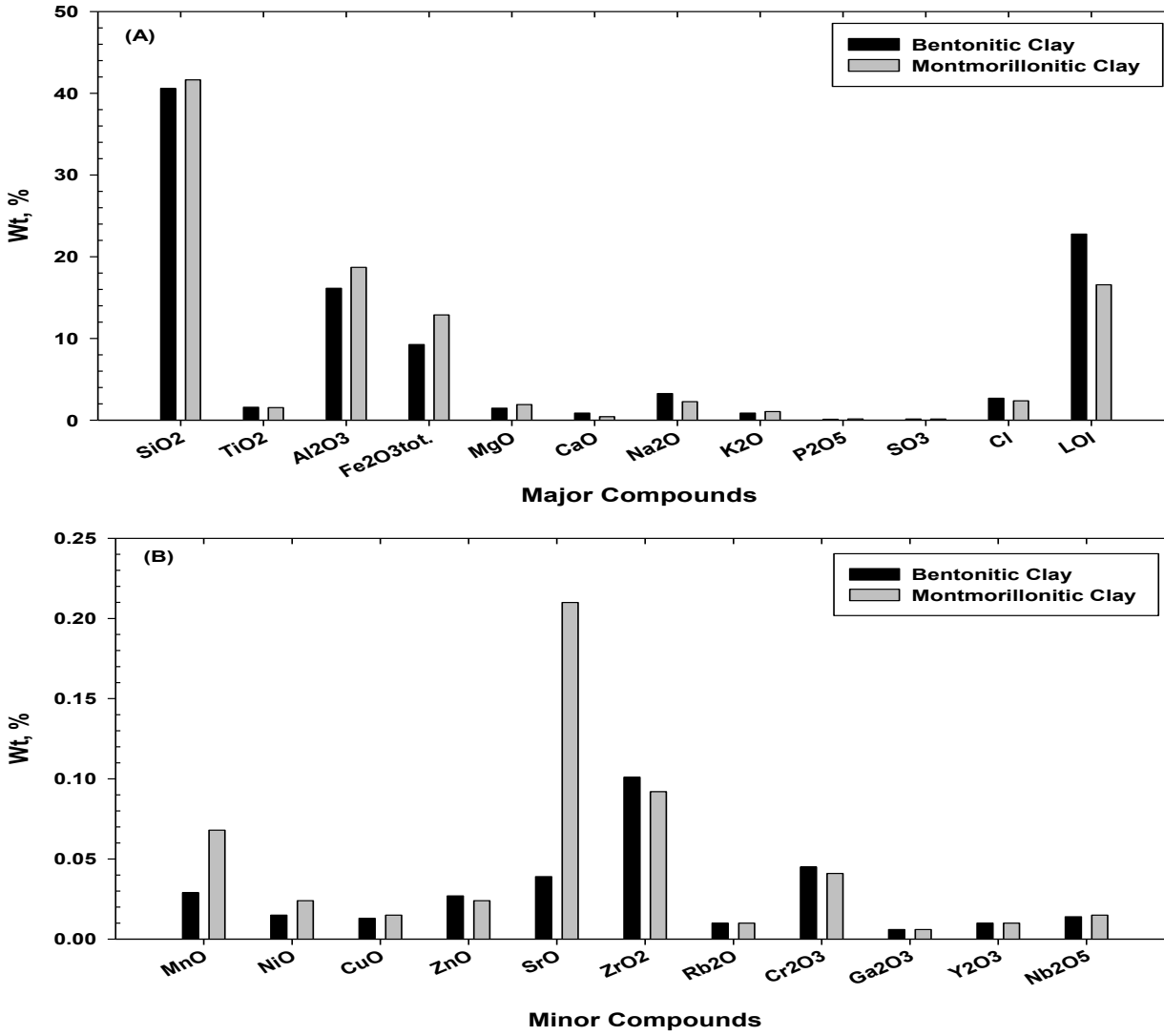

Fig. 3: XRF Charts for bentonitic and montmorillonitic clay.

with the clay particles, adding 402.4 eV to the binding energy (Benaissa & Benguella 2004).

Effect of Adsorbent Dose

Fig. 4 shows the effect of various (B) and (M) dosages on ammonium ion elimination. The ammonium ions removal rate was less than 50% when the adsorbent concentration was less than 0.3 $g.L^{-1}$. However, increasing the concentration from 0.6 to 1.2 $g.L^{-1}$ had no effect. Surprisingly, increasing the adsorbent concentration enhances the ammonium ion nitrogen removal rate. The adsorbent's active sites were less accessible when the ammonium ions concentration increased, according to one research. Because ammonium ions reached the adsorbent's lower active sites, the loading rate decreased but the loading capacity rose. Ammonium ions diffuse into the low active sites in (B) and (M) as the concentration increases, speeding up ammonia removal. The ammonium ions removal method error was less than 4%, indicating that ammonium ions intake remained constant (El-Shamy et al. 2017).

Effect of Initial pH

Fig. 5 shows adsorption data for B and M vs. contact time at 5 min intervals of up to 120 min. The number of ammonium ions absorbed by the material under investigation was determined using a cation exchange capacity and an adsorbent dosage (0.1 g.50 mL^{-1}). The authors of this article tested at pH 6. The adsorption plots of ammonium ions on (B) and (M)

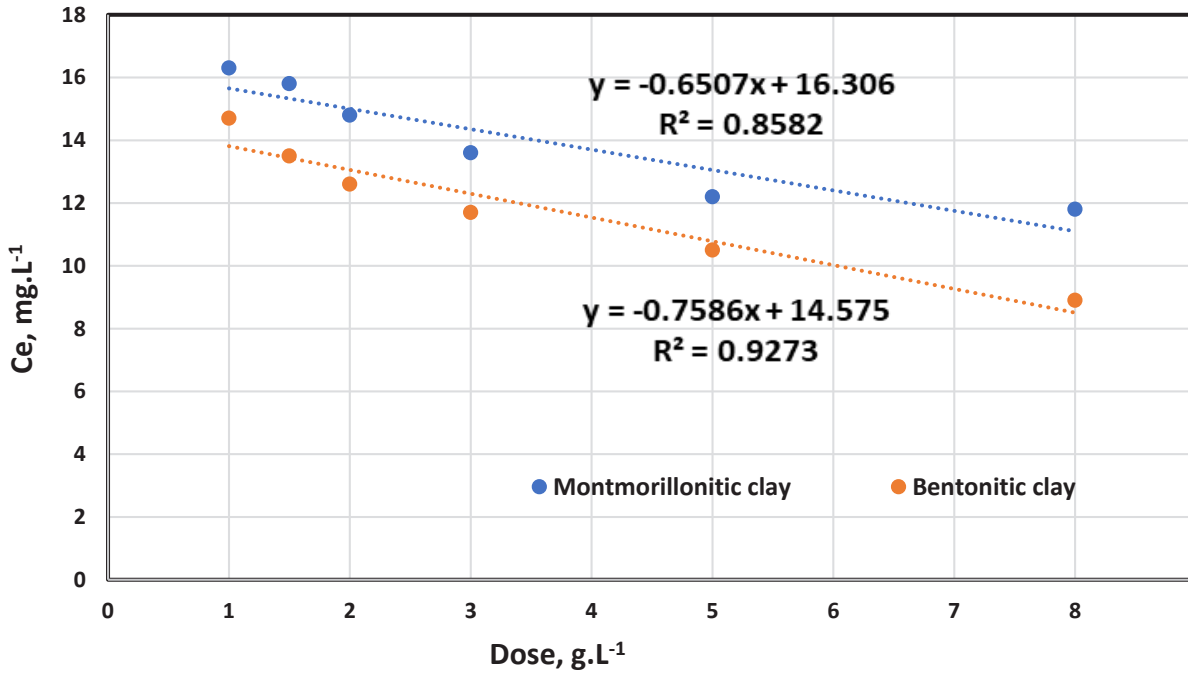

Fig. 4: Effect of weight volume % of bentonitic and montmorillonitic clay (Opt. of pH, 20 mg.L^{-1} ammonium ions, dose of clay 0.5 g.100 mL^{-1}, 100 mL solution, 1h stirring).

demonstrate three distinct phases: initial adsorption within 10 min, progressing equilibrium, and final equilibrium (El-Shamy et al. 2018).

$$(NH_2)_2CO + H_2O = 2NH_3 + CO_2 \quad \ldots(13)$$

Thus, unionized ammonia NH_3 and CO_2 are generated (CO_2). NH_4 ZZZ is formed when complex organic compounds in industrial, municipal, and animal wastes break down. Therefore, as previously explained, ammonium ions exist in two forms in aqueous solution: non-ionized (NH_4 ZZZ) and ionized (N), as represented by the equations:

$$NH_4^+ + OH^- = NH_3 + H_2O \quad \ldots(14)$$

$$NH_3 + H_3O^+ = NH_4^+ + H_2O \quad \ldots(15)$$

Ion exchange can only remove the ionized form of ammonium since it is pH-dependent. Because most ammonium install nitrogen is ionized at pH 8 and lower, these circumstances encourage elimination. In adverse circumstances, the balance changes quickly to a non-ionized form above pH 8. Because the pH of the solution affects the quantity of N removed, a series of experiments were done to determine the best pH range for ammonium removal. The ion exchange mechanism works best at pH 8 and below, with pH 6 providing maximum elimination. The equilibrium capacity is 50 meq.100 g^{-1} at pH 6, but 42 meq.100 g^{-1} at pH 8. Decreases in removal efficiency and equilibrium capacity to 35 meq.100 g^{-1} occur at pH 2. The N removal effectiveness reduces fast at pH 10, which may be explained by the fact that the ammonium ion is neutralized by the hydroxyl ion, leaving it uncharged. The performance diminishes below pH 6 due to increased hydrogen ions in the solution, which compete for exchange sites (Ho et al. 2000).

Effect of Contact Time

Adsorption of Bentonite (B) and Montmorillonite (M) vs contact time is shown in Fig. 6. The number of ammonium ions absorbed by the clay sample was monitored over time. The batch experiment of this paper was tested at pH 6. The adsorption plots of ammonium ions on (B) and (M) demonstrate three distinct phases: initial adsorption within 10 min, progressing equilibrium, and final equilibrium (Dey et al. 2019).

Kinetics of Adsorption Process

It was used to match experimental data and describe the relationship between adsorbate concentration and adsorption rate. The ammonia adsorption correlation (R^2) was 0.9997 for montmorillonitic clay. The kinetic model has a higher R^2 of 0.9985 for bentonitic clay (Fig. 7). Difficulty in experimental procedures creates unpredictable results. The qt error is large when t is fixed. qe(cal.) was found to be closer to the experimental data (qe(exp.)). The fitted kinetic model is validated by comparing the calculated qe(cal.) to the measured qe. In

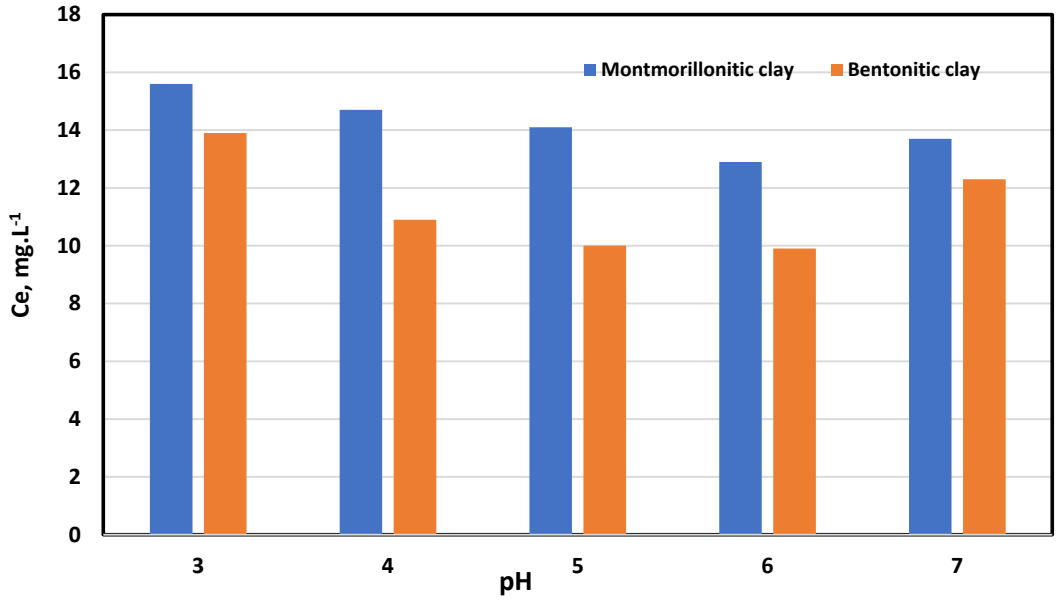

Fig. 5: Effect of pH on the adsorption capacity of bentonitic and montmorillonitic clay (Opt. of time, 20 mg.L^{-1}, a dose of clay 0.5 g.100 mL^{-1} clay, 100 mL, pH 6).

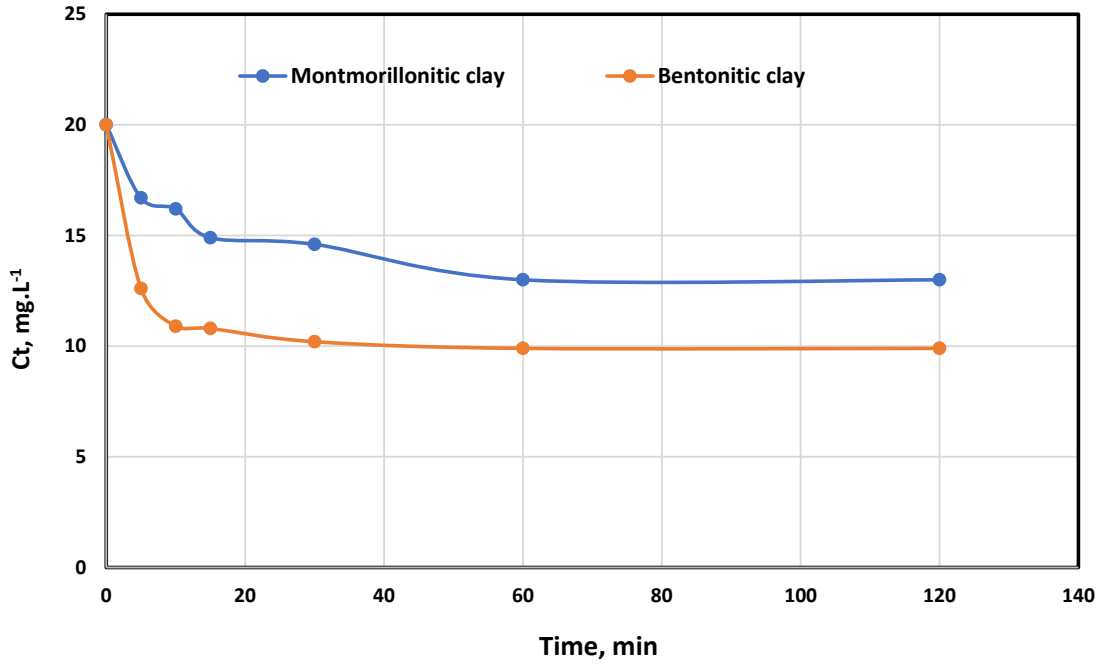

Fig. 6: Effect of contact time on the adsorption capacity of Bentonitic and montmorillonitic clay.

addition, coexisting cations influence the kinetic model for ammonia nitrogen adsorption (Aksu 2001).

Adsorption Isotherms

Fig. 8 demonstrates the batch multiple and simultaneous ammonium ions adsorption using two clay types of Langmuir isotherm. This isotherm was fitted to the ammonium ion adsorption data. C_e/Q_e versus Ce over the concentration range produced a straight curve. The adsorbed ammonium ions increased with the equilibrium concentration.

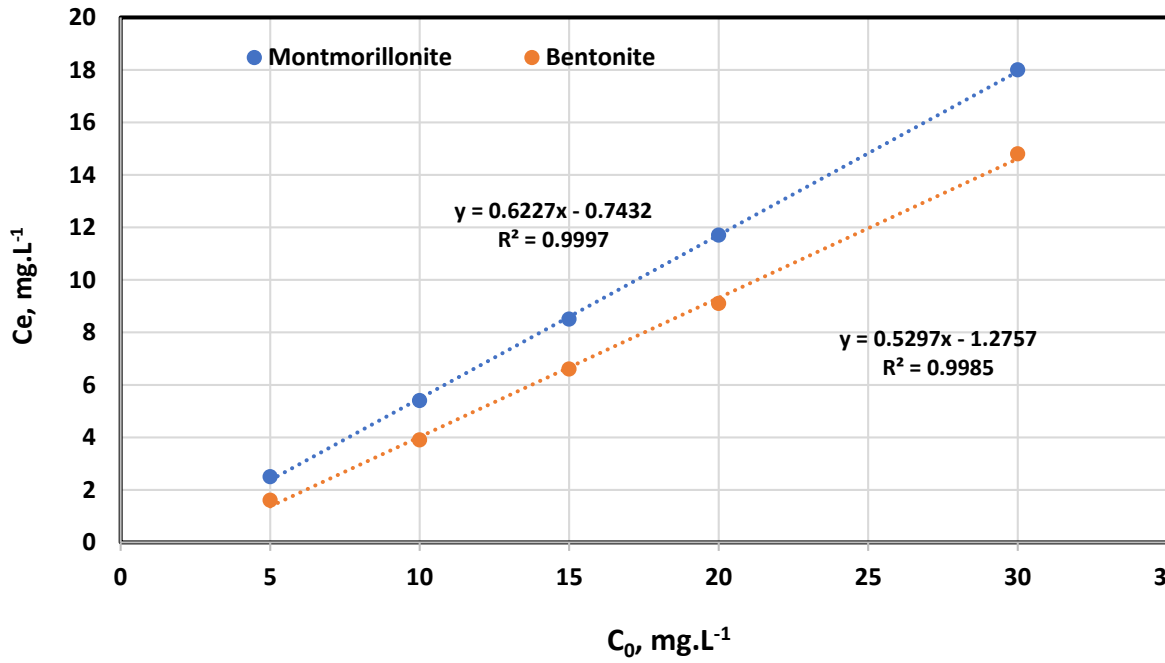

Fig. 7: Kinetics of adsorption using bentonitic and montmorillonitic clay (pH 6, a dose of clay 0.5 g.100 mL^{-1}, 1 h stirring).

Langmuir Isotherm

Fig. 8 shows the slope (b), intercept (Q_m), and coefficient of determination as well as the statistical fit of the adsorption data to the Langmuir model is shown in Table 1. It demonstrates that numerous ammonium ion adsorptions match the Langmuir isotherm model well, with $R^2 > 0.9$ for both clay types. Adsorption of ammonium ions is more favorable using Langmuir than using bentonite as an adsorbent. Similarly, ammonium ion adsorption data are applied to the Langmuir adsorption isotherm, but no linear form is obtained. The equilibrium data from the montmorillonitic clay adsorption isotherm fitted the Freundlich equation better than the Langmuir equation. The Langmuir adsorption paradigm fails in various situations because of its lack of material claim (Mehta et al. 2017).

Freundlich Isotherm

Fig. 9 shows the Freundlich isotherm for batch adsorption of ammonium ions onto clayey surfaces. In this case, the Freundlich isotherm was utilized to match the data. In Fig. 9 q_e versus C_e was displayed linearly over the concentration range. The amount of adsorbed ammonium ions q_e increased with equilibrium concentrations C_e. Table 2 displays the Freundlich isotherm model parameters from Fig. 9 and the statistical fits of the adsorption data to this equation. Because the coefficient of determination R^2 is larger than 0.9 for montmorillonitic clay, the isotherms are consistent for both montmorillonitic and bentonitic clay. Based on Fig. 9, the adsorption intensity (n) and the adsorption capacity (K_f). If the 1/n ratio is less than one, chemisorption is occurring, while co-adsorption is occurring. In bentonitic

Table 1: Langmuir data.

	Slope	Intercept	q_m	K_L	R_L	R^2
M	0.1575	4.9392	6.349206	0.031888	0.51-0.86	0.915
B	0.1813	2.3991	5.51572	0.07557	0.306-0.726	0.921

Table 2: Freundlich data.

	Slope	Intercept	n	K_F	R^2
M	0.789	-1.42	1.267427	0.241714	0.999
B	0.6714	-0.713	1.489425	0.490171	0.999

clay, ammonium ions adsorption was chemisorption and cooperative adsorption. As seen in Table 2, the Freundlich model's ammonium ion constants n vary (1.2820 - 1.1074). n represents the adsorption processes' efficiency. According to the Freundlich model, montmorillonitic clay's resorption of ammonium ions is superior to bentonitic clay (Babarinde et al. 2009).

Application of the Treatment Process for Real Drainage Wastewater

The collected drainage water from the El-Rahawy drain in Giza, Egypt, was physiochemically characterized (Table 3). After adding 10 g of clay to 1 L of collected sample and stirring for 60 minutes, the effluents were filtered and characterized physiochemically (Table 3_. More than 79 percent of COD and BOD were removed using montmorillonitic clay, while over 84 percent were removed using bentonitic clay. Both clays removed 100 % of sulfide and 100% of total phosphorus. Overall, both clays efficiently eliminated contaminants from drainage water, although bentonitic clay was more effective owing to the presence of alumina silicate and iron oxide in their structure.

Table 3: Physicochemical characterizations of the raw and treated drainage water collected from El-Rahawy drain, Giza, Egypt.

	Raw Drainage water	Treated Effluents			
		Montmorilonitic clay		Bentonitic clay	
		M	% Removal	B	% Removal
COD, $mg.L^{-1}$	108	22	79.63	13	88
BOD, $mg.L^{-1}$	62	13	79	10	84
H_2S, $mg.L^{-1}$	1.6	0	100	0	100
NH_3, $mg.L^{-1}$	3.1	0.2	94	0.1	97
TDS, $mg.L^{-1}$	920	1020	-10.87	930	-1.09
TP, $mg.L^{-1}$	0.3	0	100	0	100
TKN, $mg.L^{-1}$	23	6.8	70.4	4.9	78.7

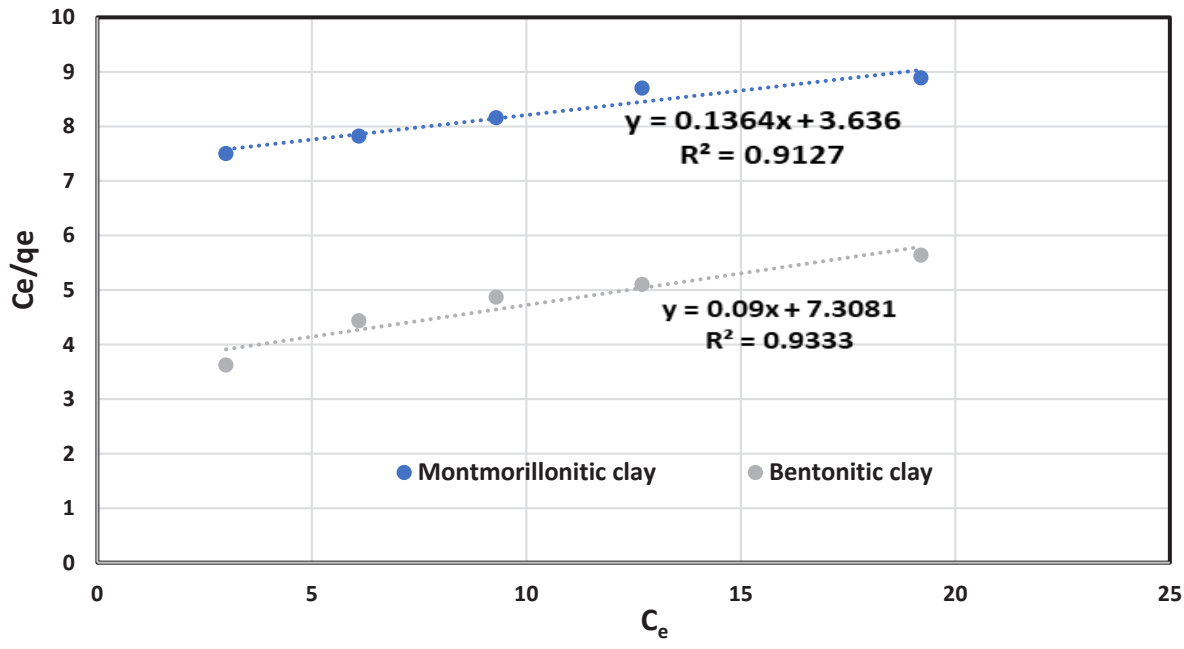

Fig. 8: Langmuir isotherm results for bentonitic and montmorillonitic clay.

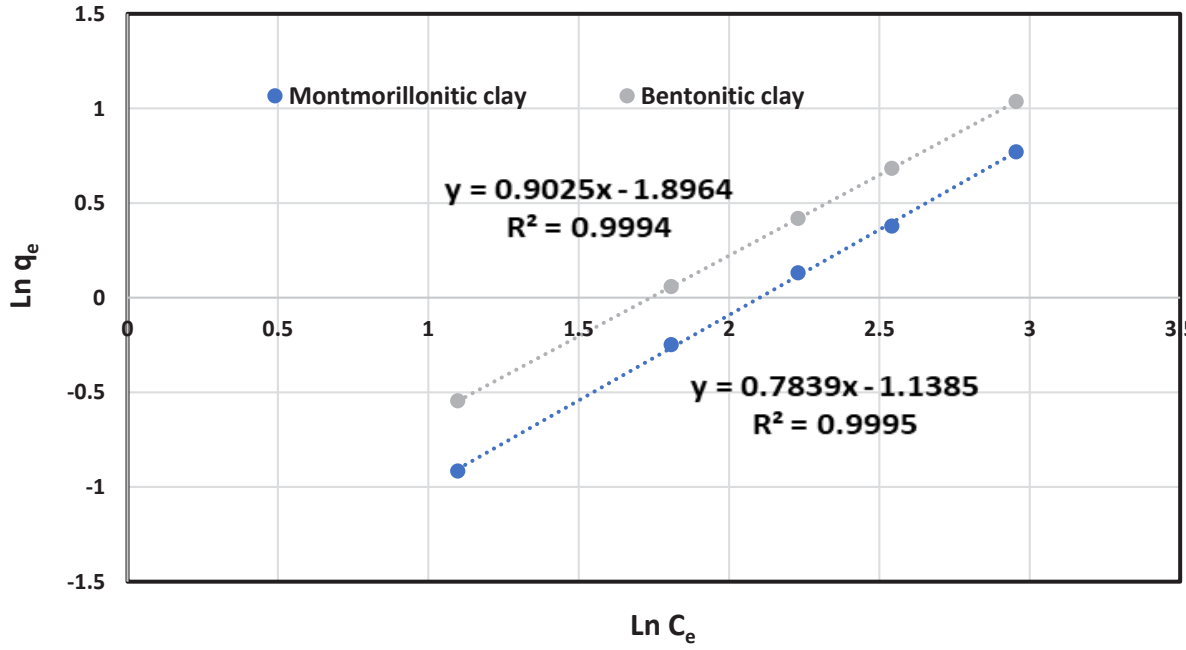

Fig. 9: Freundlich isotherm results for bentonitic and montmorillonitic clay.

CONCLUSIONS

The adsorption method in the treatment of water pollutants is considered one of the most important and simple methods because it is a simple and sensitive way to remove many water pollutants, and these pollutants include ammonium ions present in sewage water. Besides all the above advantages, it is also a low-cost and environmentally friendly method. The process of removing pollutants is through the functional groups of the absorbents, which affect the surface charge and thus the ability to adsorb. It was classified as acidic or basic, which expanded its ability to remove pollutants, as it can deal with more than one type of pollutant with high efficiency. Added to its advantages is the ability to reuse after extracting ammonium ions, thus saving costs in purchasing additional materials from the absorbent material. Many bioreactors can remove ammonium ions from the water showing how ammonium ion molecules diffuse through the liquid and solid phases when equilibrium is reached. To choose a working model, it is necessary to fit isothermal data with different models. The adsorption properties of ammonium ions, which are often found in wastewater, were studied using two clay materials. The Freundlich, isotherm model fits the experimental data best for montmorillonitic clays alone, while the Langmuir model best fits both types of clays. The isometric models indicated that the absorption is physical and chemical. Both clays succeeded in removing pollutants from wastewater, although bentonite clays were more effective due to the presence of iron oxide in their structure. Through the given results, we can say that these materials are suitable for practical application due to their ease of availability, low cost, and high efficiency in dealing with a wide range of water pollutants.

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