



Original Research Article

Removal of ammonium ions from aqueous solution using hydroxy-sodalite zeolite

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ABSTRACT

In this research study, the removal of ammonium ions from aqueous solution by hydroxy-sodalite (HS) was evaluated. The effects of the contact time, pH, initial concentration, adsorbent dose, and the presence of other competing ions on ammonium ions removal were also studied. The result showed that the pH less than 7 was the best for ammonium adsorption, and the optimum removal was achieved at pH 7. Langmuir (Two linear forms), Freundlich, Harkin-Jura, Halsey, and Elovich isotherm models were fitted to the experimental data. These data were found to fit well with the Freundlich isotherm model. Kinetic experiments showed that equilibrium was reached after 60 min and the data correlated best with the pseudo-second order model. The presence of competing ions in solution affected the ammonium ions adsorption capacity. Overall, this adsorbent has the potential to remove ammonium ions from wastewater.

Graphical Abstract



Introduction

Nitrogen is one of the macronutrients necessary for plant growth and ammonium which is one of the forms by which plants consume nitrogen. Excess ammonium nitrogen in the environment could result to eutrophication of lakes and rivers [1], spoil the quality of water and increase toxicity to aquatic organisms [2]. Excessive use of nitrogen fertilizers in agriculture is a major source of ammonium in groundwater and surface water [3]. Ammonium is also emanated from industrial wastewater and urban sewage and dissolution of solid waste [4]. Zeolites have been evaluated as potential adsorbents for the removal of ammonium ions from wastewater due to their high cation exchange capacity, high adsorption capacity and high ammonium selectivity [5]. Zeolites are crystalline microporous aluminosilicates of alkali and alkaline earth metals with well-defined pores structures. They are built up of 3-dimensional frameworks of $[\text{SiO}_4]^{4-}$ and $[\text{AlO}_4]^{5-}$ tetrahedral, linked at their corners to form different channels and cages [6]. The negative charge on the lattice is neutralized by the positive charge of cations, typically, alkali or alkaline earth metal ions located within the pores of the material.

Hydroxy-sodalite (HS) which is a type of zeolites that belongs to clathrasils group which is made of cubic array of β -cages and exhibits similar structure of sodalite $(\text{Na}_8[\text{Al}(\text{SiO}_4)]_6\text{Cl}_2)$ where framework charges are balanced with hydroxide anions [7]. Because of small pore size (2.8 Å) and high ion exchange capacity, hydroxy-sodalite has been considered as a good candidate material for a

wide range of applications such as hydrogen storage, optical materials, adsorbent and base catalyst. Furthermore, this product with the high cation exchange capacity (CEC) could be used for wastewater treatment as an inorganic ion exchanger or an ion adsorbent capable of removing cations such as NH_4^+ and toxic heavy metal ions from water and sewage [8]. However, the adsorption of ammonium ions from aqueous solutions has been focused on natural zeolites such as clinoptilolite or mordenite [2–9]. Studies on the adsorption of ammonium ions on synthetic zeolites are scanty most especially the sodalites. The aim of this study therefore, is to evaluate the adsorption capacities of hydroxy-sodalite for ammonium ions from aqueous solution. In this sense, some isotherms and kinetic models were also studied.

Experimental

Materials and methods

Ammonium chloride (BDH, analytical grade) was used in the preparation of the synthetic wastewater. The stock solution of 1000 mg/L NH_4^+ was prepared by dissolving NH_4Cl in distilled water. Working standard solutions were prepared from stock solution by dilution with distilled water. Other adsorption tests were carried out using natural mineral water to ensure the presence of competing ions in solution (The properties of natural mineral water are listed in Table 1 as supplied by Supermont Company, Muyuka, Cameroon). Initial pH values of the working solutions were adjusted with HCl and NaOH to the desired values.

Hydroxy-sodalite

Hydroxy-sodalite used in this experiment was synthesized from fine fractions of sandy clay loam soil (SCL) with low organic matter content collected from Ikasa village, Ndian Division, South West Region of Cameroon (N04° 58.321' E008° 47.970') by alkaline fusion followed by hydrothermal synthesis method as reported by Nanganoa et al. [10]. The particle size distribution of SCL was carried out by the pipette method and the chemical analysis by ICP-AES (Inductive Coupled Plasma-Atomic Emission Spectrometry). The results have been reported elsewhere [11]. 10 g of SCL raw material was mixed with 12 g of NaOH powder. This mixture was heated in a ceramic crucible in a muffle furnace at 600 °C for 1 h. The resultant material was cooled in a desiccator and ground to obtain fused material. 93.6 mL of distilled water and 0.93 g $\text{Al}(\text{OH})_3$ were added to the fused materials in a Teflon bottle. This was followed by a 24 h aging process which involves agitation on a reciprocal shaker at room temperature to obtain the aged material. The aged material was then heated in an air oven at 90 °C for 24 h in order to obtain the product. The product was filtered and washed several times with distilled water until the filtrate had $\text{pH} \leq 10$. The product was then dried in an oven at 105

°C overnight. The synthesized hydroxy-sodalite synthesized was denoted as HS. Cation exchange capacity (CEC) of zeolitic products was determined using sodium acetate procedure described by Chapman [12], zeolitic phase (s) in the solid products were identified by X-ray diffraction using a Rigaku D/Max 2500 instrument, operating on the Cu- α radiation and 2θ scanning ranging from 5° to 80° with a scanning step of 0.02° . Morphology of SCL and the end products were examined by scanning electron microscope (SEM) using an S-4700 (Hitachi) instrument. Surface area and porosimetry measurements of the zeolitic product were obtained on a Micrometrics Tri-Star surface area and porosity analyzer at 77 K by nitrogen isotherms according to BET method. The cation exchange capacity of HS was found to be 203.06 meq/100 g. The BET surface area and the total pore volume were $46.23 \text{ m}^2/\text{g}$ and $0.21 \text{ cm}^3/\text{g}$ respectively. The XRD pattern and SEM micrograph of the HS are shown in Figures 1 and 2, respectively.

Ammonium adsorption experiments

Batch adsorption experiments of ammonium adsorption onto the HS (Particles size less than $250 \mu\text{m}$) were carried out at ambient temperature (24°C) to investigate different parameters such as: 1) the effect of contact time with time ranging from 5 to 240 min, and initial concentration of ammonium nitrogen kept at 50 mg/L; 2) pH by varying from 4 to 10 and employing ammonium nitrogen concentration of 50 mg/L at equilibrium time; 3) Initial ammonium nitrogen concentrations by varying it from 25 to 125 mg/L at equilibrium time; 4) adsorbent dosages as increased from 0.1 to 1 g in 50 mg/L ammonium nitrogen solution at equilibrium time; and 5) the presence of competing ions in solution to reflect the coexistence of NH_4^+ and other ions in a real water/wastewater using synthetic wastewater solutions prepared with natural mineral water. The ammonium nitrogen concentration interval was from 25 to 125 mg/L. In all the batch adsorption, the solutions containing 25 mL adsorbate and 0.5 g adsorbent (Unless otherwise specified) were taken at the desired pH in a 250 mL capacity stoppered flasks and, then, stirred at 250 rpm at the given time intervals. The solutions were filtered and the concentrations of the supernatant were also determined by titrimetric method [13]. Control experiments were also carried out by mixing adsorbent with distilled water/natural mineral water. All the experiments were carried out at least in duplicate and the arithmetic average value reported.

Ammonium adsorption capacities q_t and q_e at time t and equilibrium were respectively calculated based on the following equations.

$$q_t = \frac{(C_o - C_t)v}{m} \quad (1)$$

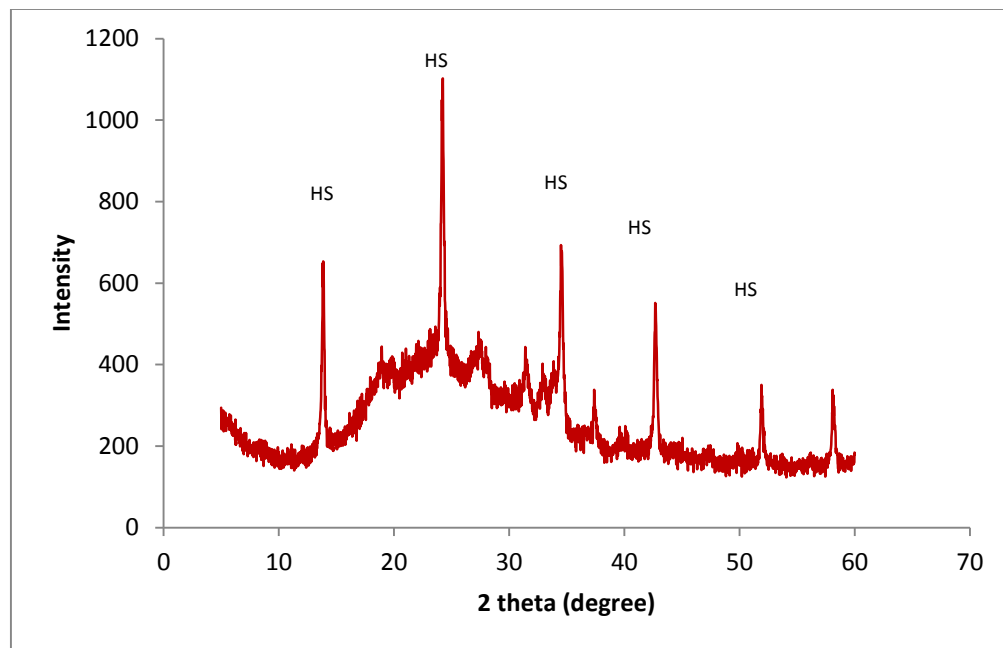


Figure 1. XRD pattern of HS

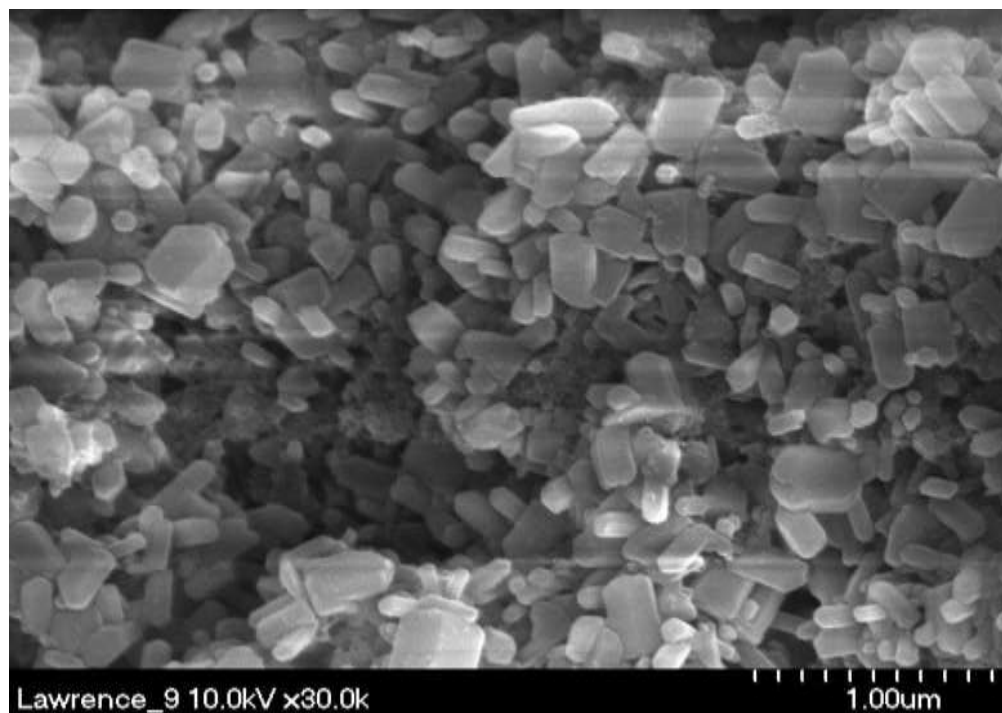


Figure 2. SEM image of HS

where C_o (mg/L) is the initial NH_4^+ concentration, C_t (mg/L) is the NH_4^+ concentrations at time t in the solution, v is the volume of aqueous solution (L) and m is the mass of adsorbent (g) used in the experiment.

Results and discussion

Ammonium adsorption studies

Effect of contact time

Figure 3 shows the ammonium uptake by the synthesized zeolite (HS) as a function of contact time at ambient temperature. The result revealed that the removal of ammonium by the HS was a very rapid process and ammonium uptake was almost completed within the first 10 min of the reaction process. Between 10 and 45 min of uptake, fluctuations could be observed and equilibrium was reached after 60 min. Previous studies have also shown that ammonium uptake by zeolite is a fast process, and uptake equilibrium is achieved at a shorter time periods of 10 and 15 min [14].

Effect of pH

The uptake of ammonium by HS was investigated at pH values ranging from 4 to 10. The result (Figure 4) showed that the ammonium removal by HS weakly increased with increasing the pH up to 7. Moreover, it was observed that the pH value around and below 7 is best suited for the adsorption process with the optimum removal being achieved at pH 7. The decrease in uptake at pH values higher than 8 might be due to the significant presence of NH_3 species as a result of the transformation of ammonium ions to ammonia [15]. The synthesized zeolite cannot remove NH_3 because of the fact that it is only the ionized form that can be removed from the solution by ion exchange/adsorption.

Effect of initial concentration

The result of ammonium ion removal by HS for different initial concentrations is shown in Figure 5. The higher the initial concentration of the ammonium ions, the higher amounts of ions are removed from the solution using HS. HS showed a better adsorption capacity as compared to the adsorption capacity of the raw material (SCL) for the different initial concentrations at pH 7 [11]. This can be attributed to the higher cation exchange capacity (CEC) of HS [16]. Franus et al. [17, 18] respectively, obtained a sorption capacity of ammonium ion on synthetic sodalite from flyash of 4.45 mg NH_4^+ /g at initial concentration of 500 mg/L.

Isotherm models

In finding a suitable isotherm model that will be best described as the adsorption process, the isotherm data were fitted to the six two-parameters isotherm models. Two linear forms of Langmuir model referred to as Langmuir I and II, Freundlich, Harkin-Jura, Halsey and Elovich isotherm models were used to describe the equilibrium isotherm data [19]. The fit between the experimental data and

the linearized forms of the isotherm equations was also compared by judging the correlation coefficients (R^2) while the average percentage errors (APE) were calculated according to Eq. 3 indicating the fit between the experimental and predicted values of adsorption capacity.

$$APE(\%) = \frac{100}{N} \sum_{i=1}^N \frac{|q_{e,exp} - q_{e,th}|}{q_{e,exp}} \quad (3)$$

where $q_{e,exp}$ is the experimental value, $q_{e,th}$ is the predicted value, and N is the number of experimental data if data from the models are similar to the experimental data, APE (%) will be a small number [20, 21]. The corresponding correlation coefficients, average percentage error (APE (%)) and the isotherm constants are calculated and presented in Table 1.

The expression of the Langmuir isotherm model is as follows:

$$q_e = \frac{q_m K_L C_e}{1 + K_L C_e} \quad (4)$$

where q_e is the amount of solute adsorbed per unit mass of adsorbent (mg/g), C_e is the equilibrium concentration of adsorbate in solution after adsorption (mg/L). q_m is the maximum adsorption capacity (mg/g) and K_L is the Langmuir constant related to energy of the adsorption (L/mg) [19–23]. Two of the linear forms of Eq. 4 are written as:

$$\text{Langmuir I: } \frac{1}{q_e} = \frac{1}{q_m K_L C_e} + \frac{1}{q_m} \quad (5)$$

$$\text{Langmuir II: } \frac{C_e}{q_e} = \frac{C_e}{q_m} + \frac{1}{q_m K_L} \quad (6)$$

The essential characteristics of a Langmuir isotherm can be expressed in terms of a dimensionless constant separation factor or equilibrium parameter, R_L , which is defined as:

$$R_L = \frac{1}{1 + K C_o} \quad (7)$$

This value indicates the shape of the isotherm to be either unfavorable ($R_L > 1$), linear ($R_L = 1$), favorable ($0 < R_L < 1$), or irreversible ($R_L = 0$) [24].

The freundlich isotherm is represented by the equation:

$$q_e = K_f C_e^{1/n} \quad (8)$$

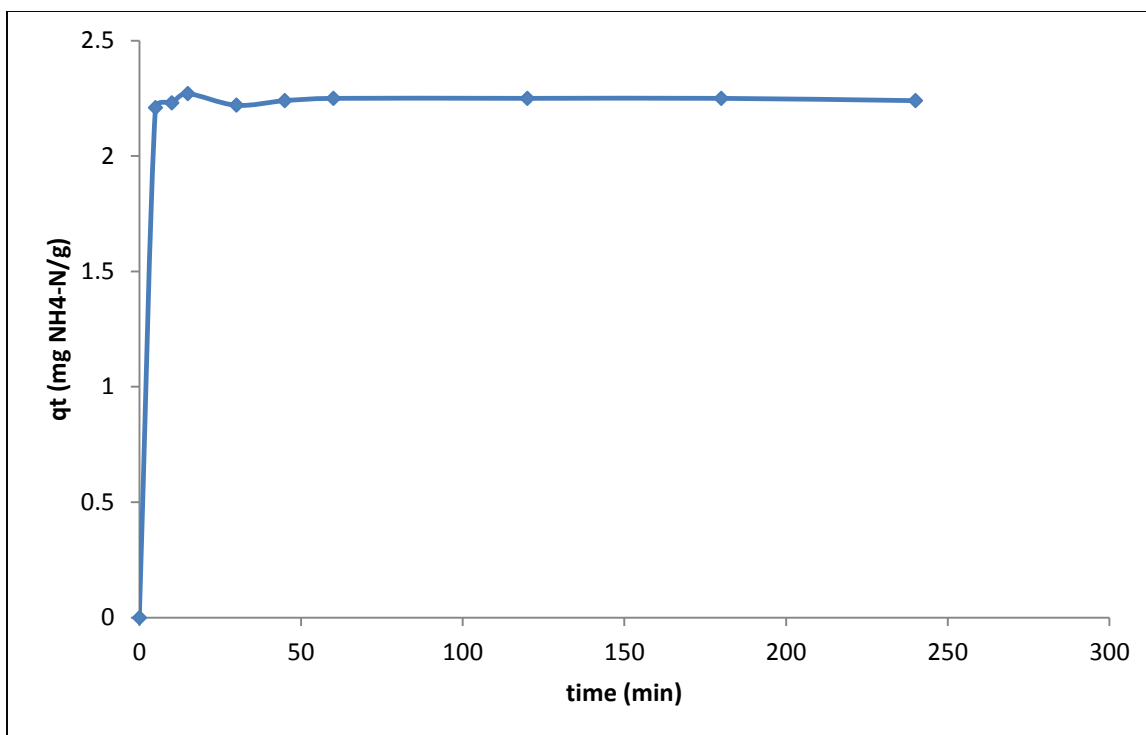


Figure 3. Effect of contact time on ammonium ion adsorption on HS at pH 6.8, initial ammonium concentration; 50 mg NH₄⁺/L

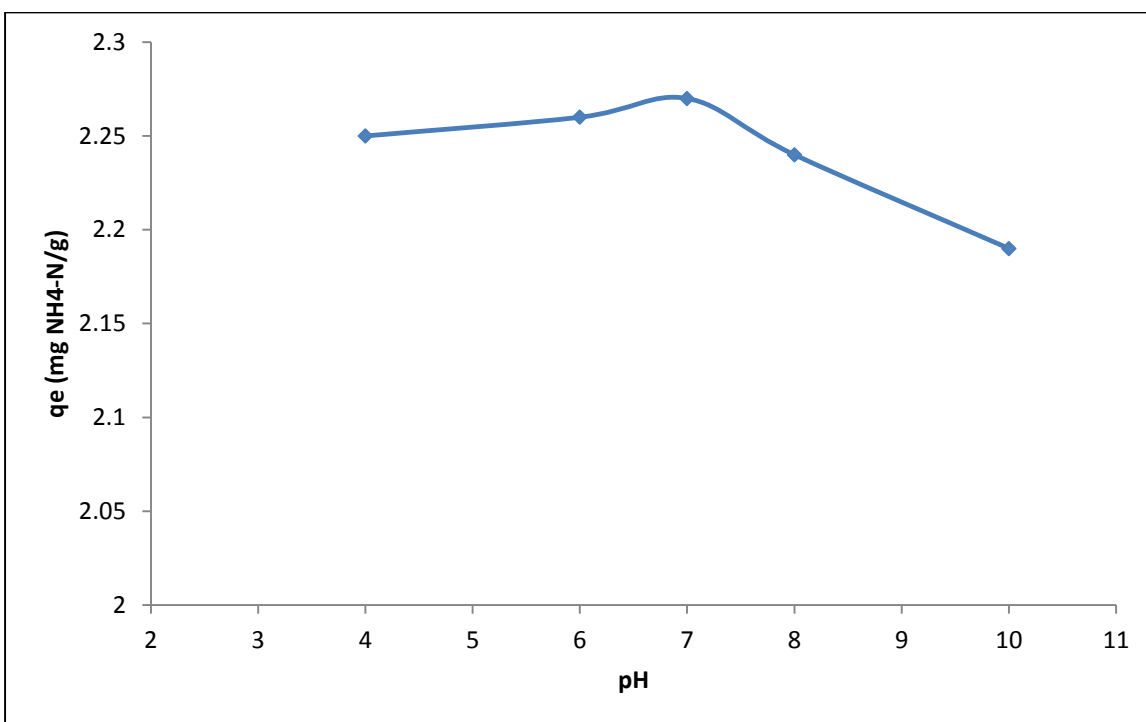


Figure 4. Effect of pH on ammonium adsorption on HS

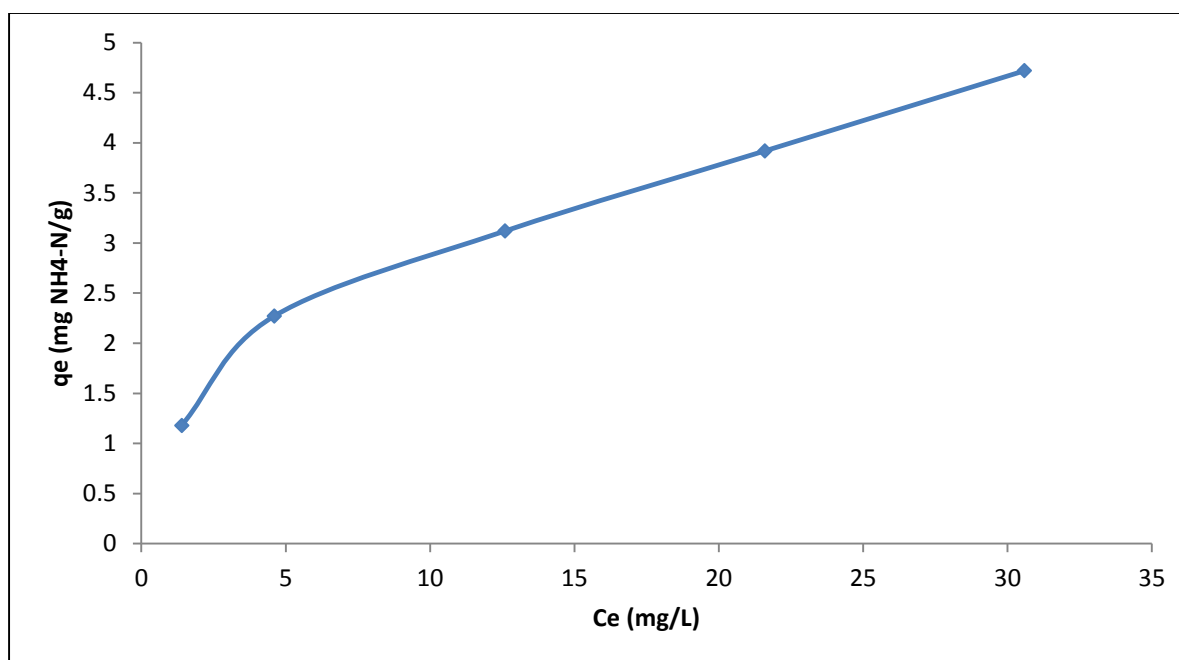


Figure 5. Effect of initial concentration on ammonium ion adsorption on HS

Table 1. Isotherm model constants for ammonium adsorption on HS

Isotherm	Parameter
Langmuir I	ZHS 2
qm (mg/g)	4.51
KL (L/g)	0.25
R ²	0.988
APE	7.04
Langmuir II	
qm (mg/g)	5.46
K _L (L/g)	0.15
R ²	0.972
APE	9.91
Freundlich	
K _F (mg/g)(L/mg) ^{1/n}	1.07
n	2.31
R ²	0.990
APE	3.95

Harkin-Jura	
A	2.10
B	1.42
R ²	0.846
APE	20.07
Halsey	
K _H	0.86
n _H	-2.31
R ²	0.990
APE	74.68
Elovich	
q _m (mg/g)	1.97
K _E	1.21
R ²	0.964

This model suggested that the sorption process involved heterogeneous sorption with different classes of adsorption sites where: q_e (mg/g) is the quantity of solute adsorbed at equilibrium, C_e is the concentration of adsorbate at equilibrium (mg/L). K_f and n are Freundlich constants related to adsorption capacity and adsorption intensity of the adsorbent respectively. The linear form is written as:

$$\ln q_e = \ln K_f + \frac{1}{n} \ln C_e \quad (9)$$

The magnitude of the exponent 'n' gives the indication of favourable condition of adsorption with values of $1 < n < 10$ [25, 26].

The Harkin-Jura adsorption isotherm is expressed as:

$$q_e = \sqrt{\left(\frac{A_H}{B_H + \log C_e}\right)} \quad (10)$$

Where A_H and B_H are the isotherm constants. The isotherm equation accounts for multilayer adsorption and can be explained by the existence of a heterogeneous pore distribution [27]. The linear form is given in Eq. 11:

$$\frac{1}{q_e^2} = \left(\frac{B_H}{A_H} \right) - \frac{1}{A_H} \log C_e \quad (11)$$

The Harkin-Jura isotherm parameters are obtained from the plot of $1/q_e^2$ versus $\log C_e$.

The Halsey adsorption isotherm model can be expressed as:

$$q_e = n \sqrt[n]{\left(\frac{K_H}{C_e} \right)} \quad (12)$$

where K_H and n are the Halsey isotherm constant and exponent respectively. The linearized form is given by:

$$\ln q_e = \frac{1}{n} \ln K_H - \frac{1}{n} \ln C_e \quad (13)$$

This equation is suitable for multilayer adsorption and the isotherm parameters are obtained by the graph of $\ln q_e$ versus $\ln C_e$ [20].

The Elovich model is based on a kinetic principle assuming that the adsorption sites increase exponentially with adsorption, which implies a multilayer adsorption [28]. It is expressed by the relation:

$$\frac{q_e}{q_m} = K_E C_e \exp\left(-\frac{q_e}{q_m}\right) \quad (14)$$

where K_E is the Elovich equilibrium constant (L/mg) and q_m is the Elovich maximum adsorption capacity (mg/g). K_E and q_m values and the correlation coefficient are obtained from the linear form of the Elovich equation (Eq. 15) by plotting $\ln q_e/C_e$ versus q_e/q_m .

$$\ln \frac{q_e}{C_e} = \ln K_E q_m - \frac{q_e}{q_m} \quad (15)$$

The Freundlich adsorption model gave the best fit ($R^2=0.990$ and APE (%)=3.95) as compared to Langmuir I and II, Harkin-Jura, Halsey and Elovich (Table 1). A better fit for Freundlich model was also obtained when ammonium ion was adsorbed by synthetic zeolites Na-X, Na-P and Sodalite synthesized from fly ash [17] and Chinese natural clinoptilolite and modified zeolite [29].

The values of R_L for NH_4^+ removal by ZHS are shown in Figure 6. The R_L values were found to vary within the ranges, 0.138–0.031 and 0.215–0.052 for Langmuir I and II, respectively, the initial NH_4^+ concentration values being 25–125 mg/L. All values are in the range of 0–1 which indicates a favorable adsorption. The n value from Freundlich model is greater than 1, which also represented

favourable adsorption ($1 < n < 10$) [30]. These proved that HS is a potential adsorbent for NH_4^+ ion from aqueous solutions.

Table 2. Chemical properties of Natural mineral water

pH	Ca^{2+} (mg/L)	Mg^{2+} (mg/L)	K^+ (mg/L)	HCO_3^- (mg/L)	Cl^- (mg/L)
7.1	30	5.9	3.8	134	1.3

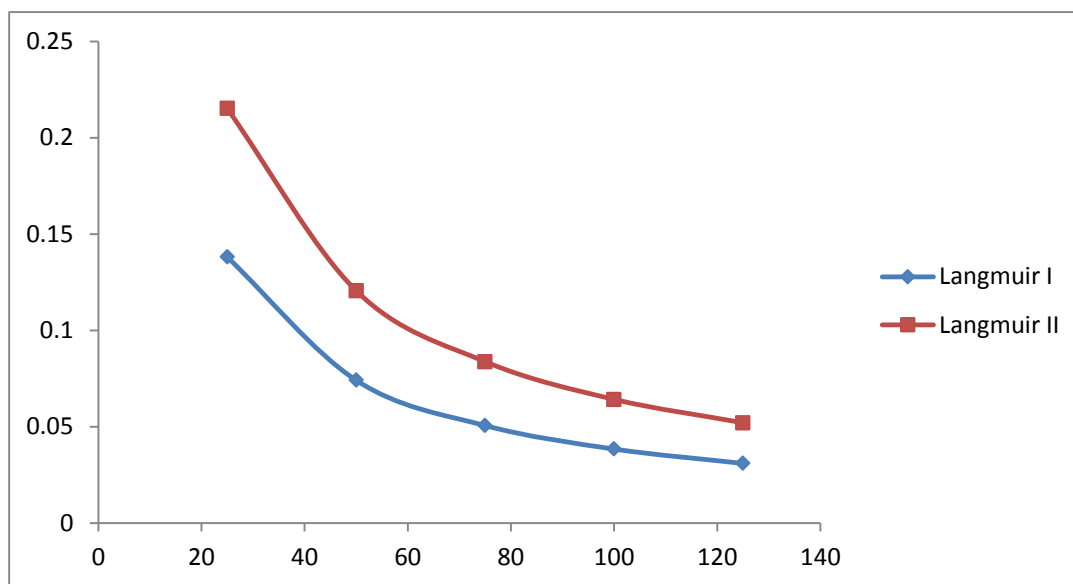


Figure 6. Variation of separation factor with initial ammonium ion concentration of HS

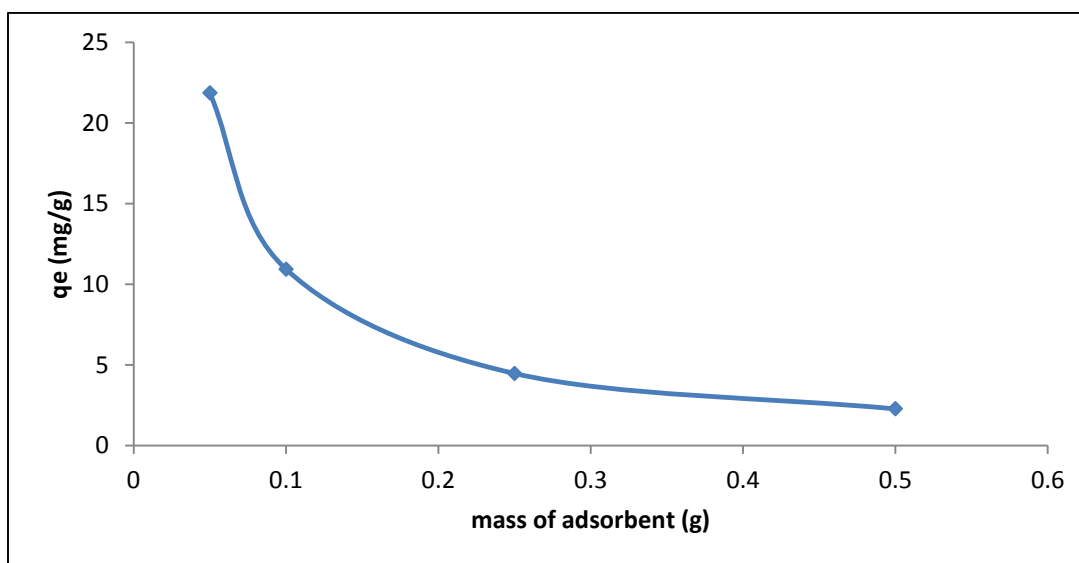


Figure 7. Effect of HS dose on uptake of ammonium ion

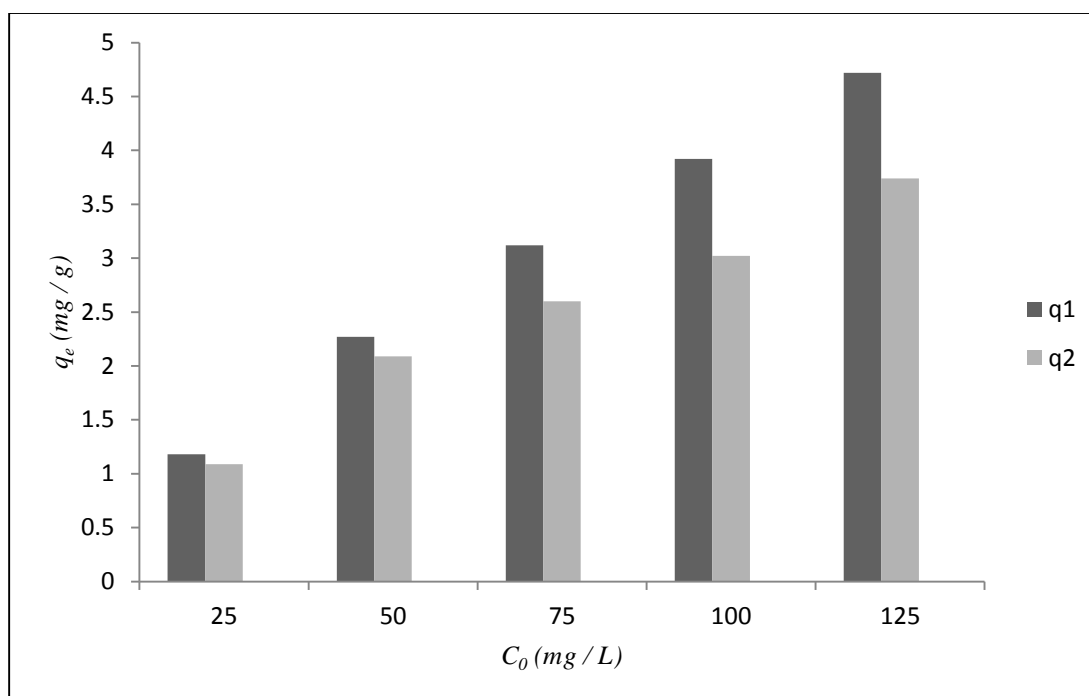


Figure 8. Comparison of ammonium ions uptake on HS with and without the presence of competing ions (q_1 : adsorption capacities without the presence of competing ions; q_2 : adsorption capacities with the presence of competing ions)

Effect of adsorbent dose

The influence of the mass of HS (0.05–0.5 g/25 mL) on the removal of ammonium ion at constant initial ammonium concentration (50 mg/L), contact time (60 min) and pH=7 is shown in Figure 7. The result showed that the amount of adsorbed ammonium decreased with increasing HS dose. The decrease in uptake capacity may be due to the fact that as the amount in grams of adsorbent is increased the total surface area available for the adsorption of ammonium ion reduces as a result of overlapping or aggregation of adsorption sites. Therefore, less ammonium ions diffused through the liquid film and intraparticle channels to the exchangeable sites of the synthesized zeolite, and more sites remained unexchanged [31, 32].

Effect of the presence of competing ions

To establish the extent to which the presence of other ions influence ammonium ion uptake, further equilibration experiments were conducted with mineral water (Properties listed in Table 2). From the results we noticed that in each initial concentration, there is a significant reduction in the equilibrium uptake of ammonium ions in the presence of the other ions (Figure 8). Thornton et al., also reported a decrease in adsorption capacity of ammonium ion onto mesolite when competing ions were present in solution [15].

Kinetic model

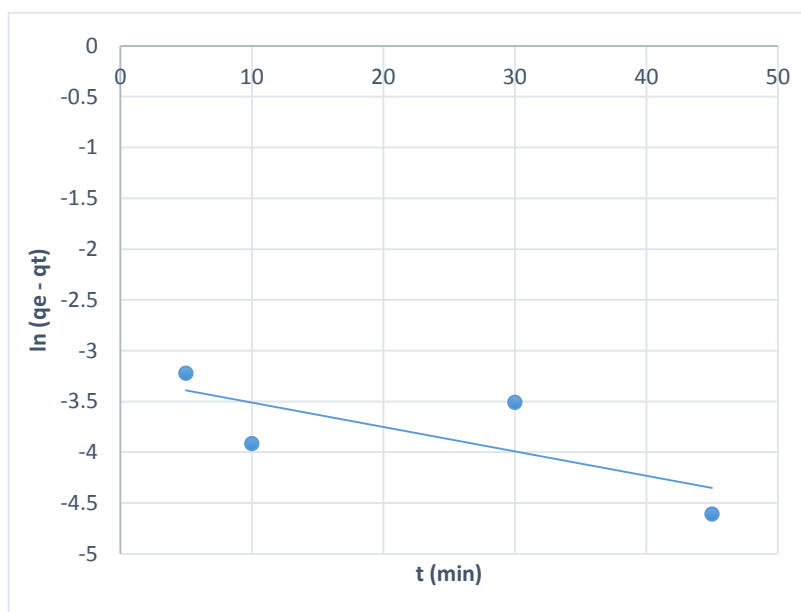
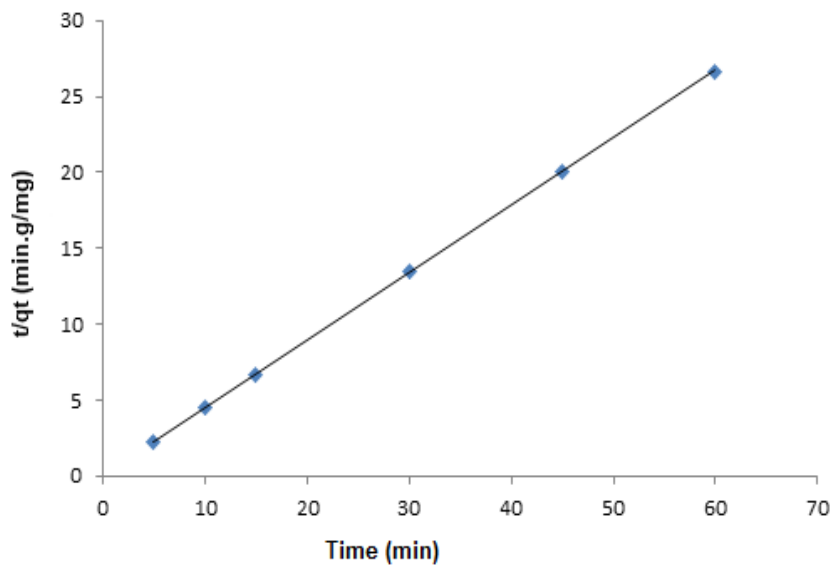
In the present research, the kinetics of ammonium ion uptake on the HS was studied. Four types of kinetic models, pseudo-first order, pseudo-second order model, Elovich and the intra particle diffusion models, were employed to fit experimental data. The correlation coefficients (R^2) values and comparison of experimental and theoretical adsorption capacity q_e were also used to determine the fitness of the kinetic models to the adsorption systems [23–33]. The mathematical equations of these models are illustrated in Table 3, where q_e and q_t are the adsorption capacity at equilibrium and at time t respectively (mg/g), K_1 and K_2 are the rate constant of pseudo-first order adsorption (min^{-1}) and pseudo second order adsorption ($\text{mg}^{-1} \text{min}^{-1}$), respectively while K_{id} is the intraparticle diffusion rate constant ($\text{mg/g min}^{1/2}$). α is the initial sorption rate ($\text{mg/g}^{-1} \text{min}$) and β is the desorption rate constant (g/mg^{-1}) during any one experiment of the Elovich kinetic model. The parameters of the aforementioned kinetic models are given in Table 4. Values of K_1 and q_e of pseudo-first order model were obtained from the linear plot of $\ln(q_e - q_t)$ against t (Figure 9), K_2 and q_e of pseudo-second-order model from the straight-line plots of t/q_t against t (Figure 10). α and β of Elovich kinetic model were obtained from q_t versus $\ln t$ (Figure 11) and k_{id} of intra-particle diffusion model from the plot of q_t versus $t^{1/2}$ (Figure 12). Based on the difference between the experimental equilibrium adsorption capacity (q_e, exp) and calculated equilibrium adsorption capacity (q_e, th), as well as the correlation coefficient R^2 , it is clearly observed that adsorption of ammonium ions onto HS can be satisfactorily described by the pseudo-second order equation. The results in Table 4 show that the correlation coefficient ($R^2=0.9999$) for the pseudo-second order model was much higher than those obtained using the Lagergren first-order model ($R^2=0.5484$), Elovich ($R^2=0.1723$) and the intraparticle diffusion model ($R^2=0.1283$). Thus, the pseudo-second order model explains the kinetic process best, indicating a two-step process of ammonium adsorption by HS.

Table 3. kinetic models and their linear forms

Model	Equation	Linear form
Pseudo-first order	$\frac{dq_t}{dt} = k_1(q_e - q_t)$	$\ln(q_e - q_t) = \ln q_e - K_1 t$
Pseudo-second order	$\frac{dq_t}{dt} = K_2(q_e - q_t)^2$	$\frac{t}{q_t} = \frac{1}{K_2 q_e^2} + \frac{t}{q_e}$
Elovich	$\frac{dq_t}{dt} = \alpha \exp(-\beta q_t)$	$q_t = \frac{1}{\beta} \ln(\alpha\beta) + \frac{1}{\beta} \ln t$
Intra-particle diffusion	/	$q_t = K_{id} t^{1/2} + C$

Table 4. Kinetic parameters and the calculated ($q_{e, th}$) and experimental ($q_{e, exp}$) of ammonium ion adsorption on HS

q_{exp}		Pseudo-first-order			pseudo-second-order		
		k_1	R^2	$q_{e, th}$	k_2	R^2	$q_{e, th}$
HS	2.25	0.02	0.548	0.04	5.79	0.999	2.25
			Elovich			Intraparticle diffusion	
		α	β	R^2	k_{id}	R^2	
HS		1.02E+100	106.38	0.172	0.004	0.128	

Figure 9. Pseudo-first-order adsorption kinetic model of ammonium ions on HS**Figure 10.** Pseudo-second-order adsorption kinetic model of ammonium ions on HS

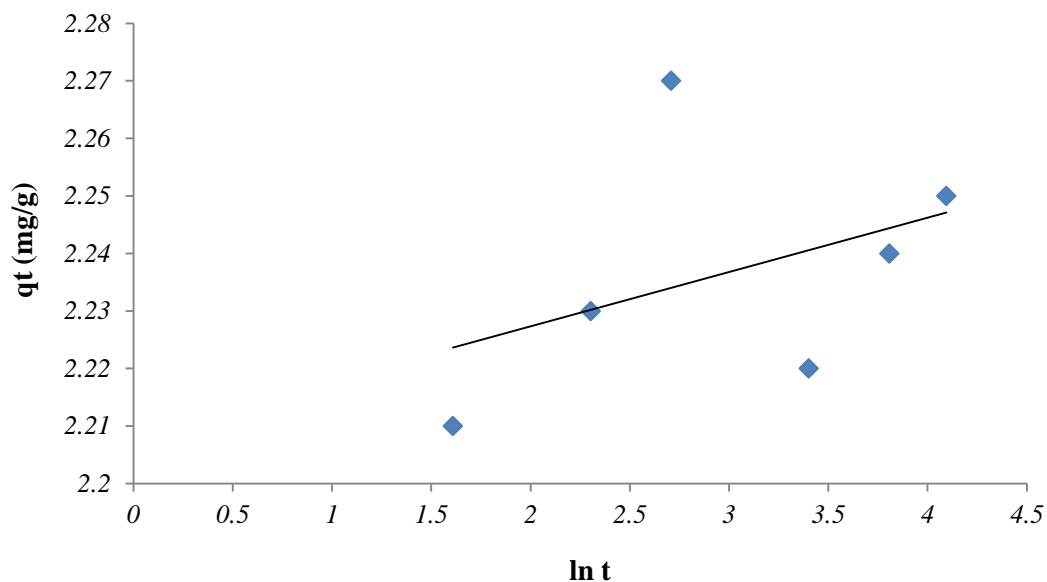


Figure 11. Elovich adsorption kinetic model of ammonium ions on HS

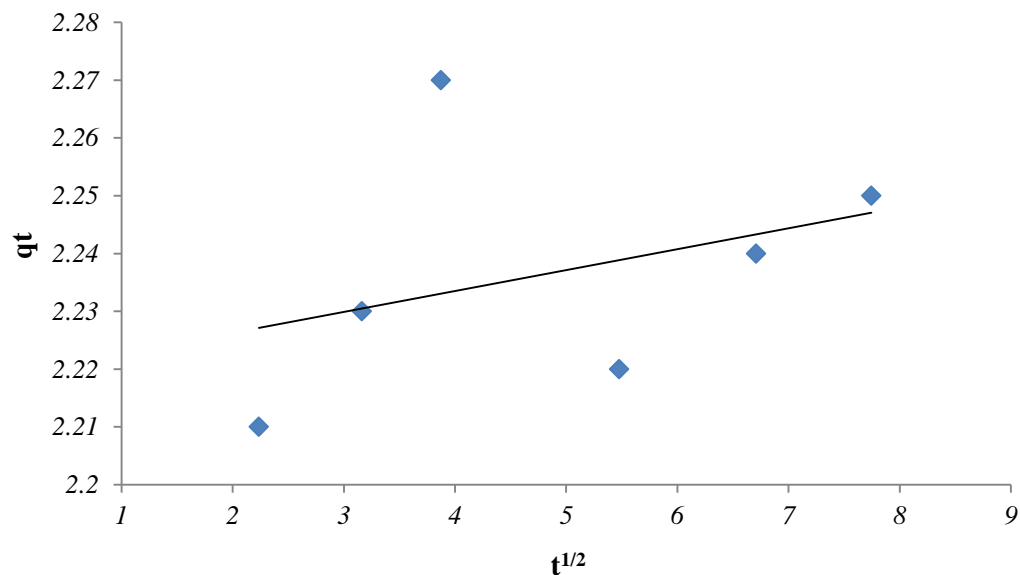


Figure 12. Intra-particle diffusion model of ammonium adsorption on HS

Conclusion

The presented research showed that the HS can be used to remove the ammonium ion from aqueous solution. The amount of ammonium ions removed from solution increased with increasing the initial ammonium concentration. Maximum ammonium uptake occurred at pH 7. The best fit of the isotherm models used was obtained by Freundlich isotherm model. The values of the separation

factor R_L from Langmuir model and adsorption intensity n from the Freundlich model suggested that the removal of ammonium by HS was favourable. Kinetic studies indicated that ammonium uptake by HS is best described by the pseudo-second order model and the effect of the presence of competing ions in solution significantly affected the uptake of ammonium. This material (HS) has potentials in the adsorption of ammonium ions from wastewater.

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Disclosure statement

No potential conflict of interest was reported by the authors.

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