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Research Article

An efficient adsorptive removal of nitrate from agricultural effluent by alkaline modified sepiolite clay using response surface approach

Emmanuel Amuntse Yerima[1], Shadrack John Aboki[1], Comfort Fatuma Bajo[2], Andeshongtso Blossom Shamaki[1], Ibrahim Adamu Ibrahim[1]*

[1]*Department of Chemical Sciences, Federal University Wukari, PMB 1020, Taraba State, Nigeria*
[2]*Department of Chemistry, Federal College of Education, Zaria-Nigeria*

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ABSTRACT

To tackle the issues related to the discharge of contaminants like nitrate from agricultural runoff, this study utilized alkaline Modified Sepiolite clay (MS) as an adsorbent for the elimination of nitrate from agricultural effluent. The optimal conditions for examining the effects of pH, temperature, dosage, and contact time on the removal process were assessed within the following ranges: pH 4-11, temperature 25-60°C, time 30-120 minutes, and adsorbent dosage 0.2-4 g, using a 100 mL adsorbate solution analyzed with Design Expert (version 13). Various characterization techniques, including Fourier Transform Infrared Spectroscopy (FTIR), Scanning Electron Microscopy (SEM), and Transmission Electron Microscopy (TEM), were used to examine the MS before and after the adsorption process. At equilibrium, the maximum percentage of nitrate removal reached 95%, which was accomplished under three specific conditions: pH = 11, time = 75 minutes, temperature = 25°C, and MS dosage = 2.1 g. The mechanism of nitrate removal by MS aligned most closely with the Blanchard pseudo-second-order kinetic model ($R^2 = 0.9253$), while the adsorption isotherm was best represented by the Langmuir Isotherm ($R^2 = 0.9944$).

INTRODUCTION

Agricultural activities are essential for food production, but they may have high effect on environmental hazards, water pollution through the release of agricultural effluents. One of the major concerns is the contamination of natural water resources through the discharge of agricultural effluents into water bodies [1]. These effluents often contain various pollutants, including compounds like nitrates (the most common nitrogen compounds in fertilizers), nitrites and phosphates which can have detrimental effects on both human health and aquatic ecosystems. The key sources of nitrates in the superficial and ground waters are also from wastewaters from agriculture, industry, and households contain various pollutants. Research has shown that one of these pollutants, particularly nitrate an essential nutrient for plant growth contributes significantly to a serious environmental issue known as eutrophication [4, 5]. Elevated amount of nitrates intake can result in health complications, like blue-baby syndrome in kids and abdominal tumor in adults [6]. Therefore, to manage nitrate in water, numerous removal techniques have been developed among which includes: adsorption, chemical de-nitrification, biological de-nitrification, ion exchange, electrodialysis and reverse osmosis [7]. However, the adsorption is well-thought-out as the most economical because of its simplicity in nature and efficiency [8, 9]. Recently, sepiolite clay an opaque combination of hydrous magnesium silicate, exhibiting off-white, grey, or creamy colour ($Mg_4Si_6O_{15}(OH)_2 \cdot 6H_2O$) finds utility as adsorbent for a number of contaminants in water systems [10, 11].

Sepiolite clay contains a silanol groups (-SiOH) which is accounts for its hydrophilicity, a high surface area and porosity responsible for its high sorptive property [12]. This study targets to evaluate the adsorption efficiency and capacity of nitrate from agricultural effluents by alkaline modified sepiolite clay as well as determine the optimal condition. Thereby; present an environmentally friendly and economically feasible solution to water pollution. This will help mitigate problems associated with water bodies, like depletion of dissolved oxygen levels, unpleasant odour and eutrophication in water.

MATERIALS AND METHOD

Sample Collection

The adsorbent, non-modified sepiolite clay (NMS) was acquired from the Central Drug House (P) Ltd. 7/28 Vardaan, Daryaganj, New Delhi-110002 (INDIA). The Agrarian effluents were gotten from Wukari, Taraba State 6(7°50′57.6′′ N, 9°49′6.5′′E) through simple random sampling adopted by Yerima et al [13]. The water samples were processed and stored in ice coolers before being conveyed to the laboratory for subsequent evaluations [14].

Adsorbent Preparation and Modification

The sepiolite powder was treated with a 0.1M NaOH solution in the ratio of (1g/100 mL) while being vigorously stirred at 90°C for 3 hours. After treatment, the alkali-modified sepiolite samples were washed five times by centrifugation using distilled water, then dried at 50°C and was labeled as modified sepiolite (MS) [15].

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* ***Corresponding Author:***
Email: yerimaemmanuel@yahoo.com (*M. A. Yarima*)

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Experimental Design

The experiment was designed by means of design expert, file version 13.0, adopting response surface, design types: box-behnken and quadratic design model. A total of 29 experiment runs was recommended by the design expert to be carried out within the varying adsorption time of 30 - 120 minutes, pH 3 – 11; adsorbent dosage 0.2 – 4 and temperature 25 – 60°C as shown in Table 1.

Table 1: Experimental Design

Exp. Runs	Temp (°C)	Dosage (g)	pH	Time (min)
1	60	2.1	11	75
2	42.5	0.2	7	120
3	60	0.2	7	75
4	42.5	2.1	7	75
5	25	2.1	3	75
6	42.5	0.2	11	75
7	25	2.1	7	120
8	25	2.1	11	75
9	42.5	0.2	7	30
10	60	4	7	75
11	42.5	2.1	11	120
12	42.5	2.1	7	75
13	42.5	2.1	7	75
14	42.5	4	7	30
15	42.5	4	11	75
16	42.5	2.1	11	30
17	25	4	7	75
18	25	0.2	7	75
19	42.5	2.1	3	120
20	42.5	4	7	120
21	42.5	0.2	3	75
22	42.5	2.1	7	75
23	60	2.1	7	120
24	42.5	4	3	75
25	42.5	2.1	3	30
26	60	2.1	7	30
27	60	2.1	3	75
28	25	2.1	7	30
29	42.5	2.1	7	75

Batch Adsorptive Removal of Nitrate from Agricultural Effluent

Mechanical shaker, weighing balance, water bath, pH meter, beakers, conical flask, filter paper, HCl (0.1M), NaOH (0.1M) for pH adjustment, plastic funnels and measuring cylinder were employed. The initial nitrate content of the agricultural effluent was analyzed by the Devarda's technique and was noted as C_o.

Agricultural effluent (100 mL) was measured into a 250 mL conical flask followed by the addition of 2.1 g of the MS (adsorbent). The solution was mixed by means of stirring rod, and its pH set at 11 with the aid of HCl (0.1M), NaOH (0.1M) and pH meter respectively. The mixture was placed on water bath (Clifton 98006 model) set at either 60°C and conditioned at the prevailing temperature before shaking for 75 minutes. The same procedure was replicated for the other 28 experiments based on the conditions recommended by the design expert displayed in Table 3 and the sample containers properly labelled following the design. The residual concentration of nitrate in the 29 filtrates were determine using Devarda's method while the combine adsorbent residue of the 29 filtrates after adsorption was labeled modified sepiolite after adsorption (MSAA).

Determination of Nitrate in Water

To a water sample of 50 mL volume in a distillation flask, 0.20 g of magnesium oxide was added. The contents were distilled into 10 mL of 2 % boric acid solution till a volume of 20 mL distillate was obtained and was titrated with 0.025 N H_2SO_4. The titre value as against ammonium was recorded. 0.20g of Devarda alloy was added into the flask and heated gently to convert nitrate to ammonia, after enough distillate for ammonium was received; 20 mL distillate was transferred into another 10 mL volume of 2 % boric acid and titrated with 0.025 N H_2SO_4 and the titre value recorded for nitrate [16].

$$3NO_3^- + 8Al + 5\,OH^- + 18\,H_2O \rightarrow 3\,NH_3 + 8[Al(OH)_4]^- \qquad \text{I}$$

$$\%NH_3 = \frac{0.014 \times \text{Normality of acid} \times 100 \times \text{titre}}{\text{Aliquot taken}} \qquad \text{II}$$

Data Analysis

The adsorption capacity (qe) and the efficiency of adsorptive removal (Re) were determined by calculating the difference between the initial and equilibrium content of nitrate, as described in equations III and IV. Here, Co represents the initial content of nitrate, Ce denotes the equilibrium content of nitrate, M is the mass of the MS (modified sepiolite), V is the volume of the solution, and Re indicates the adsorption efficiency [17].

The rate of nitrate adsorption removal was estimated by dividing the amount of nitrate adsorbed by the respective contact time shown in equation V.

$$\text{Adsorption capacity } (q_e) = \frac{C_o - C_e}{M} \times V \qquad \text{III}$$

$$\text{Removal efficiency } (R_e) = \frac{C_o - C_e}{C_o} \times 100 \qquad \text{IV}$$

$$\text{Rate of adsorption} = \frac{C_o - C_e}{t} \qquad \text{V}$$

The kinetic parameter for the adsorption of nitrate onto MS adsorbent were obtained from the respective means of contact time 25, 42.5 and 60 mins with the corresponding means of the amount of urea adsorbed $(q_m - q_t)$. The pseudo first and second order kinetic model were utilized as presented in equations VI and VII where K_1 and K_2 are rate constants at equilibrium [18, 19].

Pseudo-first order kinetic model:

$$\log(q_m - q_t) = \log q_m - \frac{k_1}{2.303}t \qquad \text{VI}$$

Pseudo-second order kinetic model:

$$\frac{t}{q_t} = \frac{1}{k_2 q_m^2} + \frac{1}{q_m}t \qquad \text{VII}$$

The constants K_1 and q_m can be determined by plotting $\log(q_m - q_t)$ against time (t), resulting in a slope of $\frac{k_1}{2.303}$ and an intercept of $\log(q_e)$ for the pseudo first order kinetic plot. Likewise, for the pseudo-second order model, the kinetic constant k_2 and theoretical q_m can be obtained from the plot of $(\frac{t}{q_t})$ versus (t).

For further analysis of the adsorption mechanism, the Langmuir and Freundlich isotherm models were utilized, as noted by Yerima et al. [19] and Egah et al. [20]. The Langmuir model is represented by the equation:

$$\text{Langmuir:} \quad \frac{C_e}{q_e} = \frac{1}{K_L q_m} + \frac{C_e}{q_m} \qquad \text{VIII}$$

A plot of C_e/q_e against C_e will yield a straight line with a slope of $1/q_m$ and an intercept of $1/(K_L q_m)$. The characteristics of the Langmuir isotherm can be described by means of a dimensionless constant called the separation factor, R_L, defined in the equation:

$$\text{Separation factor: } R_L = \frac{1}{(1 + K_L C_o)} \qquad \text{IX}$$

In the equation; K_L (L/mg) is the Langmuir constant for adsorbate-adsorbent interaction, and Co (mg/L) is the initial concentration of the adsorbate. If $R_L > 1$, the adsorption is unfavorable; if $R_L = 1$, the adsorption is linear; if R_L is between 0 and 1, the adsorption is favorable; and if $R_L = 0$, the process is irreversible.

$$\text{Freundlich: } \log q_e = \log K_F + \frac{1}{n}\log C_e \qquad \text{X}$$

A plot of $\log q_e$ versus $\log C_e$ gives a straight line with a slope = 1/n and intercept = $\log K_F$

RESULTS AND DISCUSSION

Characterization of Sepiolite Clay

The characterization of surface morphology of adsorbent was done by using SEM - JEOL, JSM 7600 F machine. SEM micrograph revealed interaction amid the adsorbate and adsorbent, indicating granules at the interface [21]. Figure 1 revealed the SEM result for non-modified sepiolite NMS at 200µm magnification, modified sepiolite MS and modified sepiolite after adsorption MSAA at 300µm and 400µm magnification. In the NMS, a large pore size is seen, in MS there is a reduction in the pore size due to the adhesion of OH^- and Na^+ to the adsorbent surface during the modification process and in MSAA there is hardly seen pore due to the adhesion of nitrate to the adsorbent during the adsorption process evidence by dense cylindrical crystals and mono-disperse speck size on the surface [22]. MS provides more reactive sites by increasing the surface-active site of the adsorbent and enhanced adsorption capacity.

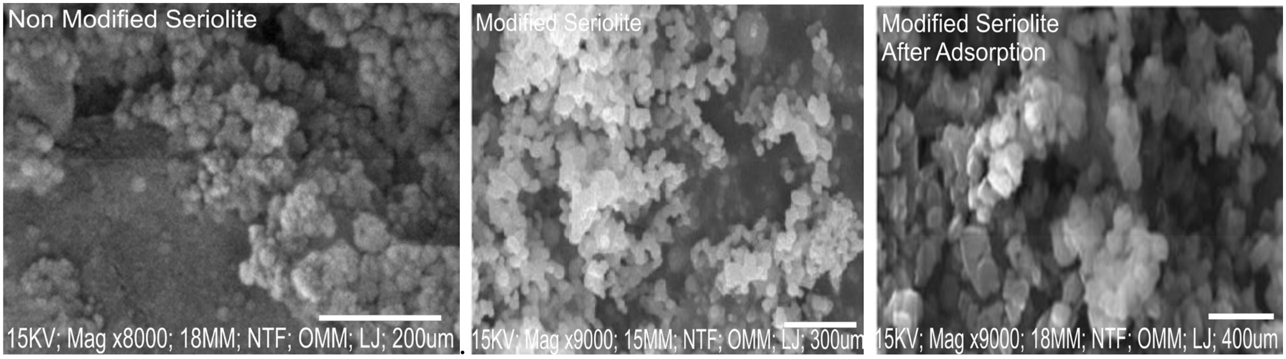


Figure 1: Scanning electron microscopy of sepiolite (un-modified, modified and after adsorption)

TEM generates image compare by utilizing the communication between the electron radiation and the sample. In the TEM image shown in Figure 2, denser regions that contain weightier elements look darker due to the scattering of electrons within the sample. The scattering of electrons from crystal planes results in diversion contrast due to orientation of the crystalline region in relation to the electron radiation. Hence, the high resolution offered by TEM, atomic orientation within crystalline structures can be observed in great detail, while areas devoid of sample along the beam path will appear bright.

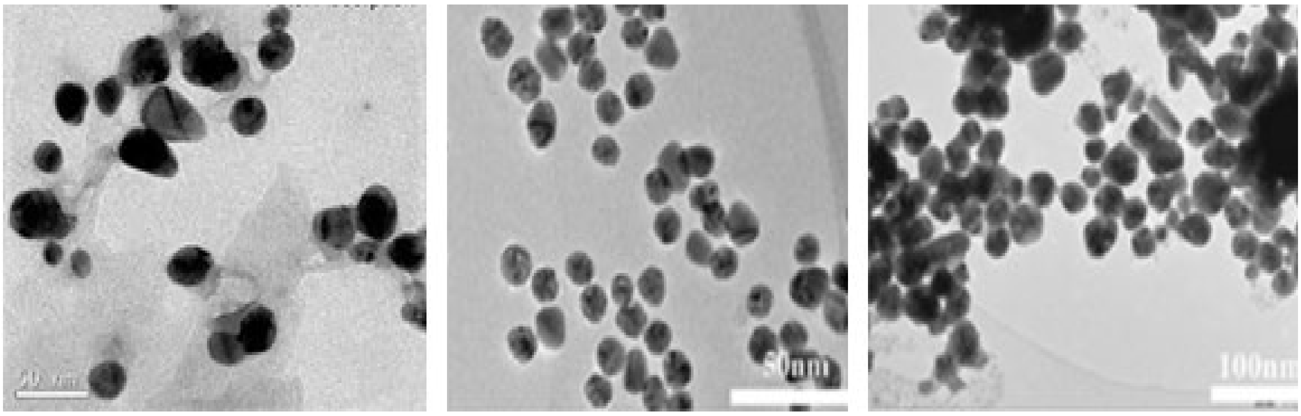
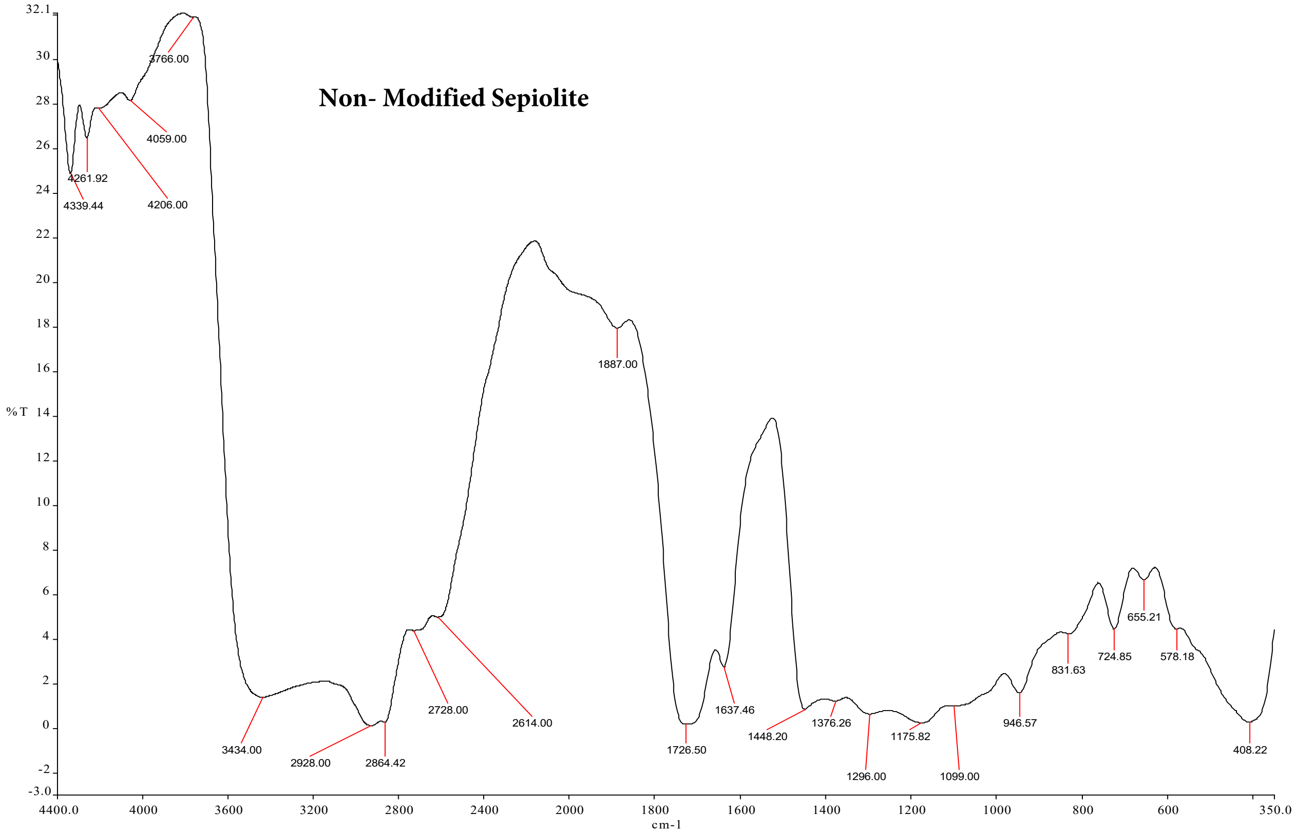
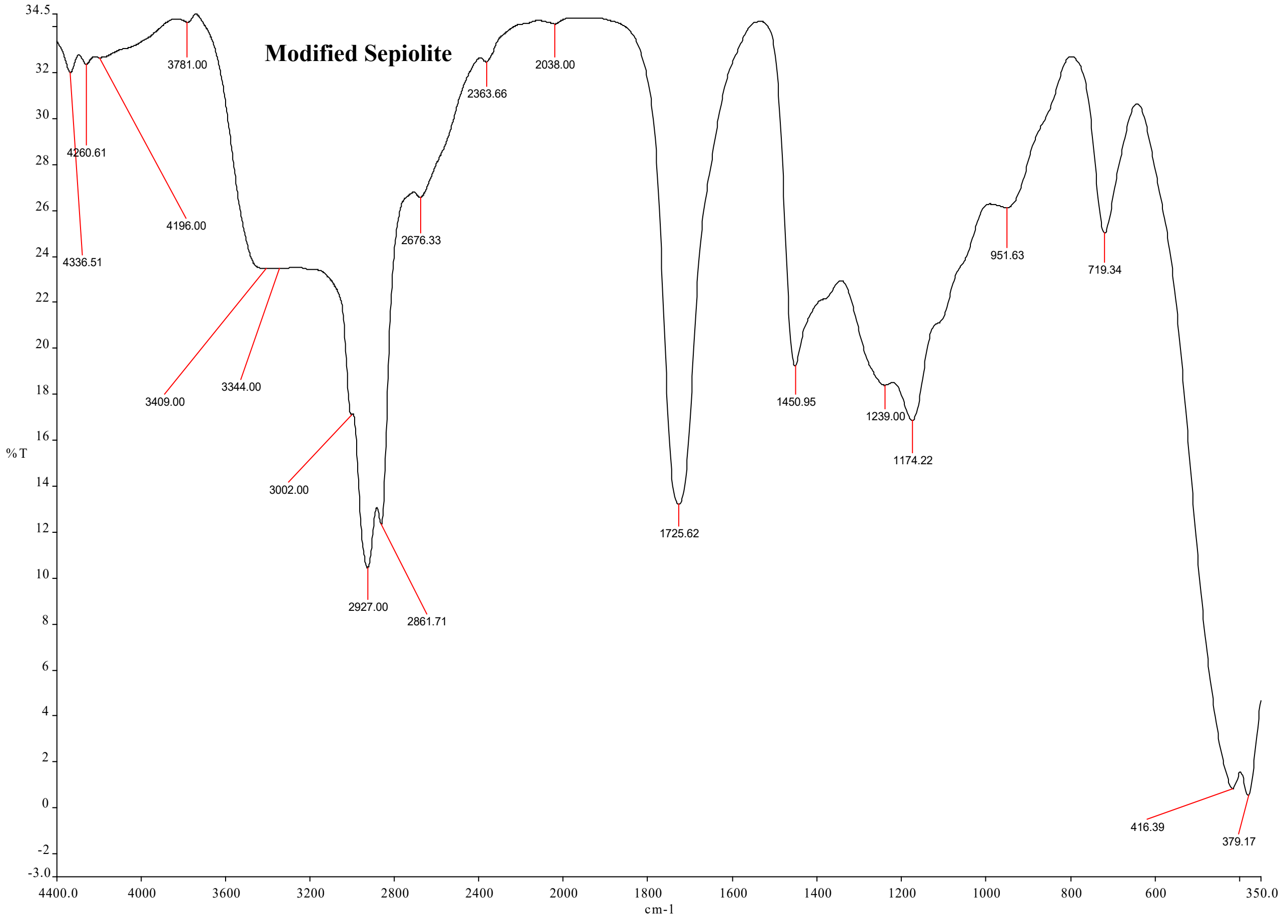


Figure 2: Transmission electron micrograph of non-modified, modified and after adsorption

The FTIR spectrum of sepiolite clay presented in Table 3 and Figure 4 shows the corresponding functional groups within the IR region. The absorbance showed peaks that certified the existence of O-H, C-H, N-H, S=O and Si-O-Si groups, among others which known to influence surface chemistry and aid adsorption of adsorbates [23, 24].


Non- Modified Sepiolite
%T
32.1
30
28
26
24
22
20
18
16
14
12
10
8
6
4
2
0
-2
-3.0
4400.0
4000
3600
3200
2800
2400
2000
1800
1600
1400
1200
1000
800
600
350.0
cm-1
3766.00
4059.00
4261.92
4339.44
4206.00
1887.00
655.21
831.63
724.85
578.18
2728.00
2614.00
1637.46
1376.26
946.57
3434.00
2928.00
2864.42
1726.50
1448.20
1175.82
408.22
1296.00
1099.00


Modified Sepiolite
%T
34.5
32
30
28
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600
350.0
cm-1
3781.00
2038.00
2363.66
4260.61
4196.00
2676.33
951.63
4336.51
719.34
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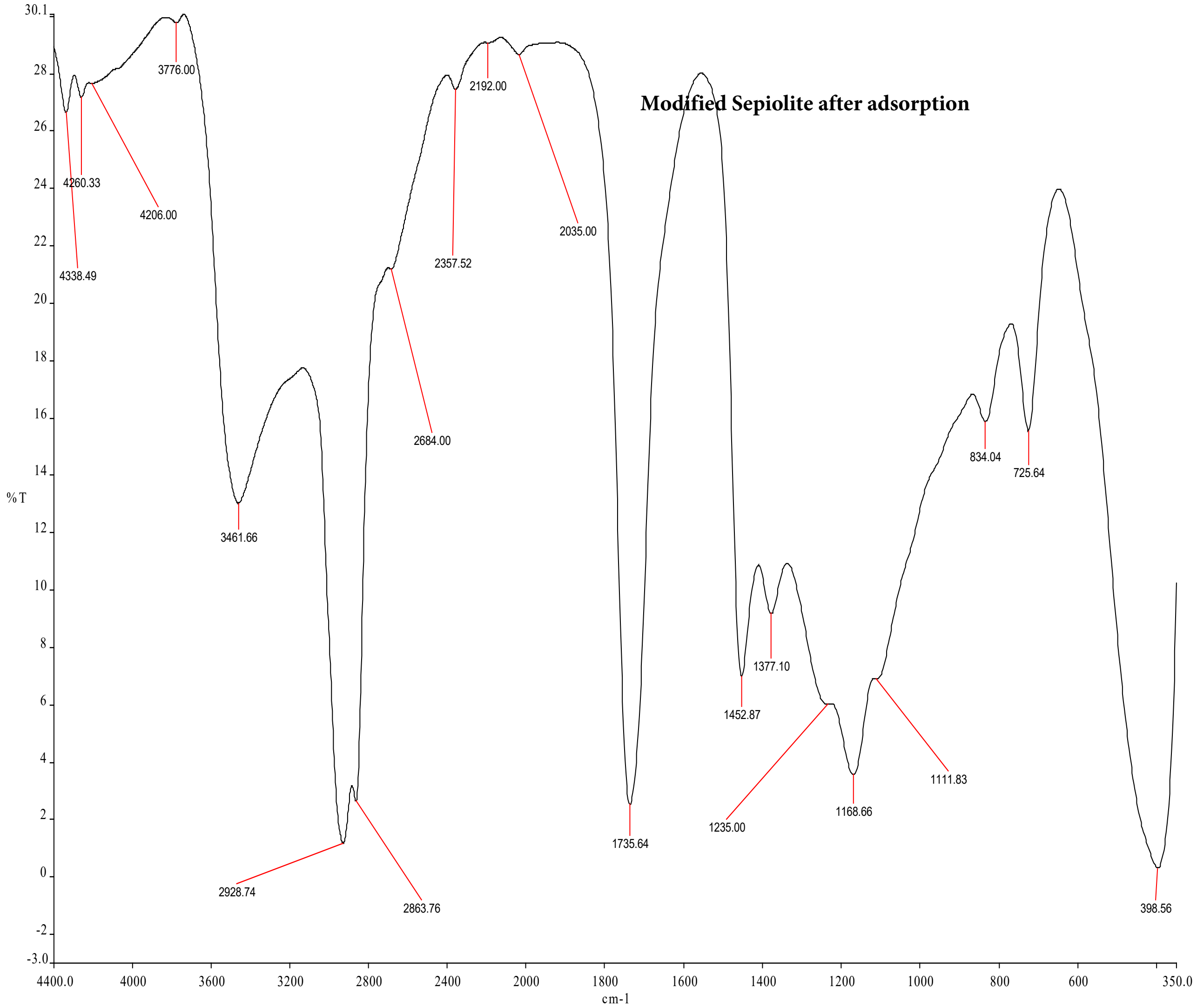

Figure 4: FT-IR Spectrum of Sepiolite (non-modified, modified and after adsorption)

Table 2: FT-IR Spectrum analysis

Frequency (cm^{-1})	Functional groups	NMS	MS	MSAA	Assignment
4000 – 3000	Alcohol	3766.00	3781.00	3776.00	Free O-H stretching
2999 – 1950	Aliphatic acid	292800	2927.00	2928.74	C-H bond symmetry stretching
1650 – 1580	Amine	1637.46			N-H bending vibration
1420 – 1330	Sulphate/sulfoxide	1376.26	1450.95	1377.10	S=O stretching
1200 – 1000	Silicate	1099.00	1174.22	1168.66	Si-O-Si stretching
950 – 905	Carboxylic acid	946.57	951.63		O-H bending
730 – 610	Alkyne	655.21	719.34	725.64	C-H bending
600 – 500	Halo-compounds	578.18			C-Cl stretching

NMS = non modified sepiolite, MS = modified sepiolite, MSAA = modified sepiolite after adsorption

Batch Adsorption Experiment

Table 3 indicates the rate of nitrate adsorbed from agricultural effluent in the 29 experiments recommended by the design expert at variable temperature, dosage, time and pH. The maximum amount of nitrate adsorbed was 36.75 mg/L with the corresponding adsorption capacity and removal percentage of 1750 mg/L and 95% respectively. The optimum removal of nitrate by the alkaline modified sepiolite clay was achieved at pH 11, adsorbent dose 2.1 g, contact time of 75 minutes, temperature of 25 °C and adsorption rate of 0.49 min^{-1}.

The corresponding removal percentage of 95% and adsorption capacity of 1750 mg/g by AMSC was higher compared to when biochar produced from quaternized sugar cane bagasse was employed; yielding an adsorption capacity of 28.3 mg/g recorded by Hafshejani et al. [25] as well as functionalized cellulose grafted epichlorohydrin recording adsorption capacity of 232.56 mg/g [26]. And the 28.82 mg/g adsorption capacity of dodecylethyldimethylammonium (DEDMA) bromide modified sepiolite reported by Adnan et al. [10].

Table 3: Amount of nitrate adsorbed, adsorption efficiency and rate

Exp. Runs	Temp (°C)	Dosage (g)	pH	Time (min)	Amount of NO_3^- adsorbed (mg/L)	Qe (mg/L)	Re (%)	R (min^{-1})
1	60	2.1	11	75	28	1333	73	0.37
2	42.5	0.2	7	120	28	14000	73	0.23
3	60	0.2	7	75	31.5	15750	82	0.42
4	42.5	2.1	7	75	31.5	1500	82	0.42
5	25	2.1	3	75	24.5	1166	64	0.33
6	42.5	0.2	11	75	35	17500	91	0.47
7	25	2.1	7	120	28	1333	73	0.23
8	25	2.1	11	75	36.75	1750	95**	0.49
9	42.5	0.2	7	30	35	17500	91	1.17
10	60	4	7	75	35	875	91	0.47
11	42.5	2.1	11	120	31.5	1500	82	0.26
12	42.5	2.1	7	75	35	1666	91	0.47
13	42.5	2.1	7	75	24.5	1166	64	0.33
14	42.5	4	7	30	21	525	55	0.7
15	42.5	4	11	75	24.5	612	64	0.33
16	42.5	2.1	11	30	24.5	1166	64	0.82
17	25	4	7	75	36.75	918	95**	0.49
18	25	0.2	7	75	35	17500	91	0.47
19	42.5	2.1	3	120	35	1666	91	0.29
20	42.5	4	7	120	24.5	612	64	0.20
21	42.5	0.2	3	75	35	17500	91	0.47
22	42.5	2.1	7	75	21	1000	55	0.28
23	60	2.1	7	120	7	333	18	0.06
24	42.5	4	3	75	7	175	18	0.09
25	42.5	2.1	3	30	21	1000	55	0.7
26	60	2.1	7	30	10.5	500	27	0.35
27	60	2.1	3	75	28	1333	73	0.37
28	25	2.1	7	30	35	1666	91	1.17
29	42.5	2.1	7	75	35	1666	91	0.47
Mean	42.5	2.1	7	75	27.75	4317.6	72.24	0.445

Qe = Adsorption capacity, Re = Removal efficiency, R = Rate of adsorption

Optimum Nitrate Adsorption Relative to Temperature and Dosage at Constant pH and Time

Figure 5 shows the 3D representations of the effect of varying temperature, dosage, pH and time on the maximum adsorptive removal of nitrate with modified sepiolite clay demonstrated by the design expert software. The trend implies that at constant pH 7 and contact time of 75 minutes, the optimum amount of nitrate adsorbed by the modified sepiolite clay was 36.75 mg/L. Also, at that condition of constant pH 7 and time (75 min), the 3D graph showed a general decrease in the amount of nitrate adsorbed with increase in adsorbent dosage and temperature.

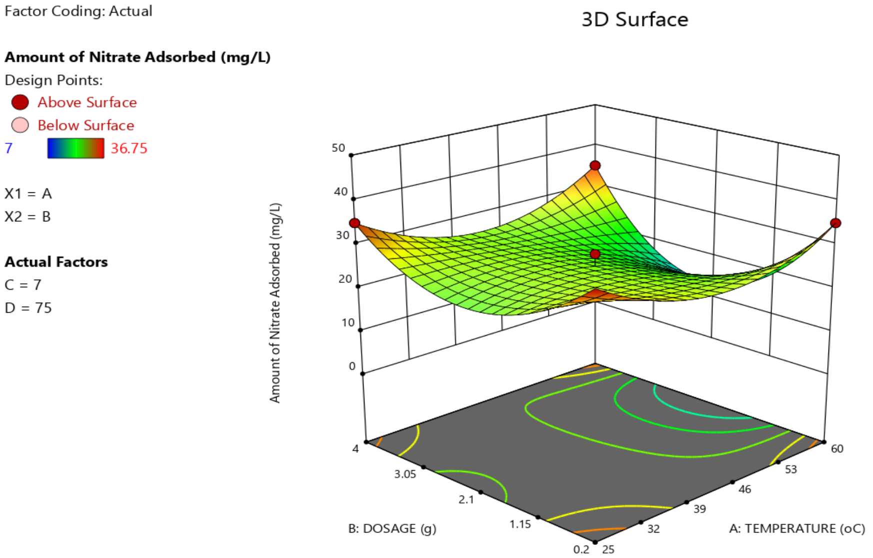

Figure 5: 3D surface showing the effect of dosage and temperature on the adsorption of nitrate

Effect of Temperature, Dosage, pH and Contact time on the Adsorption of Nitrate

The effect of temperature**,** adsorbent dosage, pH and contact time on the adsorptive removal of nitrate displayed on Figure 5, showed an increasing trend in the adsorptive removal of nitrate as the temperature increases from 25°C to 42.5°C and afterwards a continues decrease in nitrate adsorption around 60°C. This trend aligns with the affirmation that elevated temperature does affect the morphological structure of sepiolite thereby affecting its sorption capacity [27].

The dosage effect on the removal of nitrate indicates that the removal decreases with increasing dosage from 0.2g to 2.1g and 4.0g. This may be attributed to increased compact of adsorbent particles and consequently less surface area available for adsorption [28]. While the adsorption trend for nitrate removal shows continues increase with increasing pH climax at the pH of 11. The trend concords with the assertion increasing pH aids adhesion to the surface while decreasing pH aid leaching out [29].

The effect of contact time on the removal of nitrate demonstrates an increase in the concentration of nitrate adsorbed (36.75 mg/L) as the contact time increases from 30 to 120 minute. As this will affords the adsorbate and adsorbent the amble time to interact leading to adsorptive removal of nitrate.

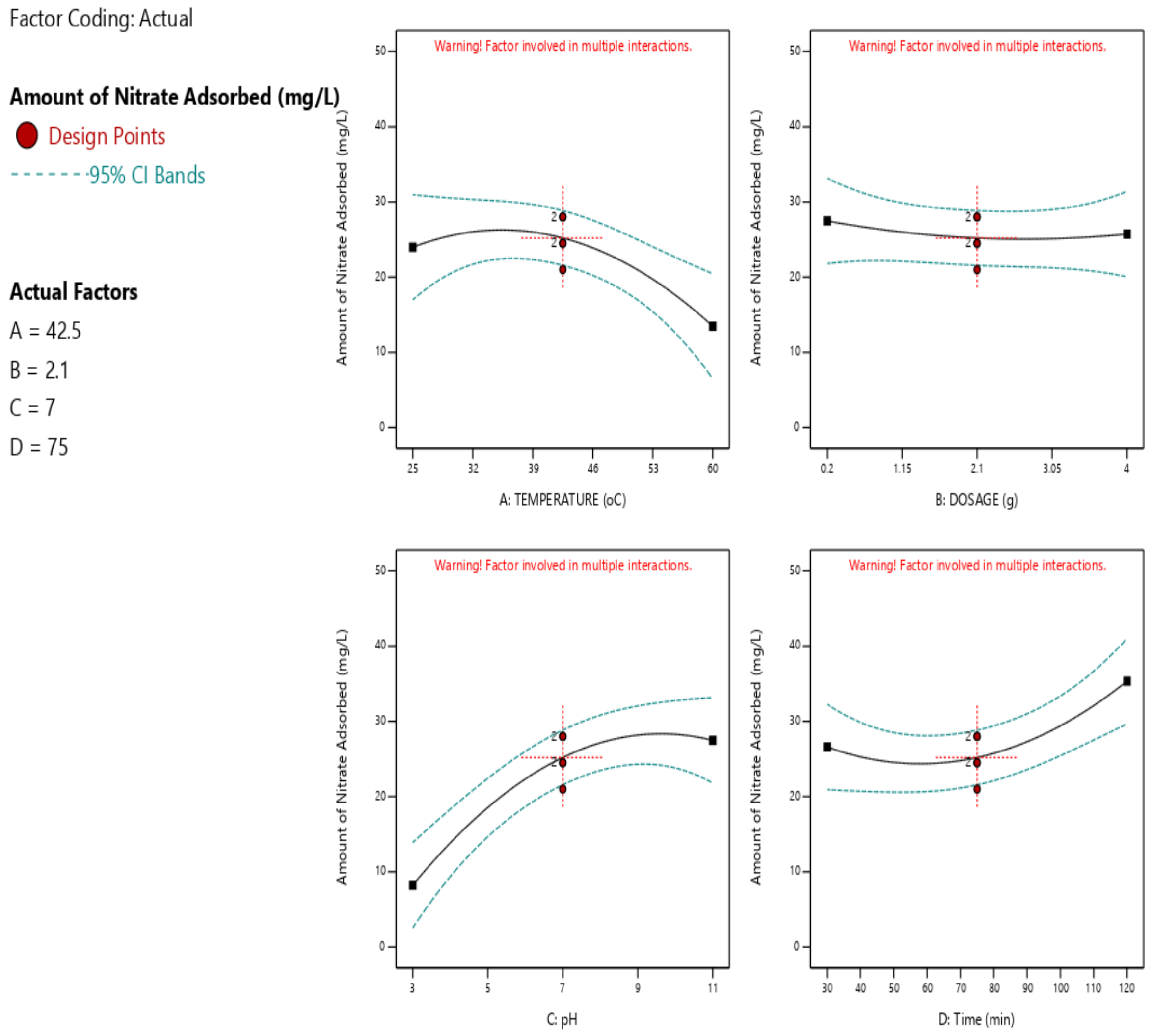

Figure 6: Effect of temperature, dosage, pH and contact time on the adsorption of nitrate

Adsorption Kinetics for the Adsorption of Nitrate

Data obtained from the effect of contact time on the removal of nitrate from agricultural effluent were imputed into the pseudo first order and second order kinetic models. The values of kinetic parameters obtained are presented in Table 4. The parameters obtained revealed that second order kinetic model best define the mechanism of nitrate removal by alkaline modified sepiolite clay by virtue of higher correlation coefficient (R^2 = 0.9253) with the corresponding rate constant K_2 of 0.0782 g.mg^{-1}min^{-1} when compared with the pseudo first order kinetics (R^2 = 0.4808) with the rate constant K_1 of 0.0129 g.mg^{-1}min^{-1}. This shows that chemisorption predominates as the rate determining step [30].

Table 4: Kinetic Parameters for Adsorption Nitrate

Model	**Parameter**	**Value**
First Order	K_1 (min^{-1})	0.0129
	Qe (mg/g)	41.3714
	R^2	0.4808
Second Order	K_2 (min^{-1})	0.0782
	Qe (mg/g)	1.0682
	R^2	0.9253

Adsorption Isotherm for the Removal of Nitrate

This describes the relationship between the adsorbate (nitrate) in the liquid phase and the nitrate adsorbed on the surface of the MS adsorbent under conditions of equilibrium and constant temperature. The adsorption isotherm for nitrate was found to fit the Langmuir Isotherm better, with a regression coefficient R^2 of 0.9944, compared to the Freundlich Isotherm, which had an R^2 of 0.8315, as shown in Table 5.

The Langmuir model's equilibrium parameter, R_L of 0.3889, indicates that the adsorption of nitrate onto MS is favorable, as suggested by the condition $R_L < 1$ (which signifies favorable adsorption), $R_L \sim 0$ (indicating irreversible adsorption), $R_L = 1$ (which indicates a linear adsorption isotherm), and $R_L > 1$ (which corresponds to unfavorable adsorption) as noted by Ibrahim et al. [31]. The Langmuir isotherm model is based on the premise that adsorption occurs at uniform sites, leading to the formation of a monolayer [32].

Table 5: Isotherm Parameters for Nitrate Adsorption

Model	Parameters	Value
Langmuir Isotherm	Qm (mg/ml)	3888
	K_L	0.0408
	R^2	0.9944
	R_L	0.3889
Freundlich Isotherm	K_F	3309
	R^2	0.8315
	$1/n$	1.3350
	n_F	0.7491

CONCLUSION

This study revealed that modified sepiolite clay exhibits excellent physicochemical properties such as polarity visible pore size of 3.6 Å x 10.6 Å, presence of surface functional groups such as silanol groups (-SiOH), which facilitate its application as adsorbent, recording 95% adsorption of nitrate from waste water with 2.1 g dosage at 25 °C and pH of 11 over a period of 75 minutes. The adsorptive removal mechanism best conforms to the Blanchard pseudo-second order and Langmuir Isotherm. A trend was observed in the effect of temperature, dosage, pH and contact time on the adsorption of nitrate. The adsorption of nitrate increases with increase in the contact time, pH and dosage of adsorbent but decreases with increase in temperature, which may be resulted from the increase in average kinetic energy of the adsorbates at higher temperature.

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Conflict of Interest

The authors declare that there are no known financial interests or personal relationships that could have influenced the work presented in this paper.

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