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Investigation of Nitrate Adsorption Potential of Natural Sand Materials from Sutlej and Beas River Basins: Equilibrium and Kinetic Studies

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Abstract

Many parts of the world are facing the problem of nitrate contamination in both surface and groundwater. Multiple health complications in humans especially in infants are caused due to elevated levels of nitrate in drinking water. Due to multiple devastating effects, nitrate mitigation strategies are among the major concern worldwide. The present study was conducted to evaluate the nitrate removal efficiency of natural and locally available sand materials. Adsorption experiments were conducted and the process parameters were optimized. The trends of results revealed that significant percent nitrate removal (76 and 81.5) was achieved with selected adsorbents under optimized conditions of pH (6.0), adsorbent dose (20 g/L), and contact time of two hours with initial nitrate concentration (200 mg/L). Best nitrate removal was observed in the acidic pH range of 4.5-7.0. Langmuir and Freundlich's isotherms were also applied to check the fitness of the data and found better fitted to the Langmuir isotherm with a high value of correlation coefficient (R^2= 0.995 and 0.998).

Keywords: Nitrate, Removal Efficiency, Sand, adsorption Isotherms and Kinetic studies

1. INTRODUCTION

Recent water quality monitoring studies conducted around the world have reported higher concentrations of nitrates in groundwater aquifers. The majority of European countries, most parts of West and Central America, Australia and China are found to have nitrates exceeding 50 mg/L NO_3-N (Ward et al. 2018; Feng et al. 2020). In India, about 21 states including intensive farming belts of U.P., Haryana, Punjab, and parts of Rajasthan, are found to contain high nitrates in their groundwater (Bhardwaj et al. 2012; Sharma et al. 2014).

Nitrate is a natural component of soil enriched with nitrogen-fixing bacteria, dead and decaying plants and animal manure. It may be added to it in the form of artificial fertilizers to increase soil fertility and crop production. The majority of this is utilized by the plants to synthesize nitrogen-containing organic compounds but the excess nitrate percolates down through the soil with rain or irrigation water and contaminates the underground aquifers. Changes in land use and disposal of untreated waste are the other factors responsible for the progressive increase in nitrate levels in groundwater, especially in the Indian subcontinent. Because of high solubility and prevailing aerobic conditions, nitrate remains persistent at higher levels in shallow aquifers for longer periods. Elevated levels of nitrate in water have greatly reduced its quality and rendered it unfit for human consumption. The WHO has set 50 mg/L as the maximum containment level in drinking water (WHO 2011). However, according to Indian standard guidelines for drinking water, it is 45 mg/L (ISI 2009).

The deleterious health effects attributed to high nitrate consumption are infantile methemoglobinemia also known as blue-baby syndrome[7,8]. Long-term consumption of elevated levels of nitrate can affect the health of adults causing respiratory tract infections, changes in the immune system, reproductive anomalies, birth defects and cancer(Espejo-Herrera et al. 2016; Ward et al., 2011 and Colman et al., 2011). Due to multiple devastating effects, nitrate mitigation strategies are among the major concern worldwide.

The present study was conducted to investigate the potential of natural, inexhaustible, low-cost sand materials for the removal of nitrates from groundwater.

2. MATERIALS AND METHODS

The model stock solution of nitrate (500 mg/L) was prepared by dissolving an appropriate amount of sodium nitrate in deionized water. The working solution of 200 mg/L was prepared by making suitable dilutions of the stock solution. All the chemicals used were of analytical grade (Merck). The concentration of nitrate before and after adsorption was determined simply by measuring absorbance at 240 nm in the UV region. All statistical analyses were performed with the help of Origin 8.0.

2.1 Sample Collection and Pre-treatment

Two types of sand samples were collected from two different locations in Punjab, the northwestern state of India. Type-I sand was obtained from the Sutlej River bed near the Moga district whereas a type-II sand sample was collected from a stone crusher site (Beasriver basin) in the Pathankot region. The adsorbent was first sieved & filtered to obtain grain size below 100 µm and was then washed several times with the distilled water. Finally, the adsorbent was dried in the oven at 250 ^{0}C for 12 hours. Adsorbent characterization studies such as FTIR, SEM, EDX, etc. were performed to obtain information about their surface characteristics including functional groups present and chemical composition.

2.2.Adsorption Experiments

Batch adsorption experiments for nitrate removal from the simulated solution were conducted in two sets. In each set of experiments, a fixed amount of adsorbate (20g/L) was added to a 100 ml model solution of nitrate (200 mg/L) in flasks of 250 ml capacity and allowed to mix by placing on a shaker set at 180 rpm for 20 minutes. The desired pH of a solution was adjusted by adding small increments of 0.1N HCl and 0.1N NaOH. Adsorption in each set was measured and recorded at regular intervals.

The percent removal and adsorption capacity of nitrate at equilibrium was calculated using the equations given below:

$$Percent\ Removal\ (R\%) = \frac{C_i - C_e}{C_i}\ X\ 100 \qquad \text{(Eq 1)}$$

Where C_i and C_e are the initial and final concentrations of nitrate in solution

$$Adsorption\ capacity\ (q_e) = \frac{C_i - C_e}{M}\ X\ V \qquad \text{(Eq 2)}$$

Where q_e (mg/g) is the amount of nitrate adsorbed, C_i and C_e are the initial and final concentrations of nitrate in solution, V is the volume of solution and M is the mass of dry adsorbent in grams.

The adsorption studies were carried out by varying the adsorption process parameters viz. pH, adsorbent dose, initial ion concentration and contact time and were optimized. All the experiments were conducted in duplicates and the mean values are reported. To study adsorption kinetics, the samples at different times were taken and analyzed to understand the rate of adsorption.

The adsorption isotherm models provide a quantitative relationship between the adsorbate concentrations of in the solution and in the adsorbent at any time at equilibrium. The classical isotherms including Langmuir, Freundlich and Temkin models were applied to check the fitness of the data.

The nitrate removal efficiency was determined by measuring the concentration of nitrate in the solution before and after the adsorption. The nitrate concentrations were determined spectrophotometrically by measuring it at 220 nm. The method is suitable for water samples containing little or no organic matter (which also absorbs at 220nm). To determine nitrate concentration by UV-visible spectrophotometer, 100 ml calibration nitrate standards for 2,4,6,8 and 10 mg/L were prepared by suitably diluting the stock nitrate solution of 100 mg/L. The samples were acidified with 1 ml HCl and the absorbance of each was measured at 220nm against reference. A calibration curve was constructed by plotting nitrate concentration versus absorbance and the nitrate concentration was determined.

3. RESULTS AND DISCUSSION

3.1 Adsorbent characterization:

Sand adsorbents selected for the present study varied greatly in their texture and appearance. The information regarding their surface characteristics and chemical composition was gained by analysing the sand materials with Fourier-transformed infrared spectroscopy (FTIR), Scanning electron microscopy (SEM) and energy-dispersive x-ray (EDX) analysis. The type-I adsorbent was silvery-white in colour whereas the type-II adsorbent was little dark brown in appearance. The variation in colour of both the adsorbents was first indicative of their differential chemical composition.

Figure 1 shows the SEM micrographs of native type-I and type–II sand adsorbents at 2300 magnification and Acc. Voltage 15.0 kV. The SEM micrographs were obtained using a Scanning Electron Microscope (Hitachi S-3400) with EDS. At first look, the SEM images of both samples showed heterogeneous surfaces with irregular and coarse type particles (Fig 1a and 1b). The noteworthy roughness and heterogeneity of these suggested that both adsorbents are porous.

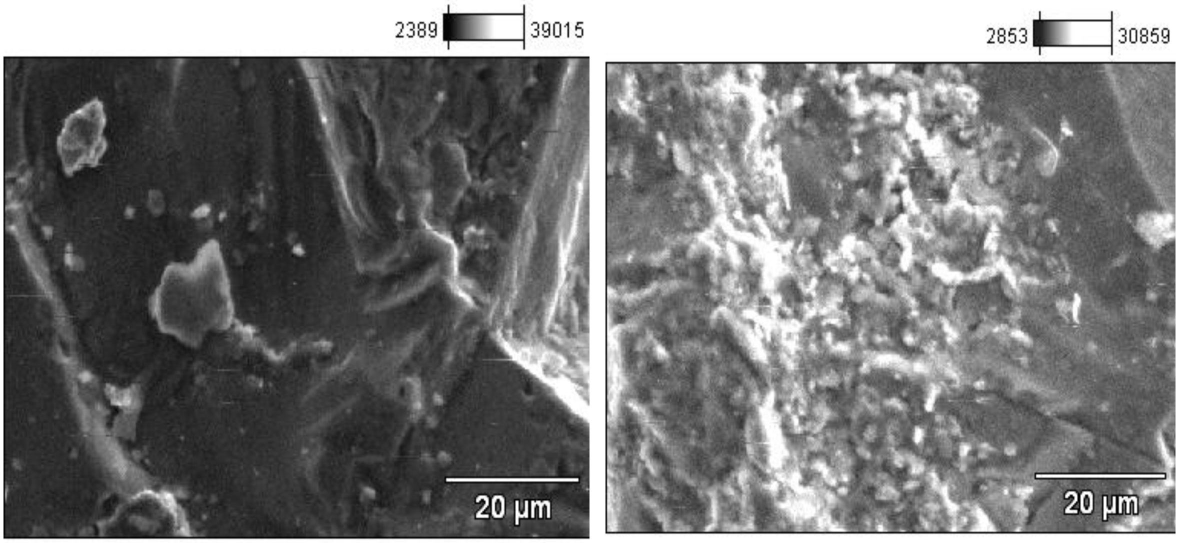


Figure 1: SEM images of type-I (a) and type-II (b) sand adsorbents

Energy dispersive x-ray spectroscopy was also done to get accurate identification of minerals in sand samples. This technique provides the visual and compositional characteristics of a specimen. The physicochemical characteristics and mineral composition of both native sand adsorbents are presented in Tables 1 and 2. From data, it is obvious that both adsorbent samples are silica and quartz types that may have eroded from mountains and carried with water to the river beds.

Table 1: Physical characteristics of sand adsorbents

Parameter	**Type-I adsorbent**	**Type-II adsorbent**
Texture	Silvery white	Dark brown
pH	7.23	7.56
Porosity	5-10 µm	4-8 µm

Table 2: Mineral composition of type-I and type-II sand adsorbents

	Type –I adsorbent			**Type-II adsorbent**		
Element	**Net Counts**	**Weight %**	**Atom %**	**Net Counts**	**Weight %**	**Atom %**
C	1389	40.10	52.78	3644	52.10	66.66
O	4380	31.85	31.47	4344	21.12	20.28
Al	206	0.30	0.18	598	0.54	0.31
Si	18083	27.43	15.44	21714	20.07	10.98
Ca	96	0.32	0.13	-	-	-
Fe	-	-	-	1077	5.97	1.64
Mg	-	-	-	225	0.20	0.12
Total		100.00	100.00		100.00	100.00

SEM and EDX analysis reveals that type-I sand is rich in silicon (Si), oxygen (O) and carbon (C) with little aluminium (Al) and hydrogen (H). The size of the sand particle varies from 5-10 µm which depicts its highly porous structure with a high surface area.

The size of the type-II sand particle also lies in the range of 4-8 µm. The carbon (C) content of adsorbent is highest followed by silicon (Si) and aluminium (Al). In addition, type-I adsorbent contains calcium (Ca) as a second minor mineral whereas type-II sand contains magnesium (Mg) and iron (Fe).

FTIR spectroscopy was performed to predict the nature of functional groups present on adsorbent surface. The technique also provides insights to the mechanism of adsorption by providing information regarding groups participating in adsorption. The IR spectra of adsorbents were obtained with an instrument FTIR NXRFT Raman Spectroscope: Thermo Nicolet 6700. Spectra were recorded in the range of 4000-400 cm^{-1} wavelength.

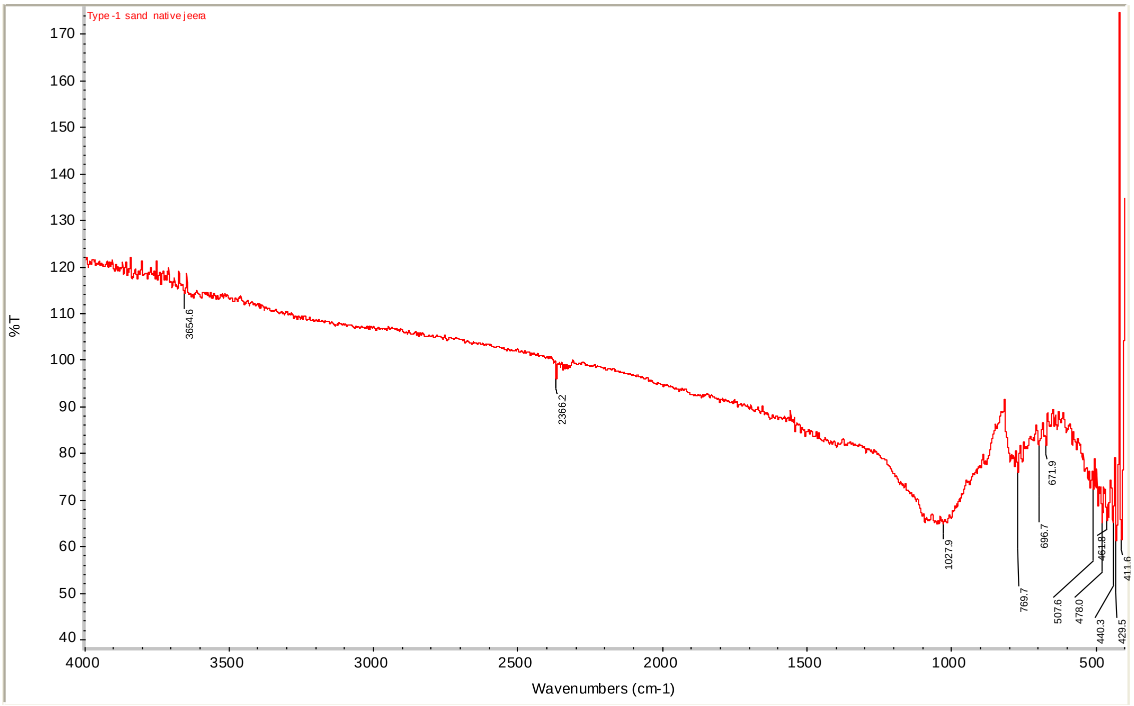


Figure 2 (a): FTIR spectrum of Type-I sand adsorbent

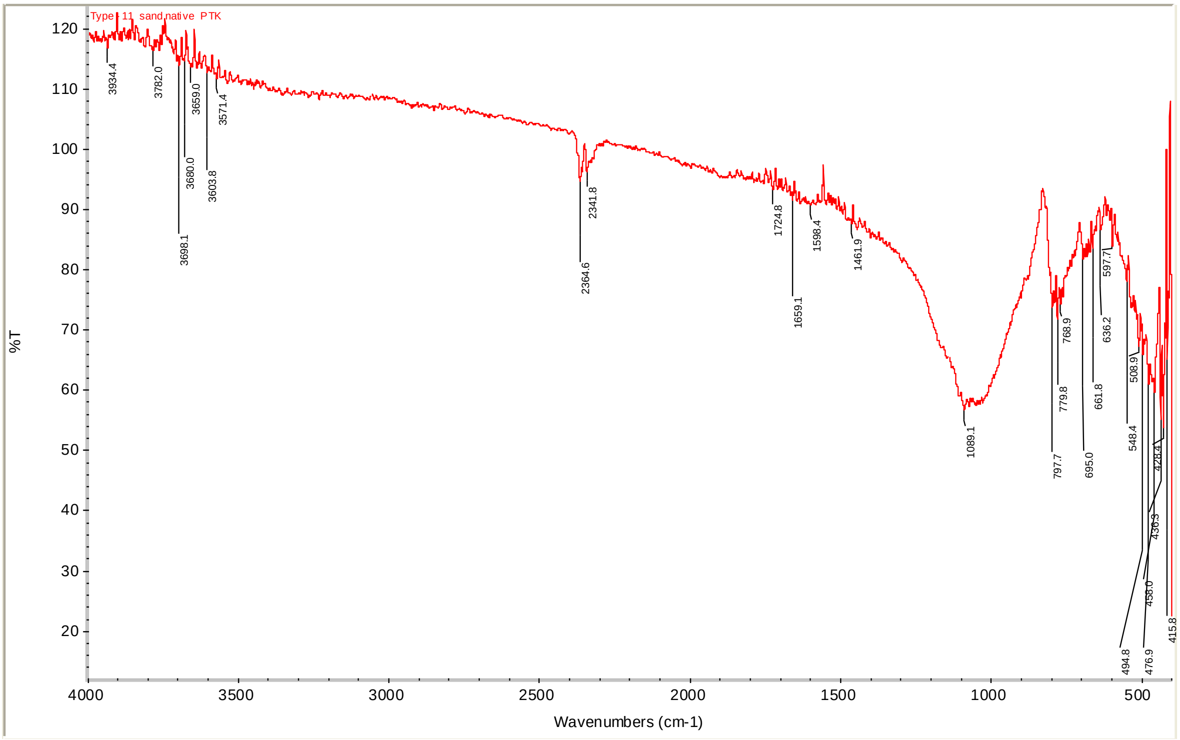
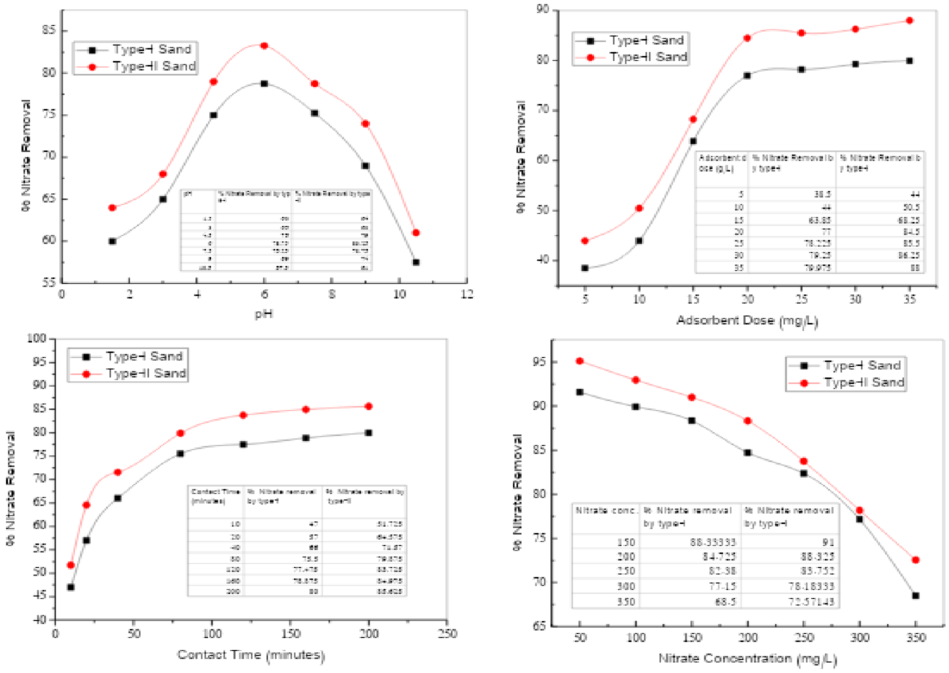


Figure 2 (b): FTIR spectrum of Type-II sand adsorbent

The FTIR spectra of type-I adsorbent showed absorption peaks at 3654, 2366 and 1028 cm^{-1}. These absorption values indicate the presence of OH groups, Si-O bonds and carbonate groups and thereby confirm the presence of silica with carbonates. This further validates the results of SEM and EDX. The presence of the crystalline nature of quartz minerals is usually diagnosed by the presence of an absorption band at 695 cm^{-1}. The absence of this band is suggestive of its amorphous nature. In the sample, the peak has appeared at 696.7 cm^{-1} which indicates that the nature of quartz is of mixed type i.e crystalline with little amorphous character[13]. In the FTIR spectrum of type-II adsorbent, peaks are observed at 3783, 3664, 3378 and 2344 cm^{-1} suggesting the presence of free and substituted –OH group, Si-O and S-H bonds. Free –SH and –OH may be taken as responsible for adsorption by this adsorbent. IR absorption bands appeared at 797, 779, 769, 695 and 458 cm^{-1} suggesting the presence of quartz. The absorption bands observed between 790–800 cm^{-1} and 690–695 cm^{-1} are widely used as the diagnostic peaks for quartz. The peak appeared at 695 cm^{-1} indicating the crystalline nature of quartz in the sample. The crystalline nature has also been confirmed by the SEM images of the sample. The region below 800 cm^{-1} is responsible for metal-oxygen-metal bonding. Peaks observed in this region may be due to metal oxides which may also partly account for adsorption.

3.2 Optimization Studies

For optimization of process parameters on nitrate removal was investigated by varying the pH between (1.5-10.5), adsorbent dose (5-35 g/L) with nitrate concentration (50-350 mg/L) at a stirring speed of 180 rpm for 90 minutes. Trends of nitrate adsorption with varied parameters are presented in figure 3.



Adsorbent dose (g/L)	% Nitrate Removal by type-I	% Nitrate Removal by type-II
5	38.5	44
10	44	50.5
15	63.85	68.25
20	77	84.5
25	78.225	85.5
30	79.25	86.25
35	79.975	88

Contact Time (minutes)	% Nitrate removal by type-I	% Nitrate removal by type-II
10	47	51.725
20	57	64.375
40	66	71.87
80	75.5	79.875
120	77.475	83.725
160	78.875	84.875
200	80	85.625

Nitrate conc.	% Nitrate removal by type-I	% Nitrate removal by type-II
150	88.33333	91
200	84.725	88.325
250	82.38	85.752
300	77.15	78.18333
350	68.5	72.57143

Figure 3: Trends of Nitrate adsorption with process parameters (a) pH, (b) Adsorbent dose, (c) Contact Time, (d) Nitrate Concentration

To investigate the effect of pH on nitrate adsorption, batch experiments were conducted at different pHs ranging from 1.5 to 10.5. The results are presented in figure 3a. With both adsorbents, nitrate showed a positive trend with pH reaching a maximum at a value equal to 6 (78% and 83%). The significant percent removal (76% and 81.5%) was obtained in acidic pH ranging between 4.5-7.5 with an optimized adsorbent dosage of 20 g/L of both type-I & type-II sand adsorbents.Therefore, all experiments were performed at pH 6.

For dosage optimization, percent adsorption of nitrate was studied at different doses varied between 5 and 35 g/L by keeping all other parameters constant: Nitrate concentration (200 mg/L), stirring speed (180 rpm), pH (6.0) and contact time (90 min) constant. Increased percent nitrate removal was observed when the adsorbent dose of both type-I and type-II sand samples was increased from 5g to 20g. Thereafter, no significant increase in removal was observed with a further rise in adsorbent dosage (Fig 3b). Maximum nitrate removal upto 80 and 88% was achieved with type-I and type-II sand adsorbents respectively.

The effect of contact time on adsorption was studied by conducting experiments at varied time intervals ranging from 20 – 180 minutes under predefined conditions of process parameters i.e. pH-6, adsorbent dose-20g and nitrate concentration equal to 200 mg/L. The nitrate adsorption occurred rapidly in the first ninety minutes and then became nearly independent of time. Both adsorbents showed similar trends of adsorption with contact time (Figure 3c).

Studies carried out with varied Nitrate concentrations (50 to 250 mg/L) revealed that very little fall in adsorption occurred in this range of parameters and therefore did not affect the adsorption significantly (Figure 3d.). Maximum adsorption (91% and 88%) was observed at pH-6 with a nitrate concentration of 50 mg/L, thereafter it declined gradually with increasing nitrate concentration. Still, significant adsorption up to 68.5 and 72.57% was achieved with nitrate concentration-350 mg/L.

Adsorption capacities obtained with varying nitrate concentrations under all standard conditions are shown in Table 3.

Table 3: Adsorption capacities obtained with varied nitrate concentrations at pH-6, A.Dose-20 g/L and Contact Time -90 mins

Nitrate concentration (mg/L)	Type-I adsorbent (q_e) (mg/g)	Type-II adsorbent (q_e) (mg/g)
50	0.22895	0.2378
100	0.4496	0.46475
150	0.6625	0.6825
200	0.84725	0.88325
250	1.02975	1.0469
300	1.15725	1.17275

3.3 Adsorption isotherms

The two classical isotherms Langmuir and Freundlich are most suitable to explain the short-term mono-component adsorption of adsorbate by the different adsorbents. Langmuir adsorption isotherm explains that the adsorbate molecules are adsorbed on the surface at certain specific sites on the surface of adsorbent. It also suggested that adsorption energy for each adsorbate site is uniform and there is no interaction between the molecules adsorbed on adjacent binding sites(Altenor et al., 2009; Loredo-Cancino et al., 2016).

The linear form of the Langmuir adsorption isotherm is represented by following equation:

$$\frac{C_e}{q_e} = \frac{1}{Q_0\, b} + \frac{C_e}{Q_0} \qquad \text{Eq.(3)}$$

where, C_e is the equilibrium concentration of adsorbate ion (μg/L), and q_e is the specific amount of adsorbate adsorbed per gram of adsorbent at equilibrium (μg/g). Q_0 (mg/g) and b (mg^{-1}) are Langmuir constants related to adsorption capacity and energy of adsorption, respectively which can be calculated from the slope and intercept of the Langmuir plot of C_e versus C_e/q_e. The Q_0 represents the maximum amount of ion adsorbed per unit gram of adsorbent to form a complete monolayer on the surface of the adsorbent.

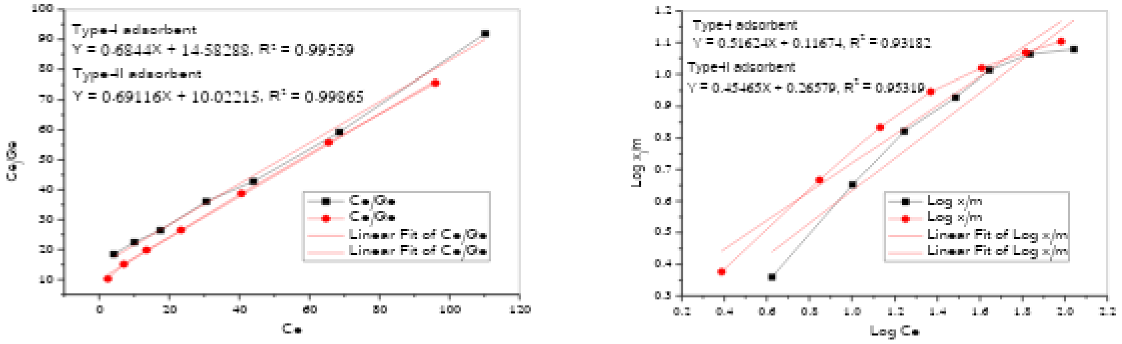


Fig 4: (a) Langmuir and (b) Freundlich plot of NO_3adsorption on type I and type II sand adsorbents

Another important feature of Langmuir isotherm is the equilibrium parameter, R_L which predicts the nature of adsorption of adsorbate on specific adsorbent. Its value can be calculated from the following equation given below:

$$\boldsymbol{R_L = \frac{1}{(1+bC_0)}} \qquad \text{Eq (4)}$$

S. No	Value of R_L	Nature of adsorption
1	$R_L > 1$	Unfavourable
2	$R_L < 1$	Favourable
3	$R_L = 1$	Linear
4	$R_L = 0$	Irreversible

Freundlich adsorption isothermrepresents the relationship between the amount of metal adsorbed per unit mass of the adsorbent (x/m) and the concentration of the metal ion in solution at equilibrium (C_e). The linear form of Freundlich may be represented by the following equation:

$$log\frac{x}{m} = \log K_f + \frac{1}{n}\log C_e \qquad \text{Eq (5)}$$

The Freundlich expression is an empirical equation based on sorption on a heterogeneous surface where energy distribution is non-uniform.

Temkin Isotherm was also applied to check the fitness of adsorption data. This isotherm is used to explain the interaction between adsorbate molecules present on adjacent sites on the adsorbent surface. The isotherm is better suited to explain the nature of adsorption based on adsorption energies.

The linear form of the Temkin equation is applied as:

$$qe = BlnA + BlnC_e \qquad \text{Eq. (6)}$$

Where A is the model constant and B is a constant related to the heat of adsorption. The values of these constants can be computed from a graph of ln C_e versus q_e

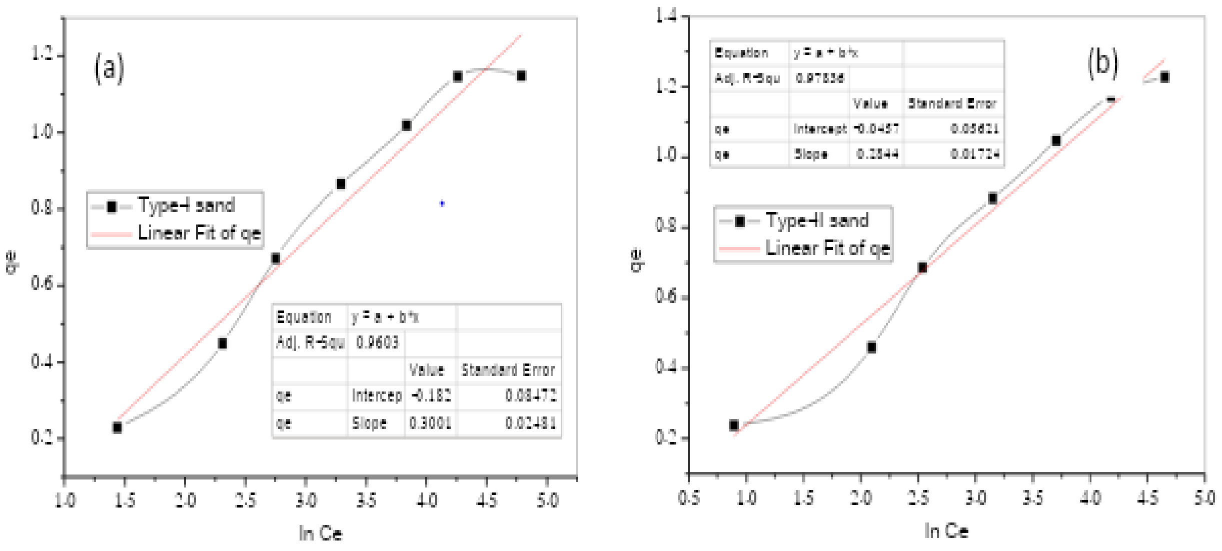


Fig 5: Temkin plot of NO_3 adsorption on (a) type-I and (b) type-II sand adsorbents

Results of isotherm studies of nitrate adsorption are presented in Figures 4a,4b, 5a, and 5b. It is obvious from the figure that nitrate adsorption by both type-I and type-II adsorbents was better fitted to the Langmuir isotherm with a high value of correlation coefficient (R^2= 0.995 and 0.998) suggesting that nitrate adsorption occurs on the surface of adsorbent as a uniform monolayer. The value of R_L for nitrate adsorption on both type-I and type-II sand adsorbents was found to be less than 1 which was suggestive of its favourable adsorption on both types of sand materials. Also low R_L value equal to 0.47 for type–I adsorbent indicated that the nitrate has more affinity for this adsorbent as compared to type-II sand adsorbent.

Table 4: Summary of adsorption isotherm parameters

Parameter	**Type-I sand adsorbent**	**Type-II sand adsorbent**
Langmuir Isotherm		
Q_0 (mg/g)	1.461133	1.44684
b (mg^{-1})	0.100195	0.068963
R^2	0.99599	0.99865
R_L	0.0470	0.0676
Freundlich Isotherm		
K_f(mg/g)	1.3083	1.9310
N	1.9370	2.1995
R^2	0.9318	0.9532
Temkin Isotherm		
A (L/m)	0.5451	0.85153
B (J/mole)	0.30012	0.28447
R^2	0.96034	0.97836

3.4 Kinetic Studies

The study of adsorption kinetics is important because it provides valuable information and insights into the reaction pathways and the mechanism of the reactions. Several kinetic models have been developed to describe the reaction order and rate constants of adsorption systems. These include first-order and second-order reversible ones, and first-order and second-order irreversible ones, pseudo-first-order and pseudo-second-order. The kinetic of adsorption data was investigated using two commonly used kinetic models; pseudo-first order and pseudo-second order. The pseudo-first order model was employed as per the equation given below:

$$\boldsymbol{\log (q_e - q_t) = \log q_e - \frac{k_1}{2.303} t} \qquad \text{Eq. (7)}$$

Where q_e and q_t are the amount of adsorbate adsorbed at equilibrium and time t, k_1 is the pseudo-first-order rate constant. By plotting the graph of log (q_e – q_t) against time (t), values of rate constants k_1 and q_e can be determined from the slope and intercept of the graph. The pseudo-second-order model was used to investigate the kinetic of adsorption by employing the following equation:

$$\frac{t}{q_t} = \frac{1}{k_2}\frac{1}{q_e^2} + \frac{1}{q_e}t \qquad \text{Eq. (8)}$$

The values of rate constant k_2 and $q_e$were determined from slope and intercept of plot t/qt versus time, t.

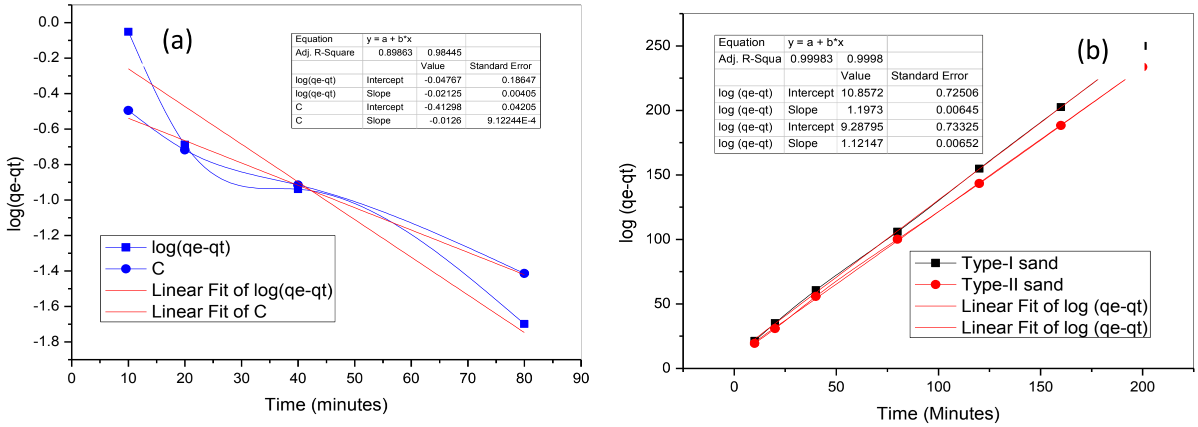


Figure 6: (a) Pseudo-first order and (b) Pseudo-second order kinetic plot of nitrate adsorption

It is apparent from the results that the pseudo-second order kinetic model with R^2= 0.999, holds good for the nitrate adsorption on both type-I and type-II sand adsorbents undertaken in the present study.

4. CONCLUSION AND FUTURE PERSPECTIVES

Both type-I and type-II sand adsorbents are capable of reducing nitrate from the water having moderate to high concentrations. The adsorption on both adsorbents was best observed in acidic pH. The equilibrium adsorption data obtained with both the adsorbents was better fitted to the Langmuir model. Results of a comparative study revealed that type II adsorbent was found to exhibit higher removal efficiencies attributed to its chemical composition. Overall findings of the study suggested that inexpensive and abundantly available sand resources can be exploited as an economically viable alternative to the expensive adsorbents for the removal of nitrate from water with low to moderate concentrations. As the results are being reported with native sand adsorbents, further removal efficiencies can be enhanced by giving thermal or chemical treatment to the sand materials used in the present study.

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DECLARATIONS

CONFLICTS OF INTERESTS

The authors declare that there are no conflicts of interest.

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This article does not include any research that the author conducted with human subjects or animals.

CONTRIBUTION OF AUTHORS

All the authors have contributed significantly to this manuscript, participated in reviewing/editing and approved the final draft for publication. The research profile of the authors can be verified from their ORCID ids, given below

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