

Research Article

Nitrate Adsorption on Clay Kaolin: Batch Tests

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Soils possessing kaolin, gibbsite, goethite, and hematite particles have been found to have a natural capacity to attenuate pollution in aqueous phase. On the other hand, the hydroxyl group in soil increases anion exchange capacity under a low pH condition. The main objective of this paper was to evaluate effects of kaolin on nitrate reduction under acidic condition. In order to analyze the kaolin adsorption behaviour under various conditions, four different concentrations of nitrate, 45, 112.5, 225, and 450 mgNO₃⁻/L, with a constant pH equal to 2, constant temperature equal to 25°C, and exposure period varying from 0 to 150 minutes were considered. The capacity of nitrate adsorption on kaolin has also been studied involving two well-known adsorption isotherm models, namely, Freundlich and Langmuir. The results revealed that approximately 25% of the nitrate present in the solution was adsorbed on clay kaolin. The laboratory experimental data revealed that Freundlich adsorption isotherm model was more accurate than Langmuir adsorption model in predicting of nitrate adsorption. Furthermore, the retardation factor of nitrate pollution in saturated zone has been found to be approximately 4 in presence of kaolin, which indicated that kaolin can be used for natural scavenger of pollution in the environment.

1. Introduction

Groundwater and soil can be polluted through various anthropogenic activities such as pesticide use and industrial processes [1]. The breaking of the ecological equilibrium between human being and the nature is the main reason of environmental contamination issue [2]. Since 1970s nitrate leaching to subsoil has become an important environmental quality and human health issue, especially due to high use of nitrogen as nutrient in agriculture [3–5]. Inorganic nitrogen is used as an essential fertilizer in intensive agriculture to produce sufficient food for growing population and food security, a substantial part of which finally infiltrates into subsurface and contaminates soil and groundwater [6–8]. Nitrate itself is relatively nontoxic, but nitrite derived by the reduction of nitrate, which can cause various human health problems such as methemoglobinemia, liver damage, and even cancers [9]. Although nitrate is highly soluble in water [10] and does not readily bind to the soil and leaches due to massive irrigation and heavy rains [11], the movement

of some anions like nitrate is retarded by variable surface-charge of soil particles [12–15]. Various soil and solution parameters which have an effect on nitrate concentration in porous medium are categorized as follows: ionic strength [16], aluminum oxide concentrations [17], competition with other anions [18], pH of soil solutions [19], soil texture and clay mineralogy [20], and organic matter content [21].

Townsend and Young [22]; Pociene and Pocius [23]; Gurdak and Qi [24]; O'Reilly et al. [25]; and a number of other researchers have reported statistical relationship between nitrate pollution and soil as well as the solution properties. According to Townsend and Young [22], the thickness of clay above the well screen has positive effect on the reduction of nitrate in groundwater. Pociene and Pocius [23] reported that (i) the amount of nitrate in clayey soil is more than sandy soil; (ii) clay and humus have impact on the leaching and concentration of nitrogen into groundwater; and (iii) relationship between the amount of clay particles and nitrogen leaching is inverse. Vulnerability of groundwater to nitrate in shallow depth tube-wells with less clay or organic

TABLE 1: Chemical composition of kaolin used in the current study.

Parameter	Result (%)
Silica as SiO ₂	45.72
Aluminum as Al ₂ O ₃	37.54
Titanium as TiO ₂	1.10
Iron as Fe ₂ O ₃	0.86
Sodium as Na ₂ O	0.02
Potassium as K ₂ O	1.29
Calcium as CaO	<0.01
Magnesium as MgO	0.51
Loss on ignition at 1025°C	13.0

matter in unsaturated zone above the groundwater table has been reported greater than other areas [24]. O'Reilly et al. [25] remarked that clay particles in saturated porous media may retard the transport of groundwater pollution. Therefore, purification of water using low cost natural and modified adsorbents such as clay minerals can be a cost-effective and sustainable approach [26].

Different soils, including kaolin, gibbsite, goethite, and hematite particles, have potential to retard nitrate contamination transport to groundwater. The hydroxyl group in these soils increases anion exchange capacity (AEC) under acidic conditions, which causes retardation of nitrate movement [8, 13–15, 27]. Based on the literature review, it can be remarked that though many efforts have been taken to investigate the behaviour of nitrate movement in different soils, the knowledge on the effect of kaolin on nitrate retention at low pH is still unknown. The objectives of the present study are (1) to assess nitrate adsorption by clay kaolin under acidic condition; (2) to find adsorption isotherm model to evaluate the adsorption capacity of kaolin; and (3) to predict retardation factor of nitrate movement in porous media in the presence of clay kaolin. It is expected that the present study will help to improve knowledge on nitrate movement in clay under acidic condition which in turn will help to prevent groundwater contamination and keep nitrate in root zone for plant uptake.

2. Materials and Methods

2.1. Soil Sample. Kaolin is one of the most common minerals in various countries around the world. Kaolin clay originates from the chemical weathering of rocks in hot and moist climates and is usually abundant in soils. For the present study, the kaolin extracted from the state of Perak, Malaysia, was collected from Kaolin Company (Malaysia). The chemical properties of kaolin used in the present study are given in Table 1.

2.2. Chemical Used. According to the World Health Organization (WHO) guidelines, the maximum limit of nitrate in drinking water is 45 mgNO₃⁻/L; excessive nitrate (>45 mg/L) in the drinking water causes health risks [28]. Studies around the world indicate that nitrate concentration in groundwater in most parts of the world has not increased more

than ten times of the maximum nitrate limit prescribed by WHO. Therefore, the range of nitrate concentration for laboratory tests was chosen from 45 up to 450 mgNO₃⁻/L. Stock solution of nitrate at concentration of 1000 mgN/L was prepared by dissolving 7.218 g of potassium nitrate (KNO₃) of analytical reagent (QReC) in one liter of distilled water. The test solutions with nitrate at concentrations, 45, 112.5, 225, and 450 mgNO₃⁻/L, were prepared by diluting stock nitrate solution to the desired concentrations.

2.3. Adsorption Study. Adsorption tests were conducted at laboratory room temperature (~25°C). A 2 g of kaolin (measured by digital balance scale model-SHIMADZO, UX 1020H) and 20 mL nitrate solution were contacted in 200-mL Erlenmeyer flask for this purpose. Initial nitrate concentration was 45, 112.5, 225, and 450 mgNO₃⁻/L. The pH of solution was adjusted by using concentrated hydrochloric acid, HCL 37%, (QReC) and was kept constant at 2 for all adsorption tests (Milwaukee-pH600). Rotary shaker (WiseShake-SHR-2D) was used to shake samples at 120 rpm. The contact time between adsorbent and solution was ranged from 15 min to 2.5 hr. Different conditions like contact time and initial concentration were also tested. After desire time, water sampling was conducted and solution was separated from the adsorbent using filter paper (Ashless filter 203). All samples were prepared using brucine sulfanilic acid reagent method in order to measure nitrate concentration. Brucine sulfanilic acid changed the color of the samples to different shades of yellow (from light to deep yellow) depending on the concentration of nitrate in the sample. The final concentration of nitrate in solution was determined by measuring the absorbance at 410 nm by UV-VIS spectrophotometer (BIO-TEK, ELx 808). All experiments were performed in duplicate and the average values were used for calculation.

2.4. Estimation of the Amount of Adsorption. The percentage and the amount of nitrate adsorption at equilibrium concentration on adsorbent surface were calculated as

$$\text{Percent of adsorption} = \frac{(C_i - C_e)}{C_i} \quad (1)$$

$$q_e = (C_i - C_e) \frac{V}{X}, \quad (2)$$

where q_e is the amount of the solute adsorbed per mass unit of adsorbent (mg/g), C_i is the initial concentration in the aqueous phase (mg/L), C_e is the concentration in the aqueous phase at equilibrium (mg/L), V is the volume of the aqueous phase (L), and X is the weight of the adsorbent (g).

2.5. Estimation of Adsorption Isotherm. An adsorption isotherm is a curve which shows the relationship between equilibrium concentration and the amount of solute on the adsorbent in the aqueous phase. In order to evaluate the nitrate adsorption capacity of kaolin, the equilibrium adsorption of nitrate was studied as a function of nitrate concentration and the adsorption isotherms. There are several models for predicting the equilibrium distribution. However,

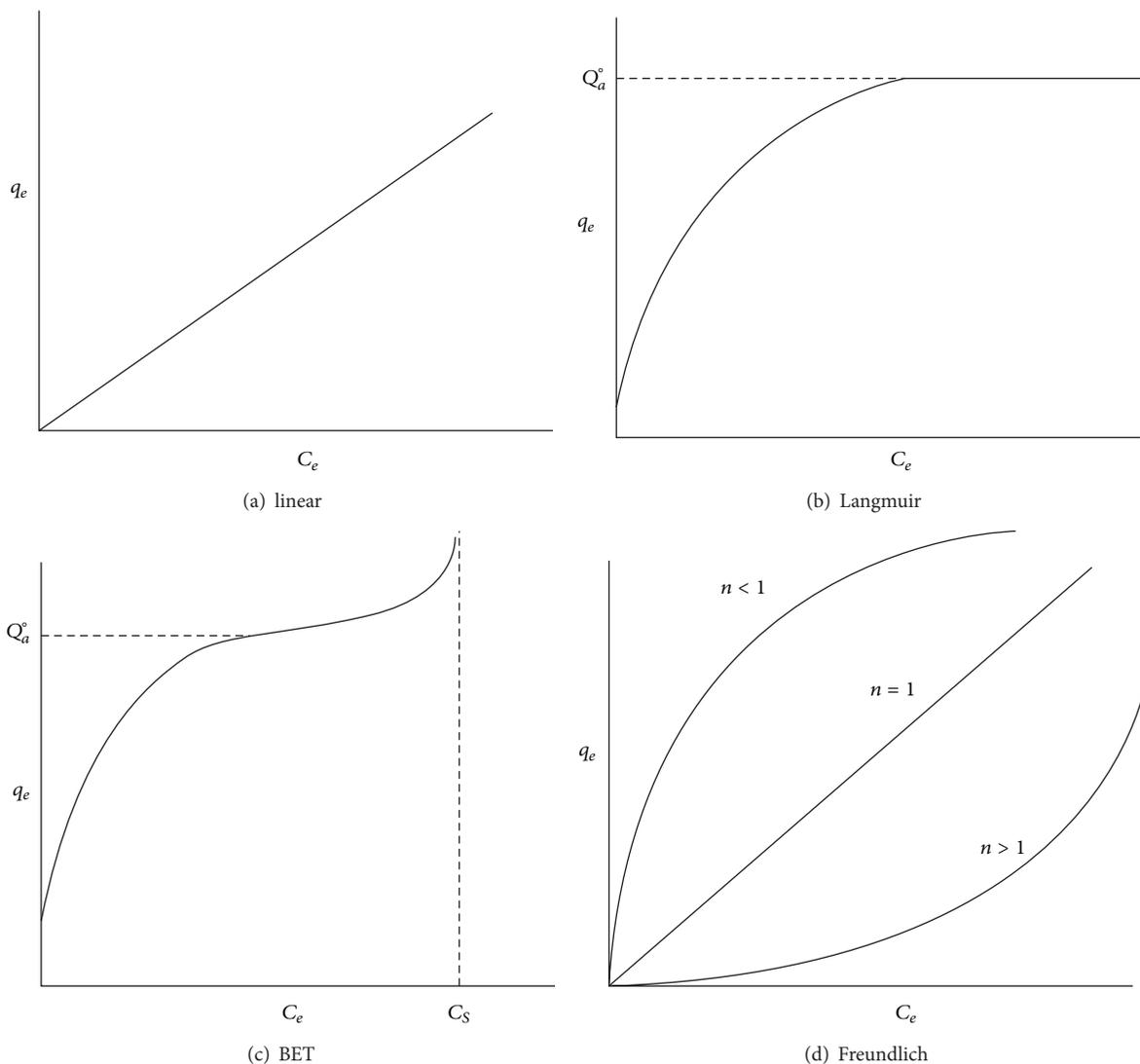


FIGURE 1: Different models of adsorption isotherm (a) linear; (b) Langmuir; (c) BET; and (d) Freundlich.

the linear, Freundlich, Langmuir, and Brunauer-Emmett-Teller (BET) are the most commonly used models, which are shown in Figure 1.

A number of researchers [7, 8, 29–31] reported that two nonlinear adsorption isotherms, namely, Langmuir and Freundlich, are the best models to explain the trend of adsorption based on the essence of adsorbents saturated with adsorbate after enough contact time.

Freundlich isotherm is an empirical equation. The nonlinear form of Freundlich's equation is written as [32]

$$q_e = K_f C_e^{1/n}, \quad (3)$$

where q_e is the amount of the solute adsorbed per mass unit of adsorbent (mg/g), C_e is the equilibrium aqueous pollutant concentration (mg/L), K_f is Freundlich constant, and n is Freundlich exponent, K_f is the indicator of adsorption capacity, and $1/n$ is a measure of intensity of adsorption.

Freundlich is an exponential equation which can be used to determine the values of parameters. The logarithm conversion is one way to determine Freundlich parameters. Therefore (3) converts to

$$\log q_e = \log K_f + \frac{1}{n} \log C_e. \quad (4)$$

By plotting $\log q_e$ against $\log C_e$, the values of Freundlich parameters can be determined. The slope of line is the exponent and the logarithm of intercept is the constant of Freundlich parameters.

Langmuir adsorption isotherm is given as

$$q_e = \frac{q_{\max} \times C_e \times K}{1 + C_e \times K}, \quad (5)$$

where q_e is the amount of the solute adsorbed per mass unit of adsorbent (mg/g); C_e is the equilibrium concentration of the aqueous phase (mg/L); and q_{\max} is the maximum

TABLE 2: Nitrate adsorption for different initial concentration.

C_0 (mgNO ₃ ⁻ /L)	C_e (mgNO ₃ ⁻ /L)	q_e (mg/g)	Percent of adsorption (%)
45	35	0.10	22.2
112.5	84	0.28	25.3
225	165	0.60	26.7
450	326	1.24	27.5

amount adsorbed. The parameters, q_{\max} and K , are Langmuir constants.

2.6. Calculation of Retardation Factor. Retardation factor is a dimensionless parameter in the governing equation of solute transport in porous media. It is defined as the relative ratio of water velocity to solute velocity [33]. Consider

$$R_d = 1 + \frac{\rho_b K_d}{n}, \quad (6)$$

where R_d is the retardation factor (dimensionless), ρ_b is the bulk density of soil sample (g/cm³), K_d is the adsorption or distribution coefficient (cm³/g), and n is the porosity (cm³/cm³).

The adsorption or distribution coefficient K_d can be estimated as

$$K_d = \frac{q_e}{C_e}. \quad (7)$$

3. Results and Discussion

3.1. Effect of Initial Concentration. The effect of initial nitrate concentration on equilibrium adsorption was investigated at different initial nitrate concentrations. Four different initial concentrations, mentioned in Section 2.2, were used to assess the capability of kaolin on nitrate adsorption. The percentage and the amount of nitrate adsorption at equilibrium concentration for all initial concentrations were calculated using (1) and (2), which are shown in Table 2.

Table 2 revealed that initial nitrate concentration has an effect on the amount of nitrate adsorption at equilibrium. The equilibrium adsorption values increased from 0.1 mg/g to 1.24 mg/g with an increase of initial concentration from 45 to 450 mgNO₃⁻/L, respectively. However, the increasing rate of nitrate adsorption decelerated with further increase in nitrate concentration. This behavior is due to the increase in the driving force of the concentration gradient, as further increase in the initial nitrate concentration overcomes the limitations of low adsorption by adsorbent [7]. Such phenomenon is common in a batch test with either constant adsorbent dose or varying initial adsorbate concentration or vice versa [34]. Therefore, it can be remarked that nitrate adsorption process at higher initial concentration is enhanced compared to the case at lower initial nitrate concentration. Percentages of nitrate adsorption at different initial nitrate concentrations are shown in Figure 2. The percentages of nitrate adsorption

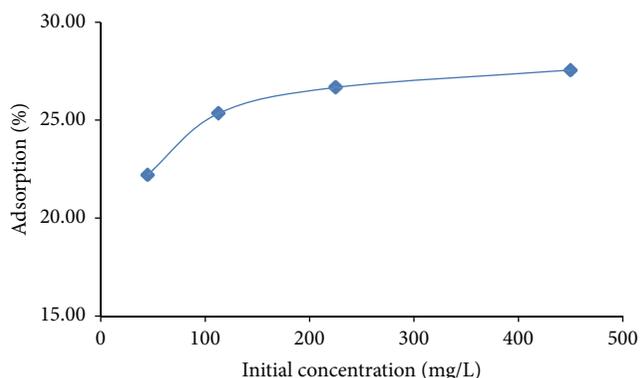


FIGURE 2: Percentage of nitrate adsorption (%) in aqueous phase, at $T = 25^\circ\text{C}$, $\text{pH} = 2$, $v = 20$ mL, $X = 2$ g, and different C_0 .

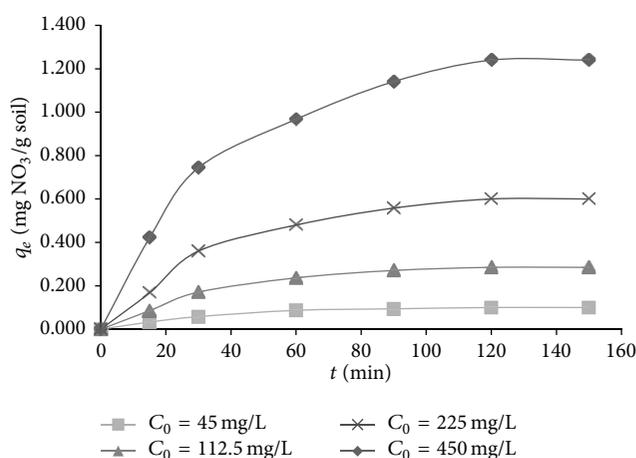


FIGURE 3: Effect of contact time on nitrate adsorption by clay kaolin in samples, at $T = 25^\circ\text{C}$, $\text{pH} = 2$, $v = 20$ mL, $X = 2$ g, and different C_0 .

in aqueous phase were approximately 22.2% and 27.5% for the lowest and highest initial nitrate concentrations, respectively. Figure 2 shows that the slope of curve in lower nitrate concentrations was steeper than at higher nitrate concentrations. Constant adsorbent, 2 g, for all tests and much availability of nitrate anion at higher concentrations have caused flattening of the slope of the curve.

3.2. Effect of Contact Time on Nitrate Adsorption. Water sampling was conducted at different times from the start of experimental test as mentioned in Section 2.3 to measure the nitrate adsorption by adsorbent at any nitrate concentration. Equation (2) was used to calculate the amount of nitrate adsorption at a particular time of water sampling. The amount of adsorptions, q_e , versus related times was depicted for different nitrate concentrations and present in Figure 3. The figure represents the effect of contact time on nitrate adsorption for various samples.

The rate of nitrate adsorption (Figure 3) can be divided into two parts; one represents rapid nitrate adsorption and

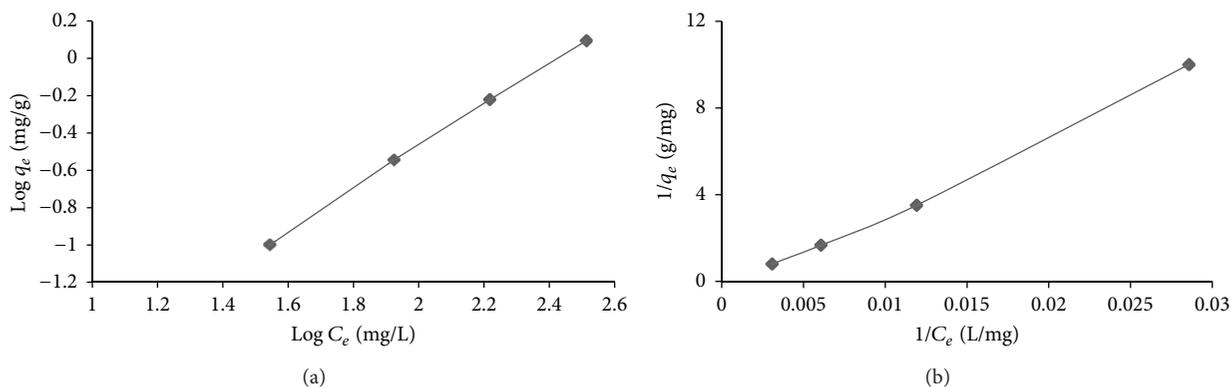


FIGURE 4: (a) Linearized Freundlich; (b) Langmuir adsorption isotherm on experimental data, at $T = 25^{\circ}\text{C}$, $\text{pH} = 2$, $v = 20\text{ mL}$, $X = 2\text{ g}$, and different C_0 .

another represents slow nitrate adsorption. Duration time for rapid nitrate adsorption was approximately 50 minutes. During this period, the tendency of adsorbent to adsorption was high and therefore, the slope of adsorption curve was steep. Slow adsorption rate was between 50 and 120 minutes. During this period, the slope of adsorption curve was gradually flattened, and the nitrate adsorption was gradually decreased until reaching zero. This usually occurs as the external surface of kaolin is rapidly saturated by nitrate anion and therefore the adsorption rate reduces [7]. Approximately, 120 minutes was taken for nitrate adsorption into kaolin and reaching equilibrium in the experiment.

3.3. Adsorption Isotherm. Based on the method mentioned in Section 2.5 and using the data given in Table 2, the plots of $\log q_e$ versus $\log C_e$, and $1/q_e$ versus $1/C_e$ were depicted to show the Freundlich and Langmuir isotherms, respectively, in order to find the best model of adsorption isotherm that can describe experimental data (Figure 4). The results showed that Freundlich adsorption isotherm had better matching on data with $R^2 = 0.99$ than Langmuir adsorption isotherm with $R^2 = 0.97$. Linearized Freundlich adsorption isotherm was used to calculate K_f and n which is shown in Figure 4(a). The parameters of Freundlich's equation obtained from Figure 4(a) were 0.0018 and 0.887 for K_f and n , respectively. Freundlich equation based on mentioned conditions and criteria to calculate nitrate adsorption capacity was obtained as

$$q_e = 0.0018C_e^{0.887}. \quad (8)$$

The value of $1/n$ in the current study was 1.127. The intensity of adsorption, $1/n = 1$, means reaction between solute and adsorbent is linear; $1/n < 1$ means the shape of nonlinear adsorption isotherm is convex and adsorption is unfavorable at lower equilibrium concentration; and $1/n > 1$ means the shape of nonlinear adsorption isotherm is concave and the adsorption is more favorable at lower equilibrium concentration. Therefore, the value of $1/n$, 1.127, indicates that adsorption of nitrate by kaolin particles was dominated in aqueous phase.

3.4. Retardation Factor. A physical scale model was constructed in the Hydraulics Laboratory at Faculty of Civil Engineering, Universiti Teknologi Malaysia, for laboratory experiment. Experimental tests were conducted using the above mentioned laboratory model with a constant seepage velocity rate and initial nitrate concentration. The values of seepage rate and initial nitrate concentration were kept constant at $v = 0.0047\text{ cm/s}$, $C_0 = 112.5\text{ mgNO}_3^-/\text{L}$, respectively. Mixed sand (85%) and kaolin (15%) were used as porous medium. The porosity, bulk density (ρ_b), hydraulic conductivity, and longitudinal dispersion coefficient values of the media were obtained through laboratory soil column experiments. The results showed that the porosity of the media was $0.41\text{ cm}^3/\text{cm}^3$, bulk density was 1.5 g/cm^3 , hydraulic conductivity was 0.0153 cm/s , and longitudinal dispersion coefficient was $0.0037\text{ cm}^2/\text{s}$. The value of absorption or distribution coefficient (K_d) was calculated as $0.918\text{ cm}^3/\text{g}$ using (7). Finally, substituted laboratory data in (6) showed that the retardation factor was approximately 4. The result showed that kaolin under experimental condition can retain nitrate in soil. Therefore, it can be remarked that kaolin can play a significant role in nitrate adsorption to prevent leaching in groundwater.

4. Conclusion

Batch experimental tests on nitrate adsorption showed that kaolin particles are capable of adsorbing nitrate pollution and retaining it in soils. The mean percentage of nitrate reduction in solution was 25% for initial concentration. Nitrate contamination was retarded in porous medium by kaolin particles. The retardation factor was calculated approximately 4 under acidic condition. It can be concluded from this study that existing kaolin particles in soil can retain and attenuate nitrate pollution to prevent groundwater contamination which in turn can help to achieve sustainability in groundwater use. Finally, it can be remarked that easy availability of various types of clay kaolin and their ability to adsorb and retain nitrate [34, 35] will create more interest to develop new natural adsorption method of pollutant removal from solution.

Conflict of Interests

The authors declare that there is no conflict of interests regarding the publication of this paper.

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