Denitrification of water using a low-cost adsorbent

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Abstract. The recent studies about water quality indicated a clear increase in the concentrations of many pollutants in the sources of freshwaters, such as nitrate, phosphate, and inorganic pollutants. This increase is attributed to the expansion of the global population, industries, and climate changes. These studies represented a call for the need for efficient treatment for water and wastewater. The current study investigates the possibility of using activated red mud (ARM) as an adsorbent to eliminate nitrates from water/wastewater. The operational parameters such as treatment times, pH, and doses of ARM were tested to assess their effects on the Nitrate removability by ARM. The optimum treatment times, pH, and dose for best removal within this study were 60 min, 7, and 75 g/L for treatment times, pH, and doses, respectively. Freundlich and Langmuir isotherm models and kinetic models including the pseudo-first-order, the pseudo-second-order, and interparticle diffusion using non-linear regressions were utilized to analyze the observed data which were gained from batch equilibrium tests. The obtained results revealed that the equilibrium data have good and reasonable fitness and agreement with the Langmuir isotherm models. The highest adsorption capacity of ARM was 1.79423925mg/g. The maximum percentage of removal was found to be about 71% at ambient temperature.

1. Introduction
Pollution of water sources became a common phenomenon since the industrial revolution due to the increase in the industrial activities that consume a significant amount of water and discharge a large volume of this water as wastewater to the sources of water [1, 2]. A similar increase was observed in the agriculture activities and human population that also consume a significant amount of water and discharge a large volume of this water as wastewater to the sources of water [3-5]. For example, the irrigation process usually
results in the discharge of huge volumes of wastewater that is rich in nitrate, phosphates, pesticides, and metals [6-8], while industrial activities result in the production of wastewaters that are rich in a countless number of pollutants; such as phenols [9], fluoride [10, 11], dyes [12-14], metals [15-17], complex organic matter [18], phosphate/nitrate [19-23], pathogens [24-26] and inorganic pollutants [27, 28]. Besides the activities, the climate changes, which mainly result from man-made sources [29-31], have resulted in a serious increase in water consumption [32-34] and wastewater pollution [35-37]. For example, the cement industry is the main source of climate change-inducing gases [38-41], and basic wastewaters [42-46]. Among these pollutants, nitrate and phosphate brought significant attention due to their direct effects on water quality and aquatic life [23, 47].

Generally, moderate concentrations of nitrate are found in most of the natural water but, the overusing of fertilizers increases its concentration. The presence of nitrate in high concentration leads to detrimental effects on the environment such as eutrophication in water in addition to causing infectious diseases [47]. Many certain diseases could be caused by excessive amounts of nitrates in drinking water such as blue-baby diseases (methemoglobinemia) and other diseases [48]. To avoid the high nitrate intake effects and keep public healths; the World Health Organization (WHO) has limited the acceptable nitrate concentrations in drinking water to be 50 mg/L [8]. The traditional treatment approaches, such as coagulation, chlorination, and filtrations are not efficient for nitrate removals from water. For that reason, other techniques have been utilized to eliminate the excess amounts of nitrate from water such as biological remediation, adsorption process, ion exchange, electrodialysis technique [8, 48]. Additionally, using many various adsorbents like activated carbon, synthetic ion exchanger, and slag for nitrate elimination [15, 47].

Recently, considerable researches have been carried out to investigate various types of low-cost materials as adsorbents such as the bark of trees, alum sludge, red mud, and others to get rid of toxic substances. These works focused on the adsorption method because it is cheap, reliable and its by-products could be recycled instead of disposing into expensive landfills [49-51]. Red mud is a metal oxide adsorbent, it has a special structure with many various metal oxides, and these oxides have hydroxylated surfaces which develop a charge in a humid environment. It is unwanted intermediates that are produced during alkaline-leaching of bauxite in the Bayer process where half-ton of this strongly alkaline material (pH≈12–13) is annually produced in Seydis¸ehir Aluminum Plant [52]. The current paper aims at the investigation of the potential use of the activated red mud for nitrate removability from drinking water by adsorption. Also, the properties of Nitrate batch adsorption are described in this work focusing on different operational parameters; such as treatment time, pH, and dosage of the adsorbent. Kinetics and isotherms were applied to analyze the observed data.

2. Equilibrium of sorptions processes
The sorption models relate the sorbed chemicals onto the surfaces of the adsorbents \( (q_e, \text{mg.g}^{-1}) \) with the final concentrations of these chemicals \( (C_e, \text{mg.L}^{-1}) \). It is calculated at specified values of pH and temperatures and shows the fitnesses with other isotherm models, such as Freundlich and Langmuir [53].

2.1. Freundlich Model
This model can be applied for sorption of multilayer and surfaces of heterogeneous and its general formula could be represented as follows [53]:

\[
q_e = K_F C_e^{1/n}
\]  

(1)

where \( K_F \) is an indicator for the highest amount of sorbed pollutants onto the adsorbent, and \( 1/n (<1) \) is the sorption intensities.
2.2. Langmuir Model
The following developed formula of the Langmuir model is capable of the homogenous surface and monolayer sorptions [53]:

\[ q_e = \frac{q_{\text{max}} b C_e}{1 + b C_e} \]  

(2)

where \( q_{\text{max}} \) indicates the highest adsorption capacities (mg.g\(^{-1}\)); and \( b \) is the intensity of the contaminant to the solid phase.

3. Kinetic Models
To design the suitable sorption process, the rates of the transferred solutes from the solution to the solids phase is utilised for this purpose. These rates are estimated using:

3.1. Pseudo-first-order model
The following equation is applied to describe the sorptions rates as a function of the time [54]:

\[ \frac{dq}{dt} = k_1 (q_e - q_t) \]  

(3)

By substituting \( q_t = q_e \) at \( t = t \) and \( q_t = 0 \) at \( t = 0 \), Eq.3.3 is integrated to produce the following model:

\[ \ln(q_e - q_t) = \ln q_e - k_1 t \]  

(4)

The above equation could be rearranged as follows:

\[ q_t = q_e (1 - e^{-k_1 t}) \]  

(5)

where \( q_t \) (mg.g\(^{-1}\)) is the amounts of the sorbed pollutant on the solid matrix at the time of \( t \), \( q_e \) is the equilibrium time, and \( k_1 \) denotes the rate constant of Pseudo-first-order (1.min\(^{-1}\)).

3.2. Pseudo-second-order model
The main assumption used to derive this model is the monolayer of contaminants attached to the surfaces of the sorbents, and the second assumption is the same sorption energy for sorbents and not interacted sorbed chemicals. This model is shown in the following equation:

\[ \frac{dq}{dt} = k_2 (q_e - q_t)^2 \]  

(6)

where \( k_2 \) is the rate constant of Pseudo-second-order (g/mg min).

Eq.5 is integrated with the same assumptions of the previous model and the Pseudo-second-order equation can take the forms in Eq.6:

\[ \frac{1}{(q_e - q_t)} = \frac{1}{q_e} + k_2 t \quad \text{or} \quad q_t = \frac{t}{\left( \frac{1}{k_2 q_e^2} + \frac{t}{q_e} \right)} \]  

(7)

4. Materials and Methods

4.1. Adsorbent and contaminant
Red mud sample was collected from a local aluminium plant in Iraq. Generally, it has a grain size of less than 10 μm, and 21.1m\(^2\)/g surface area, the chemical composition of the collected sample, in Table 1, was measured via an X-ray fluorescence spectrometer (XRF).

The raw alkaline red mud was sanitized by dissolving the red mud in distilled water with 2/1 liquid to solid ratio on a weight basis, and stirring it continuously until the equilibrium pH reached 8.0–8.5 and then it was dried in an oven at 105 °C. After that, the red mud was subjected to acidic activation process as follows: 10
g of the prepared mud was washed by water and dried then it was boiled in 20 wt.% HCl with 200 ml volume for 20 minutes. After the filtration process for the produced acid slurry, the residue was washed with deionized water to get rid of the remaining acid and soluble compounds of Al and Fe. After drying the residue at 40 °C as a final step, it was used in the experiments. The nitrates solutions have been diluted from the stock solution (1 g.L⁻¹) to the wanted concentrations. Specific ions-electrode (Mettler Toledo) was used to measure the concentration of nitrate using (2M (NH₄)₂SO₄) solution as an ionic strength adjuster (ISA) to eradicate the interference influence of complexions. The ISA solution was added at a ratio of 100:2. An Orion EA940 ion meter was used to measure the pH nitrate concentration.

| Table 1. Average physical and chemical composition of a red mud sample. |
|-----------------|-----------------|
| Oxide           | Composition (wt%) |
| Al₂O₃           | 16.8            |
| Fe₂O₃           | 58.7            |
| TiO₂            | 4.8             |
| SiO₂            | 4.9             |
| Na₂O            | 14.8            |

4.2. Batch Study

Batch tests were carried out to attend the single equilibrium and kinetic information for interactions between the sorbent and nitrate. The optimum conditions such as treatment Time, initial pH, and adsorbent dosage, for initial concentration (Cₒ) (100 mg/L) which attend high efficiency for the remediation process will be specified by these data. The batch tests include preparing a series of flasks with 250 mL volume and polluted water, with a volume of 100 mL and an initial concentration of 100 mg/L, which is put in each flask. Different doses of adsorbent were added during the agitation process, where the flasks were agitated at 150 rpm for 60 minutes. Afterward, the filtration process for flasks’ solutions was implemented to eliminate the solid particles. To measure the remaining nitrate concentration (Cₑ) in the filtered solution, an ion electrode (Mettler-Toledo) was used for this purpose. The mass balance principle could be employed to calculate the adsorbed quantity of the pollutant per unit mass of the adsorbent. Adsorption study was carried out with the contact time range (15-120 min), pH (3-10). For the best conditions, the amounts of adsorbed pollutants onto the adsorbent (qₑ) were calculated by (Wang et al., 2009):

\[ qₑ = \frac{(Cₒ - Cₑ)V}{m} \]  

where \( V \): the volumes of water in the flasks (L) and \( m \): the sorbent mass (g) in the flasks. The adsorption isotherm is drawn between calculated \( qₑ \) and \( Cₑ \). The adsorption efficiency (\( R \)) is measured by the differences between \( Cₒ \) and \( Cₑ \) with respect to the initial concentration as follows:

\[ R = \frac{(Cₒ-Cₑ)}{Cₒ} \times 100 \]  

5. Results and discussion

5.1. Dosage of ARM

The influences of adsorbent doses on Nitrate adsorption were investigated where, the amount of ARM was varied from 15 to 75 g/L for batch tests at 20±1°C, while the other parameters (\( Cₒ, \) pH, agitation speed, contact time ) were remained constant at 100 mg/L, 7, 150 rpm and 1 hour respectively. The Nitrate removability is represented in figure1 as a function of various doses of ARM. An enhancement in Nitrate removability was noticed as the adsorbent dosage increased from 15 to 75 g/L for a constant initial concentration of Nitrate. This explains that the maximum adsorption starts after adding a certain amount of
adsorbent (75 g/L), and hence the amount of Nitrate engaged to the ARM. Even with adding extra amounts of ARM, the residual concentration of Nitrate in solution remains constant.

5.2. Equilibrium Time

one of the batch test requirements keeping the treatment time at a constant value to achieve the equilibrium concentrations. The influence of treatment time on Nitrate removability using 45 g/L of ARM for batch tests at 20±1°C is shown in figure 2. This figure reveals that a dramatic increase in Nitrate removability has been observed with treatment time growth. Obviously, at the initial step, the adsorption rate was fast but after that, it began to slow down gradually. The decreasing sorption places on the adsorbent surfaces explain this slowness of the adsorption process. At a contact time of 60 min, about 71% Nitrate removal was achieved. However, by increasing the treatment time (more than 60 min), a fixed Nitrate concentration was noticed. This means that the residual Nitrate concentrations did not change remarkably after a contact time of 60 min and up to 120 min. This contact time was used to conduct the sorption experiments in other batches.

![Figure 1](image1.png)

**Figure 1.** Effects of ARM dosage on the removals efficiency of Nitrate, time: 60 minutes, agitation speed: 150 rpm, pH:7, temperature: 20±1°C, initial nitrate concentration: 100 mg/L

![Figure 2](image2.png)

**Figure 2.** Nitrate removability by ARM as a function of treatment time, Agitation speed: 150 rpm, dose: 45 g/L, temperature: 20±1°C, pH:6, initial nitrate concentration: 100 mg/L.
5.3. Initial pH of the solutions
The pH of the solutions strongly controls the adsorption of anions where OH⁻ ions engage with anion adsorption, therefore the neutral or acidic mediums are the favourable environments for the adsorption process. The pH of the solution is an influential variable as it has a vital role in controlling the adsorption process of the adsorbent with an aqueous interface. The study of nitrates adsorption on the activated red muds was implemented by varying the pH values from 3 to 10 and the gained outcomes are shown in figure 3. Clearly, nitrate elimination grew in the pH range 3–7. It can be noticed that the attained outcomes for maximum adsorption have a correspondence with the outcomes of the study of nitrate elimination using sepiolite and activated sepiolite (about pH 7) [8]. Figure 3 also shows that the nitrate removability declines as the solution turns to be alkaline (pH above 7), this decline is attributed to the increase in the competition between nitrate and hydroxide ions. This confirmed by the high measured OH⁻ concentration in solutions with pH values more than 7.

Figure 3. Effects of initial pH on Nitrate removability by ARM, Time: 60 minutes, agitation speed: 150 rpm, pH:7, temperature: 20±1 °C, initial nitrate concentration: 100 mg/L.

5.4. Kinetic and Isotherm analysis
According to figure 4, good fitness can be observed between the kinetic data and Pseudo-first-order, Pseudo-second-order, and Weber-Morris models. Non-linear regression techniques in Microsoft Excel 2016 are used in these fittings to calculate the constants of these models as shown in Table 1. Clearly, the sorption of nitrate fits the Pseudo-first-order model. This indicates that the major mechanism for the sorption processes is physical sorptions. Due to the difficulty of achieving the characterisation of the sorption mechanism depends on the kinetic models mentioned above, this target was commenced by the intra-particles diffusions model that is based on the theory of Weber and Morris (1962) [20]. This model is an empirical formula where sorbed amount is varied as a function of $t^{1/2}$ rather than $t$ as shown in the following equation:

$$q_t = k_{int}t^{0.5} + C \quad (10)$$

where $k_i$ (mg/g hr$^{1/2}$): the constant of step $i$ and it represents the slope of relationship that relates $q_t$ to $t^{1/2}$. $C$ represents the intercepts of step $i$ where $c$ indicates the thickness of the boundary layer. This means that the effective boundary layer becomes greater when the value of the intercept grows larger. The intra-particle diffusion takes place when the relationship of $q_t$ versus $t^{1/2}$ becomes linear. If the linear plot goes via the origin, the rate-determining processes are characterised by the intra-particle diffusions; otherwise, other mechanisms must be included with it. In general, the mechanism of adsorption includes the following steps:
(i) mass transfer of adsorbates materials from the bulk-phase to particle surfaces, (ii) adsorption processes on surface sites, and (iii) intraparticle diffusions of the adsorbates molecules to an adsorption site by pore diffusions and/or surfaces diffusions. Step (ii) is often assumed to be extremely fast, as a result, that the large molecules, with long contact times to equilibrium, are always adsorbed by diffusion as an external film resistance and/or internal diffusion mass transport or intraparticle diffusion control it. To find if intraparticle diffusion controls an adsorption process, a classical approach is used for that by plotting the adsorbed amount versus the square root of time, $t^{1/2}$. If the plot is linear and goes through the origin point, that means the intraparticle diffusion is the controller of the adsorption process. Figure 4 shows the adsorbed amount of nitrate as a function of $t^{1/2}$ for the adsorbents. It can be noticed that the observed data follow the intraparticle diffusion model, with a linear section that doesn’t go through the origin which refers to the absence of intraparticle diffusion control on the tetracycline adsorption on these adsorbents. Sorption tests are carried out under conditions of pH = 7, speed = 150 rpm and contact time = 60 min for different concentrations. The Freundlich and Langmuir isotherms are shown in Table 2 that lists their constants that are calculated by using nonlinear fitting of these models with sorption measurements through the application of the ‘Solver’ option in the Microsoft Excel 2016. Based on this figure and coefficient of determination ($R^2$) in Table 2, the Langmuir isotherm is better than the Freundlich model in the description of sorption measurements.

| Table 1. Constant of kinetic and isotherm models for the sorption of nitrate. |
|---------------------------------|---------|---------|
| Model                  | Parameter | Value       |
| Frendlich              | $K_F$ (mg/mg)(L/mg)$^{1/n}$ | 0.995835279 |
|                       | $n$       | 6.967956633 |
|                       | $R^2$     | 0.750404162 |
|                       | SSE       | 0.119342171 |
|                       | $q_{max}$(mg/g) | 1.79423925 |
|                       | $b$(L/mg) | 0.57864471 |
| Langmuir              | $R^2$     | 0.943493455 |
|                       | SSE       | 1.785421191 |
|                       | $q_e$(mg/g) | 1.673476391 |
|                       | $k_1$(1/min) | 0.045181434 |
| Pseudo-first order    | $R^2$     | 0.947017909 |
|                       | SSE       | 0.012180156 |
|                       | $q_e$(mg/g) | 1.979559369 |
|                       | $k_2$(g/mg min) | 0.027566571 |
| Pseudo-second order   | $R^2$     | 0.866572687 |
|                       | SSE       | 0.028804745 |
| Intra-particle diffusion | $k_{int}$(mg/g min$^{0.5}$) | 0.1095       |
|                       | $R^2$     | 0.8145      |
To enhance the removal of nitrate using the activated red mud could be enhanced by modifying the surfaces of the mud particles by adding some chemical that has good efficiency for adsorption of nitrate. Additionally, sensors, like those used by other researchers [55-58], could be used to monitor the saturation status of the particles’ surfaces or to monitor the residual concentrations of nitrate in the solution.

6. Conclusion

The possibility of utilising Activated red mud (ARM) to eliminate Nitrate from aqueous solutions by adsorption was investigated. The Activated red mud (ARM) shows a good efficiency for Nitrate removability, the maximum adsorption capacity of ARM was 1.79423925mg/g. The removability process was remarkably affected by the contact time, initial pH of the solution, and adsorbent dosage. The process kinetics was found to fit the Pseudo-first order rate equation. The next studies in this field of water and wastewater treatment technologies could be focused on the enhancement of the properties of the adsorbents’ surfaces to increase the adsorption capacity. Also, some studies could be focused on the development of smart adsorption units based on sensors.

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