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Adsorption of Vanadium from Iraqi Crude Oil on Nano Zeolite and Alum Sludge

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Abstract

Vanadium is considered one of the heavy metals that can be found in crude oil. Since vanadium is toxic to specific refining catalysts, the amount of vanadium in crude oil can affect its selling price. At the same time, it is responsible for equipment corrosion when temperatures reach high enough. This paper investigates vanadium adsorption by two adsorbents named nano-zeolite and alum sludge (residual from a sedimentary tank in a water treatment plant). A batch adsorption unit was selected to determine the effect of different operating factors such as adsorbent dose, agitation speed, temperature, and contact time in vanadium removal. The highest removal efficiency was 89% and 67%, performed at 2 g of nano-zeolite and 4.5 g of alum sludge, respectively, at 500 rpm agitation speed and 80˚C temperature with a contact time of 480 min for nano-zeolite and 420 min for alum sludge. Adsorption isotherm data was fitting to Langmuir and Freundlich models with R^2 of 0.9665, 0.9727 for nano-zeolite and 0.9907, 0.9964 for alum sludge, respectively. Pseudo-first-order and pseudo-second-order models show excellent conformity to adsorption kinetic data with R^2 of 0.9617, 0.96624 for nano-zeolite, and 0.97494, 0.96515 for alum sludge, respectively.

Keywords: Adsorption; Alum sludge; Batch mode; Nano-zeolite; Vanadium

1. Introduction

As conventional light oil reserves dwindle worldwide, more heavy oils are being produced and refined [1]. Heavy oil is more complex to extract and process due to its higher viscosity and density, requiring advanced technology and higher costs to produce fuels and petroleum products. However, heavy oil is a vital energy source, especially as global demand for fossil fuels increases [2]. However, this petroleum wealth includes abundant metals and complex materials that may harm oil refinery equipment and machinery [3]. Metals, especially nickel, iron, copper, vanadium, etc., are abundant in heavy oils, and these oils also have nitrogen, oxygen, and sulfur hetero atoms [4]. The development of effective methods for the pre-concentration and purification of petroleum metal is a necessary condition for further progress in both fundamental and convenient research regarding the characteristics of these compounds and their role in the genesis and refining of oil. This condition must be met before further progress can be made in either area [5]. Vanadium is one of many heavy metals found in varying amounts in crude oil, and it negatively affects oil refining processes. Refining oil containing vanadium can cause equipment corrosion and damage catalysts used in refining processes, leading to higher maintenance and production costs [6]. For example, since all metallic impurities in crude oil are in liquid at temperatures below 650 °C, they contribute to the formation of corrosive compounds during combustion. Combustion-related corrosion of diesel engine valves and turbos, gas turbine blades, and boiler heat exchangers can be avoided by neutralizing these compounds (besides solid encrustations) [7]. Therefore, removing vanadium and heavy metals from crude oil before refining is essential to maintain process efficiency and ensure the quality of the final products [8]. Many methods are used for recovering heavy metals from aqueous solutions or other fluids, such as coagulation-flocculation, membrane, ultra-filtration, advanced oxidation process, ion exchange, chemical precipitation, biological

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method, adsorption, … etc. [9]. Each of these methods has obstacles and drawbacks to treating heavy metals, like high cost, needing pre-treatment, being unable to treat low concentrations, requiring a wide area, or consuming high energy [10]. Conversely, adsorption technology effectively and efficiently treats a milieu contaminated with heavy elements such as soil, water, air, and crude oil [11]. Adsorption has proven its ability to remove many pollutants as well as heavy elements, such as dyes [12], biological stains [13], organic materials [14], inorganic toxins [15], pesticides [16], drugs [17], medicines [18], hardness [19], and eutrophication elements [20], etc. This high treatment capacity is the efficiency of adsorption media, characterized by their high surface area and unique properties such as activated carbon, alumina, and zeolite [21]. Despite the benefits obtained from using this technology, the progress of work on it has revealed some of the negatives it includes, including the high cost of the known adsorption media and the necessity of reactivating them after the accumulation of pollutants on their surface in addition to losing part of their mass with each reactivation process [22]. These obstacles prompted researchers and specialists in the environmental field to search for effective and cheap alternatives to produce common adsorption media or use them directly as adsorption media [23]. Among the most important alternatives that have attracted attention as promising materials that have shown clear efficiency in addition to their low cost and negligible toxicity are agricultural and industrial wastes such as rice husks [24], banana peels [25], orange peels [26], lemon peels [27], mandarin peels [28], watermelon rinds [29], eggshells [30], sunflower hulls [31], used tea leaves [32], tree leaves [33], aquatic plants such as the water hyacinth [34], algae [35], aluminum foil [36], etc. The accumulation of toxic waste from these materials after the adsorption process has led to the development of a sustainable approach to waste management, which is the concept of zero residue level, which is based on considering these wastes as raw materials for preparing useful materials according to the compounds or elements they contain, such as acetone [37] or bioethanol [38] or as a concrete additive [39] or as a radiation absorbent in hospitals [40] or as a natural fertilizer [41] or as a catalyst [42] or as toxic material for undesired animals [43] or as a rodenticide [44] or nanomaterial [45]. Because of the danger of vanadium to devices and equipment in oil refineries, the current investigation aims to study the ability of two types of traditional and non-traditional materials to directly absorb heavy elements from crude oil. The efficiency of the process was studied using a batch adsorption system and at different operating conditions to determine the capacity of nano-zeolite and alum sludge as media to remove vanadium from Iraqi crude oil.

2. Experimental Work

2.1. Crude Oil Used

The composition of crude oil tested by spectral M. Table 1 illustrates the characteristics of crude oil.

2.2. Adsorption Materials

Nano-zeolite and alum sludge were adsorbents to remove vanadium from Iraqi crude oil. Table 2 shows the properties of nano-zeolite (from the manufacturer). Alum sludge collected from the sedimentary tank in the Baaqubah water treatment plant, dried at 105 ˚C for 24 hours, and grinded by a mill for particles the size of (1-400) µm. The composition of alum sludge was tested by energy-dispersive X-ray spectroscopy, as shown in Table 3.

Table 1. Characteristics of crude oil used in this study

Metal	Conc. (ppm)	Metal	Conc. (ppm)
Na	0.59	Si	0.29
K	0.13	Cr	0.42
Li	0.03	Ni	24.43
V	96.8	Cu	0.00
Mg	0.10	Al	1.41
Ca	0.21	Fe	0.94
Pb	0.91	Mn	0.00
Zn	0.02	C	331

Table 2. Nano Zeolite properties (From the manufacturer)

Property	Specifications
Appearance (color)	White
Appearance (form)	Powder
Surface area	<186 m^2/g
Formula weight	294.19 g/mol
SiO_2 content	~ 6 %
Al_2O_3 content	~ 48 %
NaO content	≥ 0.2 %
Pore volume	≥ 0.16 cm^3
Particle size	≤ 100 nm

Table 3. Alum sludge composition

Components	Percentage (%)
Al_2O_3	40.1
Fe_2O_3	21.7
SiO2	10.8
CaO	6.4
K_2O	5.3
Na_2O	4.6
MgO	3.5
SO_3	1.3
P_2O_5	0.8
Others	6.3

2.3. Batch Adsorption Experiments

Various doses of nano-zeolite in experimental work ranged from 0.1-2.5 g/50 ml of crude oil. The alum sludge used in this study ranged from 0.1-5.0 g/50 ml of crude oil. The adsorbents were mixed with crude oil samples with a vanadium concentration of 96.8 ppm. The adsorption process is done for the samples (adsorbents and crude oil) in a shaking water bath at an agitation speed of 500 rpm. Various contact times ranging from 10-480 min were used to estimate the effect of time on vanadium removal. The sample was later filtered, and spectroil M measured the residual concentration of vanadium with a calibration curve prepared according to [46]. The efficiency removal is estimated by equation (1), while the capacity of adsorption is determined by equation (2) at equilibrium:

$$\%R = \frac{C_0 - C_e}{C_0} \times 100 \quad (1)$$

$$q_e = (C_0 - C_e) \times \frac{V}{m} \quad (2)$$

Where %R percent removal of vanadium; C_0 and C_e: the concentration of vanadium before and after adsorption, measured by (ppm); V: the volume of crude oil, measured by (ml); m: is the mass of adsorbent measured by (g); q_e: the capacity of adsorption measured by (mg/g).

3. Adsorption Isotherm Models

The isotherm model provides valuable information for comprehending the adsorption mechanism [47]. Isotherms describe the release or movement of a material from aqueous permeable media to a solid phase at constant pH and temperature described by an adsorption isotherm, which is a valuable curve [36]. Two models tested are Langmuir and Freundlich to describe the adsorption process. It was assumed in the Langmuir model that adsorption occurs exclusively at discrete sites, without any interfaces between the molecules that have been adsorbed, and that the adsorption energy is constant for all molecules, independent of the amount of surface area exposed [28]. The linear equation for this model is expressed in equation (3):

$$\frac{C_0}{q_e} = \frac{1}{q_m b} + \frac{C_0}{q_m} \quad (3)$$

Where: qm: The maximum monolayer adsorption capacity (mg.g^{-1}), b: is the adsorbate-adsorbent affinity coefficient (mg^{-1}).

The Freundlich isotherm model empirically describes heterogeneous systems [3]. The linear equation of this model is expressed in equation (4):

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

Where: K_f denotes Freundlich factor in (mg.g^{-1})/(l.mg^{-1})$^{1/n}$, n is intensity factor.

K_F and n result from intercept and slope, respectively, from the plot of $ln\ ln\ q_e$ and $ln\ ln\ C_e$.

4. Adsorption Kinetic Models

The term "adsorption kinetics" refers to the study of how quickly a substance can be adsorbed and how long the adsorbates remain at the solid/liquid boundary [10]. The mechanism and the adsorption rate are determined from the kinetic constants [19]. Many models theorized to explain the adsorption kinetic. These models take on various forms according to the adsorbent's nature, the adsorbates, and the intermolecular interactions [46]. In this study, two models tested to describe the process kinetics are Pseudo first order (PFO) and Pseudo second order (PSO). PFO assumes that the change in the rate of adsorbate uptake at a given reaction time is proportional in a straight line to the difference in concentration and rate of removal. Lagergren's PFO equation is expressed in equation (5):

$$\frac{dq_t}{dt} = K_L(q_e - q_t) \quad (5)$$

Where q_e and q_t are the equilibrium and time t metal ion adsorption concentrations in mg/l on the sorbent, and K_L in (min^{-1}) is the rate constant [38].

At the intervals [t from 0 to t] and [qt from 0 to qe], get:

$$log\ log\ (q_e - q_t) = log\ log\ q_e - \left(\frac{K_L}{2.303}\right) t \quad (6)$$

K_L and q_e can be calculated from the intercept and slope of the plot of t vs. log $(q_e - q_t)$, respectively. PSO rate equation is:

$$\frac{dq_t}{dt} = K_S(q_e - q_t) \quad (7)$$

Where: K_S is PSO rate constant in (g.mg^{-1}.min).

When the equation integrates from 0 to te; qt from 0 to qe, the linear equation yields:

$$\frac{1}{(q_e - q_t)} = \frac{1}{q_e} + K_S\, t \quad (8)$$

5. Results and Discussion

5.1. The effect of Adsorbents dose on Vanadium Adsorption

Fig. 1 shows the relationship between adsorbent dose and efficient removal of vanadium at an agitation speed of 500 rpm, temperature of 80˚C, and contact time of 480 min. From the figure above, it is apparent that the removal efficiency increased with the increase of adsorbent dose; the highest efficiency removal was achieved at 2 and 2.5 g of nano-zeolite with 89%, but 67% of vanadium was removed at 4.5 and 5 g of alum sludge. After that, there was no discernible rise in the amount of adsorbed substance, which most likely occurred because the adsorption sites on the adsorbent had reached their maximum capacity [26].

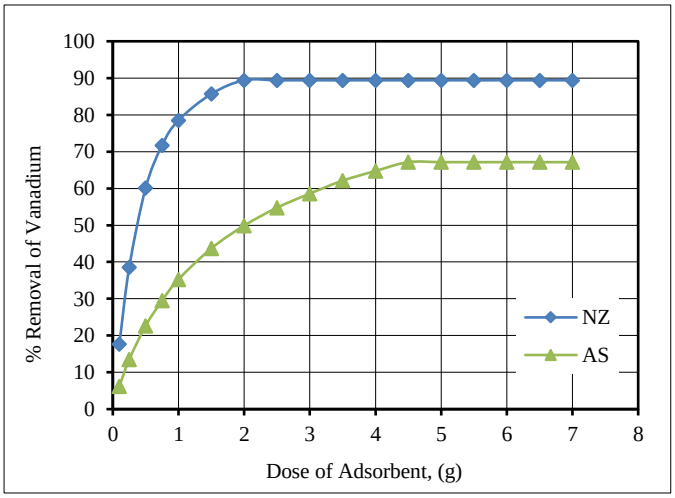

Figure 1. Effect of adsorbents dose on vanadium removal

5.2. The Effect of Contact Time on Vanadium Adsorption

Fig. 2 illustrates the relationship between contact time and vanadium removal efficiency. The relationship between the contact time and the removal efficiency is a direct relationship; that is, the longer the contact time, the higher the removal efficiency.

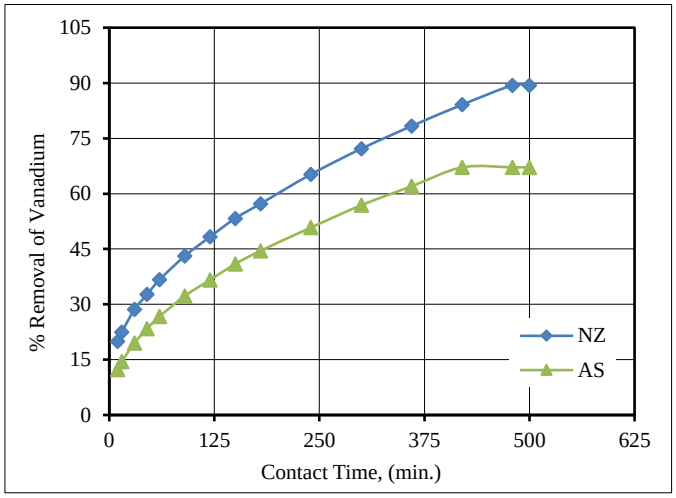

Figure 2. Contact time effect on vanadium removal

The highest removal efficiencies were satisfied at 480 and 500 min for nano-zeolite and 420 min for alum sludge, and no increase in the adsorption rate occurred after that time. It is noted that the increase in removal efficiency occurs quickly at the beginning; the availability of uncovered surface area on the adsorbent may be responsible for the faster removal rate at the beginning [15].

5.3. Vanadium Adsorption Isotherm Study

Langmuir isotherm model plot in Fig. 3 and Fig. 4 for nano-zeolite and alum sludge, respectively. Meanwhile, Fig. 5 and Fig. 6 show the Freundlich isotherm model for nano-zeolite and alum sludge, respectively. On the other hand, Table 4 shows the isotherm parameter for adsorbents.

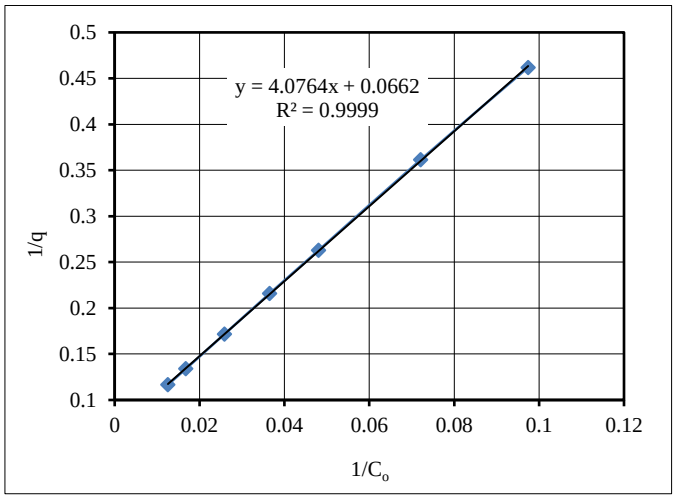

Figure 3. Langmuir model for nano-zeolite

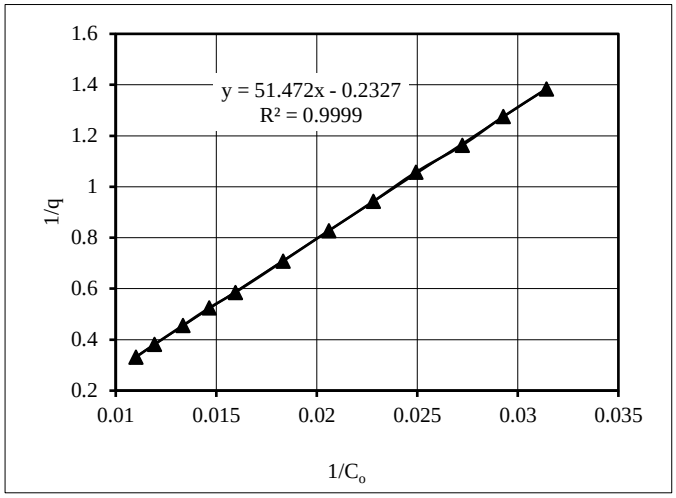

Figure 4. Langmuir model for alum sludge

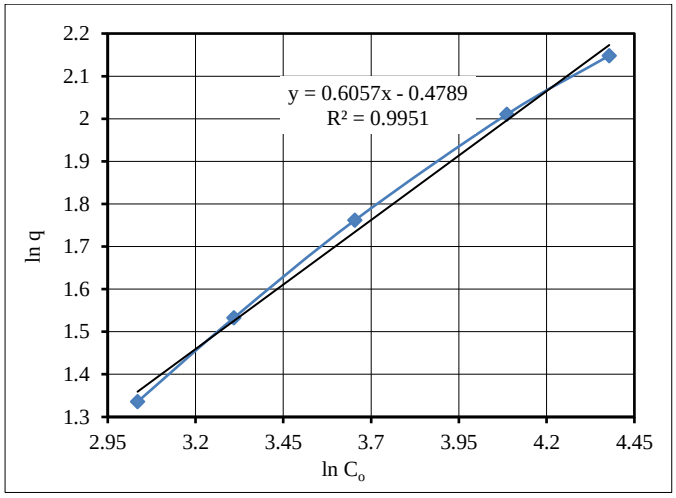

Figure 5. Freundlich model for nano-zeolite

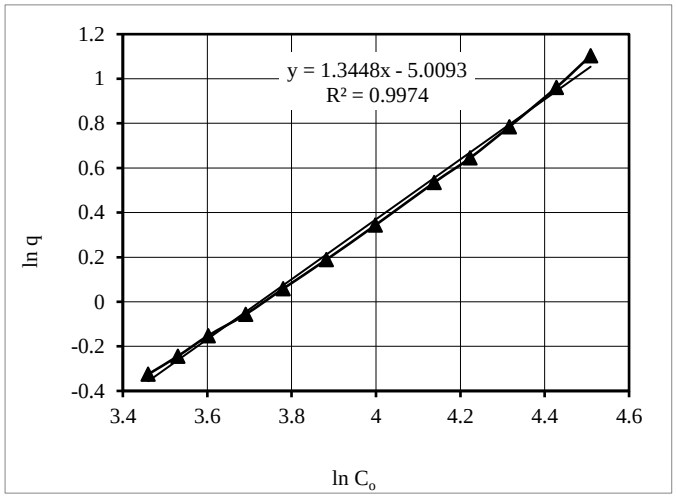

Figure 6. Freundlich model for alum sludge

It's clear that from Fig. 3-6 and Table 4, the maximum adsorption capacity achieved its highest value when vanadium adsorbed on nano-zeolite at a concentration of 20.9398 mg/g. Langmuir and Freundlich isotherm models are suited for adsorption capacity, with correlation coefficients of 0.9665 and

0.9727 for nano-zeolite and 0.9907 and 0.9964 for alum sludge, respectively. The nature of the isotherm shape for nano-zeolite is favorable because the value of R_L is between 0 and 1. At the same time, alum sludge is unfavorable due to the value of R_L being more significant than 1 [19].

Table 4. Isotherm Parameter for nano-zeolite and alum sludge

Adsorbent	model	Parameter	Value
Nano-Zeolite	Langmuir	q_{max}	20.9398
		K_L	0.0101
		R_L	0.5051
		R^2	0.9665
			0.1081
	Freundlich	K_F	1.3659
		n	0.9727
		R^2	
Alum Sludge	Langmuir	q_{max}	3.6750
		K_L	-0.0051
		R_L	1.0523
		R^2	0.9907
	Freundlich	K_F	0.000008
		n	0.7322
		R^2	0.9964

5.3. Vanadium Adsorption Kinetic Study

Two adsorption kinetic models analyzed the kinetics of vanadium adsorption: pseudo-first-order (PFO) and pseudo-second-order (PSO). Fig. 7 illustrates PFO for nano-zeolite and Fig. 8 for alum sludge. In a related context, Fig. 9 shows the PSO model for nano-zeolite, while Fig. 10 shows the PSO model for alum sludge. Table 5 show the results obtained from the kinetic study.

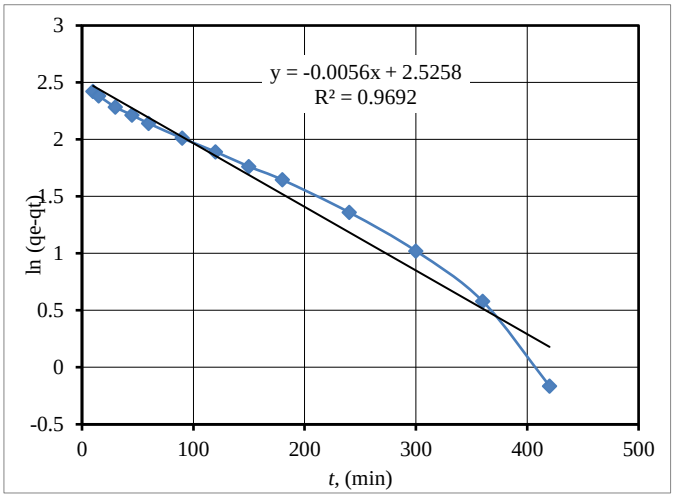

Figure 7. PFO model for nano-zeolite

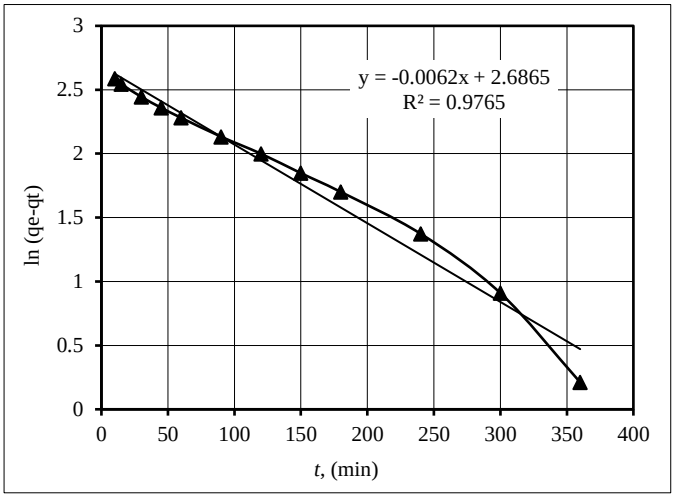

Figure 8. PFO model for alum sludge

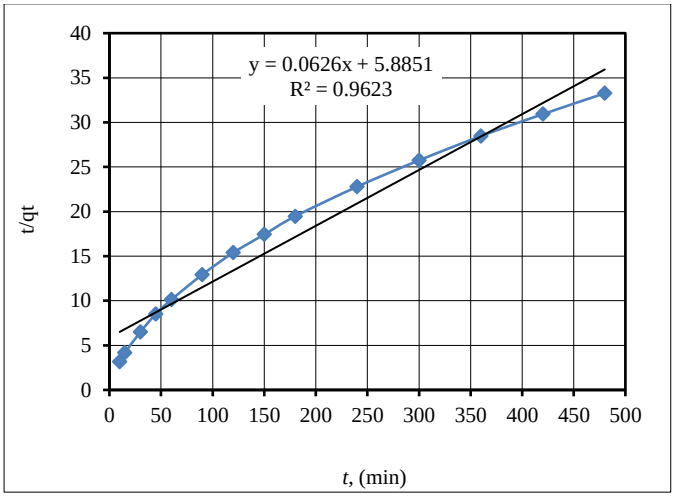

Figure 9. PSO model for nano-zeolite

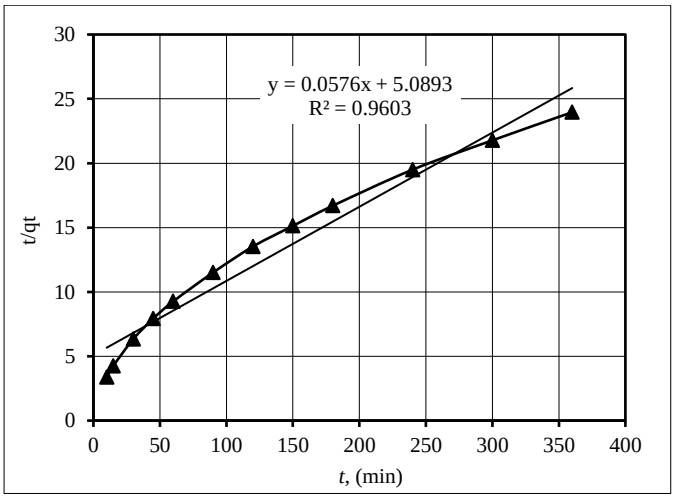

Figure 10. PSO model for alum sludge

Table 5. Adsorption kinetic parameters

Model	Parameter	Nano-Zeolite	Alum Sludge
PFO	q_e (mg/g)	12.5012	18.51201
	K_s (min^{-1})	-0.000013	0.009496
	R^2	0.96917	0.97494
PSO	q_e (mg/g)	16.4541	24.05951
	K_s ($g.mg^{-1}.min$)	0.00061	0.041563
	R^2	0.96624	0.96515

The adsorption kinetic of adsorbents shows excellent conformity to PFO and PSO with R^2 of 0.9617, 0.96624 for nano-zeolite, and 0.97494 and 0.96515 for alum sludge, respectively. For nano-zeolite and alum sludge, the theoretical adsorption capacities are the closest approximation to the experimental capacities of PFO. The theoretical adsorption capacity for nano-zeolite is nearest to PSO, but alum sludge is nearest to PFO.

5. Conclusions

The percent of vanadium removal increases with adsorbent dose (nano-zeolite and alum sludge), and the optimum dosage ranges from 2 - 2.5 g for nano-zeolite and 4.5 - 5 g for alum sludge for 50 ml of crude oil. The percentage of removal also increases with the increase in contact time. The optimum contact time was 480 for nano-zeolite and 420 min for alum sludge. The data fit the Langmuir and Freundlich isotherm models for two adsorbents. PFO and PSO provide the most accurate description of vanadium's adsorption onto nano-zeolite and AS. The isotherm shape has a favorable nature with $0 < R_L > 1$ for nano-zeolite but an unfavorable nature with $R_L > 1$ for alum sludge. The batch adsorption system may be replaced with a fixed adsorption system to allow more contact between crude oil and adsorbents. Alum sludge is a low-cost adsorbent that can be reused to remove vanadium from industrial wastewater from factories that use vanadium in their manufacture.

Conflict of interest:
The authors declare that there is no conflict of interest regarding the publication of this paper.

Author Contribution Statement
The Author Contributions section should specify the exact contributions of each author in a narrative form. For instance:
Abbas M.N. proposed the research problem and supervised the findings of this work.
Khudair S.Y.: executed the experiments and performed lab work.
Alsarayreh A.A.: Technically wrote the manuscript, discussed the results, and contributed to preparing the final manuscript.

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