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CONTENTS

Original scientific article

STUDY OF ISOTHERMAL, KINETIC, AND THERMODYNAMIC PARAMETERS FOR SORPTION OF VANADIUM

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Abstract

This study evaluated the equilibrium, kinetics, and thermodynamics of vanadium sorption from aqueous solution onto nanocomposite functionalized with diethylene triamine in batch conditions. The effects of temperature, solution pH, and initial concentration of vanadium were examined. The maximum sorption capacity of vanadium on the tested sorbent was achieved at pH 6. The sorption capacity increased with increasing temperature and initial concentration. The equilibrium adsorption data were analyzed using Langmuir, Freundlich, Temkin, Jovanovic, Toth, Sips, Khan, and Redlich-Peterson isotherm models. The kinetics data were studied using pseudo-first-order, pseudo-second-order, the fractional power, Elovich, and Avrami models. A non-linear fitting method was used to compare the best fitting of the equilibrium and kinetic data. The sorption equilibrium data were best represented by the Langmuir, Khan, Toth, and Redlich–Peterson isotherms. The adsorption kinetics was estimated to follow the pseudo-second-order kinetic model. The mechanism of vanadium sorption was analyzed with intra-particle, Bangham, Boyd, and liquid film diffusion models. It was observed that the sorption process was controlled by the film-diffusion as well as the pore-diffusion. Thermodynamic parameters (change of standard enthalpy ($\Delta H°$), standard entropy ($\Delta S°$), and standard free energy ($\Delta G°$)) suggested that the sorption of vanadium onto functionalized nanocomposite was a spontaneous and endothermic process.

Keywords: sorption, vanadium, kinetics, isotherms, thermodynamics.

Introduction

Vanadium (V) is a transition element that exists in several oxidation states in the range from +2 to +5, while tetravalent and pentavalent vanadium are the most commonly used forms (Ścibior et al., 2020). Due to its good physicochemical properties, vanadium is widely used in many industries such as petrochemical, electrochemical, and metallurgical industries (Luo et al., 2017). It plays an important role in contemporary science thanks to its application in producing ceramics, alloys, glasses, and redox batteries (Dakroury et al., 2022; Peng, 2019). An important source of vanadium is represented by minerals, such as vanadinite, patronite and carnotite (Luo et al., 2017). Also, it is used in steel production to improve strength, malleability, and resistance (Ścibior et al., 2020). The rapid growth of the industry has resulted in environmental pollution by vanadium. At the trace level, V has positive effects on plants and algae. On the other hand, huge amounts of V could be released into the environment through industry, contaminating wastewater, groundwater, and soils (Rivas et al., 2019; Ścibior et al., 2020). Vanadium compounds toxicity increase with increasing valence. Through the

food chain, vanadium could reach the human body and cause various health diseases and disorders (Barceloux & Barceloux, 1999). During the production of heat and electricity, exposure to vanadium occurs. Much attention has been focused on the removal of vanadium pollution. Various methods have been employed for removing vanadium from aqueous solutions, such as solvent extraction, sorption, chemical precipitation, and ion exchange (Li et al., 2011; Mthombeni et al., 2015). Among the mentioned methods, sorption is the most economical and effective method due to its flexibility, low cost, simple operation, and high efficiency. Various natural and synthetic materials have been used as sorbents for the removal of vanadium from aqueous solutions, such as natural soil colloids (Luo et al., 2017), di-2-ethylhexyl phosphoric acid (Li et al., 2011), amine extracts (Yang et al., 2016), polymers (Rivas et al., 2019), etc.

In this work, nanocomposite functionalized with diethylene triamine was used as a sorbent for the removal of vanadium. The effects of initial pH value, operative temperature, and initial concentration of vanadium in solution onto sorption capacity were investigated. In addition, the isotherms, kinetics, and thermodynamics of the sorption process were discussed.

Materials and Methods

In the present study, all the chemicals were analytical reagent grade. A stock solution of vanadium (100 mg/dm^3) was prepared by dissolving a NH_4VO_3 (Merck, Germany) in deionized water (Milli-Q Millipore, conductivity 18 MΩ/cm). The batch sorption experiments were performed in 100 ml erlenmeyer flasks by contacting 10 g/L of sorbent with a certain concentration of vanadium solution. The effects of process parameters on sorption capacity were investigated by varying initial pH values (3 - 8), initial concentration of the metal solution (1 - 100 mg/dm^3), and temperature (298 - 343 K). The residual vanadium concentration in the aqueous solution was determined by ICP-OES (Thermo Scientific iCAP 6500, USA). All experiments were repeated in duplicate. Based on obtained results, the kinetic, isotherm, and thermodynamic parameters were determined. The sorption capacity at a given time (Q_t, mg/g) and at equilibrium (Q_e, mg/g) were calculated using equations 1 and 2, respectively:

$$Q_t = \frac{(C_0 - C_t)V}{m} \quad (1)$$

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

where C_0 (mg/dm^3) is the initial concentration of vanadium solution, C_t (mg/dm^3) and C_e (mg/dm^3) are the concentrations of vanadium solutions at time *t*, and at equilibrium, respectively, V (dm^3) is the volume of the aqueous phase, and m (g) is the mass of the sorbent.

Results and discussion

The pH value of vanadium solution is a very important parameter of the sorption process because it affects the surface chemistry of the sorbent and vanadium ions specification. The effect of pH values of vanadium solution on sorption capacity was investigated in an initial pH range between 3 and 8 at T = 298 K using 10 g/dm^3 sorbent dose for 60 min. It was observed from Figure 1a that as the initial pH value was increased, the capacity of vanadium sorption increased and the maximum sorption capacity was reached at pH 6. With a further increase in the initial pH value, the sorption capacity decreased. It is known that the dominant form of vanadium at pH lower than 4 is VO^{2+}, while neutral and anionic forms exist at pH above 4 (Guzmán et al., 2002). At pH around 6, amino groups on nanocomposite were protonated, thus, the interaction between anionic forms of vanadium and active

sites of sorbent was more efficient. The influence of initial metal concentration was examined at pH = 6, T = 298 K for 60 min. The increase of initial vanadium concentration from 1 to 100 mg/dm^3 resulted in an increase in sorption capacity from 0.07 mg/g to 4.97 mg/g (Figure 1b). This can be accounted to the increase in the number of vanadium ions to the constant amount of active sites on the sorbent. To investigate the effect of temperature, the sorption capacity was determined at 298, 310, 329, and 343 K -at pH value 6, with a sorbent dose of 10 g/dm^3 for 60 min. The results are presented in Figure 1c. As can be seen, the sorption capacity slightly increases with increasing the temperature. The observed behavior can be attributed to the increase in the rate of diffusion of vanadium ions to the active sites on the sorbent (Atsar et al., 2021).

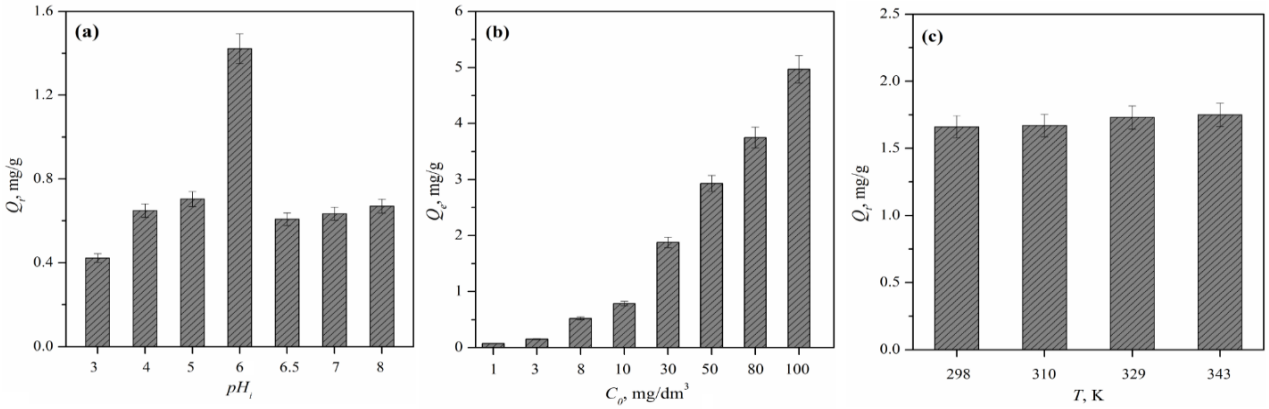

Figure 1. Effect of (a) initial pH, (b) initial concentration and (c) temperature on the sorption capacity of vanadium

To examine the binding interaction between sorbent and vanadium ions, experimental data at equilibrium were analyzed using two-parameters models, namely Langmuir, Freundlich, Temkin, and Jovanovic as well as three-parameters isotherms, namely Redlich-Peterson, Sips, Toth and Khan (Marković et al., 2014; Nastasović et al., 2022). The non-linear regression was used for fitting the curves by applying the Microsoft Excel Solver function. The isotherm models were analyzed using three statistical error functions, namely coefficient of determination (R^2), the sum of square error (SSE), and Chi-square (χ^2) (Kuete et al., 2020). The obtained parameters for two-parameters and three-parameters isotherm models are presented in Tables 1 and 2, while isotherm fitting curves are portrayed in Figure 2.

With maximal R^2 and minimal SSE and χ^2, the Langmuir model better described the experimental data compared to other two-parameters isotherm models, indicating monolayer sorption and the homogenous nature of sorbent. All three-parameters isotherm models showed good agreement with experimental data since their R^2 values were high ($\geq$ 0.993). The obtained results indicate that three-parameters isotherms better describe the sorption process than two-parameters models. Also, it can be seen that the Redlich-Peterson and Khan isotherm models have the same and maximal R^2 value (0.994) and minimal values of SSE and χ^2 (0.097 and 0.035, respectively), indicating that sorption of vanadium was a hybrid process and does not follow ideal monolayer adsorption.

Table 1. Parameters of two-parameters isotherm models for the sorption of vanadium onto amino-functionalized nanocomposite

Isotherm model	Parameter	Value	R^2	SSE	χ^2
Langmuir	K_L, L/mg	0.02	0.992	0.066	0.094
	$Q_{max,L}$, mg/g	8.63			
Freundlich	K_F, L^n mg^{1-n}/g	0.27	0.988	0.093	0.042
	n	1.37			
Temkin	b_T, kJ/mol	1.45	0.943	1.053	3.451
	K_T, L/mg	0.27			
Jovanovic	K_J, L/g	0.02	0.991	0.104	0.152
	$Q_{max,J}$, mg/g	7.63			

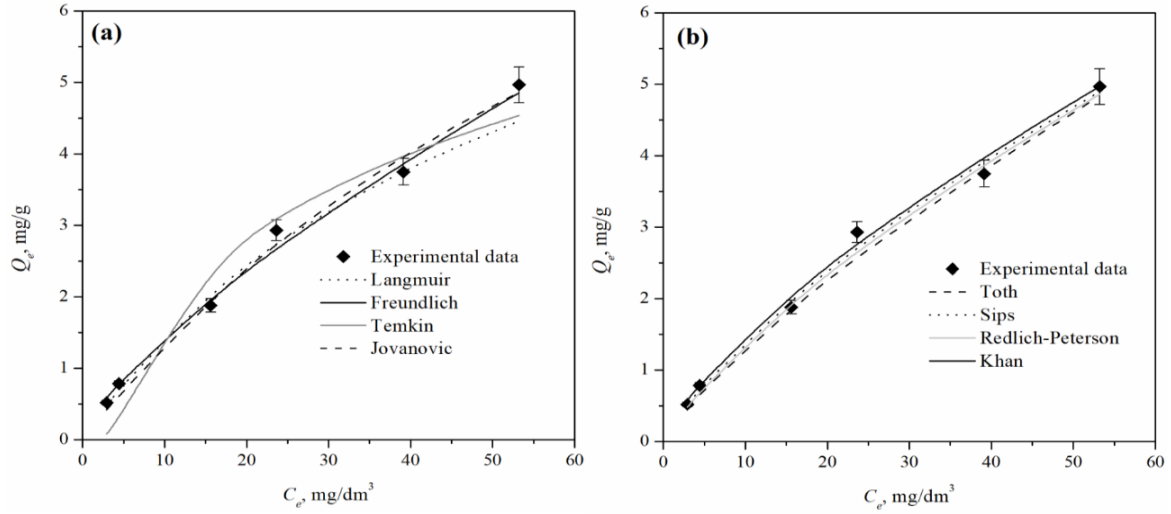

Figure 2. Predicted curve non-linear fits for the (a) two-parameter and (b) three-parameter isotherms of vanadium sorption by amino-functionalized nanocomposit

Table 2. Parameters of three-parameters isotherm models for the sorption of vanadium onto amino-functionalized nanocomposite

Isotherm model	Parameter	Value	R^2	SSE	χ^2
Redlich-Peterson	A, L/g	0.32	0.994	0.097	0.035
	B, L/mg	0.50			
	g	0.40			
Sips	$Q_{m,S}$, mg/g	28.86	0.993	0.100	0.036
	$K_S \cdot 10^3$, L/mg	2.76			
	β	0.83			
Toth	$Q_{m,T}$, mg/g	70.19	0.994	0.103	0.040
	$K_T \cdot 10^3$, L/g	3.29			
	t	0.42			
Khan	$Q_{m,K}$, mg/g	0.76	0.994	0.097	0.035
	K_K	0.28			
	n_K	0.30			

To investigate the kinetics of the vanadium sorption onto nanocomposite functionalized with diethylene triamine five models were used, namely pseudo-first-order, pseudo-second-order, Elovich, Avrami, and Fractional power kinetic models (Nastasović et al., 2022). The non-linear regression method was used to determine the kinetic parameters. The three statistical error functions (R^2, SSE, and χ^2) were applied to test the best fitting kinetic models. The estimated kinetic parameters are presented in Table 3, while kinetics fitting curves are shown in Figure 3.

Table 3. Kinetic parameters for sorption of vanadium onto amino-functionalized nanocomposite

Kinetic model	Parameter	Value	R^2	SSE	χ^2
Pseudo-first-order	k_1, 1/min	0.14	0.787	0.267	0.395
	Q_e^{cal}, mg/g	1.42			
Pseudo-second-order	k_2, 1/min	0.18	0.998	0.001	0.001
	Q_e^{cal}, mg/g	1.48			
Elovich	α, mg/g min	4.91	0.992	0.004	0.003
	β, g/mg	5.35			
Avrami	k_{AV}, 1/min	0.07	0.997	0.002	0.002
	Q_e^{cal}, mg/g	10.49			
	n	0.17			
Fractional power	k_{FP}, mg/g min	0.71	0.888	0.076	0.084
	v, 1/min	0.16			

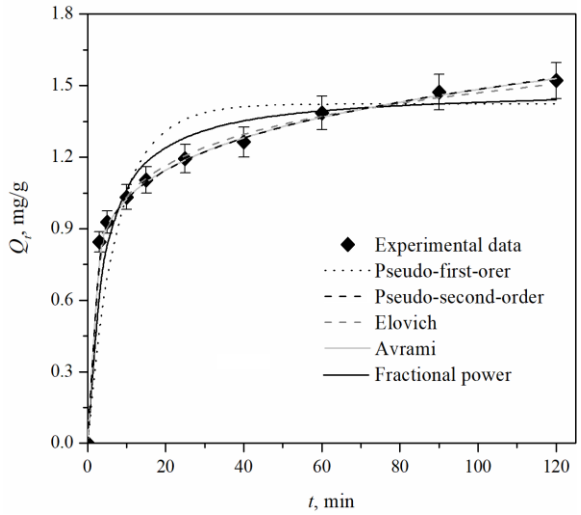

Figure 3. Non-linear kinetic models fitting for sorption of vanadium by nanocomposite functionalized with diethylene triamine

Based on statistical error functions presented in Table 3, the non-linear model of pseudo-second order is best suited to describe the sorption of vanadium. The Q_e^{calc} value (1.48 mg/g) from the pseudo-second-order was close to the Q_e^{exp} value (1.52 mg/g), meaning that chemisorption plays an important role in the vanadium sorption onto amino-functionalized nanocomposite.

The liquid film diffusion, intra-particle diffusion, Bangham, and Boyd models (Nastasović et al., 2022) were used to understand the sorption mechanism. The results are presented in Figure 4 and Table 4. As can be seen from obtained results, the high values for R^2 for liquid film diffusion, intra-particle diffusion, Bangham, and Boyd models, as well as multilinear intra-particle diffusion plot, suggest the simultaneous influence of film and pore diffusion onto vanadium sorption by the investigated sorbent.

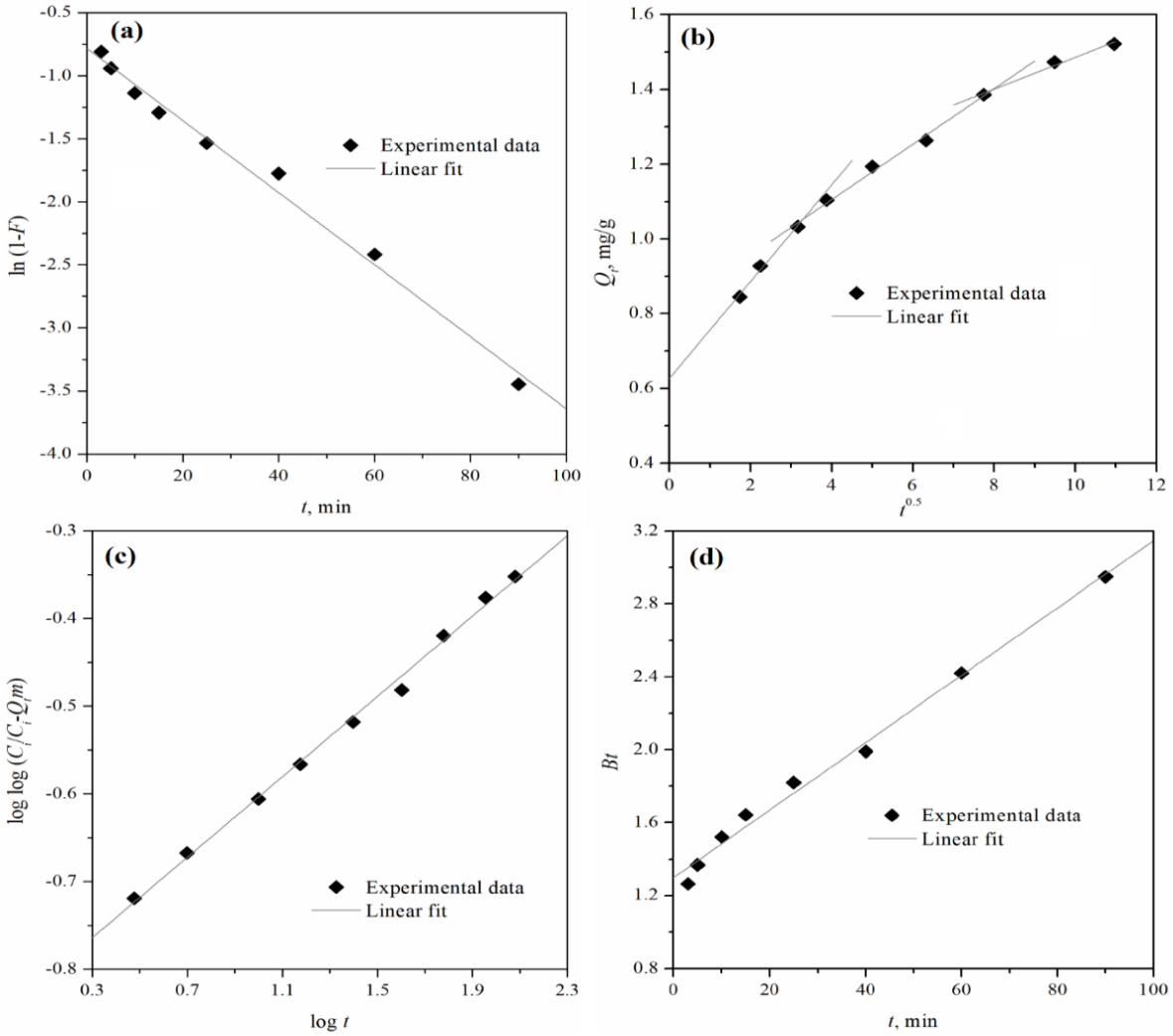

Figure 4. Linearized plots of (a) liquid film diffusion, (b) intra-particle diffusion, (c) Bangham and (d) Boyd models for the sorption of vanadium onto nanocomposite functionalized with diethylene triamine

Table 4. Parameters for sorption mechanism of vanadium onto amino-functionalized nanocomposite

Parameters	Values	Parameters	Values
Intra-particle diffusion		Liquid film diffuison	
$k_{id,1}$, mg/g min$^{0.5}$	0.13	$k_{LFD} \cdot 10^2$, 1/min	2.86
$C_{id,1}$	0.63	C_{LFD}	-0.79
R_1^2	0.990	R^2	0.990
$k_{id,2}$, mg/g min$^{0.5}$	0.07	Bangham model	
$C_{id,2}$	0.81	$k_B \cdot 10^3$, 1/g	0.68
R_2^2	0.992	α	0.23
$k_{id,3}$, mg/g min$^{0.5}$	0.04	R^2	0.996
$C_{id,3}$	1.06	Boyd model	
R_3^2	0.987	R^2	0.991

The standard Gibb's free energy change ($\Delta G°$), the standard enthalpy change ($\Delta H°$) and the standard entropy change ($\Delta S°$) were calculated from Van't Hoff equation (Marković et al., 2017). The thermodynamic parameters are given in Table 5. The positive values of $\Delta S°$ (0.31 kJ/K mol) typify a high degree of randomness and reversible process. The positive $\Delta H°$ value (69.5 kJ/mol) suggests that the sorption is endothermic and chemisorption in nature (Dada et al., 2020). The negative $\Delta G°$

values at all studied temperatures indicate that sorption of vanadium is thermodynamically favourable and spontaneous.

Table 5. Thermodynamic parameters for vanadium sorption by amino-functionalized nanocomposite

T, K	$\Delta G°$ (kJ/mol)	$\Delta H°$ (kJ/mol)	$\Delta S°$ (kJ/Kmol)	$T\Delta S°$ (kJ/mol)
298	-22.98	69.5	0.31	92.55
313	-26.71			96.28
328	-31.61			102.18
343	-36.95			106.53

Conclusion

This study has investigated the possible application of amino-functionalized nanocomposite as sorbent for the removal of vanadium from aqueous solutions. The sorption capacity was dependent on the initial pH value of vanadium solution, temperature as well as initial metal concentration. Isotherms models confirmed that sorption of vanadium was a hybrid process and did not follow ideal monolayer adsorption. The sorption followed the pseudo-second-order kinetics model, as confirmed by statistical error functions. The mechanism investigation has shown that intra-particle diffusion was not the only rate-limiting step. Thermodynamic parameters suggested that the sorption of vanadium onto functionalized nanocomposite was a spontaneous, thermodynamically favourable, and endothermic process.

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