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The isotherm and kinetic study of Nickel and Chromium adsorption by pumice powder from aqueous environments

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ABSTRACT

Nickel and chromium are toxic heavy metals that have serious adverse effects on human health and the environment. The removal efficiency of nickel and chromium from aqueous solution was evaluated using pumice powder as an adsorbent. After preparing pumice powder, batch experiments were performed at different times and adsorbent dosages (pH=7, heavy metals concentrations = 50 mg/L, and stirring/minute= 200rpm). Obtained products were adsorbed using Langmuir and Freundlich isotherms, pseudo-first order and second-order models, intra-particle diffusion, and Elovich kinetics. Results indicate that nickel adsorbs more than chromium and adsorption of both nickel and chromium follows the Freundlich model. Correlation coefficients obtained from Freundlich model and pseudo-second order kinetic are R2=0.995, R2=0.996 and R2=0.999, respectively, which shows that the Freundlich expression and the kinetics of pseudo-second order provided a better fit to the experimental data of pumice powder. Also, the removal efficiency was increased significantly by increasing the adsorbent dose and contact time (R2<0.001). According to the obtained value (0 – 1) for dimensionless constant separation term of Langmuir model (RL) and adsorption intensity (1/n), pumice is a proper adsorbent for the removal of nickel and chromium from aqueous environments.

Introduction

Nickel and chromium are toxic heavy metals that have serious adverse effects on human health and the environment [1]. High concentrations of heavy metals in industrial effluent are regarded as one of the most important environmental problems [2]. These heavy metals are gradually accumulated in natural water ecosystems and their presence in ecological systems causes several adverse effects. Heavy metal pollution arises from various sources, such as nature, and different industries including production/ manufacturing related to electroplating, battery, paint, stabilizers, metallurgical, chemical industry, smelting of ores, paper factory, oil refineries, and chemical fertilizers that discharge different heavy metals into the environment [3]. Therefore, the removal of noxious pollutants is necessary to meet the standards limits [4]. Researchers have made use of several methods, such as ion exchange, ultrafiltration, reverse osmosis, electrodyalysis, chemical precipitation, evaporation, solvent extraction, membrane processes coagulation, flocculation, biological treatment, chemical oxidation and adsorption to remove or reduce heavy metal application [5]. Indeed, effectiveness and inexpensiveness play an important role in selecting each of these removal methods. These factors, therefore, move researchers toward methods of having low cost and high efficiency [6, 7]. Accordingly, each of these methods contain some advantages and disadvantages, dependent on some issues, including: preparation of raw materials, flexibility, effectiveness of processes, cost, technical or maintenance problems, incomplete metal removal, facility requirement, expensive monitoring systems, large amounts of chemical and energy requirement, sludge and other wastes production. However, among several chemical and physical methods, the adsorption method is thought to be superior to other methods [8]. Annually, various artificial adsorbents are commercially produced; however, owing to unavailability or high cost issues, they are not sufficiently applied [9]. Yet, among natural and artificial adsorbents, raw materials seem to be appropriate in term of low cost and availability [10]. In fact, to remove heavy metals from aqueous solutions, adsorption onto pumice powder has been shown to be an appropriate method because of it is inexpensive and available [12]. Several studies revealed that pumice is a material with a porous structure and large surface area causing sorption onto this substance [11]. The current research is an attempt to evaluate the pumice powder efficiency, as an adsorbent, and to remove nickel and chromium from aqueous solution. For comparison purposes, adsorption isotherm which include Langmuir, Freundlich and pseudo-first order, pseudo-second order, intra particle diffusion and Elovich kinetics are applied to the process.

Materials and Methods

Batch experiments were conducted as follows. Pumice granules, purchased from mines located in Kurdistan, were washed with distilled water and dried in an oven at 103°C for 6 hours to remove moisture. Thereafter, they were placed into a muffle furnace at the 550°C for1 hour to remove organic impurities. The pumice granules were crushed and sieved through a 50 mesh sieve to achieve particle sizes of 0.297 mm.

After adsorption, all synthetic solutions contained heavy metal [Cr^{+3}‘Ni^{+2}), with standard concentrations [with regard to their concentration in industries’ effluent] (50 mg/l), were prepared using stock solution purchased from Merck Company (Germany). Experiments were conducted using a batch reaction process and pumice powder dosage of 2, 4, 6, 8 and 10g weight. Then, 100 ml of a solution, containing 50 mg/l of heavy metals, was prepared. Also, a predetermined amount of pumice powder was added to this solution [100 ml) in a 500 ml measuring flask. Then, it was placed on a magnetic stirrer with 200 rpm. Stirring was performed at different contact times (15, 30, 45, 60 and 75 minutes). The pH of all mixtures was set to 7.0 using Hcl and HNO_3. After this process, 50 ml of prepared solution was centrifuged at 2000 rpm for 15 min. Finally, the metal concentration of solutions was determined using ICP 7300 DV (US). The adsorbent characteristic was determined through FTIR, XRD and SEM. FTIR was performed using a spectrometer (WQF-510) that scans in the range of 400 to 4000 cm^{-1} with a spectral resolution of 4 cm^{-1}. Micrographs of a KBr pellet of pumice stone are shown in Fig 1. Table 1 indicates the chemical composition of pumice powder samples determined with XRD method (Shimadzu XRD–6000). SEM image of absorbent was performed using a Philips XL30 which given in Fig. 2.

Results

To determine the optimum dose of adsorbent, pumice powder dose range (2, 4, 6, 8 10 g) was considered as a variable and other parameters as constants. The experiment revealed that the removal efficiency of both nickel and chromium in a fixed contact time of 1 hour increased significantly from 81.74 and 76.98 to 100% ($P < 0.001$) (Fig 3).

Table 1. Chemical composition of pumice powder

Composition	%
SiO_2	67
$Al2O_3$	15.6
K_2O	5.4
Fe_2O_3	1.1
Na_2O	3.7
CaO	3.9
MgO	0.2
etc.	3.1

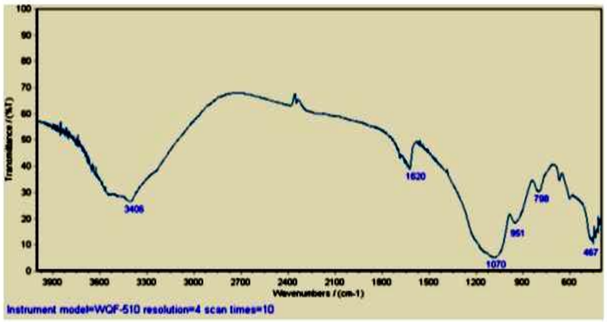

Figure 1. FTIR spectra of pumice powder

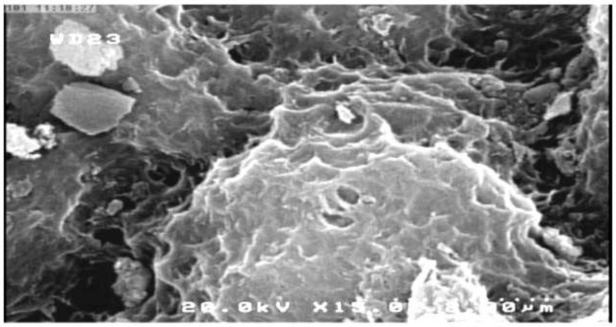

Figure 2. Microscopic scan of electronic adsorbent of pumice powder

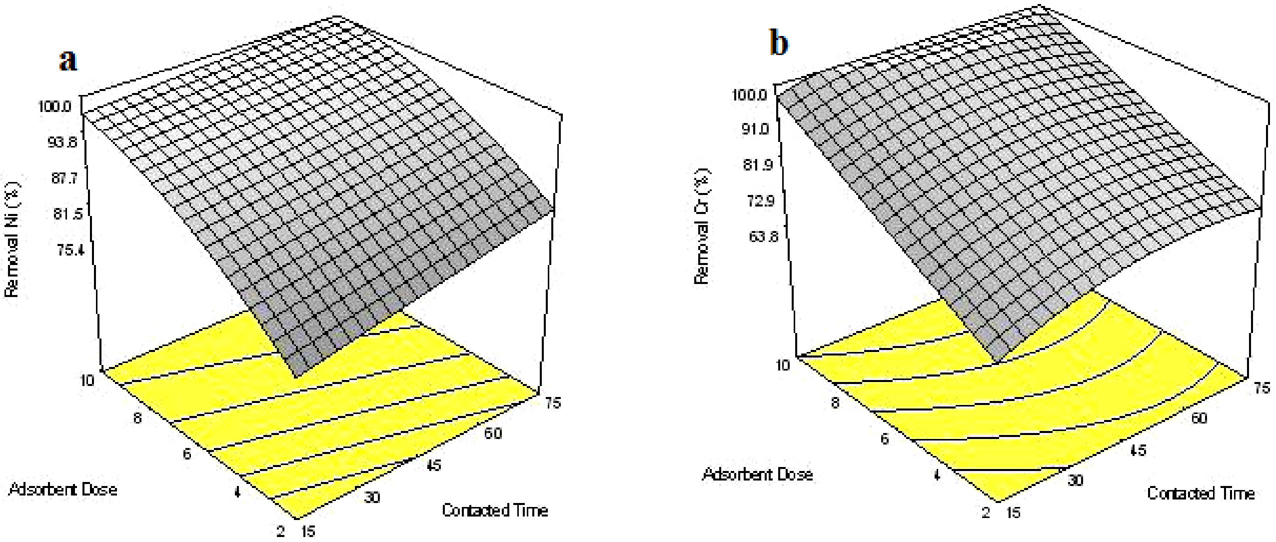

Figure 3. Effect of adsorbent dosage and contact time on efficiency of nickel (a) and chromium (a) removal process (Con=50 mg/l, PH=7, rpm=200)

The optimum time was determined as follows. At first, contact time range (15, 30, 45, 60, 75 minutes) was regarded as a variable and others as constant parameters. It was indicated that through increasing contact time that the removal efficiency of chromium and nickel in predetermined optimum dose of pumice powder (4g) increased from 82.97 to 97.87% and 73.28 to 86.63%, respectively ($P < 0.001$) (Fig 3).

In order to examine the equilibrium constant (Fig 4 and 5), 1000 mL mixture solution of heavy metals with the same concentration (50 mg/l) was prepared with distilled water. Then, 100 ml of this solution was transferred to 500 ml measuring flasks. After adding pumice powder to dosage of 2, 4, 6, 8 and 10 grams, they were placed on a magnetic stirrer with 200 rpm. The pH value of the solution was set to 7.0. This process continued until achieving the process equilibrium state, which took 60 minutes. The concentrations of heavy metals in adsorbent phase were calculated using following equation [13] (Eq. 1).

$$Q_e = \frac{(c_0 - c_e)v}{m} \qquad (1)$$

Where:

q_e *is the* equilibrium concentration of heavy metals in adsorbent (mg/g)

C_o and C_e (mg/l) are the concentration of heavy metals at initial and equilibrium state.

V (l) is volume of heavy metals solution

m (g) is the weight of pumice powder

Adsorption isotherm is principally noteworthy to express how solutes interact with adsorbent, and it is essential in optimizing the use of adsorbents. Creating appropriate interactions for equilibrium and optimizing in designing a surface adsorption system for removal of adsorb is of critical importance. It should be noted that important adsorption isotherms include Langmuir and Freundlich.

The Langmuir isotherm is based on a hypothesis that estimates a single-layer coating of adsorbent on the outer surface of the sorbent. The absorption rate is proportionate to the drive force of the adsorbent on the surface area. Driving force and surface area are referred to the concentration of solution and surface area available to the adsorbent, respectively [4].

Langmuir Isotherm Assumptions:

- In all areas of the adsorbent surface, absorption energy is constant and similar
- Adsorb particles are not able to move on the adsorbent surface.
- Maximum absorption occurs when the adsorbent surface covered with monolayer of adsorbed molecules .
- Absorption on one part of the adsorbent surface is not affected by a molecule of adjacent places .
- In all parts of adsorbent, absorption of molecules takes place similarly .
- While reaction occurs between adsorbent and adsorb, adsorption temperature of all molecules decreases linearly [14].

Linear equation of Langmuir isotherm is

$$\frac{1}{q_e}=\frac{1}{Q_e}+\frac{1}{bQ_m c_e} \tag{2}$$

Where:

q_e is equilibrium concentration of heavy adsorbed metals (mg/g)

C_e (mg/l) is the concentration of heavy metals at equilibrium state

b (l/mg)is Langmuir constant in relation to energy of adsorption

Q_m (mg/g) is maximum absorption capacity

Table 2. Langmuir separation factor of absorption process

R_L Factor	Absorption
RL=0	Irreversible
0<RL<1	Favorable
RL=1	Linear
RL>1	unfavorable

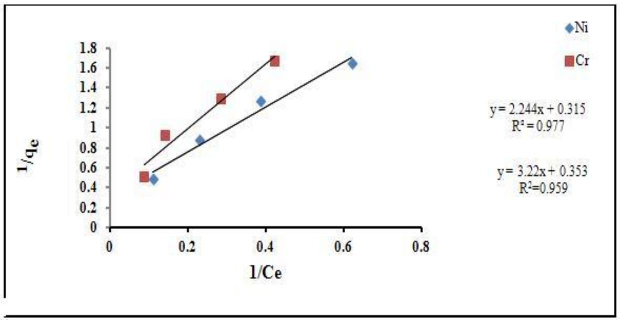

Figure 4. Langmuir adsorption isotherm of nickel and chromium on pumice

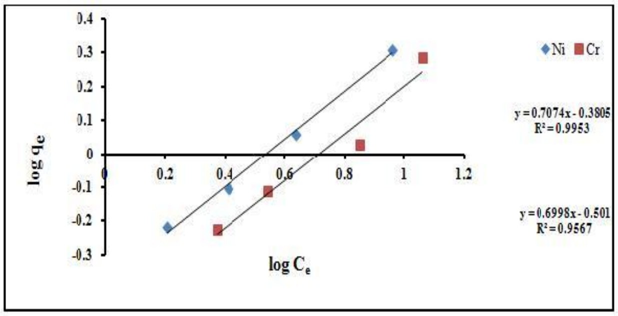

Figure 5. Freundlich adsorption isotherm of nickel and chromium on pumice

In order to calculate Langmuir constant (b) and maximum absorption capacity (Qm) through gradient and width of a line equation centered at the origin, two curves need to be drawn; $1/qe$ versus $1/C_e$. Langmuir separation factor (RL) is an important factor of Langmuir adsorption process, which is in the range of (1–0). Equation of RL isotherm is:

$$RL=\frac{1}{(1+bC_0)} \tag{3}$$

Where:

RL is Langmuir separation factor

C_o (mg/l) is initial concentration

b (mg/l) is Langmuir constant

Table 2 indicates Langmuir separation factor of an absorption process [15]:

The Freundlich isotherm is based on monolayer absorption on the heterogeneous surface of adsorbent containing unequal amount of energies. Not only it is not limited to a monolayer adsorption, but also it is applied for multi-layer absorption. Unlike Langmuir isotherm, temperature level of this isotherm decreases logarithmically. The linear equation model of Freundlich isotherm is as followed [16]:

$$\log q_e = \log k_f + \frac{1}{n}\log C_e \qquad (4)$$

Where:

q_e (mg/g) is equilibrium concentration of heavy metals in adsorbent

C_e (mg/L) is the concentration of heavy metals at equilibrium

n,K_f is constant of adsorption capacity(L/mg) (1/*n*) and absorption intensity (mg/g), respectively.

Here, *n* and K_f are Freundlich constants parameters; *n* is an indication of how propitious the adsorption process is and K_f is the adsorption capacity of the adsorbent. To obtain the value of *n* and K_f using gradient and width of a line equation of Freundlich isotherm centered at the origin. Two curves need to be drawn: *log* q_e versus *log* C_e. However, *n* values (1 to 10) indicate the type of isotherm, which is either favorable ($n > 1$) or unfavorable ($n < 1$). Also adsorption intensity between $0 < (1/n) < 1$ indicates that the heavy metal adsorption is favorable [17]. **Table 3**, represents the calculated parameters of isotherm models.

Kinetic models are used to evaluate the reaction rate of adsorption (Fig 6 - 9) [18]. These models are applicable for designing and optimizing chemical processes that are based on removing pollutants from effluent. This study investigated kinetic reactions based on pseudo-first-order and second-order model for the adsorption mechanism. Therefore, to obtain kinematic model, contact time is a variable while additional parameters are held constant (absorption rate of heavy metals in different times of 15, 30, 45, 60 and 75 min; fixed pumice dose 6g; and rpm=200).

The pseudo-first-order kinetic equation is

$$\frac{dq_t}{d_t} = k_1(q_e - q_t) \qquad (5)$$

The linear form of this expression (with t=0, t=t, q=0 and q=q) is:

$$\log\left(1-\frac{q_t}{q_e}\right) = -\frac{k_1}{2.302}t \qquad (6)$$

Where: q_e is the concentration of heavy metals adsorbed per unit mass of pumice at equilibrium state (mg/g).

q_e is the amount of heavy metals adsorbed on pumice at constant time (mg/g)

K_1 is the pseudo first order constant rate (min^{-1})

Pseudo-first-order kinetic model was calculated by plotting $\log\left(1-\frac{q_t}{q_e}\right)$ versus t and K_t.

Pseudo-second-order kinetic equation equals to:

$$\frac{d_q}{d_t} = k(q_e - q)^2 \qquad (7)$$

The linear form of this model is :

$$\frac{1}{q_e - q_t} = \frac{1}{q_e} + k_t \qquad (8)$$

Note that, the above mentioned equations could be rewritten as follows:

$$\frac{t}{q_e} = \frac{1}{h} + \frac{1}{q_e}t \qquad (9)$$

$$h = k_2 q_e^2 \qquad (10)$$

Where: q_t and q_e are the concentration of heavy metals in t and equilibrium times(mg/g)

h Initial absorption rate t→ 0 (mg g^{-1} min^{-1})

K_2 constant rate of Pseudo-second-order kinetic (g mg^{-1} min^{-1}). **Table 4**, shows the kinetic constants.

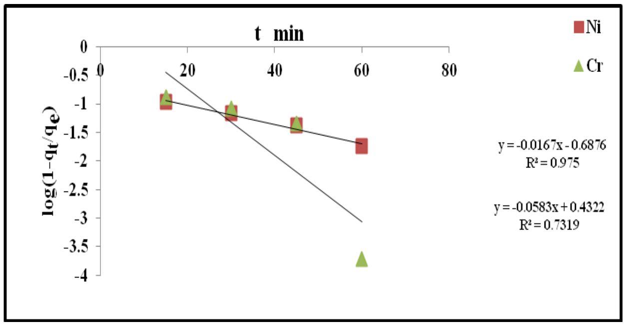

Figure 6. Pseudo-first-order kinetic adsorption of nickel and chromium on pumice

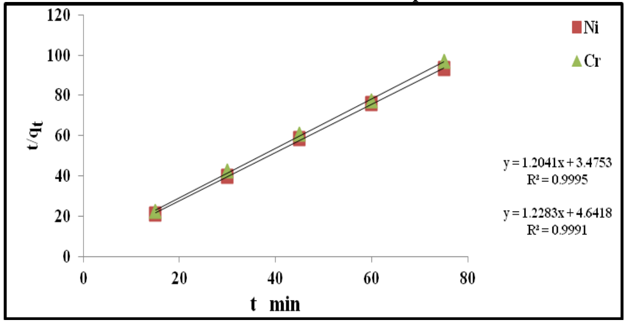

Figure 7. Pseudo-second-order kinetic adsorption of nickel and chromium on pumice

Table 3. Calculated parameters of isotherm models

Heavy metals	Freundlich R²	Freundlich 1/n	Freundlich n	Freundlich K_f (mg/g)(L/mg)$^{(1/n)}$	Langmuir R²	Langmuir R_L	Langmuir b (L/mg)	Langmuir Q_m (mg/g)
Ni^{+2}	0.9953	0.70771	1.413	2.40	0.9775	.124	0.140	3.172
Cr^{+3}	0.9567	0.70028	1.428	3.169	0.959	.154	0.11	2.828

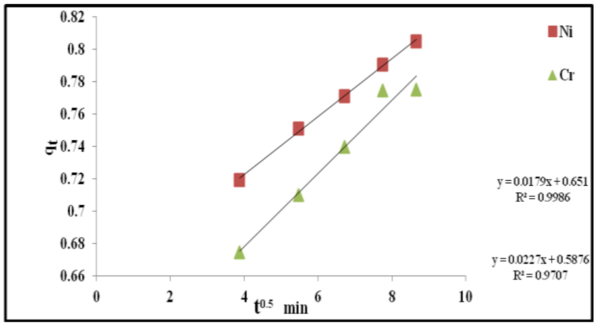

Figure 8. Intra-particle diffusion kinetic for nickel and chromium on pumice

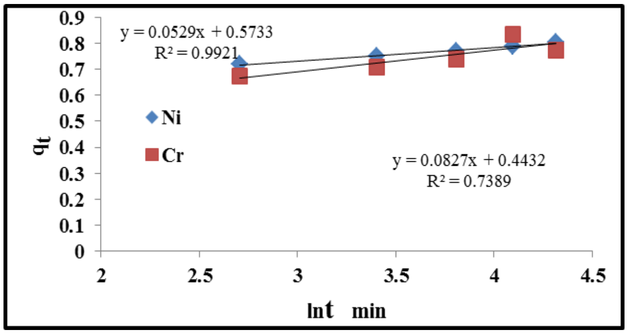

Figure 9. Elovich adsorption kinetic for nickel and chromium on pumice

Table 4. Calculated parameters of kinetic models

Adsorbate	Pseudo-first-order $K_{1(min^{-1})}$	Pseudo-first-order R²	Pseudo-second-order K_2 (g mg^{-1} min^{-1})	Pseudo-second-order R²	Intraparticle diffusion K_i	Intraparticle diffusion I	Intraparticle diffusion R²	Elovich β	Elovich a	Elovich R²
Ni^{+2}	0.0384	0.975	0.417	0.999	0.017	0.651	0.998	58.8	0.85	0.992
Cr^{+3}	0.134	0.732	0.325	0.999	0.022	0.587	0.735	37	0.85	0.738

Pseudo-first-order kinetic model is calculated by gradient and width of two curves, t/q_t versus t and K_2, centered at the origin.

Pseudo-first-order kinetic model by plotting t/q_t versus t and K_2 was calculated from the intercept and slope.

Intra-particle diffusion kinetic models examine the mechanism of adsorption on porous adsorbent and determine the controlling level of adsorption rate, which affects the adsorption process. The linear form of this model is :

$$q_t = k_i t^{0.5} + I \qquad (11)$$

Where:

K_i is intra-particle diffusion rate on pumice (mg/g. min $^{0.5}$).

I is a constant rate dependent on the thickness of boundary layer (mg /g).

Elovich adsorption kinetics equation equals to :

$$q_t = \left(\frac{1}{\beta}\right)\ln(\alpha\beta) + \left(\frac{1}{\beta}\right)\ln t \qquad (12)$$

Where:

α is Elovich constant (mg/g.min)

β is Elovich constant (g/mg)

Discussion

The findings of the present study indicate that the highest removal rate of adsorbent materials occurs in the initial 15 minutes. This means that wide adsorption sites of using adsorbent are available in a given initial time [19]. Through this removal ratio of $Ni^{+2} > Cr^{+3}$. Katalfuma et al., (2003) found similar results in their study. It was

also found that the removal efficiency rate was relatively higher in term of increasing the adsorbent dose and contact time [20]. However, the removal efficiency rate was increased further due to increasing the adsorbent dose, rather than increasing the contact time. The adsorption rate with increasing initial contact time in a given mass of sorbent is raised and then gradually reaches equilibrium. This is because the absorbing surfaces of a given particular mass of adsorbent involve actual sites to absorb metal concentration of effluent. These sites would be occupied by adsorbent ions within short period of time owing to increasing adsorbent dose and contact time. Absorbed metal concentration carried out into the pores of the adsorbent; consequently, the absorption rate will be decreased [21]. However, metal absorption highly depends on physicochemical characteristics of the absorbent and the adsorbate. The results show that the pumice characteristics contain 67% and 15.6% of SiO_2 and AL_2O_3, respectively. In another study, Li et al., (2006) concluded that adsorbent size, pore distribution of adsorbent and chemical characteristics of their surface have a significant effect on removal efficiency [22]. In addition, Yenisoy-Karakas et al., (2004) indicated that the chemical characteristic of the adsorbent surface plays a significant role in removing metals ions rather than adsorbent pore size [23].
study, the researchers also observed a metal Attempts made by previous researchers revealed that the presence of metal oxides in aqueous solutions considerably affect the adsorption process since it forms groups of agent factor on the surface area [24]. While pumice placed in water, its contained metal oxides would be covered by water molecules with the following formula [25]:

$$Si-OH+OH^- \leftrightarrow Si-O^- \qquad (13)$$

$$Si-OH+H^+ \leftrightarrow SiOH^{+2} \qquad (14)$$

Where in acidic condition the formula changes to (Eq. 15):

$$H^+ + Si^{+2} \leftrightarrow HSi^{+3} \qquad (15)$$

According to Moracia, metals absorption on pumice is expressed in the following (Eq. 16):

$$>SiOH + M_e^{+2} \leftrightarrow \left(SiO^- - M_e^{+2}\right) + H^+ \qquad (16)$$

Where, M_e is any alkaline or earth alkaline metal which is exchanged with the metal ion of solution [11]. Some other researchers, such as Baradachky (2009), found that the removal efficiency of pollutants is related to hydroxyl groups attached to the silica structure. In addition, additional factors including electrostatic force and ion exchange are expressive in adsorption of metals by pumice [26]. Also, physicochemical characteristics of adsorbs, such as metals hydrated radius, ion radius, and pH level affect their absorption rate.

Because the metals could be presented as hydrated complexes with water molecules in aqueous solutions [27]; more adsorption in nickel compared to chromium may be due to having larger ionic (0.74 A^0) and smaller hydrated radius (3.244 A0) of nickel than chromium [ionic radius (0.62 A^0), hydrated radius (4.61 A^0). Since, with regard to ionic and hydrated radius, the order of these metals are $Ni^{+2} > Cr^{+3}$ and $Cr^{+3} > Ni^{+2}$, respectively [28]. In all experiments, the pH level was equal to 7. Accordingly, pumice surface is negatively charged, so metals with larger ionic radius and smaller hydrated radius are further absorbed by pumice [24]. On the other hand, lower removal efficiency of chromium could be due to its small size and particular structure. Also, at elevated pH level, chromium changes to CrO_4^{2-} which moves away from produced ion (SiO^{2-}) of absorbent. Whereas in acidic conditions (low pH value), the hydrated radius ($HCrO_4$) are larger and they are absorbed by Si^{+3} which is dominated in this condition. The removal efficiency of chromium is sizeable under low pH conditions [29]. In addition to hydrated and ionic radii of metals, metals could be adsorbed through electrostatic force, other researchers such as Moracia et al., (2010) and katalfouma et al.,(2006) found that metal removal results from ionic exchange process between alkaline and earth alkaline metals (Na^+ ،K^+،Mg^{+2} and Ca^{+2}) or other available metals in solutes[15, 20]. Obtained isotherm adsorption data show that the adsorption of both nickel and chromium obeyed the Freundlich model (R2=0.995 and R2=0.996) (Fig. 4 and 5). It was found that the adsorption process taken place in multi-layered adsorbents. However, n values (1 to 10) indicate the type of isotherm to be either favorable $n > 1$ and unfavorable $n < 1$. Thus, heterogeneous adsorbent surface is less important. Also, adsorption intensity $0 < 1/n < 1$

indicates that heavy metals adsorption is a favorable process [30]. Therefore, considering *RL*, pumice is an appropriated adsorbent for the removals of pollutants [31]. Analyzing kinetics of adsorption showed that data obey kinetic reaction to the following order: pseudo-first order < Elovich < = Intra- particle diffusion < second-order (Fig. 6–9). This means that proper adsorption obeys the second-order kinetic model and the absorption process is dependent on the adsorbent concentration because the second-order kinetic model is based on the capacity of absorption [32]. Linear equation of intra-particle diffusion of kinetic indicates that it plays an important role in the adsorption process by pumice powder [33].

Conclusion

Results showed adsorption process was obeyed the freundlich isotherm that confirming the multi-layer sorption of metals. Also, considering the obtained value (0 – 1) for dimensionless constant separation term of Langmuir model (RL) and adsorption intensity (1/n), pumice is a proper adsorbent for the removal of nickel and chromium from aqueous environments.

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