Desalination and Water Treatment
www.deswater.com

doi: 10.5004/dwt.2019.23367

142 (2019) 205–212
February

Adsorption kinetics and isotherms of Ni (II) and Zn (II) heavy metals onto a natural adsorbent: expanded perlite

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Received 25 June 2018; Accepted 27 October 2018

ABSTRACT

The diversity of wastewaters varies depending on the development of industry and the application of different production processes in each area. Heavy metals that cause toxic effects in the wastewater are left untreated by many sectors. These heavy metals are then transported by natural resources (rainwater, etc.), which have a negative effect on the natural environment and disturb the balance of life. Even in very low concentrations in water, heavy metals can cause toxic effects that can lead to illnesses and even deaths in living organisms. The technologies and methods used to remove heavy metals are inadequate. An investigation into the adsorption with expanded perlite on nickel and zinc was therefore conducted to ascertain which has the most important poisoning effect. Specifically, the adsorption of Ni (II) and Zn (II) ions with expanded perlite was investigated through measures of pH, contact time, initial metal concentration, and the adsorption effect of the adsorbent dose. The results showed that the Ni (II) ion conforms with the pseudo-second-order kinetic model, in which the Zn (II) ion equilibrates within 10 min and within 5 min. The initial metal concentration was chosen to be 5 mg/L for both ions, and the expanded perlite dosage was 0.4 g for Ni (II) and 0.5 g for Zn (II). In adsorption isotherm studies based on metal concentrations, the Ni (II) ion conforms to the Freundlich isotherm model with $R^2 = 0.91$ and the Zn (II) ion conforms to the Langmuir isotherm model with $R^2 = 0.97$.

Keywords: Heavy metals adsorption; Water treatment; Natural adsorbent; Expanded perlite

1. Introduction

Heavy metals are usually formed through industrial activities and accumulate in sediments mixed with rivers, lakes, and underground waters. Unlike wastewaters containing organic compounds, wastewaters containing heavy metals cannot be removed by biodegradation. There are many methods available for removing heavy metals from aqueous solutions, the most effective of which is adsorption. In a research, silver, copper, and mercury were removed from aqueous solutions with expanded perlite. The adsorption process for metal ions was in accordance with the pseudo-second order. When selecting the Langmuir isotherm model, the maximum adsorption capacity of expanded perlite was found to be 8.46 for Ag (II), 1.95 for Cu (II), and 0.35 mg/g for Hg (II) [1]. In another study, walnut sawdust was used as an adsorbent material in the adsorption of lead, cadmium, and nickel. Contact time, initial metal concentration, and the effect of temperature were all measured. The optimum point was measured in 60 min. Pb (II) = Cd (II) > Ni (II) was the equation used to determine removal efficiency [2]. In studies investigating the adsorption formations used to remove Ni (II) ions from aqueous solutions, the aim was to combine existing research on potentially effective adsorbents. Additionally, equilibrium adsorption isotherms and kinetic and thermodynamic data have been measured for Ni (II) ions of different adsorbents under various optimal conditions (pH, equilibrium contact time, and adsorbent dosage).

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Agricultural solid wastes, natural materials, and biosorbents have shown superior adsorption capabilities for Ni (II) ions. Overall, more than 190 research studies have shown a superior adsorption ability for agricultural solid waste, natural materials, and biosorbents containing Ni (II) ions [3]. Belova et al. aimed to understand the adsorption behavior of nickel by comparing synthetic limestone and calcite to synthetic and biogenic calcite. The stability constants for the Ni (II) surface complex were log $k = -1.12$ on calcite and log $K = -0.43$ and -0.50 on two limestone samples. The study confirms that synthetic calcite and chalk contain nickel ions, but Ni (II) is more binding than biologically calcined synthetic powder due to the presence of trace amounts of polysaccharides and clay nanoparticles in biogenic calcite [4]. Kalantari et al. investigated an application of novel nano-adsorbent with an ability in heavy metal ion adsorption; researchers highlighted that the low dosage of magnetic nano-adsorbent could adsorb high concentrations of heavy metals. The adsorption process was also carried out in less than 2 min [5]. And these researchers investigated another research of rapid adsorption of heavy metals by Fe_3O_4/talc nanocomposite. Optimal conditions with initial heavy metal concentrations of 100, 92, and 270 mg/L, 120 s of removal time, and 0.12 g of adsorbent amount resulted in 72.15%, 50.23%, and 91.35% removal efficiency for Cu (II), Ni (II), and Pb (II), respectively [6]. Shing et al. evaluated the effects of lime bracketing (1%, 2%, and 3%) on the bioavailability and extractability of heavy metals during 30 days of fertilization of water hyacinth, cattle powder, and sawdust (6:3:1). The results showed that the total concentration of heavy metals increased during the fertilization process. Due to the addition of calcium, the initial pH value of gubernin effectively increased, while filtering of the toxicity properties of diethylene triamine pentaacetic acid (DTPA) in the water content decreased. Water-soluble metals (Ni, Pb, and Cd) and DTPA extractable metals (Pb and Cd) were not found in the water-soluble fraction. With the addition of the calcian, water significantly reduced the bioavailability and extractability of heavy metals when plumage was added. They also used a Tessier sequential extraction method to investigate the changes in heavy metal species (Zn, Cu, Mn, Fe, Pb, Ni, Cd, and Cr) during composting of water hyacinth (*Eichhornia crassipes*). The results showed that the total metal concentration increased during the composting process. No reducible and oxidizable fractions of Ni, Pb, and Cd were found during the water hyacinth composting. Cu and Cd concentrations were very low compared with other metals, and the percentage of carbonate fractions was similar to other metals although this can be changed. Singh concluded that the proper proportion of cattle grafting significantly reduced mobile and easily obtainable fractions during composting [7,8]. Various parameters such as pH, initial Zn (II) ion concentration, temperature, flow rate, and contact time were all measured when investigating the removal of Zn (II) ions with chitosan-coated diatomaceous earth (CCDE) droplets. The maximum adsorption capacity of the Zn (II) ion to the CCDE droplets in batch studies was 127.4 mg/g. Adsorption was adapted to the pseudo-second-order kinetic and Langmuir isotherm models [9]. The adsorption of Ni (II) ions on activated carbon is based on spent coffee (SAC) and coffee husk (HAC). Experiments were performed using the parameters of adsorbent dose, Ni (II) concentration, and pH. The adsorption of Ni (II) ion was evaluated using different adsorption isotherm models (Langmuir, Freundlich, Single Compound Adsorption for Radonkevich and Dubinin—Single Layer Model) and predetermined thermodynamic and kinetic parameters. The maximum adsorption capacities using the Langmuir isotherm model were found to be 57.14 and 51.91 mg/g for HAC and SAC, respectively [10]. The aqueous solution adsorption of Ni (II) ions by chitosan (CTS) and triethylenetetramine-derivatized particles (CCTS) has been investigated in many adsorption systems. The maximum adsorption capacities of chitosan and chitosan derivatives were found to be 58.09 and 91.44 mg/L, respectively. Adsorption experiments of Ni^{2+} with chitosan derivatives strongly correlate with the Langmuir isotherm model with a high coefficient ($R^2 > 0.99$). This suggests that single-layer adsorption occurs at the surface of chitosan derivative adsorbents, while Freundlich adsorption isotherm is more suitable for chitosan [11]. In a research on heavy metal removal from leachate using illite, they investigated the adsorption of Cu (II) and Zn (II) in leachate of industrial wastes. Experimental studies showed that illitin is effective in the removal of Cu (II) and Zn (II) as an adsorbent and can be used as an alternative to other adsorbents in the treatment of leachate water due to its low cost. It has been found that illitin, an inexpensive material that provides an alternative to activated carbon, has sufficient binding capacity to remove Cu (II) and Zn (II) from leachate [12]. Mishra et al. conducted kinetic and isothermal studies of two types of soil and metal ions to measure the contact time, adsorbent dosage, and metal concentration in Cu (II) and Ni (II) experiments on red and black soils. The maximum removal rate for Cu (II) by red soil was 97.3% and for Ni (II) by black soil was 99.9%. Determination of the kinetic and adsorption isotherms reveals the adsorption experiments with the pseudo-second-order equation and the Freundlich isotherm model, respectively [13]. Mostaedi et al. investigated the removal of cadmium and nickel from aqueous solution using expanded perlite. Researchers found that the maximum removal efficiency of Cd (II) is 88.8% at pH 6 and exposure to 10 g/L expanded perlite, while for Ni (II) it is 93.3% at the same pH and exposure to 8 g/L adsorbent. For the adsorption of both metals, the Freundlich isotherm model fitted the equilibrium data better than the Langmuir isotherm model [14].

2. Materials and methods

2.1. Adsorbent: expanded perlite

In this study, expanded perlite was used as an adsorbent. The expanded perlite was obtained from the Izmir region in Turkey. Its physical and chemical properties are shown in Table 1 below.

2.2. Surface area experiment of expanded perlite

The surface area, adsorption, desorption, and average pore size of expanded perlite were measured using the Brunauer-Emmett-Teller (BET) method and shown in Table 2. BET surface area analysis was carried out with a Quantachrome Nova Touch LX4 instrument at the Kastamonu University Research Laboratory.

Table 1
Physical and chemical properties of expanded perlite [15]

Material	Percentage (%)
SiO_2	71–75
Al_2O_3	12.5–18
Na_2O	2.9–4
K_2O	4–5
CaO	0.5–0.2
Fe_2O_3	0.1–1.5
MgO	0.03–0.5
Free humidity	Max. 0.5
pH	6
Softening point	890°C–1,100°C
Melting point	1,280°C–1,380°C
Color	White and gray

2.3. SEM and EDS analyses of expanded perlite

2.3.1. Scanning electron microscopic analysis

As seen in Fig. 1, the scanning electron microscopic (SEM) and energy-dispersive X-ray spectroscopic (EDS) analyses were also analyzed using the FEI Quanta FEG 250 instrument at the Kastamonu University Research Laboratory.

2.3.2. EDS analysis

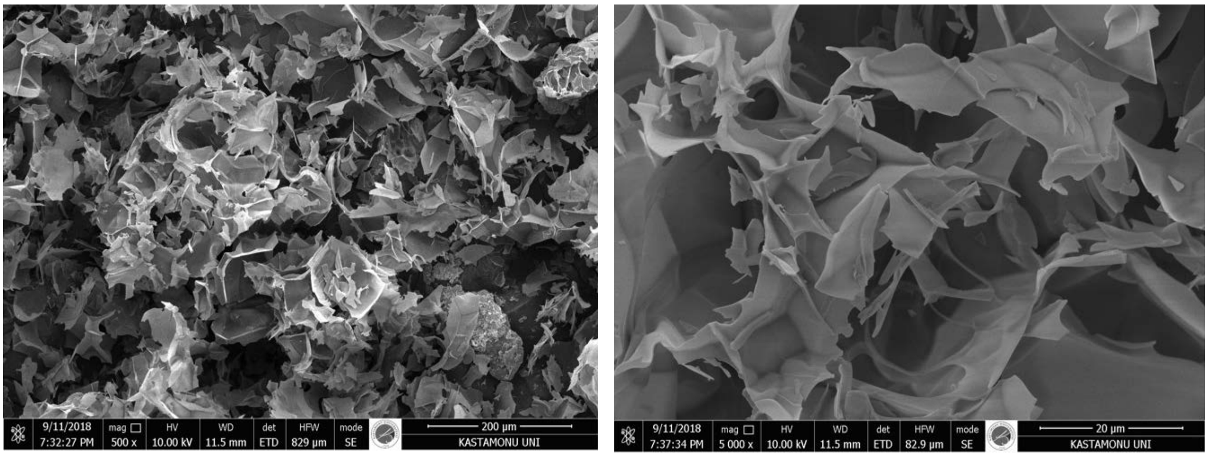
In the EDS analysis taken on the expanded perlite (Fig. 2), it is seen that there were 31.61% Si and 50.86% O elements. Al, Na, K, and Au elements were also determined in the analysis. As a result of the analysis, the high proportion of oxygen (O) element indicates the formation of a possible oxide compound (such as SiO_2, Al_2O_3, NaO, and KO) with other elements.

2.4. Adsorption study

Adsorption studies with expanded perlite were conducted according to the Batch method. Adsorption experiments were carried out at room temperature to investigate contact time, adsorbent dosage, initial metal concentration, pH, adsorption isotherms, and kinetic models. Ni^{+2} and Zn^{+2} (1,000 mg/L stock solution of $NiCl_2 \cdot 6H_2O$ and $ZnSO_4 \cdot 7H_2O$ (Merck, Darmstadt, Germany)) heavy metal salts were prepared and diluted to obtain the desired concentrations. All aqueous solutions were prepared from deionized water (ELGA Purelab Option-Q). The prepared samples were mixed at 500 rpm for 5-, 10-, 20-, and 30-min time intervals with a magnetic stirrer (2mag MIX 15 Eco) by adding 0.1, 0.2, 0.3, 0.4, and 0.5 g of expanded perlite in 500-mL beakers, respectively. Examples were then constructed on the Perkin Elmer Optima 5300 DV Optical Emission Spectrophotometer (ICP-OES). The efficiencies were then calculated using the following formula:

Table 2
Element analysis of expanded perlite

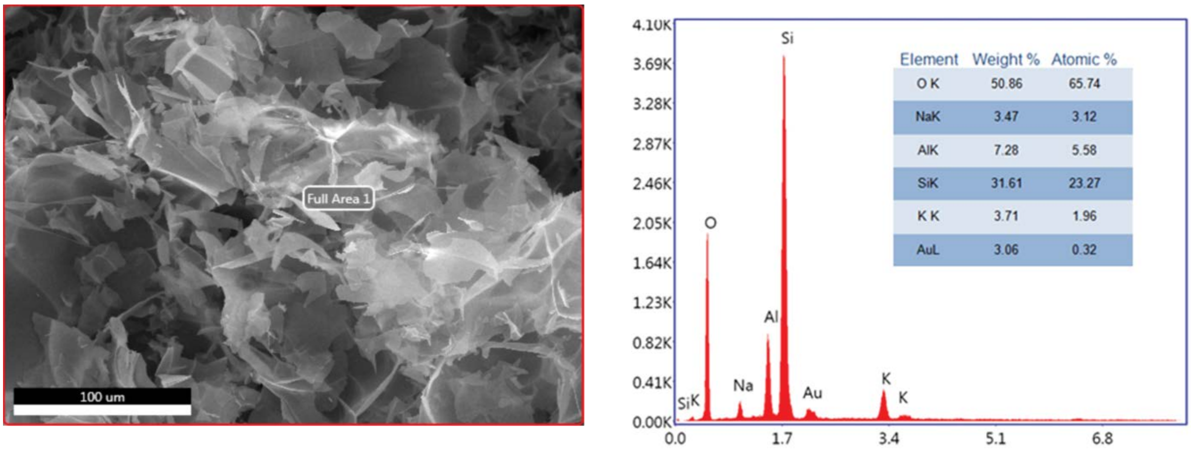
Element	Weight %	Atomic %	Net Intensity	Error %	*K* ratio	*Z*	*R*	*A*	*F*
O K	50.86	65.74	340.78	9.09	0.15	1.05	0.97	0.28	1
Na K	3.47	3.12	38.08	12.60	0.01	0.96	0.99	0.3	1
Al K	7.28	5.58	208.53	6.89	0.04	0.94	1.01	0.57	1.01
Si K	31.61	23.27	1004.42	5.18	0.19	0.97	1.02	0.63	1
K K	3.71	1.96	101.04	6.10	0.03	0.9	1.05	0.82	1.02
Au L	3.06	0.32	11.14	41.05	0.03	0.59	1.23	1.07	1.31



(a) SEM image of Expanded Perlite 500 ×

(b) SEM image of Expanded Perlite 5,000 ×

Fig. 1. SEM images of expanded perlite.



Element	Weight %	Atomic %
O K	50.86	65.74
NaK	3.47	3.12
AlK	7.28	5.58
SiK	31.61	23.27
K K	3.71	1.96
AuL	3.06	0.32

(a) SEM image of Expanded Perlite

(b) Lsec:28.0 0 Cnts 0.000 keV Det:Octane Pro Det

Fig. 2. EDS analysis of Expanded Perlite (EP).

$$\text{Efficiency}(\%) = \frac{E_i - E_e}{E_i} \times 100 \tag{1}$$

Where E_i = initial concentration (mg/L); E_e = exit concentration (mg/L)

3. Data analysis

3.1. Adsorption isotherm models

In the adsorption isotherm studies, expanded perlite was added to Ni (II) and Zn (II) solutions at initial concentrations ranging from 1 to 100 mg/L. Langmuir, Freundlich, and Temkin isotherm models were then applied to the obtained data.

The Langmuir isotherm describes single-layer adsorption to the homogeneous surface of adsorbent in any interaction between adsorbed Ni (II) and Zn (II) ions. It is expressed by the following Eq. (2).

$$\frac{1}{q_e} = \frac{1}{q_m K} + \frac{1}{C_e} \tag{2}$$

Due to the interaction between the adsorbed ions, single-layer adsorption occurs at the heterogeneous surface of the adsorbent and the Freundlich isotherm is then applied using the following Eq. (3).

$$\log C_s = \log K_f + \frac{1}{nf} \log C_e \tag{3}$$

The Temkin isotherm is expressed as shown in Eq. (4).

$$q_e = B \ln A + B \ln C_e \tag{4}$$

3.2. Adsorption kinetics

To explain the adsorption kinetics of Ni (II) and Zn (II) ions on the expanded perlite, Lagergren pseudo-first-order and pseudo-second-order kinetic models were applied to the experimental data.

The Lagergren pseudo-first-order kinetic equation is as follows:

$$\log\left(q_{\text{den}} - q\right) = \log\left(q_{\text{den}}\right) - \frac{k_1}{2.303} t \tag{5}$$

The experimental data were applied to the pseudo-second-order kinetic model given in Eq. (6).

$$\frac{t}{q_t} = \left[\frac{1}{k_2 q_e^2}\right] + \left(\frac{1}{q_e}\right) t \tag{6}$$

4. Results and discussion

4.1. BET surface area

The surface area of the expanded perlite, adsorption, desorption, and average pore width were all measured. The results show that 1 g of the surface area of expanded perlite was 375.49 m^2/g and the average pore size was 0.823 nm (Table 2).

4.2. pH effect

The effect of pH on the adsorption of Ni (II) and Zn (II) with expanded perlite was measured at an initial heavy metal concentration of 5 mg/L and at 25°C. The changes in pH value are shown in Fig. 3.

As shown in Fig. 3, the adsorption efficiency increases at pH = 5, while adsorption is at its lowest at pH = 2. The optimal pH-dependent removal efficiencies for Ni (II) ions are pH = 7, 85%; pH = 9, 82%; and pH = 11, 84% and for Zn (II) ions are pH = 7, 83%; pH = 9, 85%; and pH = 11, 86%. Because removal efficiency is lower at higher pH values, additional H^+ ions left in the aqueous solution cause the expanded perlite surface to become positive, affecting the repulsive force between the

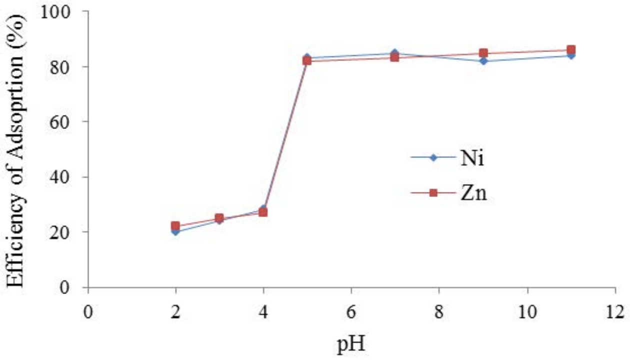


Fig. 3. pH effect.

molecules and causing the amount of heavy metal adsorbed to decrease.

4.3. Contact time

Adsorption efficiency was investigated for the removal of Ni (II) and Zn (II) ions in terms of the time of contact. The initial concentration was chosen to be 5 mg/L for both heavy metals. In the Ni (II) ion experiments, the turning point at which optimal removal was obtained was 10 min. Expanded perlite of 0.4 g was then added to remove 93% of the initial concentration of Ni (II) ions. In the Zn (II) ion experiments, the turning point was 5 min; 91% removal efficiency was then obtained by adding 0.5 g of expanded perlite as shown in Fig. 4.

4.4. Effect of initial concentration

In the adsorption experiments with expanded perlite, the effect of the initial heavy metal concentration was achieved at 25°C with a pH ranging from 5 to 6.5. The effect of the initial concentration on removal efficiency is shown in Fig. 5.

The removal efficiencies for the initial Ni (II) heavy metal concentrations of 1, 5, 10, 25, 50, and 100 mg/L were 96.5%, 93.12%, 75.98%, 44.72%, 22%, and 21.7%, respectively. For Zn (II) concentrations, they were 97.6%, 91.96%, 59.43%, 54.88%, 45.16%, and 13.84%, respectively. Therefore, as the heavy metal concentration increased, the removal efficiencies decreased. This is because the amount of adsorbent was insufficient. Recovery at concentrations of 50 and 100 mg/L resulted in very low yields.

4.5. Effect of adsorbent dosages

Concentrations of Ni (II) and Zn (II) and adsorption percentages are shown in Fig. 6. The initial concentration for both heavy metals was 5 mg/L. As shown in Fig. 6, the percent adsorption values increased at certain rates for both heavy metals. The optimal removal efficiencies after a certain value were 0.4 g (93.2%) and 0.5 g (91.96%) of expanded perlite for Ni (II) and Zn (II), respectively.

4.6. Isotherm studies

Adsorption experiments were conducted in a laboratory environment. Ni (II) and Zn (II) adsorption isotherms on the expanded perlite were investigated according to Freundlich, Langmuir, and Temkin isotherm models in Table 3. Adsorption of 10 different heavy metal concentrations with expanded perlite showed that the Ni (II) ion was compatible with the Freundlich isotherm model (R^2 = 0.91) and the Zn (II) ion was compatible with the Langmuir isotherm model

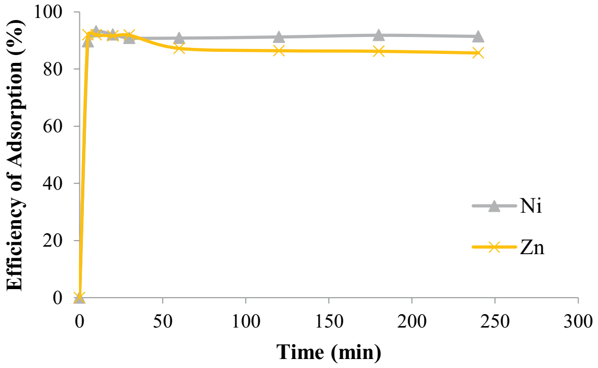


Fig. 4. Contact time (initial concentration: 5 mg/L, room temperature 25°C, pH = 5 for Ni (II) and pH = 6.5 for Zn (II). Adsorbent dose: 0.4 g for Ni (II) and 0.5 g of expanded perlite for Zn (II).

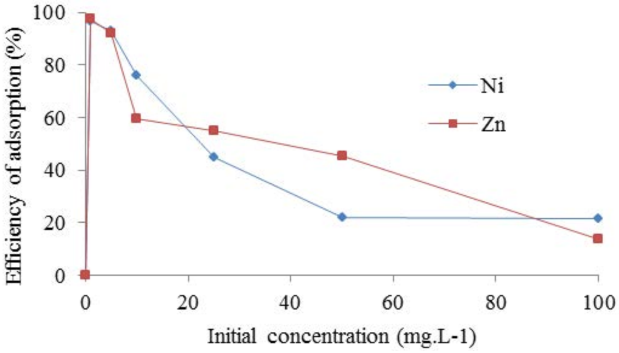


Fig. 5. Effect of initial concentration (room temperature 25°C, pH for Ni (II) and Zn (II): 5–6.5). Dose of adsorbent: 0.4 g for Ni (II) and 0.5 g for Zn (II).

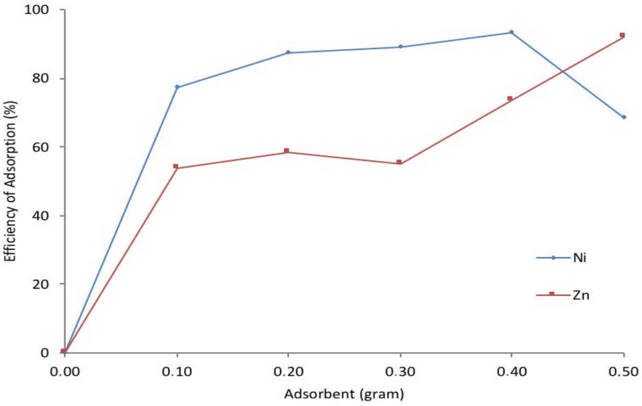
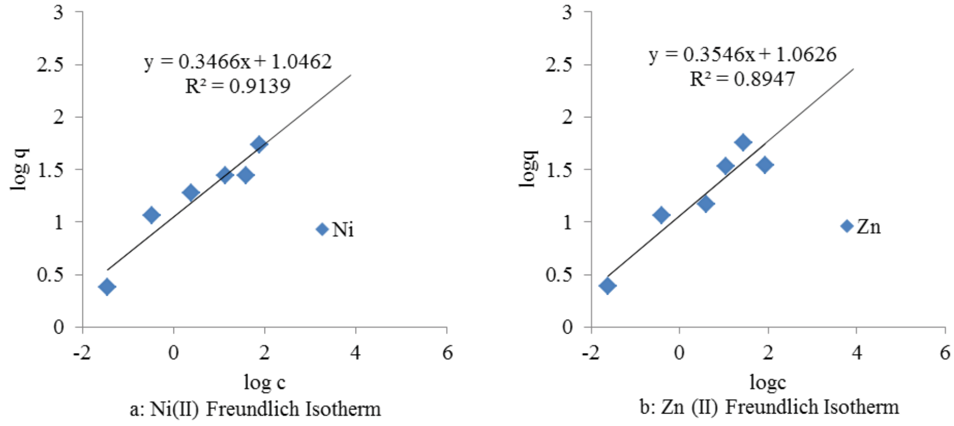


Fig. 6. Effect of adsorbent (pH: 5 for Ni (II) and 6.5 for Zn (II), initial concentration for both ions: 5 mg/L, rinsing time for Ni (II) was 10 min and Zn (II) was 5 min).

(R^2 = 0.97). The isotherms obtained are shown in Figs. 7–9. The isotherm constants are presented in Table 4.

For the Freundlich isotherm, correlation values of 0.89 for Zn (II) ions in Fig. 7(b) and 0.91 for Ni (II) ions in Fig. 7(a) were obtained. The adsorption of Freundlich isotherms also indicates that the surface may have heterogeneous properties and that adsorption is physically taking place.

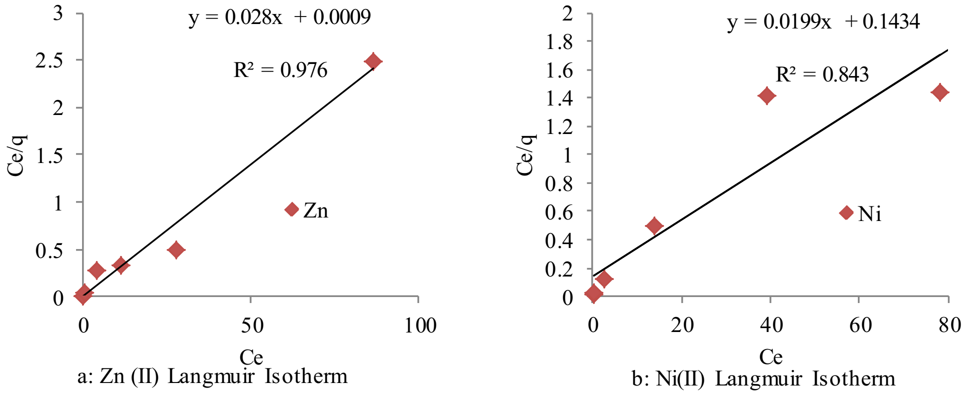
As shown in Fig. 8, in the Langmuir isotherm model, the correlation coefficient value of adsorption for Ni (II) on expanded perlite was 0.84 in Fig. 8(b) and for Zn (II) on expanded perlite it was 0.97 in Fig. 8(a). Langmuir isotherm model conformity can be interpreted as showing that the surface of the adsorbent is homogeneous and covered with a single layer.



a: Ni(II) Freundlich Isotherm

b: Zn (II) Freundlich Isotherm

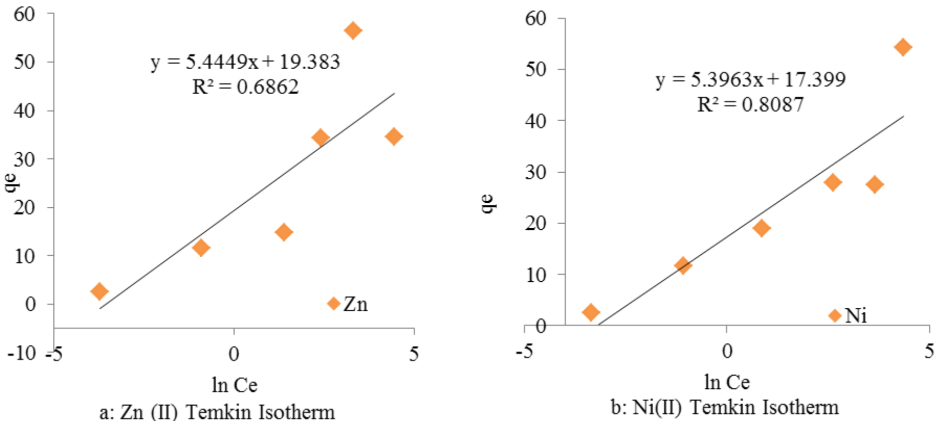
Fig. 7. The adsorption of Ni (II) and Zn (II) ions on the expanded perlite by the Freundlich isotherm model.



a: Zn (II) Langmuir Isotherm

b: Ni(II) Langmuir Isotherm

Fig. 8. Langmuir isotherm of adsorption of Ni (II) and Zn (II) ions on the expanded perlite.



a: Zn (II) Temkin Isotherm

b: Ni(II) Temkin Isotherm

Fig. 9. Temkin isotherm of adsorption of Ni (II) and Zn (II) ions on the expanded perlite.

Table3
Isotherm constants

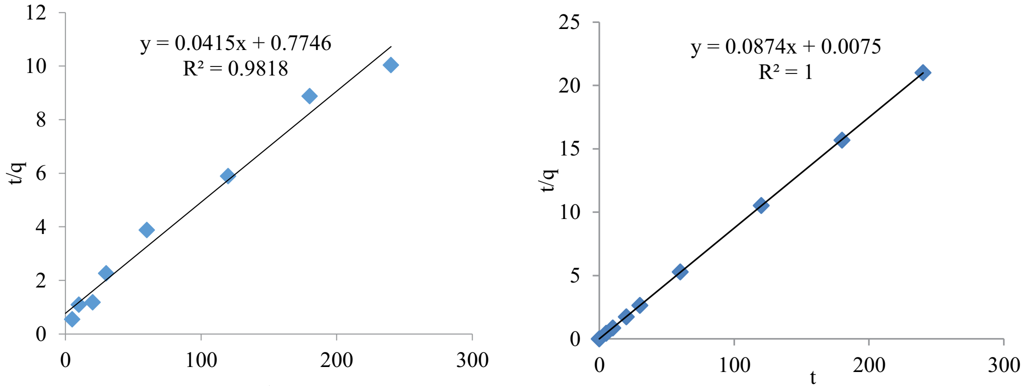
Freundlich isotherm		Langmuir isotherm		Temkin isotherm	
Zn (II)	Ni (II)	Zn (II)	Ni (II)	Zn (II)	Ni (II)
$K_f = 1.002$	$K_f = 11.122$	$Q_o = 2.475$	$Q_o = 50.25$	$B = 5.4449$	$B = 5.3963$
$n_f = 35.714$	$n_f = 2.885$	$b = 2.18$	$b = 7.2$	$A_t = 34.813$	$A_t = 25.028$
$R^2 = 0.89$	$R^2 = 0.91$	$R^2 = 0.97$	$R^2 = 0.84$	$R^2 = 0.68$	$R^2 = 0.80$

Table 4
Analysis of expanded perlite according to BET surface area

BET analysis	Expanded perlite
Surface area, m²/g	375.9
Pore volume, cc/g	0.4605
Total pore volume, cc/g	0.4826
Average pore size, nm	0.823
Molecular weight, g	28.0134

Table 5
Adsorption kinetic parameters

Pseudo-first-order kinetics		Pseudo-second-order kinetics	
Zn (II)	Ni (II)	Zn (II)	Ni (II)
$K_1 = 0.00182$	$K_1 = 0.00161$	$K_2 = 0.0222$	$K_2 = 1.019$
$q_e = 0.437$	$q_e = 0.271$	$q_e = 24.096$	$q_e = 11.441$
$R^2 = 0.34$	$R^2 = 0.12$	$R^2 = 0.98$	$R^2 = 1$



a: Zn(II) The pseudo second-order kinetic

b: Ni(II) The pseudo second-order kinetic

Fig. 10. Pseudo-second-order kinetic graph of the adsorption of Ni (II) and Zn (II) heavy metals on expanded perlite.

In the adsorption study on the expanded perlite, the correlation values of Ni (II) and Zn (II) ions for the Temkin isotherm were 0.80 in Fig. 9(b) and 0.68 in Fig. 9(a). Adsorption Temkin isotherm compliance suggests that the expanded perlite surface was coated with heavy metal and that, as the coating increased, the adsorption temperature decreased. It has therefore been found through adsorption that the Temkin isotherm conforms that the expanded perlite is covered with heavy metal and, as the coating increases, the adsorption temperature decreases. The same interpretation cannot be applied to Zn (II) ions, as this result can be interpreted as a decrease in adsorption heat by the Temkin isotherm model in the Ni (II) adsorption experiments.

4.7. Adsorption kinetics

The kinetics of Ni (II) and Zn (II) heavy metal adsorption with expanded perlite were investigated from the pseudo-first-order to the pseudo-second-order models. The adsorption kinetics at different concentrations were shown at 25°C and pH values ranging from 5.6 to 6. The data obtained are shown in Fig. 10, and the kinetic parameters are presented in Table 5.

The results show that the number of correlations from the first pseudo-kinetic model for reactions is very low and does not match. In the second kinetic model, the correlation coefficients for Zn (II) and Ni (II) are 0.98 in Fig. 10(a) and 1 in Fig. 10(b). Adherence to the pseudo-second-order kinetic model of adsorption indicates that there may be chemical activation between expanded perlite and heavy metals.

5. Conclusion

In this study, expanded perlite was used as an adsorbent for the adsorption of Ni (II) and Zn (II) heavy metals. The effects of adsorption contact time, pH, adsorbent dosage, and inlet concentration were all investigated. Adsorption isotherm and kinetic studies were also conducted. The results show that the adsorption efficiency increases with adsorbent dosage, pH, and contact time, while an increase of heavy metal concentration causes a decrease in adsorption. The kinetic studies show that the adsorption of Ni (II) and Zn (II) heavy metals on expanded perlite conforms to the pseudo-second-order kinetic model but does not fit the pseudo-first-order kinetic model. Langmuir isotherms were found to be compatible with the adsorption of Zn^{2+} heavy metal with expanded perlite, and Freundlich isotherms were found to be compatible with the adsorption of Ni^{2+}. Therefore, the expanded perlite is preferred because it is a cheap and abundant material that can be used as a suitable adsorbent for the adsorption of Ni (II) and Zn (II) heavy metals.

Acknowledgement

This study was carried out as part of project BEBAP 2017-02 and was supported by the Scientific Research Projects Coordinator at Bitlis Eren University, Bitlis, Turkey. I would also like to thank my graduate student Ümit Korkutata.

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