



Adsorption isotherms and thermodynamic Study of Cu (II) and Ni (II) removal using commercial zinc oxide nanoparticle

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Abstract

This present work done on the adsorption isotherm study for the removal of Cu (II) and Ni (II) ions from aqueous solution using industrial zinc oxide nanoparticles. Various techniques such as AFM, XRD and FT-IR were employed to characterize the surface. From the XRD analysis, each reflection peak with the relative intensity of different planes confirmed the presence of ZnO and the spectrum revealed that the particles size was about (27.43 nm), which is in good agreement with those estimated from AFM and mean that the particle diameters are in the nanometer range. Adsorption experiments were studied to investigate the effect of contact time, adsorbent dose, initial concentration, and temperature on the adsorption capacity. The equilibrium data were evaluated by the Langmuir, Freundlich, Elovich and Halsey models of adsorption. The removal of Cu (II) and Ni (II) by ZnO nanoparticles follows the Freundlich and Halsey isotherm which fits well with the experimental data ($R^2 = 0.9718, 0.9942, 0.9525$ and 0.9941 respectively). The maximum percentages of Ni (II) and Cu (II) removal were found to be above 96.03 % and 96.45 % as well. Finally, the thermodynamic parameters (ΔH , ΔS , ΔG°) were calculated and indicated that the adsorption process is exothermic, spontaneous, and less random when metal ions overlap with the commercial zinc oxide nanoparticles. The higher percentage removal at higher dosage value of ZnO could be due to an increased active sites for the adsorption of metals ions with an increased amount of adsorbent dosage.

Keywords: adsorption, Cu (II), Ni (II), commercial zinc oxide, nanoparticle.

1. Introduction

It is known with the development of technology that the problems of the ecosystem are increasing. One of the main problems is heavy metal pollution, so the removal of heavy metals from sewage and industrial waste has become a very important issue [1]. Heavy metals are major pollutants in land, marine, industrial and even treated wastewater. The most specific sources of heavy metal pollutants are industrial wastewater from mining, mineral processing, organic chemicals, pharmaceuticals, tanneries, pesticides, rubber and plastics. Heavy metals are transported by runoff water and pollute water sources downstream from the industrial site. It is essential to remove toxic heavy metals from wastewater prior to disposal to avoid health risks. Most of the heavy metals discharged in wastewater have been found to be carcinogenic and toxic and pose a serious threat to human health [2]. Physical and chemical methods can be used to remove heavy

metal ions that are hazardous to health from wastewater [3]. Widely used methods for removing inorganic and organic waste that cause environmental pollution and are harmful to human health include ion exchange, chemical precipitation, membrane filtration and adsorption. The Adsorption process is widely used because of its high efficiency in the removal of heavy metal ions. Such ions, organic pigments, radioactive elements and materials employed in the adsorption of non-ionic pollution and paint are removed by adsorbents such as zinc oxide [4]. Zinc oxide (ZnO) is one such important metal oxide NP which has gained the scientific spotlight currently. The unique set of characteristics of ZnO NPs such as catalysis, medicinal effects, photochemical capability, fungicidal, antibacterial and UV filtration has made ZnO a multi-functional agent and more promising for wastewater remediation [5].

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The aim of the present investigation is to study the adsorption isotherms of copper (II) and nickel (II) ions removal on commercial zinc oxide nanoparticles and also determine the thermodynamic parameters of adsorption process.

2. Materials and Methods

2.1 Materials used

Standard solutions of nickel (II), copper (II) ions were prepared at a concentration of 1000 mg/L of $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ and $\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ in deionized water. Also, different concentrations (20-40-60-80 and 100) mg /L of standard stock solutions were prepared for each metal ion. Then, using atomic absorption, the concentrations were determined and the absorbance of the solutions measured with wavelength of up to 232 nm for nickel and 324.7 nm for copper ions.

2.2 Adsorbate

A standard stock solutions of nickel (II) and copper (II) ions (1000) mg/L were prepared from the salts of $\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ and $(\text{CuSO}_4 \cdot 5\text{H}_2\text{O})$ in deionized water. Many concentrations (20, 40, 60, 80 and 100) mg/L were prepared from this standard stock of each metal ions. The absorbance of these solutions was measured at a wavelength λ_{max} (232 nm) for nickel and (324.7 nm) for copper in order to determine the concentrations of metals by atomic absorption.

2.3 Study of the adsorption

Several volumetric flasks containing 50 ml of nickel (II) or Copper (II) ions solution with C_o of (100 mg/L) for each one, pH of 6, then (0.1 g) of commercial zinc oxide was added into each flask. These materials are placed in a water bath shaker device at speed (185 rpm) and room temperature (298 K) at different time intervals (10, 20, 30, 40 and 50) min. Then samples are filtered before analysis to prevent nanoparticles interference with analysis. The concentrations of metal solutions were determined using atomic absorption. Studied factors influencing the adsorption of the two metals onto commercial zinc oxide are: contact time, adsorbent quantity, initial concentration, temperature. The two metal removal percentage was calculated using the equation below [6,7].

$$R \% = \frac{(C_o - C_e) * 100}{C_o} \dots\dots (1)$$

Where: (R %) is the percentage removal of the two metals, (C_o) is the initial concentration (in a single

system) of metal ions (mg/L), (C_e) is the concentration of copper and nickel ions after removal (mg/L).

3. Characterization of adsorbent

3.1 Characterization of commercial zinc oxide nanoparticle

3.1.1 X-ray diffraction

The X-ray diffraction pattern of the prepared oxide was recorded using XRD-6000 with CuK ($\lambda = 1.5406 \text{ \AA}$) that has an accelerating voltage of 220/50 HZ which is produced by SHIMADZU company. XRD technology was used to determine and confirm the crystalline structure of nanoparticles through the use of commercial zinc oxide in Figure 1 (a). By recording the full width half-maximum (FWHM) of any of the most prominent peaks, the average size of the nano crystallites is determined using Debye-Scherrer's equation ($D = \frac{0.9 \lambda}{\beta \cos \theta}$), $k = 0.99$ for spherical particles. The estimated particle size of the commercial zinc oxide is 27.43 nm. The presence of sharp peaks in XRD samples and particle size of less than (100) nm refers to the nanocrystalline nature of the surfaces. The particle sizes were calculated from Debye-Scherrer formula in given below [8]:

$$D = \frac{0.9 \lambda}{\beta \cos \theta} \dots\dots (2).$$

Where: (D) is the crystallite size, (λ) is the wavelength of radiation, (θ) is the Bragg's angle, (β) is the full width at half maximum (FWHM).

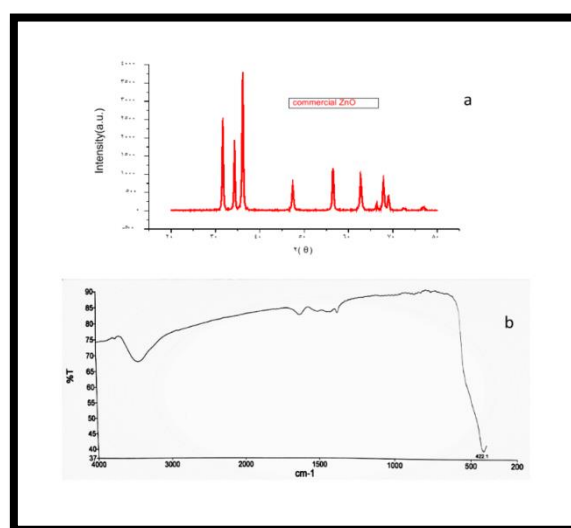


Figure 1: (a) XRD, (b) FT-IR of commercial zinc oxide nanoparticle.

3.1.2 Fourier transforms infrared spectrum analysis (FT-IR)

Infrared spectroscopy is used to detect the presence of functional groups attached to the compounds, and Figure 1 (b) shows the infrared spectrum of the commercial zinc oxide. The metal oxides give general absorption bands in the fingerprint region, i.e. less than 1000 cm^{-1} arising from inter-atomic vibrations. The peak is observed as a broad band in the confined area between $3300\text{--}3400\text{ cm}^{-1}$. This band is due to the vibration of the stretching of the (O-H). The Peak located at 422.1 cm^{-1} is a distinctive peak for ZnO, which corresponds to the expansion mode for ZnO [9].

3.1.3 Atomic Force Microscope

The AFM analysis is used to provide information about the morphology of the surface and average grain size. Figures (2) explain images of AFM of the commercial zinc oxide and (CLE.ZnO.NP) with (size = $200.0, 200.0\text{ nm}$), and ability analytical (pixel = $256,256$), respectively. The Figures (2A) are AFM images in three dimensions (3D), Figure (2 B) are AFM images in two dimensions (2D), and Figure (3) explains the grain distribution diagram of commercial zinc oxide nanoparticles. It is found that the mean roughness is (5.083 nm), for both surfaces. The root mean square (RMS) is (1.24 nm) [10].

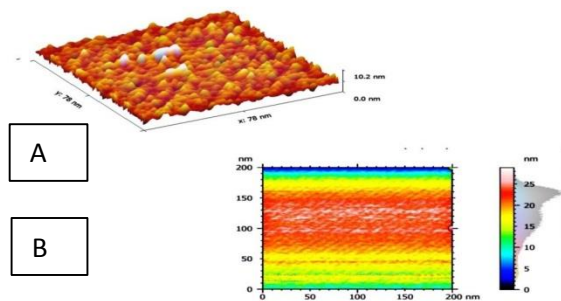


Figure 2: AFM images of commercial zinc oxide nanoparticle

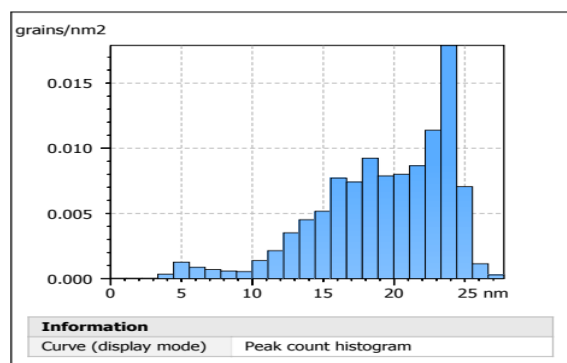


Figure 3: Granularity cumulating distribution of commercial zinc oxide nanoparticles.

4. Removal Cu (II) and Ni (II) ions on (commercial zinc oxide) surface

4.1 Contact time effect

The adsorption of nickel and copper ions on a commercial zinc oxide surface reached 30 minutes, according to the experimental results obtained and shown in Figure 4 (a). Initially, a decrease in the percentage of removal of Cu (II) and Ni (II) ions (commercial zinc oxide) was observed, then a gradual increase in the percentage of removal of ions was observed and a cause of the initial rapid increase in the rate. The modifier in later stages is the availability of excess adsorption sites on the adsorbents. Ion exchange followed by the slow chemical reaction of the active groups of metal ions may be a reason for the high rate of initial absorption in sample [11], and the remaining vacant surface sites are difficult to fill due to repulsive force. Metal ions have to traverse deeper and more in pores, encountering greater resistance [12].

4.2 Adsorbent quantity effect

Figure 4 (b) shows the amount of adsorbent on the absorption of nickel (II) and copper (II) ions using (commercial zinc oxide) as a surface at a concentration of 100 mg/L . The results of the experiment showed an increase in the removal rate with an increase in the amount of adsorbent. Due to the increased surface area, the percentage of desorption increases as adsorption occurs, and the optimum amount of adsorption that can be used to remove nickel (II) and copper (II) ions is (0.1 g). After that, the removal percentage will increase a little [13].

4.3 Temperature effect

The effect of temperature on the extent of adsorption of copper and nickel ions on commercial zinc oxide was studied at different temperatures ($298, 308, 318$ and 328 K), weight 0.1 gram of the surface at an initial concentration of 100 mg/liter . The contact time was constant at 30 min . Figure 4 (c) shows the general shape of the nickel (II) and copper (II) ions, (commercial zinc oxide). The data showed that the percentage of removal decreases with increasing temperature, and this proves that the removal of individual metals is exothermic in nature for each of the nickel-copper ions present at the surface (commercial zinc oxide) As the temperature increases, a decrease in the absorption rate may weaken the strength of the interaction between the active sites on the adsorption surface and the single metal ions [14].

4.4 Initial concentration effect

The adsorption of the single metals from an aqueous solution onto (commercial zinc oxide) was studied first at optimum conditions, using different initial

concentration of aqueous solution of (20, 40, 60, 80 and 100 mg/L) for all metal ions. The results shown in Figure 4 (d) that the impact of the initial concentration indicate little decrease in the removal with increasing of the initial concentration of nickel (II) and copper (II) ions on the surface (commercial zinc oxide). The small decrease in the percentage of the removal at higher concentration could be attributed to the limited number of active sites in zinc oxide nanoparticles adsorbent, which become more saturated with an increases in the concentration of metal ions, and removal of nickel (II) and copper (II) ions on commercial zinc oxide is high because of the small nanoparticle size and the high surface area that contains a lot of active adsorption sites which will be available [15].

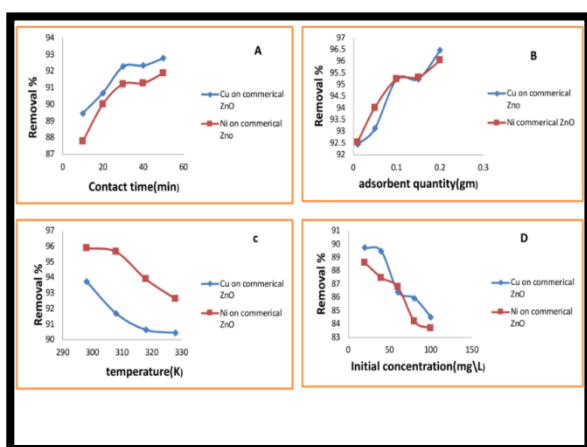


Figure 4: Show effect of (A) contact time, (B) adsorbent quantity, (C) temperature and (D) initial concentration.

5. The adsorption isotherm

In order to studied adsorption interaction were used four greatest communal adsorption isotherms, Langmuir, Halsey, Freundlich and Elovich.

5.1 Langmuir isotherm

The Langmuir equation, which is valid for monolayer adsorption onto a completely homogeneous surface with a finite number of identical sites and with negligible interaction between adsorbed molecules, shown as in figure 5. The equation of Langmuir is assumed by [16] :

$$\frac{C_e}{Q_e} = \frac{1}{ab} + \frac{C_e}{a} \dots \dots (3).$$

Where: Q_e : The quantity adsorbed at equilibrium in (mg/g). C_e : the equilibrium concentration of adsorbate in (mg/L). a : The Langmuir constant which is a measure of adsorption capacity in (mg/g).

b : also the Langmuir constant which is a measure of energy of adsorption in (L/mg).

5.2 Freundlich isotherm

Freundlich isotherm equation (4) was applied on adsorption of Cu^{+2} and Ni^{+2} ions on commercial zinc oxide by plotting of $\log Q_e$ against $\log C_e$ [17].

$$Q_e = K_f C_e^{1/n} \dots \dots (4).$$

The Freundlich isotherm constants (K_f) which is the adsorption capacity of the adsorbent, and (n) which is the adsorption intensity being calculated from the slope and intercept of the plot of ($\log Q_e$) versus ($\log C_e$) as shown in figure 6 .

5.3 Elovich isotherm

The Elovich equation model depends on a kinetic principle that the adsorption sites greatly increase with adsorption, which implies multilayer adsorption. Equation of Elovich isotherm is assumed by [18]:

$$q_e \setminus q_m = K_E C_e^{q_e/q_m} \dots \dots (5).$$

Where: q_e : (mg/g) is the amount of absorbed material at equilibrium. q_m : (mg/g) is the Elovich maximum adsorption capacity. K_E : (L/mg) is the Elovich equilibrium constant. C_e : (g/L) is the equilibrium concentration.

If the adsorption obeys the Elovich equation, Elovich maximum, adsorption capacity and Elovich constant can be calculated from the slopes and intercept of the plote $\ln (q_e \setminus C_e)$ versus q_e as shown in figure 7.

5.4 Halsey isotherm

The Halsey isotherm model can be used to evaluate the multilayer absorption system for metal ions at a relatively large distance of the surface. the equation of Halsey isotherm is assumed by:

$$q_e = \frac{1}{n_H} \ln K_H - \frac{1}{n_H} \ln C_e \dots \dots (6).$$

where: K_H and n_H are the Halsey constants. which can be obtained from the slope and the intercept of the linear plot $\ln q_e$ against $-\ln (1/C_e)$ respectively. As shown in figure 8.

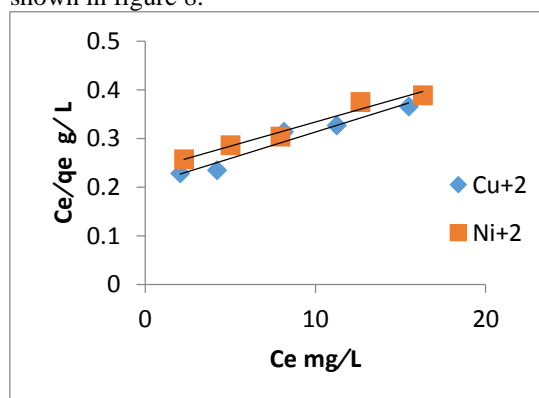


Figure 5.Langmuir adsorption isotherms for removal Ni^{+2} and Cu^{+2} ions.

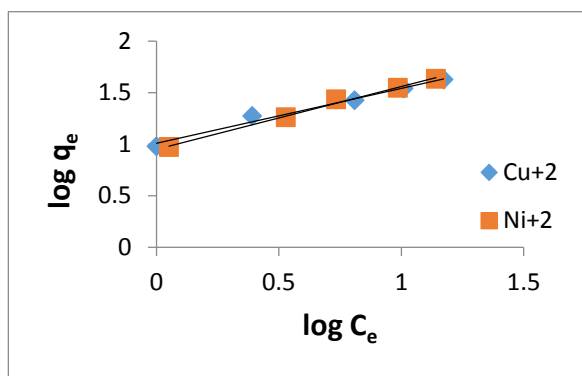


Figure 6. Freundlich adsorption isotherms for removal Ni²⁺ and Cu²⁺ ions.

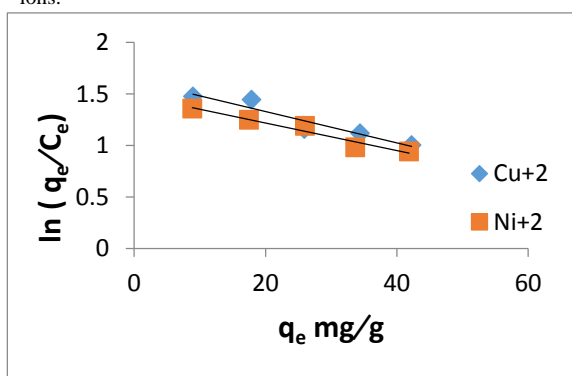


Figure 7 Elovich adsorption isotherms for removal Ni²⁺ and Cu²⁺ ions.

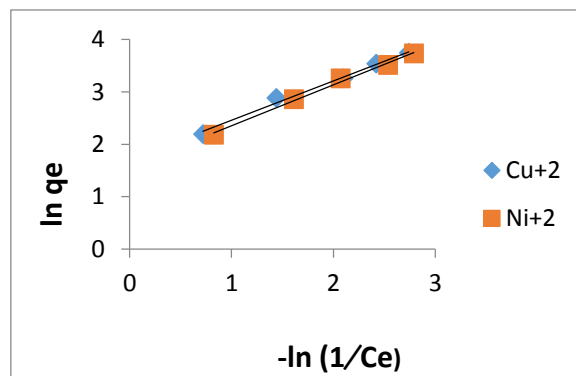


Figure 8., Halsey adsorption isotherms for removal Ni²⁺ and Cu²⁺ ions.

Table (1) explain the Langmuir, Freundlich, Elovich and Halsey constants and R² for adsorption of Cu²⁺ and Ni²⁺ ions in the presence of a variable initial ion concentrations. By the values of R² the Freundlich and Halsey isotherm was found to be the best one that could describe the Cu²⁺ and Ni²⁺ removal.

Table 1. Explain the Langmuir, Freundlich, Elovich and Halsey constants and R² for the adsorption of Cu (II) and Ni (II) ions with commercial zinc oxide nanoparticle.

Models & Metals		Commercial zinc oxide	
Langmuir	a(mg\g)	b(L\g)	R ²
Cu ²⁺	91.743	0.0533	0.9505
Ni ²⁺	100	0.04	0.9718
Freundlich	n	K _f (mg/g)	R ²
Cu ²⁺	1.3292	5.5055	0.9908
Ni ²⁺	1.2802	4.8250	0.9942
Elovich	q _m (mg\g)	K _E	R ²
Cu ²⁺	65.789	0.975	0.9169
Ni ²⁺	75.187	0.980	0.9525
Halsey	n _H	K _H	R ²
Cu ²⁺	1.3303	4.1457	0.9909
Ni ²⁺	1.2802	3.7684	0.9942

6. Thermodynamic study

The effect of temperature on removal of metals in a single system on adsorbent nanoparticles at different temperatures (298, 308, 318 and 328) K was investigated. This study will help change in evaluation of the basic thermodynamic function enthalpy, change ΔH (kJ/mole), (change of free energy ΔG , (kJ/mole), and entropy, and change ΔS (J/mole. K). Equilibrium of adsorption constant, K is explained thermodynamically by the Van 't Hoff equation given below:

$$\ln K = \frac{\Delta H}{RT} + \frac{\Delta S}{R} \dots\dots\dots (7).$$

The equilibrium constant, K were calculated at any different temperature by below equation [17].

$$K = \frac{Q_e \times m(g)}{C_e \times v(L)} \dots\dots\dots (8).$$

Where: Q_e : The adsorption capacity of metals ion, (mg/g). m : The quantity of metal oxide nanoparticles, (g). C_e : The concentration equilibrium after removal of metals in, (mg/L). V : Volume of aqueous solution contains copper (II) and nickel (II) ions, (L). Table (2) illustrates (K) values for adsorption of copper (II) and nickel (II) ions on (commercial Zinc oxide) at different temperatures.

Gibbs free energy change (ΔG°) can be calculated from relationship [19]:

$$\Delta G^\circ = -RT \ln k \dots\dots\dots (9).$$

Where: ΔG° : The standard free energy change, (kJ/mole). R : The universal gas constant, (8.314×10^{-3} J/mol. K). T : Temperature, (K). K : Thermodynamic equilibrium constant.

Table 2: Effect of temperature on equilibrium constants for the adsorption of copper (II) and nickel (II) ions on commercial zinc oxide nanoparticle.

Surface	T (K)	1/T (K ⁻¹)	C _e (mg/L)	Q _e (mg/g)	K	ln K
Cu ⁺²	298	0.00335	6.259	46.870	14.976	2.706
	308	0.00324	8.321	45.839	11.017	2.399
	318	0.00314	9.367	45.31	9.675	2.269
	328	0.00304	9.552	45.224	9.469	2.248
Ni ⁺²	298	0.00335	4.113	47.943	23.312	3.149
	308	0.00324	4.354	47.823	21.967	3.089
	318	0.00314	6.108	46.946	15.371	2.732
	328	0.00304	7.377	46.311	12.555	2.530

Values (ΔH) and (ΔS) can be calculated from the slope and intercept by drawing ($\ln K$) versus ($1/T$), as shown Figures (9) and (10) and explain below equations [20]:

$$\text{Slope} = -\Delta H/R \dots\dots\dots (10).$$

$$\text{Intercept} = \Delta S/R \dots\dots\dots (11).$$

Tables (2) display the data for the effect of copper (II) and nickel (II) ions removal on the adsorbent and Figures (9) and (10), shows the Van't Hoff plots for adsorption on commercial zinc oxide.

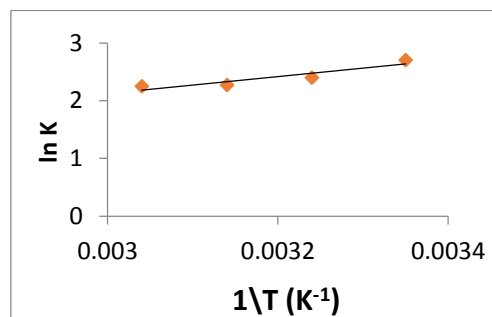


Figure 9: Van't Hoff plot for adsorption of Copper (II) ions on commercial zinc oxide.

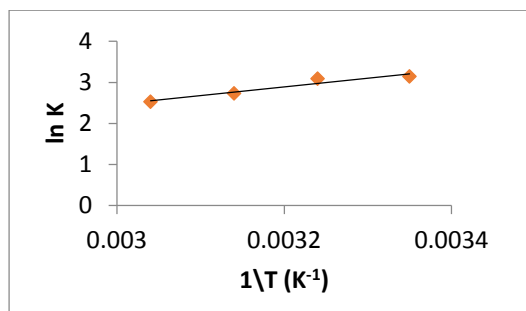


Figure 10: Van't Hoff plot for adsorption of nickel (II) ions on commercial zinc oxide.

The ΔG value of the metal ion removal on two adsorbents being negative, showed that the adsorption processes were spontaneous and ΔG values also determine the rate of the adsorption reaction, $\Delta G < 0$ indicates physisorption process. The rate increases as ΔG increases. The value of ΔH is negative, indicating that the adsorption process was exothermic in nature. The negative value of ΔS , indicates decreases in randomness at the (solid-solution) interface due to adsorption of copper (II) and nickel (II) ions on commercial zinc oxide nanoparticle.

Table 3. Show value of thermodynamic function for adsorption of Cu^{+2} and Ni^{+2} ions.

	T/ K	ΔG KJ/ mol	ΔH KJ/mol	ΔS KJ/ mol
Cu^{+2}	298	-6.7043		
	308	-6.1431		
	318	-5.9989	-12.2506	-9.1100
	328	-6.1302		
Ni^{+2}	298	-7.8018		
	308	-7.9100	-17.7695	-32.827
	318	-7.2230		
	328	-6.8992		

Conclusion

Zinc oxide nanoparticle was used as an absorbent material to remove heavy metals, where the percentage of removal of both nickel (II) and copper (II) ions was (96.45%, 96.03%) respectively. The Langmuir, Freundlich, Halsey and Elovich models were also applied to the experimental data. It was found that Freundlich and Halsey models fitted the experimental data well for adsorption onto commercial zinc oxide nanoparticles. As well, the negative values of the thermodynamic functions of Cu^{+2} and Ni^{+2} ions adsorption indicated that the adsorption processes are spontaneous and exothermic and there was less randomness at the solid-solution interface.

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