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Effective Adsorptive Removal of Ni2+ from Aqueous Solutions using MgO Adsorbent: A Study of Kinetics and Adsorption Model

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Abstract: **The threats to humans' and animals' health are increased by even very modest concentrations of metallic ions in water. Despite the fact that there are numerous treatment options are employed to remove heavy metals from wastewater such as chemical precipitation, ion exchange, membrane filtration, electrochemical treatment, coagulation, flocculation, adsorption and co-precipitation. So many researchers used the method of adsorption which is a highly effective and economical technique for the removing of toxic heavy metals from the wastewater. This paper through some light on the removal of toxic heavy metals ion (nickel (II)) from wastewater using magnesium oxide as adsorbent. Equilibrium models are applied to calculate the Langmuir and Freundlich and kinetics isotherms of the reaction, whereas the isotherm models confirm the experimental data. The sorption of nickel ion (II) onto magnesium oxide was strongly adsorbed dependent on contact time, initial of nickel (II), temperature, pH and adsorbent dosage. Thermodynamic parameters can affirm as ΔG° negative value, and a ΔS° positive value shows that the adsorption of nickel (II) ions by magnesium oxide was spontaneously happened. A positive result indicates endothermic adsorption for ΔH°. Finally, the pseudo-second-order equation provided the adsorption data.**

Keywords: **Magnesium Oxide, Nickel, kinetic model, heavy metals, Thermodynamic, Adsorption.**

I. INTRODUCTION

Fresh water is very important to human life. Water can be over the ground like lakes, rivers, and wetlands and could be underground. Water can be polluted by many chemicals discharged from industries, so we need to take a wide look at healthy freshwater habitats. Heavy metals are considered the main pollutants in marine, ground and surface waters caused by human activity [1]. Water is a vector for transporting heavy metals from their sources to our bodies via the food chains. Although heavy metals may be found in very small traces, which cannot be harmful, their toxicity appears by bioaccumulation in the organisms till reaching critical concentrations that make them dangerous. To respect the quality standards of water and food, eliminating heavy metals from wastewater is necessary [2].

The problem of heavy metals in water is widespread, as most are poisonous organisms, are not biodegradable, and usually build up in biota, putting the immune system in peril and causing threatening life on earth and cancer. The cohabitation of pollutants like heavy metals in water supplies could be due to frequently geological sources or growing discharge of wastewater from various industries, such as chemicals, plating, fertilizer tanning, metallurgical, mining, welding, metal finishing, alloy manufacturing, etc. [3]. Numerous methods, including chlorination, solvent attraction, chemical oxidation, membranes, coagulation and finally, adsorption, are employed to get rid of dangerous substances through industrial waste. Still, these are deemed high costs and demand high operating costs. Studies were undergone to find cost-effective techniques for eliminating pollutants from wastewater [4].

Adsorption is one of the greatest methods for its high effectiveness and is effective for removing almost all impurities; the procedure is simple and inexpensive [5]. The nickel symbol is Ni, it has the atomic number 28. It is the 24th most plentiful component of the crust and represents 3% of the earth's composition. It is a hard, malleable, silvery-white glossy metal from the transition metal family [6].

Metallic nickel is considered from lightest heavy metal. Using metals in equipment in industrial activities such as electroplating, and alloys is the main source of nickel. Sometimes nickel is found in groundwater because of dissolution from nickel-bearing rocks [7]. Nickel can be easily used in addition to other metals to form alloying, providing high-temperature stability, flexibility, toughness, strength, and corrosion resistance to water, alkali and air. Therefore, it is frequently used in different manufacturing processes [6].

Mucous membranes and Enduring skin contact may cause itchy and occasionally produce allergies. Nickel monoxide or metallic can be chronic inhalation and produce specific asthma and tubular dysfunction cases. Ingestion of nickel salts can yield nausea, vomiting, and diarrhoea. Also, Eczema from skin exposure to nickel is the most frequent Impact. Organics with nickel, for example, tetra-carbonyl-nickel, are highly toxic and carcinogenic, causing mainly lung cancer [7]. In present work, Magnesium oxide is used to remove Nickel (II) from polluted waters by adsorption.

II. MATERIALS AND METHODS

A. Preparation of nickel Stock Solution:

Nickel standard solution of concentration 1000 mg/l was prepared using 4 g from Nickel Sulfate added to 1000 milliliters of pure distilled water. Diluting Ni^{+2} ions stock solution from 1000mg/l was diluted to different concentrations with distilled water under the law of dilution. All chemicals used in experiments were very pure (99.9% purity). Solutions pH adjusted to the desired value by adding 0.1N drops of NaOH or 0.1 drops of N H_2SO_4 at a fixed temperature of 25 °C. Adsorbent: Magnesium Oxide purity of 99% was used.

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B. *Adsorption experiments*

Experiments were carried out using stirring magnesium oxide with a digital magnetic stirrer with temperature sensor PT 1000 (MS-H-Pro) and 250 ml of an aqueous nickel sulfate solution. Different temperatures, different initial concentrations for Ni (II), different weights of magnesium oxide and various pH levels of the fluid were the studied parameters. pH was adjusted by adding 0.01N HCl. HANNA pH 211- Romania. 25 ml were taken from the adsorption solution at different intervals for Ni (II) analysis. The samples were collected on a regular basis. They were subsequently examined using an atomic absorption spectrophotometer. Varian Flame AA240 Fs; Graphite GTA 120 Swiss.

The following equation is used to calculate the Ni removal percentage:

$$\%Removal = \%Adsorption = \frac{C_0 - C_t}{C_0} \times 100 \qquad (1)$$

The Ni (II) retention of concentration in the adsorbent phase (qe) magnesium oxide is calculated through the following equation2)

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

where:
C°: metal ion original concentration (mg/l|).
Ce: metal ion concentration (mg/l) at equilibrium or their final concentration in solution.
V: volume (l).
m: weight of adsorbent (g).

III. RESULTS AND DISCUSSION

A. *Effect of pH*

Forming of adsorbed species is largely based on the solution's pH. Metal ions' original concentration was 50 mg/l, and the contact time for adsorption was 90 min to reach complete equilibrium. The study of equilibrium at various pH ranges between 2 and 8 is used to comprehend how pH affects the adsorption of nickel ions (Figure 1).

It is evident from Figure 1 that the maximum (qt) for Ni2+ at pH=5 was 19.04 mg/g. It was found that the pH increases with increasing (qt) till pH =5, then decreasing by increasing the Ph (8). The adsorption of Ni decreased at low pH as a result of the competition between H+ ions and Ni2+ cations to maintain the free zone on the adsorbent surface caused by the higher concentration of H+ ions. Adsorbent surface deprotonation and electrostatic attraction forces between the adsorbent and adsorbate are brought on by higher pH values, which increases capacity. In contrast, pushing forces between adsorbents and adsorbate rise at lower pH due to forming more positively charged areas. (9) Alomá et al. (2012) described that at pH 5.0. Ni2+ ions are predominant, and a higher pH is not recommended due to precipitation [10].

B. *Effect of contact time:*

As shown in Figure 2, the adsorption of Ni^{2+} in the first 10 minutes is rapid. After twenty minutes, adsorption rates gradually fell and reached equilibrium at 60 to 90 minutes. The results show that the maximum (qt) of Ni^{2+} is 19.04 mg/g at contact time = 90 minutes. Since there are many free active sites available for the adsorption process, very high adsorption rates were seen initially, and the adsorption equilibrium was gradually achieved [10].

C. *Effect of Ni+2 concentration onto MgO*

The effect of Ni2+ concentration on MgO was performed at contact time (20 min), pH = 5, stirring speed (300 rpm), temperature 25°C, and adsorbent dose (0.5 g/250 mL) for the following concentrations: 50 and 100. and 150, 200 and 300 ppm. The adsorption capacity of Ni2+ qt (mg/g), shown in Fig. 3, increased with increasing Ni2+ concentration. Also, it turned out that the maximum (qt) of nickel was 54.325 mg / g at concentration = 300 ppm [11, 12].

D. *Effect of adsorbent dose*

Adsorption capability qe(mg/g) and adsorbent dosage of Ni2+ was studied at the time of adsorption for 90 min for an original concentration of 50 ppm Ni2+ ions at a temperature of 25°C. Different adsorbent dose (magnesium oxide) = (0.1, 0.3, 0.5, 0.7 and 1 g/250ml).

Figure 4 indicates that as the amount of adsorbent utilized rises, the adsorption capacity of Ni2+ increases (qe(mg/g)); this is expected because, for a constant original concentration of sorbate, the increasing in adsorbent material gives a higher surface area of adsorptive sites., i.e. An increase in the number of exchangeable sites or surface area ensured improved Ni2+ absorption [13].

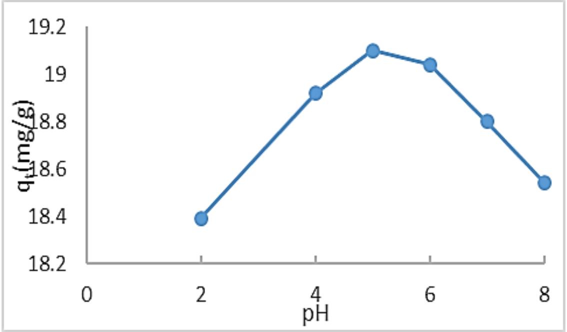

Figure 1. Effect of pH on the amount of metal adsorbed (Ni (II)) onto magnesium oxide adsorbent (original concentration = 50 mg/l, magnesium oxide wight = 0.5g/250 ml, speed of stirring = 300 rpm T=25°C and adsorption time = 90 min)

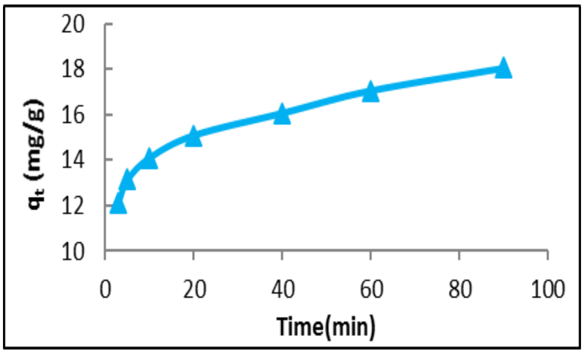

Figure 2. Contact time effect on the adsorption of Ni+2 onto MgO at (original concentration = 50 mg/l, magnesium oxide wight = 0.5g/250 ml, speed of stirring =300 rpm T= 25°C and adsorption time = 90 min, pH =5)

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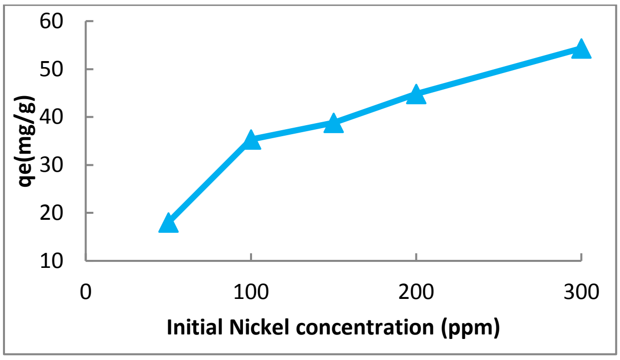

Figure 3. Ni+2 ion original concentration effect on the adsorption of Ni+2 onto MgO at (magnesium oxide dose = 0.5g/250 ml, speed of stirring =300rpm T= 25°C and time = 90 min, pH =5)

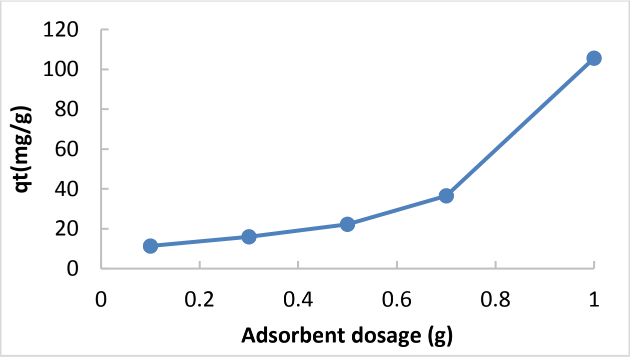

Figure 4. magnesium oxide dose on the adsorption of Ni+2 onto MgO at (stirring speed =300rpm T= 25°C and adsorption time = 90 min, pH =5, Ni+2 ion initial concentration=50 ppm)

Figure 4 indicates that as the amount of adsorbent utilized rises, the adsorption capacity of Ni2+ increases (qe(mg/g)); this is expected because, for a constant original concentration of sorbate, the increasing in adsorbent material gives a higher surface area of adsorptive sites., i.e. An increase in the number of exchangeable sites or surface area ensured improved Ni2+ absorption [13].

E. Effect of temperature

Figure 5 gives the effect of temperature on the adsorption qe(mg/g) of Ni2+ from wastewater using magnesium oxide. Different temperatures were used in the study at (20,25, 30, 35 and 40 °C) with a 50 ppm initial concentration of Ni2+ ions in the presence of (1 g/250 ml) for magnesium oxide. The figure showed that in 90 min, the adsorption capacity qe(mg/g) increased by raising the temperature because the Ni (II) is activated at higher temperatures, improving adsorption at the adsorbent's coordination sites, and the Ni (II) moves more quickly. Formation of novel activation sites on the adsorbent surface and acceleration of some previously slow stages.

IV. ADSORPTION OF KINETICS

A. Pseudo–First Order Model

The first known example of adsorption rate is most likely the pseudo-first-order equation. Using equation(3), (Lagergren, 1898) proposed a pseudo-first-order kinetic model.

$$\ln(q_e - q_t) = \ln(q_e) - K_1 t \qquad (3)$$

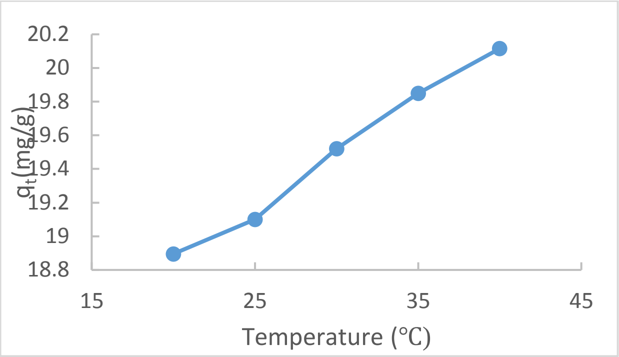

Figure 5. Temperature effect on the adsorption of Ni+2 onto MgO at (stirring speed =300rpm, magnesium oxide wight = 0.5 g /250 ml , contact time = 90 min, pH =5, Ni+2 ion initial concentration=50 ppm)

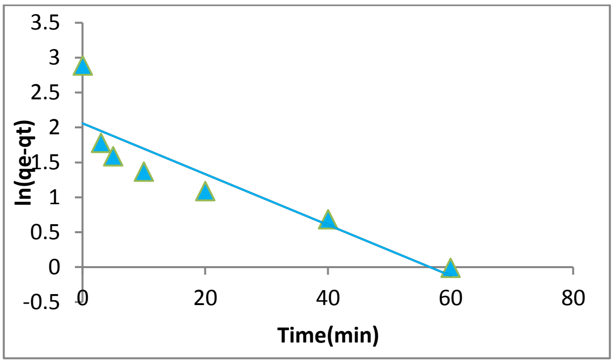

Figure 6. Pseudo-first order for Ni+2 for the adsorption onto MgO at (original concentration = 50 ppm, magnesium oxide dose = 0.5g/250 ml, speed of stirring =300rpm T= 25°C and adsorption time = 90 min, pH =5)

The form in equation (3) for condition of t=0, qt= 0 and t

$$q_t = \frac{(C_0 - C_t)\times v}{m} \qquad (4)$$

$$q_e = \frac{(C_0 - C_e)\times v}{m} \qquad (5)$$

The initial adsorbate concentration, which varies from system to system, determines the value of K1. It typically falls as the original adsorbate concentration in the bulk phase rises. The drawing of ln (qe-qt) against t is given in figure (6) that provides a linear connection, and the slope and intercept of the graph can be used to calculate k1 and qe.

B. Pseudo–Second Order Model

Equation (6) can be used to test the second-order kinetics:

$$\frac{dq_t}{dt} = K_2(q_e - q_t)^2 \qquad (6)$$

where: k2: the adsorption's pseudo-second-order rate constant (mg g-1 min-1).

A linear expression in the structure of equation (7), in which the adsorption kinetics may also be represented, is produced by separating the variables, integrating, and applying the conditions (qt=0 at t=0 and qt= qt at t=t) The linear zed–an integral form of the model.

The integral form shown in equation (7) indicates that the relationship between time and the ratio between the time / adsorbed amount of metal ion (t/ qt) should be a linear function of time.

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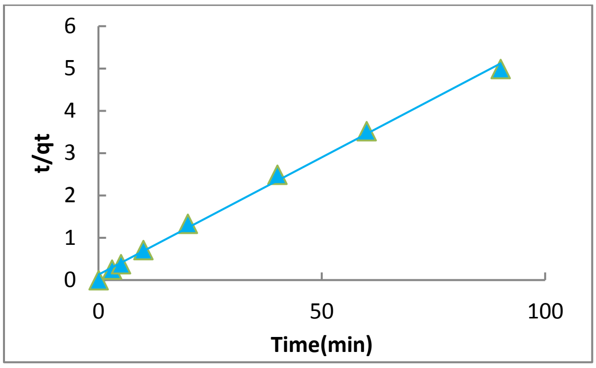

Figure 7. Pseudo-second order for the adsorption of initial concentration of Ni+2ion =50ppm, dose of MgO =0.5g/250ml, stirring speed 300rpm, contact time 90 min ,pH 5 at 25 ˚C.

$$\frac{t}{q_t} = \frac{1}{K_2 {q_e}^2} + \frac{1}{q_e} t \qquad (7)$$

C. Weber and Morris model

The internal diffusion of most liquid systems controls the adsorption rate, making the intra-particle diffusion model extremely interesting. A general illustration of kinetics can be found in equation (8), where the predicted value of the exponent is 0.5 and the intercept is related to mass transfer across the boundary layer.

$$q_t = K_m t^{0.5} + C \qquad (8)$$

where km (mg/g min1/2) is the intra-particle diffusion rate constant.

Figure 8, in agreement with this concept, the plot of qt versus t0.5 results in a straight line with a slope of km and an intercept of C when the intraparticle diffusion is included in the adsorption process. The boundary layer thickness can be roughly estimated from the values of C, the larger the C value, the thicker the boundary layer. If the plot's lines cut through the beginning.

In this study, Pseudo-second order and Weber and Morris kinetic models were the best rate-determining models with R2 > 0.97 for the adsorption of Ni (II) on MgO (Table 1). The maximum adsorption of Ni (II) (qe) for Pseudo–Second Order and Weber and Morris kinetic models were 18.0398 mg/g (K2 = 0.0238 mg/mg.min) and 11.4889 mg/g (Km = 0.60 mg/mg.min), respectively.

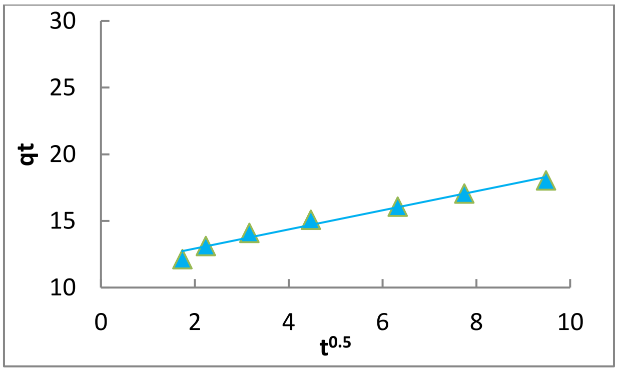

Figure 8. the intraparticle diffusion model of adsorption for Ni (II) (original concentration of Ni+2ion =50ppm dose of MgO =0.5g/250ml,stirring speed 300rpm , contact time 90 min ,pH 5 at 25 ˚C

Table 1. Kinetic models and other parameters on adsorption of Ni (II) original concentration 50ppm, the dose of MgO = 0.5 g/250ml, stirring speed 300rpm; contact time 90min, pH=5 at 25˚C

Kinetic models	Parameters	0.5g
Pseudo 1st order equation	q_e (EXP.) (mg/g)	18.05
	q_e (calc.)	5.598
	R^2	0.8014
	$K_1(min^{-1})$	0.0363
Pseudo 2nd order equation	q_e (calc.)	18.0398
	R^2	0.997
	K_2 (g/mg.min)	0.0238
Weber and Morris model	q_e (calc.)	11.4889
	R^2	0.975
	K_m (mg $g^{-1}min^{-1}$)	0.60

Similar results were reported by Gonen et al. [13] showed that pseudo-second-order kinetics and intraparticle diffusion models were the best rate-determining models with R2 > 0.99 for Ni (II) biosorption using orange peel. In addition, Arshadi et al. [14], in the adsorption study of Ni (II), Cd (II), Cu (II) and Co (II) ions on barley straw ash, confirmed that the rate-kinetic specific form of pseudo-second order.

D. Thermodynamic Parameter:

The standard Gibbs free energy (ΔG°), standard enthalpy change (ΔH°), and standard entropy change (ΔS°) were several of the major thermodynamic parameters that were calculated using the data on adsorption equilibrium at various temperatures (15-18). Equation (9) was used to compute the Ni+2 adsorption standard Gibbs free energy (G°).

$$\Delta G^0 = RTln\, K_e \qquad (9)$$

Using equation (10), the adsorption equilibrium constant, Ke, was determined for each temperature.

$$K_e = \frac{q_e}{c_e} \qquad (10)$$

Where:
qe (mg/g): the amount of Ni^{+2} ions adsorbed onto the solution at equilibrium.
Ce(mg/l): the equilibrium concentration ion in the solution.
R (j/mol.K): the gas constant 8.314.
T(K): the absolute temperature.

ΔH˚ and ΔS° were determined from the slope and intercept, respectively, of the van' t Hoff's plot of ln (Keq) versus 1/T; equation (11) as illustrated in figure (9).

$$\ln K_e = -\left(\frac{\Delta H^0}{RT}\right) + \left(\frac{\Delta S^0}{RT}\right) \qquad (11)$$

The values of ΔG°, ΔH˚ and ΔS° from the Table show that the positive value of ΔH indicates that the exchange of the divalent metal ions requires a significant amount of energy. Positive values of ΔH° demonstrate the endothermic nature of the adsorption and govern the physical adsorption potential. Similar results were reported by Arivoli et al. (19). they find that a very low value of ΔH° for the nickel ion and the adsorption is physically on the adsorbent. The positive values of ΔS suggest that some structural changes occurred on the adsorbent and confirmed that affinity exists between the adsorbent and adsorbate (20). The negative value of ΔG° means that adsorption the sorption of Ni (II) onto MgO is feasible and spontaneous.

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Table 2. Thermodynamics parameters

Ni^{+2} Concentration 50 ppm Remove by MgO	T(K)	293	298	303	308	313
	1000/ T(K^{-1})	3.41	3.35	3.29	3.24	3.19
	q_e	18.895	19.1	19.52	19.85	20.115
	C_e	10.44	9.8	9.19	8.55	7.97
	K_e	1.80986	1.94898	2.124	2.3216	2.5238
	$\ln K_e$	0.593	0.6673	0.753	0.8422	0.9258
	ΔG°(kj/mol)	-1445.29	-1654.12	-1897.85	-2157.68	-2410.35
	ΔH˚(Kj/mol)	1.52308				
	ΔS°(Kj/mol.K)	48.02443				

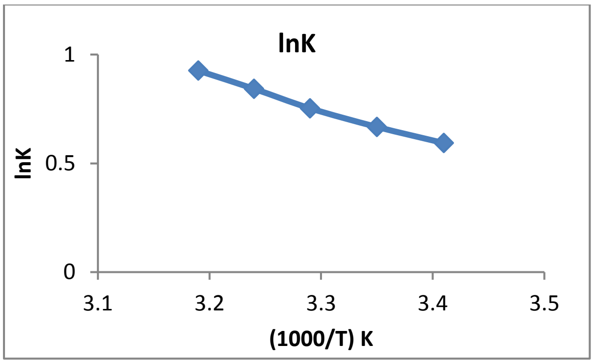

Figure 9. Van't Hoff's plot for adsorption Ni (II) original Ni+2 ion concentration=50 ppm at pH = 5, MgO adsorbent weight = 0.5 g /250 ml, speed of steering = 300 rpm, adsorption time 90 min at various temperatures.

V. ISOTHERMAL MODELS

A. Langmuir adsorption isotherm:

The Langmuir model presupposes that the Langmuir model presumes that there is no interaction between the two species and that ion uptake happens on a homogeneous surface through monolayer adsorption [21-25]. Equation (12) expresses the Langmuir.

$$\frac{c_e}{q_e} = \frac{1}{q_{max}\times b} \times \frac{c_e}{q_{max}} \qquad (12)$$

Where:
q_e: the equilibrium Ni (II) concentration on the adsorbent (mg/g).
Ce: the equilibrium Ni+ (II) in the solution.
q_{max}: the monolayer adsorption saturation capacity of the adsorbent.
b: the Langmuir constant.

The values of slope ($1/q_{max}$) and intercept ($1/q_{max}.b$) at initial Ni(II) ion concentration are given by a plot of Ce/qe versus Ce, as illustrated in Figure (10). The linear plot in Figure 10 shows the Langmuir isotherm.

B. Freundlich adsorption isotherm:

Freundlich isotherm of adsorption. The experimental data are often well-fit over a wide range of concentrations by one of the most popular mathematical models. This isotherm provides an expression that considers the exponential distribution of active sites' energy and surface heterogeneity(26-28). The Freundlich model's nonlinear form is written as:

$$q_e = K_f (c_e)^{1/n} \qquad (13)$$

The linear form of the Freundlich model is expressed as:

$$\log q_e = \log(K_f) + \left(\frac{1}{n}\right) \log c_e \qquad (12)$$

where n is a constant associated with the sorption intensity, which varies with the heterogeneity of the adsorbents, and K_f is the Freundlich constant reflecting adsorption capacity. The Figure illustrates a plot of log qe vs log Ce that results in a straight line with a slope of 1/n and an intercept of log K_f. Figure (11).

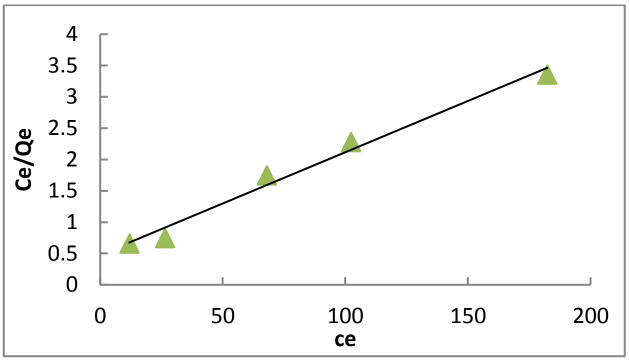

Figure 10. Langmuir adsorption isotherm for Ni+2 ion adsorption dose of bulk MgO (0.5 g/250 ml) at pH =5, 300rpm and contact time 90 min

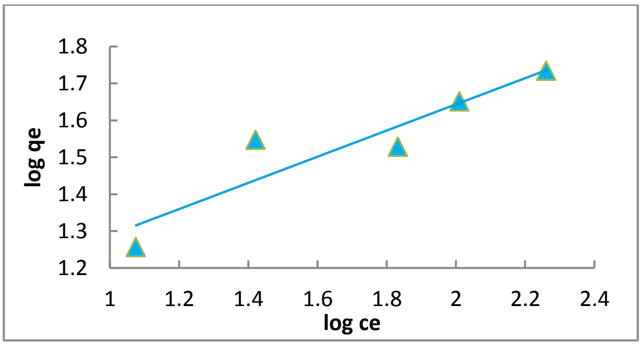

Figure 11. Freundlich adsorption isotherm, for Ni+2 ion adsorption, MgO dose =0.5 g/250 ml, at pH =5 , 300rpm and contact time 90 min.

VI. CONCLUSION

Magnesium oxide served as a cheap adsorbent to study the adsorption of nickel ions from an aqueous solution under various experimental conditions in a batch procedure. The results show that magnesium oxide is efficient at eliminating nickel ions from aqueous solutions. The initial concentration of nickel ions in the solution raised, as did the contact time, and so did the amount of nickel ions that were adsorbed. The outcomes also demonstrated that an increase in adsorbent dosage increases the amount of nickel ions adsorbed. Additionally, the adsorbent created in this study has the best pH of 5. Pseudo-second-order and Weber and Morris kinetic models were the best rate-determining models with R2 > 0.97 for the adsorption of Ni (II) on MgO. The values of ΔH°, ΔS° and ΔG° results shows that the MgO employed has a

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considerable potential as an adsorbent for the removal of nickel ion.

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Conflicts of Interest: There are no ethical issues applied to this research and no conflict of interest.

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