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ORIGINAL RESEARCH PAPER

Kinetics, and Thermodynamic Studies of Nickel Adsorption from Aqueous Solutions Using Melamine-modified Nanographene Oxide

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Abstract: Today, one of the most important issues in water resources is pollution caused by heavy metals. Nickel is widely used in various industries. Nickel is one of the most important pollutants in the environment and its removal is very important. There are several methods for separating heavy metals from aqueous media. Among all methods, adsorption is a low-cost and simple method for removing heavy metals at low and medium concentrations. The aim of this study is to investigate the adsorption efficiency of nickel ions from aqueous solutions using melamine modified nanographene oxide. Melamine-modified nanographene oxide was investigated by Fourier-transform infrared spectroscopy (FTIR) Tensor 27 Mode. The pseudo-first-order and pseudo-second-order kinetic models were investigated. Finally, thermodynamic parameters such as enthalpy and entropy change as well as Gibbs free energy were calculated. In the kinetic studies, according to the linear regression coefficient of 0.3084 for the pseudo-first-order, and 0.9970 for the pseudo-second-order model, it was found that the pseudo-second-order model better describes the adsorption process of nickel by the melamine-modified nanoadsorbent. The maximum adsorption capacity and removal percentage occur at a concentration of 200 mg/l, during 150 min, which is equal to 1915.75 mg/g and 99.82%, respectively. The adsorption reaction of nickel ions on the surface of melamine-modified nanographene oxide was spontaneous and endothermic with an increasing entropy. Nanographene oxide modified by melamine has a special surface and high potential and is a suitable adsorbent for removing nickel from aqueous solutions.
Keywords: Adsorption, Isotherms, Kinetics, Nickel, Melamine, Graphene oxide

1. Introduction

Today, due to industrial and agricultural activities in the country, we are more exposed to heavy metals (jedokun A. T and Bello O. S. 2016). The metals in industrial effluents include lead, nickel, copper, zinc and cadmium, which can have negative effects on humans and the environment, even in small amounts (Madaeni. S. S et al. 2013). When industrial pollutants enter the environment due to the solubility of metal ions in the aquatic environment, living organisms begin to absorb these substances and endanger their health (Barakat M 2011). When these metals accumulate in the body, they have a negative effect on different parts of the body such as liver, nervous system, kidneys, skin and gastrointestinal tract and also cause cancer in different parts of the body (Brown P A et al. 2000). Due to effects of these metals, it is necessary to reduce the concentration of these metals to the level of set standards and if they have economic value, they should be reused and recovered (Strivastava S K et al. 1997). Nickel is a heavy metal commonly found in effluents (Basso M et al. 2002). Nickel concentration in industrial effluent is between 3.40 and 900 mg/L and the maximum allowable concentration of nickel available in drinking water is 50 mg /L (Maibach H. I. and Menné T, 1989). Wastewater from the battery industry and silver refineries release large amounts of nickel into the environment (Hasar H. 2003). High concentrations of nickel can cause lung and bone cancer. Heavy metals cause major environmental problems. Heavy metals pose a threat to human health due to their high toxicity, even in low concentrations. Therefore, It is necessary for these elements to be removed from the sewage before being discharged into the environment (Al-Rub F. A et al. 2004). Various methods are used to remove heavy metals such as: ion exchange, chemical precipitation, filtration, electrochemical purification, adsorption and membrane technologies (Fan H.L et al. 2017; Omrani and Fataei, 2018). In recent research, nanoparticles have been used to remove organic compounds and good results have been observed (Nouri dodaran P et al 2019) Here we used modified nanographene oxide to remove heavy metals, which is a surface modification method. Despite its special properties such as chemical inertness, high current density, transparency and good hydrophobicity, graphene oxide has received a lot of attention (Peng W et al. 2017). Research by Mohammadnia et al. (2017) states that graphene oxide nanoparticles due to their high potential, the presence of a large amount of oxygen and the presence of carboxyl groups in graphene oxide plates provide a large surface area for the removal of heavy metals from aqueous solutions. Enriqe perez Ramirez et al. (2016) used graphene to remove organic pollutants and heavy metals from water, and the results of using graphene and graphene-based materials in adsorption and photocatalysis showed that these materials have a good future for water treatment. Nanostructured particles are used for immediate treatment of sediments for water treatment (Gooran ourimi and nezhadnaderi, 2020). Al-Ain et al. (2020) investigated the use of magnetic graphene for water treatment. The maximum adsorption capacities for lead, copper, chromium, and nickel were 200, 24.330, 62.893, 63.694 and 51.020 mg/g, respectively. The adsorption processes were spontaneous and endothermic. The reaction corresponded with the Langmuir adsorption isotherm and the pseudo-second-order kinetic model.

Graphene oxide (GO) is an efficient adsorbent for the removal of heavy metals in the aqueous environment due to the high functional of oxygen groups, high specific region and strong hydrophobicity. However, it has limitations, including aggregation and difficult separation and restricting the environmental application. GO alone is not a good adsorbent, for this reason, melamine is used to modify GO (Zhang et al. 2020). Melamine has a negative charge due to the presence of amine groups and therefore adsorbs metal cations.

The aim of this study was to effectively adsorb nickel heavy metal using modified nanographene oxide. To achieve this goal, a number of laboratory goals have been defined:

1-Efficiency and adsorption power of modified oxide nano geraphene for removal of nickel ions.

2- Investigation of thermodynamic and kinetic parameters affecting the removal of nickel heavy metal ions.

2. Materials and Methods

To prepare solutions containing nickel, salt nitrate of $Ni(NO_3)_2$ was used and to adjust the pH, 0.1 HCl and NaOH solutions were used, which were purchased from Merck, Germany. Re-ionized distilled water was used to dilute the solutions. Graphene and melamine (2,4 and 6-triazine 1,3 and 5-triamine) were also purchased from the German Company Merck. Instruments used for testing were: pH-meter model AZ 8653 made in Taiwan to check the pH value, digital scale model (Bands Bs-3003) with accuracy of 0.001 g for weighing, shaker incubator model Ikaks model (4000 IC) made in Germany, which was used to mix adsorbents and solvent pollutants, and a US-made (Hermle Z300) centrifuge at 4000 rpm was used to separate suspended particles from the solution, and a British-made Uniam919 flame atomic adsorption spectroscopy (AAS) was used.

2.1. Preparation of Modified Nanographene Oxide Adsorbent:

Graphene nanostructure synthesis was performed by Hummer method. The 360 ml of sulfuric acid and 40 ml of phosphoric acid as well as 4 g of graphite was first mixed and stirred slowly, gradually adding 18 g of potassium permanganate to the reaction. When the reaction temperature reached about 35 to 40 °C, the container was transferred to the oil bath at 50 °C and stirred for 12 h, then allowed to stay at room temperature. The container under the hood was then transferred to a Bécher containing 3 ml of hydrogen peroxide and 400 ml of ice water to remove unreacted potassium permanganate. By using a centrifuge, the solids were removed and washed with 200 ml of water, and again remove the solids by centrifuge, washed with 200 ml of ethanol and 200 ml of 30% chloride acid twice to remove all metal ions and acids. The material was dried by freezing drying and was placed in vacuum oven at 50 °C overnight to remove residual moisture and the product was graphene oxide (Qare Biglu, M et al. 2016). To increase the efficiency of graphene oxide, it was mechanically converted to nanographene oxide and melamine (4, 2 and 6-triazine, 3, 1 and 5 triamine) as co-precipitators was used as modifier.

2.2. Preparation of standard nickel solution:

first, a stock solution of 1000 mg /L nickel was prepared by dissolving 3.196 g of $Ni(NO_3)_2$ salt in a 1000-ml flask. 1000 mg of nickel with a molecular mass of 58.693 g/mol was obtained from nickel salt with a molecular mass of 189.693 g/mol and 3.1196 g. The calculated salts of nickel nitrate were weighed using a scale with an accuracy of 0.001g and poured into a 1000 ml volumetric flask and made up to volume with deionized distilled water. To adjust the pH, 0.1 HCl and NaOH solutions and pH meter model AZ 86552 were used and all adsorption tests were performed in a batch system with 2 replications.

2.3.Thermodynamic studies of nickel adsorption:

To study the thermodynamic of adsorption process, the three main parameters of Gibbs free, ΔG_0 (kJ mol^{-1}), enthalpy change, ΔH_0 (kJ mol^{-1}), and ΔS_0 entropy change (J mol^{-1} K^{-1}) were evaluated.

$$\Delta G° = -RT\ln K_0 \quad (1)$$

$$\Delta G_0 = \Delta H_0 - T\Delta S_0 \quad (2)$$

(2)

$$\ln K_0 = \frac{(\Delta s°)}{R} - \frac{(\Delta H°)}{RT} \quad (3)$$

where T is absolute temperature (K), R is ideal gas constant (8.314 J mol^{-1} K^{-1}), K_0 is the thermodynamic equilibrium constant. The values of enthalpy change ($\Delta H°$) and entropy change ($\Delta S°$) were calculated from the slope and intercept of the plot $\ln K_0$ versus 1/T (Rezaei et al. 2011).

2.4. Nickel adsorption kinetics:

Kinetics studies the speed of the removal process heavy metals and the effect of reaction time on the removal efficiency of the contaminant. The pseudo-first-order kinetic is based on the adsorbent capacity and the pseudo-second-order kinetic controls the surface adsorption based on the solid phase adsorption (Robati D et al. 2016).

The linear form of the pseudo-first-order kinetic model is as follow:

$$\ln(q_e - q_t) = \ln q_e - kt \quad (4)$$

which k shows the adsorption rate constant of pseudo-first-order (min^{-1})

The linear form of the pseudo-second-order kinetic model is as follow:

$$\frac{t}{q(t)} = \frac{t}{q_e} + \frac{1}{k_2 q_e^2} \quad (5)$$

where, q_e is the amount of metal adsorbed during equilibrium (mg g^{-1}), $q_{(t)}$ indicates the amount of metal adsorbed at time (mg g^{-1}), and k_2 is the adsorption rate constant of pseudo-second-order (g mg^{-1} min^{-1}) (Yakout S. M, and Elsherif E. 2010).

Results

3.1. Adsorbent Properties

3.1.1. FTIR

The FTIR analysis was used to determine the superficial groups of nanographene oxide. Figure 1 indicates the FTIR spectra of nanographene oxide prior to the nickel adsorption (purple curve), and after nickel adsorption (pink curve).

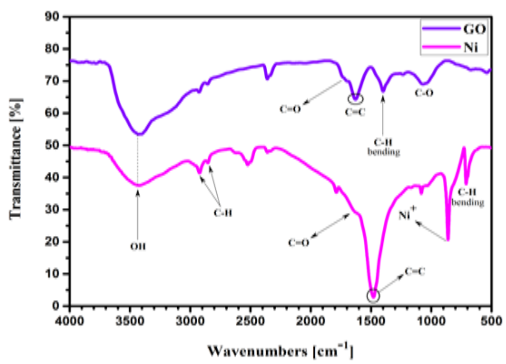


Figure 1: The FTIR spectra before (purple curve), and after (pink curve) the adsorption of Ni^{2+} ions on melamine-modified nanographene oxide

As shown in Figure 1, the wide peak (the left side) in the region of (3400-3500 cm^{-1}) is related to the –OH (hydroxyl) functional groups in the FTIR spectrum of adsorbing nickel ions by melamine-modified nanographene oxide. The peak (the right side) in the region of (800-900 cm^{-1}) is related to the Ni functional groups adsorbing by nanographene oxide. As shown in the spectra image, the hydroxyl or amino, carbonyl as well as carbon-carbon double bonds groups show good adsorbent conditions for the Ni^{2+}adsorption.

3.1.2. The X-ray Diffraction (XRD) Analysis:

Japan's XRD, Ultima IV Model was used to investigate the crystalline structure of graphene oxide nanoadsorbent. The spectra in the range of 5 to 15 and 25 to 35 indicate the crystalline and non-amorphous adsorbent structure, respectively. Figure 2 shows the XRD pattern of the adsorbent.

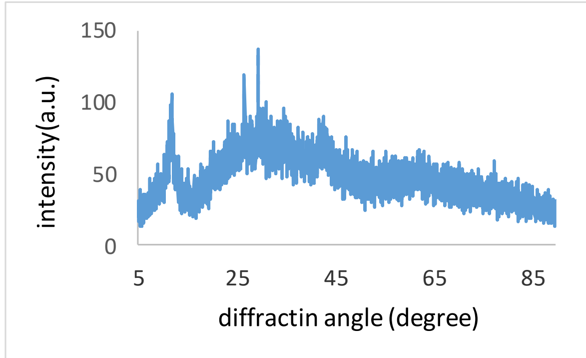
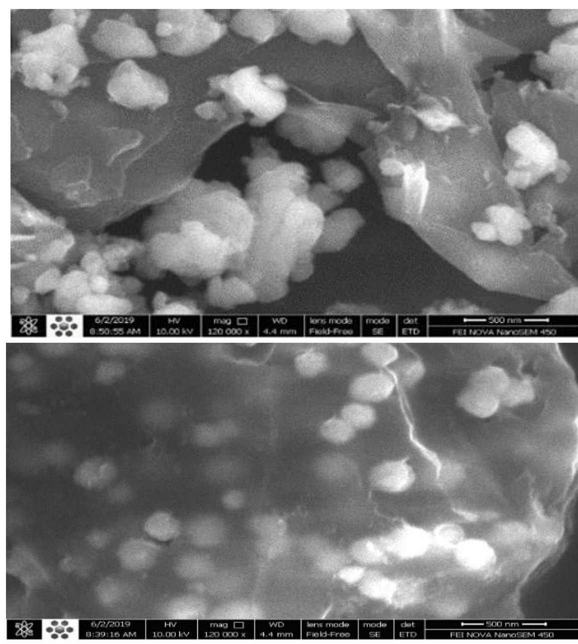


Figure 2: The X-ray diffraction analysis (XRD) of modified nanographene oxide

3.1.3. FE-SEM

Figure 3 shows (FE-SEM) images before and after the adsorption of nanographene oxide. The synthesized adsorbent surface has high porosity and suitable nanoparticle pore size. The porous structure indicates the high adsorption property this adsorbent. As shown in the figure, particles with dimensions of a few micrometers were seen with small holes that indicate the accuracy of the synthesis of graphene oxide plates synthesized.



a

B

Figure 3: FE-SEM (a) modified nanographene oxide prior to adsorption, and (B) modified nanographene oxide after nickel

3.2. Thermodynamic model for nickel adsorption

Thermodynamic parameters of the nickel adsorption process were performed and according to Figure 4 and Table 1 it was determined that the enthalpy of the reaction (ΔH) is positive, so the process is endothermic and positive value of ΔS indicates an increase in disorder and ΔG is positive, meaning a non-spontaneous reaction at 10 °C. A negative value of ΔG at high temperature indicates that the reaction is spontaneous with increasing temperature, so this experiment is endothermic and spontaneous at higher temperature.

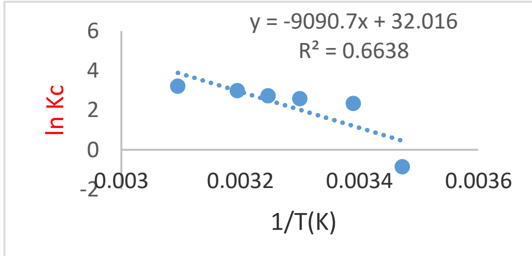


Figure 4: Van 't Hoff plot for the Ni ions' adsorption onto the modified nanographene oxide

Table 1. Thermodynamic parameters of adsorption of Ni^{2+} by modified nanographene oxide

Thermodynamic parameters	ΔH (kJ mol^{-1})	ΔG (kJ mol^{-1})	T(K)	Θ (°C)	ΔS (J mol^{-1} k^{-1})
	75.580	0.251 -1.080 -2.943 –5.072 -6.403 -7.734 -10.396	273 288 295 303 308 313 323	10 15 22 30 35 40 50	266.18

Thermodynamic analysis showed that the values of ΔG are negative, ΔH is positive and ΔS is positive, which indicates that the reaction is spontaneous and endothermic. ΔG is initially positive at 15 °C, however, becomes negative at subsequent temperatures, so it reacts non-spontaneously at 15 °C and spontaneously at high temperatures.

3.3. Kinetics model of nickel adsorption

To find the factors affecting the reaction speed, a kinetic evaluation was performed and Figures 5, and 6, and Table 2 show the results of the experimental data with pseudo-first-order kinetic model and pseudo-second-order kinetic models. The correlation coefficient for pseudo-first-order kinetics model is 0.3084 and pseudo-second-order kinetics model is 0.9997. Due to the higher value of pseudo-second-order kinetics model, it was observed that the pseudo-second-order model better shows the adsorption process of nickel ions by the synthesized adsorbent.

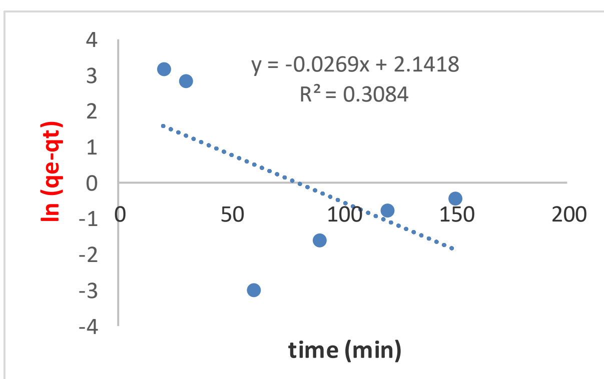


Figure 5: pseudo-first-order kinetic model of nickel adsorption by modified nanographene oxide

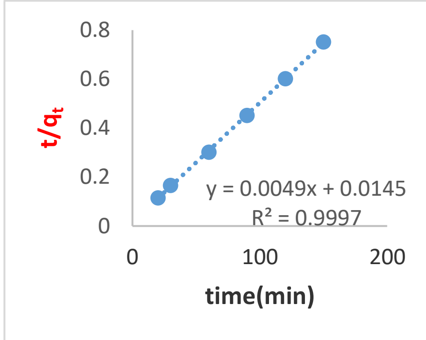


Figure 6: pseudo-second-order kinetic model of nickel adsorption by modified nanographene oxide

Table 2. Kinetics parameters obtained nickel ions adsorption onto the modified nanographene oxide

kinetic models	k_2	k_1	R^2
pseudo-first-order	-	0.0269	0.3084
pseudo-second-order	0.001656	-	0.9997

4. Discussion

In the present study, it was found that the process of nickel removal by modified nanographene oxide is possible stoichiometrically. The pseudo-first-order kinetic model is penetration from within a layer based on the capacity of the solid. Adsorption occurs through the process of diffusion and physical adsorption. The pseudo-second-order kinetic model is the deceleration phase which controls the adsorption process and is based on the solid phase adsorption process. Chemical adsorption can controle the adsorption process, too (rastgar et al 2020). The correlation coefficient of the pseudo-second-order kinetic model shows that the adsorption of nickel in the modified nanographene oxide is controlled by chemical adsorption, which involves strong surface reactions between metal ions and oxygen-containing groups on the graphene surface. The results show that the adsorption of nickel by modified graphene oxide is in accordance with the pseudo-second-order kinetic model with a correlation coefficient of 0.99. Also, researchers have conducted similar studies to investigate the effect of kinetics on nickel removal. Researchers have done similar studies to investigate the kinetic effect on heavy metal removal (Rajaei et al 2020). Ebrahimzadeh Rajaei et al. (2012) Investigated the adsorption kinetics, equilibrium and mechanism of Ni (II) and Cd (II) ions on MSTL. The results showed that the data are accordance to the pseudo-second-order kinetics, which is consistent with the results of this study. Khalili Arjaghi et al. (2020) removed metallic contaminants of mercury and arsenic from water using synthesized iron oxide nanoparticles. The results showed that the removal process in both metals is spontaneous and exothermic. Both metals corresponded to the pseudo-second--order model. Rajai et al. (2013) have used *Typha latifolia* L. fine root powder to adosorb Cu and Zn ions from aqueous solutions. Experimental data matched pseudo-second-order kinetic model that were consistent with the results of this study, too.

Thermodynamic hypotheses with physicochemical parameters show adsorption rate, surface properties as well as adsorption mechanism by adsorbent. Thermodynamic studies showed that the removal of nickel on graphene oxide is endothermic and the adsorption rate increases when the ambient temperature increases. As the ambient temperature increased from 10 to 50 °C, the nickel removal efficiency increased and indicated that the reaction was endothermic. ΔG is initially positive at 10 °C, ie the reaction is non-spontaneous. From 10 to 50 °C, its value becomes negative, indicating that the reaction is spontaneous and is stoichiometrically possible. ΔS° is positive which indicates that the Irregularity will also increased. This also suggests some structural changes in the adsorbate and the surface of adsorbent. Researchers have done similar studies to investigate the thermodynamic effect on heavy metal removal (Arjaghi et al. 2021).

Esdaki et al. (2019) removed nickel (II) ions from aqueous solutions using iron oxide (III) nanoparticles. Thermodynamic studies showed that the reaction is endothermic and the spontaneity of the adsorption process is controlled by the entropy factor. Their studies have shown that the presence of graphene oxide or nickel often causes the reaction to become endothermic and spontaneous and that was consistent with the results of this study. Amiri et al. (2019) removed chlorpyrifos from aqueous solution using a chitosan graphene oxide composite. The results showed that the process with pseudo-second kinetics and the equation data correspond to the Langmuir isotherm model. Evaluation of thermodynamic parameters showed that the process of chlorpyrifos removal was endothermic and spontaneous that was consistent with the results of this study.

5. Conclusions

ΔH has a positive value that indicates that the reaction is endothermic, and a positive value of ΔS indicates an increase in irregularity, and ΔG is positive at 10 °C, not spontaneous, and negative at 15 to 50 °C indicates that the nickel adsorption process on the modified graphene oxide nanoparticles is spontaneous. Kinetic analysis showed that the pseudo-second-order kinetic model better shows the adsorption process of nickel by the adsorbent. As the ambient temperature increases, the adsorption rate also increases. In general, the results of this study showed that nanogeraphene oxide has a very high ability to remove the heavy metal nickel from aqueous solutions. As a general conclusion, it can be said that melamine-modified nanogeraphene oxide with its large surface area, hydrophobicity, high negative charge density, ease of fabrication and high adsorption can be used as an effective adsorbent for metal removal.

6. Conflict of interest

The authors declare that they have no conflict of interest.

7. Additional Information And Declarations

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This paper is the result of a dissertation entitled “removal of heavy metals (lead and nickel) from aqueous solutions using melamine modified graphenenano oxide’’ and approved by Gorgan University of Agricultural Sciences and Natural Resources.

Grant Disclosures

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Competing Interests

The author declare there is no competing interests, regarding the publication of this manuscript.

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