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Isotherm and Thermodynamic Investigation on Removal of Pb(II) from Aqueous Solutions by Graphene Oxide and Magnetic Graphene Oxide: A Comparative Study

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Abstract

One of the most effective approaches for removing heavy metal ions from an aqueous solution is adsorption. However, the synthesis of adsorbents with intended selectivity and performance is a major obstacle in the fight against water pollution. The objective of this investigation is to address the constraints of Graphene Oxide (GO) as an adsorbent by synthesizing Magnetic Graphene Oxide (MGO). Both GO and MGO were synthesized, and their adsorption capacity for heavy metal ions such as lead (Pb (II)) was investigated. The maximum adsorption capacities (Q_{max}) of GO and MGO under optimal conditions (pH = 6, temperature = 50°C, and contact time = 20 minutes) were determined to be 46.19 mg/g and 49.8 mg/g, respectively. This indicates that magnetizing graphene oxide with iron oxide nanoparticles not only addresses its separation challenges but also enhances its adsorption capacity. Thermodynamic analyses indicated that the adsorption process was spontaneous and endothermic.

Keywords: Adsorption; Nanomaterials; Lead; Graphene Oxide (GO); Magnetic Graphene Oxide (MGO); Isotherm; Thermodynamic variable

Introduction

Heavy metals pose a significant hazard to the environment and human health due to their toxicity and propensity for bioaccumulation. The abundance of heavy metals, including lead, in water has emerged as a significant issue in recent years. Nanomaterials, due to their extensive surface area, ease of regeneration, and capacity for chemical modification, offer a distinctive platform for the filtration of contaminated water (Baby et al., 2019). A range of effective adsorbents has been shown for the remediation of heavy metal-contaminated water. The use of graphene oxide (GO), a carbon-based nanomaterial, has garnered widespread attention (Yan et al., 2014). GO originates from the oxidation of graphene, leading to an increase in interlayer spacing from 0.335 nm to 0.625 nm (Sitko et al., 2013).

It consists of a hexagonal network of covalently bonded carbon atoms with oxygen-containing functional groups such as hydroxyl, epoxy, lactone, quinone, phenol, anhydride, carbonyl, ether, and carboxyl groups attached to various sites. The basal planes are decorated mostly with hydroxyl and epoxide groups, in addition to carboxyl and carbonyl groups located at the edges (Guerrero-Fajardo et al., 2020; Zhao et al., 2011). GO possesses some special characteristics such as high surface area (2620 m²/g), large Young's modulus, high optical transmittance, imperishable hydrophilicity, and thermal conductivity (Gopalakrishnan et al., 2015; Koduru et al., 2019). Methods that are most commonly employed for the synthesis of GO include the Brodie method, Hofmann



method, Staudenmaier method, and Hummers method and its improved and modified forms. In these methods, graphite powder is first made to react chemically with acids (HNO_3 , H_2SO_4 , and HCl , etc.) followed by the intercalation of alkali metals compounds such as KClO_3 , NaNO_3 , and KMnO_4 , into the graphitic layers, and this intercalation further aids in the breaking of graphitic layers into small fragments (Singh et al., 2016). Lone pairs of electrons found in oxygen-containing functional groups on the surface of GO allow it to efficiently attach to a metal ion to generate a metal complex, therefore rendering GO potential sorbents of metal ions (Sitko et al., 2013).

After reaching adsorption saturation, fast separation of sorbents from water is necessary for the recovery of both adsorbent and adsorbate. Nevertheless, high dispersibility, hydrophilicity, and agglomeration of GO during application and storage limit its direct usage as an adsorbent since it becomes difficult to separate GO even after adsorption saturation has taken place (Liu et al., 2015, Sun et al., 2015). GO magnetization utilizing iron oxides represents one potential way to mitigate the issues. Magnetic nanoparticles have recently demonstrated significant efficacy in the expedited and simplified regeneration of adsorbents from liquid media when subjected to an external magnetic field (Yan et al., 2014).

Nano-sized iron oxides exhibit superparamagnetism, low toxicity, and chemical inertness (Jawed et al., 2020). The magnetized GO may be readily isolated with an external magnetic field (Lingamdinne et al., 2019). However, it remains uncertain whether magnetizing GO enhances, impairs, or has no impact on adsorption. This study examines the manufacture and use of graphene oxide (GO) and modified graphene oxide (MGO) for the adsorption of lead ions from aqueous solutions. Lead is among the most harmful heavy metal ions. It usually gathers in bones and other important organs and causes dyslexia, renal disease, brain damage, autism, paralysis, and dyspnea (Martin and Griswold 2009). Moreover, notably in the area of Punjab, its content in waterways has been found to exceed the allowed level of 0.01 ppm. Thus, trying to address this issue right away becomes really crucial.

Material and Methods

Extra pure fine graphite powder (10-30 nm in size) was procured from Loba Chemicals, sulfuric acid (H_2SO_4), potassium permanganate (KMnO_4), phosphoric acid (H_3PO_4), ferrous ammonium sulfate hexahydrate $[(\text{NH}_4)_2\text{Fe}(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}]$, ammonium ferric sulfate dodecahydrate $[(\text{NH}_4)_2\text{Fe}(\text{SO}_4)_2 \cdot 10\text{H}_2\text{O}]$, aqueous ammonia (NH_4OH) (weight 25%), hydrogen peroxide (H_2O_2), and HCl were procured from Molychem. All chemicals were of analytical grade and used without any further purification.

GO was prepared by the modified Hummer's method. MGO was prepared by the co-precipitation of iron oxide nanoparticles on the surface of GO. The complete procedure for production followed by characterization using N_2 adsorption-desorption analysis, TEM, and FTIR has been discussed in detail in previously reported work. Structural characterization exhibited higher surface area for Magnetic Graphene Oxide ($108.3750 \text{ m}^2/\text{g}$) compared to GO ($35.8220 \text{ m}^2/\text{g}$). (Grover and Jeet, 2023).

Adsorption studies

Batch adsorption experiments were conducted using both adsorbents to evaluate the influence of factors such as pH, temperature, and contact time on the adsorption of lead ions in an aqueous solution. A lead solution at a concentration of 10 ppm was utilized for the batch adsorption tests. The wavelength of maximum absorption, λ_{max} , was determined to be 516 nm. A calibration curve was constructed between concentration and absorbance using this wavelength. The concentration of $\text{Pb}(\text{II})$ ions in each solution was subsequently ascertained from the calibration curve utilizing the absorbance of each solution. The percentage of $\text{Pb}(\text{II})$ eliminated was determined from the initial and final concentrations using the following equation (Hadavifar et al., 2014).

$$\text{Percentage removal} = \frac{C_i - C_f}{C_i} \times 100 \quad (1)$$

Where C_i and C_f are the initial and final concentrations in mg/l .

Adsorption Isotherm Study

The adsorption equilibrium data were analyzed by fitting it to different isotherm models. The adsorption isotherm step is crucial for optimizing the efficiency of adsorbents and studying the solute-adsorbent interactions (Tan IAW and Hameed BH, 2010). The current study employed isotherm models, specifically the Freundlich and Langmuir isotherms.

Thermodynamic Parameters

This study calculates the changes in Gibbs energy (ΔG°), enthalpy (ΔH°), and entropy (ΔS°) to anticipate the adsorption process. The thermodynamic distribution coefficient can be determined using the following equation:

$$K = \frac{Q_e}{c_e} \quad (2)$$

The values of entropy (ΔS°) and enthalpy (ΔH°) are calculated from the intercepts and slopes and of a plot of $\ln K$ versus $1/T$ using the relation:

$$\ln K = \frac{\Delta S^\circ}{R} - \frac{\Delta H^\circ}{RT} \quad (3)$$

Gibbs free energy is calculated using:

$$\Delta G^\circ = -RT \ln K \quad (4)$$

where $R = 8.3145 \text{ J}\cdot\text{mol}^{-1}\cdot\text{K}^{-1}$ is the ideal gas constant, K is the equilibrium constant at different temperatures, and T is the absolute temperature (Mei et al., 2010; Zhao et al., 2010).

Results and discussion

Effect of pH

The Pb(II) adsorption capacity of the synthesized samples was examined over a pH range of 3 to 9, while maintaining a constant temperature of 25°C , with the findings illustrated in Figure 1. It was observed that for all adsorbents, adsorption increased with rising pH up to a specific threshold, after which it declined with further increases in solution pH. All adsorbents exhibited peak adsorption capacity at a pH of 6.0. This adsorption tendency related to varying pH levels can be explained as follows. In the presence of metal ions in an aqueous solution, hydration occurs, resulting in the formation of diverse complexes at different pH levels (Liu et al., 2019). The distinct forms of lead in an aqueous solution are Pb^{2+} , $\text{Pb}(\text{OH})^+$, $\text{Pb}(\text{OH})_2$, and $\text{Pb}(\text{OH})_3^-$. The electrostatic interactions between these states and the surface charge of the adsorbent at a specific pH are crucial to the adsorption process (Sitko et al., 2013).

During the adsorption process of graphene oxide, the oxygen-containing functional groups undergo deprotonation, resulting in the release of some H^+ ions into the solution. The adsorption of lead ions onto the surface of graphene oxide (GO) occurs due to the exchange of Pb^{2+} ions with $-\text{H}$ groups on the GO surface. At low pH levels, the primary lead species present are Pb^{2+} and $\text{Pb}(\text{OH})^+$, but at elevated pH levels, the dominating species is $\text{Pb}(\text{OH})_3^-$. Previous studies have determined the pH_{PZC} of GO to be between 3.8 and 3.9 (Sitko et al., 2013). Consequently, at solution pH levels exceeding this range, the surface of GO acquires a negative charge, rendering the predominant species $\text{Pb}(\text{OH})_3^-$ at elevated pH values challenging to adsorb onto the negatively charged surface of GO. This explains the gradual decline in adsorption observed above a solution pH of 6. The reduced adsorption capacity seen at pH 3-4, as illustrated in Figure 1, can be ascribed to the competition between Pb^{2+} and H^+ ions for identical adsorption sites, or it may result from the limited dissociation of the surface functional groups of GO. A pH exceeding 5 is advantageous for the adsorption of lead ions, as the surface of graphene oxide (GO) is negatively charged within this range, while the predominant lead species remain positively charged, namely Pb^{2+} and $\text{Pb}(\text{OH})^+$. Consequently, the interactions between lead ions and GO intensify (Zhao et al., 2011, Sitko et al., 2013, Guerrero Fajardo et al., 2020).

The adsorption capabilities of MGO were significantly enhanced at all pH levels compared to GO, but the trend remained rather consistent. The pH_{PZC} of MGO was approximated at 4.5 (Hur et al., 2015). Consequently, the surface charge of MGO is positive at pH levels below this threshold, which may account for the reduced Pb removal capacity. At acidic pH, the surface functional groups become protonated, resulting in a positively charged MGO surface that induces electrostatic repulsions with the prevalent Pb^{2+} and $\text{Pb}(\text{OH})^+$ ions in this range. At a pH exceeding 4.5, the functional groups of MGO undergo deprotonation, resulting in attractive interactions between the specified Pb species and the MGO surface, hence enhancing the Pb removal capability. Beyond a pH of 6.0, the adsorption capacity decreased once more, as the prevailing lead species in this range is $\text{Pb}(\text{OH})_3^-$ resulting in repulsive interactions between the deprotonated negatively charged MGO surface and lead ions. Comparable findings were reported by Qurat-UI-Ain et al., 2020 and Jin Hur et al., 2015.

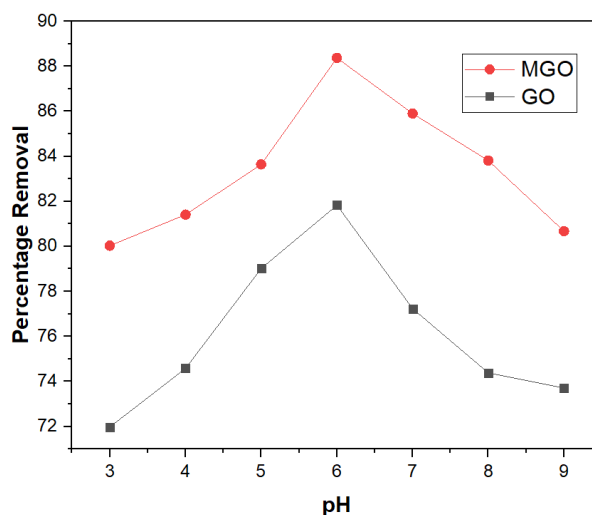


Fig. 1. Effect of pH on lead adsorption by GO and MGO

Effect of temperature

The impact of temperature was examined over a range 30°C to 50°C at the optimal pH of 6.0 using a 10 ppm Pb²⁺ solution. The percentage removal of Pb(II) increased almost linearly (as depicted in Figure 2) with rising temperature for both adsorbents as the temperature was increased from 30°C to 50°C, indicating that the adsorption of Pb(II) ions on the synthesized adsorbents was more favorable at higher temperatures. The removal percentages at 30°C for GO and MGO were determined to be 80.84% and 87.07%, respectively. As the temperature increased from 30°C to 50°C, the removal percentage increased. The enhanced adsorption of Pb(II) ions at elevated temperatures can be attributed to the migration of metal ions into the active sites of the adsorbent (Guerrero-Fajardo et al., 2020). The elevation in diffusional processes and surface area at elevated temperatures may result from the rupture of some internal bonds, hence increasing the number of adsorption sites (Rehman et al., 2019; Mohan et al., 2006; Beheshti et al., 2016). At elevated temperatures, metal ions undergo dehydration, which enhances their solubility in aqueous solutions and subsequently improves their adsorption (Zhao et al., 2011; Guerrero-Fajardo et al., 2020). A similar trend was observed in the removal of Pb (II) ions by Jia and Lu (2014) using few-layered graphene oxide, and by Mahdavi et al., 2013 employing titanium dioxide nanoparticles.

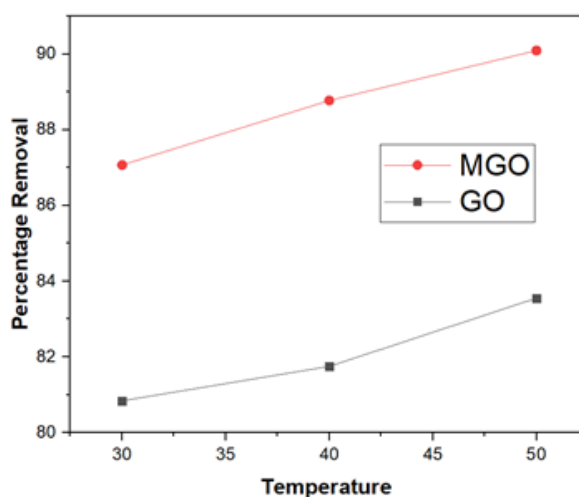


Fig. 2. Effect of temperature on lead adsorption by GO and MGO

Effect of contact time

The influence of contact duration was examined under ideal conditions of pH and temperature (i.e., pH 6.0 and temperature 50°C). The adsorption of Pb(II) ions was noted to rise significantly with contact time, reaching its peak within the first 20 minutes for the adsorbents (illustrated in Figure 4). Subsequently, the percentage removal of Pb(II) ions showed no substantial increase. This may have occurred due to the initial abundance of adsorption sites for metal ion uptake. As equilibrium is approached with prolonged contact time, the adsorption sites become saturated, resulting in a deceleration of the adsorption rate. This may also be attributed to the repulsive interactions

between the pre-adsorbed Pb(II) ions on the adsorbent surface and those present in the solution (Dixit et al., 2017)

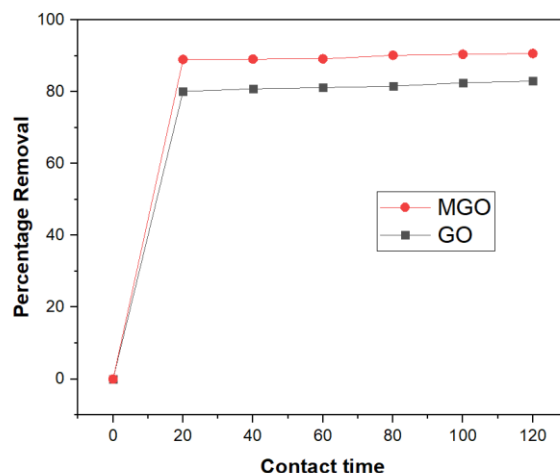


Fig. 3. Effect of contact time on lead adsorption by GO and MGO

Adsorption isotherm modelling

The Langmuir and Freundlich adsorption isotherms were selected to characterize the equilibrium between the adsorbent and adsorbate. The Langmuir isotherm model assumes a homogeneous distribution of active sites on the adsorbent surface. The Freundlich, conversely, accounts for surface heterogeneity.

The linearized Langmuir and Freundlich isotherm equations used in this study are given below:

$$\frac{c_e}{Q_e} = \frac{1}{Q_m K_a} + \frac{c_e}{Q_m} \quad (5)$$

Linear equation of the Langmuir isotherm

Where " Q_e , Q_m and K_a " represent the quantity of Pb (II) ions adsorbed at equilibrium in (mg/g), the maximum amount of metal ions adsorbed in mg per gram of the adsorbent, and equilibrium sorption constant in L/mg which refers to the bonding energy of adsorption. (Liu et al., 2019)

Here, the parameter Q_e was calculated using the following expression

$$Q_e = \frac{(C_i - C_e) \cdot V}{m} \quad (6)$$

The Freundlich equation is expressed as:

$$\log(Q_e) = \frac{1}{n} \log(c_e) + \log(k_f) \quad (7)$$

The linear form of Freundlich isotherm equation

Where K_f is the Freundlich isotherm constant which indicates the adsorption capacity, and $1/n$ is an empirical constant which is dependent on environmental conditions (Mohy-Eldin et al., 2010). The Langmuir and Freundlich isotherm plots for both adsorbents (GO and MGO) are shown in Figures 4 and 5, respectively.

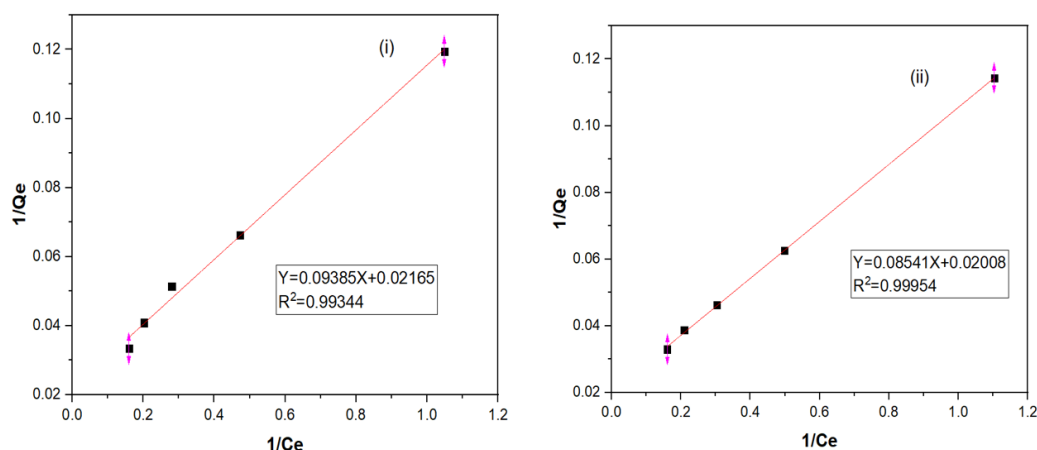


Fig. 4. Langmuir isotherm plot of (i) GO and (ii) MGO

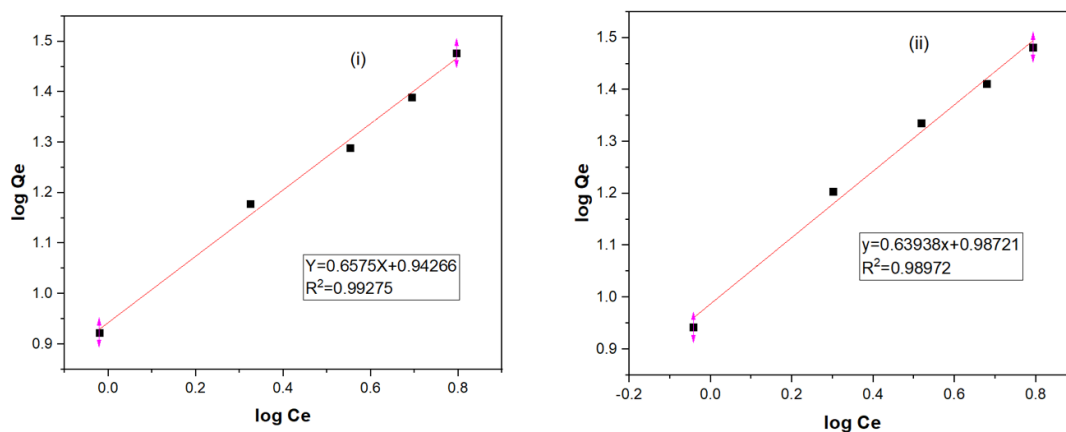


Fig. 5. Freundlich Isotherm plot of (i) GO and (ii) MGO

The calculated Langmuir and Freundlich isotherm parameters are presented in Table 1. The coefficients of correlation (R^2) were higher for the Langmuir isotherm model than for the Freundlich model for both GO and MGO, indicating that the Langmuir isotherm model was more appropriate for the adsorption of Pb(II) ions. The depicted results also illustrated the monolayer coverage of Pb(II) ions on the adsorbent surfaces. The K_L values of the Langmuir model were consistently below 1 for all adsorbents, indicating that the adsorption was favorable. The determined Q_{max} values were 46.19 mg/g for GO and 49.8 mg/g for MGO, indicating that MGO is a superior adsorbent for Pb(II) ion adsorption.

Table 1. Calculated Langmuir and Freundlich parameters for Lead Adsorption by the prepared adsorbents

Adsorbent	Q_{max} (mg/g)	Langmuir		Freundlich		
		K_L (L/mg)	R^2	$1/n$	K_f (L/mg)	R^2
GO	46.18938	0.230687	0.99344	0.6575	8.763145	0.99275
MGO	49.8008	0.235101	0.99954	0.63938	9.709794	0.98972

Thermodynamic studies

Thermodynamic investigations were carried out to monitor the energy fluctuations during the adsorption process. At three distinct temperatures—that is, 30, 40, and 50°C—the thermodynamic parameters were computed using the following formulae. The thermodynamic plots are shown in Figure 6.

$$\ln K = \frac{\Delta S^0}{R} - \frac{\Delta H^0}{RT} \quad (8)$$

$$\Delta G^0 = -RT \ln K \quad (9)$$

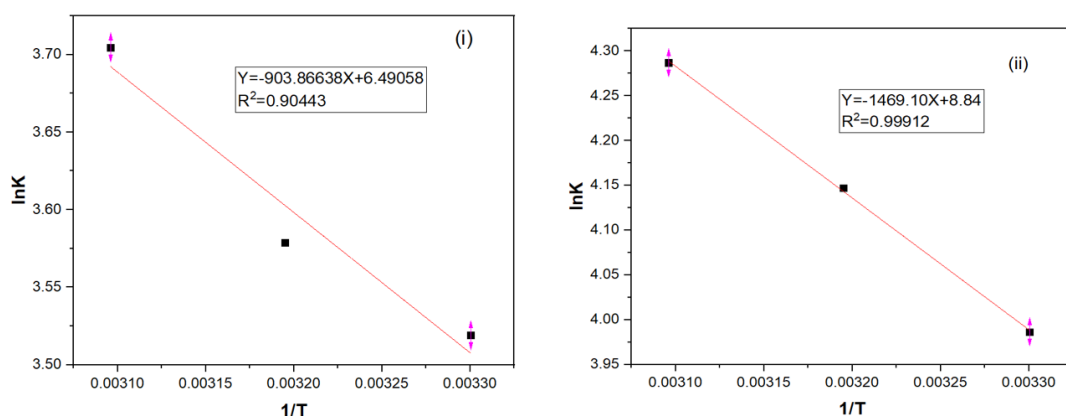


Fig. 6. Plot of $\ln K$ vs $1/T$ to predict thermodynamic parameters for the adsorption of Pb(II) ions onto (i) GO, and (ii) MGO

The calculated thermodynamic parameters are given in Tables 2 and 3. Positive values of entropy change ΔS^0 (Figure 6) indicate that the adsorption process is spontaneous, and there is an increase in the randomness of the solution-solid interface system. The positive values of ΔH^0 indicate that

the adsorption process is endothermic, as supported by the increase in Pb(II) ion adsorption with rising temperature. The negative values of ΔG° reveal that the adsorption process was feasible and spontaneous. Moreover, the more negative ΔG° values with increasing temperature show that the adsorption process is endothermic, as further supported by positive values of ΔH° .

Table 2. Thermodynamic parameters (ΔH° , ΔS°) for Pb (II) adsorption on the adsorbents

Adsorbent	GO	MGO
$\Delta H^\circ(\text{kJmol}^{-1})$	7.5145	12.21414
$\Delta S^\circ(\text{kJmolK}^{-1})$	53.96268	73.469

Table 3. Thermodynamic parameters (ΔG°) for Pb (II) adsorption on the adsorbents

Adsorbent T(K)	GO		MGO	
	lnK (lmg^{-1})	ΔG° (kJmol^{-1})	lnK (lmg^{-1})	ΔG° (kJmol^{-1})
303	3.519	-8.865	3.987	-10.043
313	3.579	-9.313	4.147	-10.791
323	3.705	-9.948	4.287	-11.511

Conclusion

Experiments were conducted to examine the adsorption of Pb(II) by the synthesized GO and MGO, considering three variables: pH, contact duration, and temperature. The highest adsorption capacities for Pb(II) were observed at 46.19 mg/g for GO and 49.8 mg/g for MGO under optimal conditions (pH = 6, contact time = 20 minutes, and temperature = 50 °C). This demonstrates that MGO exhibits enhanced efficiency in adsorbing lead ions from aqueous solutions compared to GO. Moreover, MGO has the added advantage of being easily separable from aqueous solutions using standard magnets and may be regenerated after the adsorption process. The equilibrium was achieved promptly within the initial 20 minutes of the experiment. These features collectively make MGO an ideal adsorbent for the development of real-time filters designed to remove lead ions from aqueous solutions. Thermodynamic investigations demonstrated that the adsorption of Pb(II) ions onto both GO and MGO was spontaneous and endothermic. The equilibrium adsorption data were well described by the Langmuir isotherm model for both adsorbents, indicating monolayer adsorption of Pb(II) ions on the surfaces of GO and MGO.

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Author Contributions

KG and KJ conceived the concept, wrote and approved the manuscript.

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Availability of data and materials

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Competing interest

The authors declare no competing interests.

Ethics approval

Not applicable.



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