RESEARCH PAPER

Quantitative Analysis and Adsorptive Removal of Glyphosate from Water Using MgO Nanostructures

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ABSTRACT

The MgO nanoparticles are inexpensive, non-toxic, environmentally benign, and conventional adsorbent materials. They are a fantastic option for eliminating harmful compounds from the environment because of their straightforward adsorption procedure. In this study, the glyphosate was extracted from the soil using MgO nanoparticles, the surface was characterized by using XRD and SEM techniques. Glyphosate was a non-selective systemic pesticide used to manage weeds. Aquatic creatures are extremely hazardous to glyphosates. With time, there is a greater chance that glyphosate may undergo a minor chemical breakdown or absorption due to its high affinity for binding to soil particles. The Freundlich and Langmuir equations may be used to explain the isotherm data collection. It has been used to examine the temperature, adsorbent dosage, and contact time using UV-visible spectrophotometry. Langmuir's isothermal analysis provided excellent data match. At an 82.08% removal rate, the time was unchanged at 20 minutes. The adsorption's kinetic parameter was identified. The adsorbent process is exothermic, spontaneous, and entropy-decreasing when its free energy of Gibbs, enthalpy, and entropy are negative. According to the findings, magnesium oxide nanoparticles are an effective adsorbent for glyphosate in its aqueous solutions.

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INTRODUCTION

Adsorption is an important method for the removal of contaminants according to its high efficiency, flexibility, low cost, simplicity, and activity toward a wide range of contaminants [1,2]. Nanoparticles have a high affinity with adsorbents due to high porosity, their specific surface area, and active surface [3]. The adsorption method of pesticides on nano-surfaces is widely used. Previous studies indicate that the Nano Metal Oxides have a high affinity for adsorption towards metals. So, they used to remove some toxic metals from water. Many studies show that MgO nanoparticles are traditional adsorbent substances, eco-friendly, low cost, and non-toxic [4,5]. Other studies have reported the superior characteristics of nano-materials as a result of their size and they show extremely superior

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performance in many fields [6-8]. The adsorption method of pesticides on Nano-surfaces is widely used. Industry and agriculture are the sources of introducing pesticides into the environment, so there are many studies that used Nanoparticles to remove pesticides from the environment by adsorption method as shown in Fig. 1. [9-11]

Glyphosate, a non-selective and systemic herbicide, effectively controls weeds by preventing the creation of aromatic amino acids, which are vital for protein synthesis in vulnerable plants. Because of its strong adsorption to soil, glyphosate is unlikely to leach excessively or be absorbed by non-target plants. [12-15]. Glyphosate is unlikely to enter the water through the surface because it binds strongly to the soil unless the soil is washed away by runoff [16]. The studies found that over 50% of the glyphosate was still present 14.4 km downstream due to its direct discharge into the waters of an irrigation canal [17]. Fig. 2 shows that the biodegradation pathway of glyphosate does not always lead to complete detoxification, as it produces AMPA, a persistent pollutant in the environment. The accumulation of glyphosate and its derivatives in soil and water disrupts microbial activity, reduces soil fertility, and negatively impacts non-target organisms such as earthworms and beneficial fungi. Glyphosate can also migrate

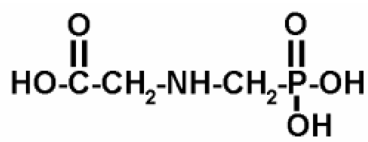

Fig. 1. Glyphosate chemical formula.

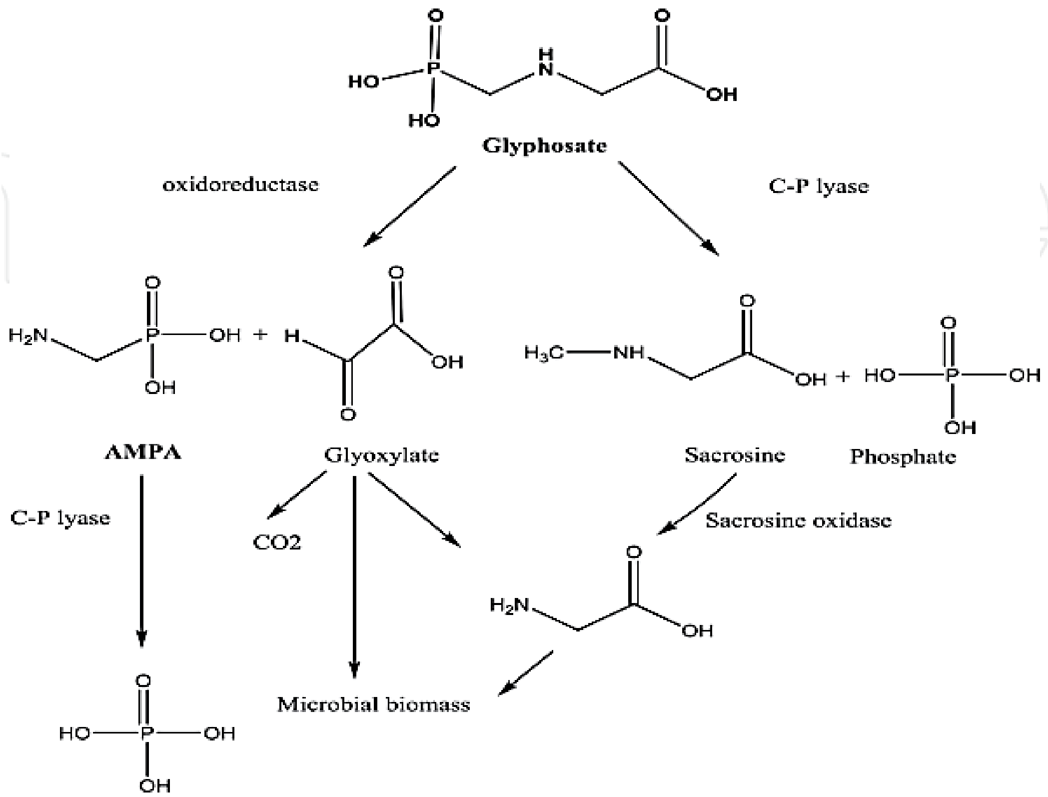

Fig. 2. Glyphosate biodegradation pathways [18].

into groundwater, increasing the risk of water pollution and affecting aquatic ecosystems by inhibiting the growth of algae and aquatic plants. Studies indicate that chronic glyphosate exposure can cause long-term environmental disruptions due to its slow degradation and the persistence of its intermediate products [18].

MATERIALS AND METHODS

Materials

All the chemicals are of high purity, commercially available AR grade.

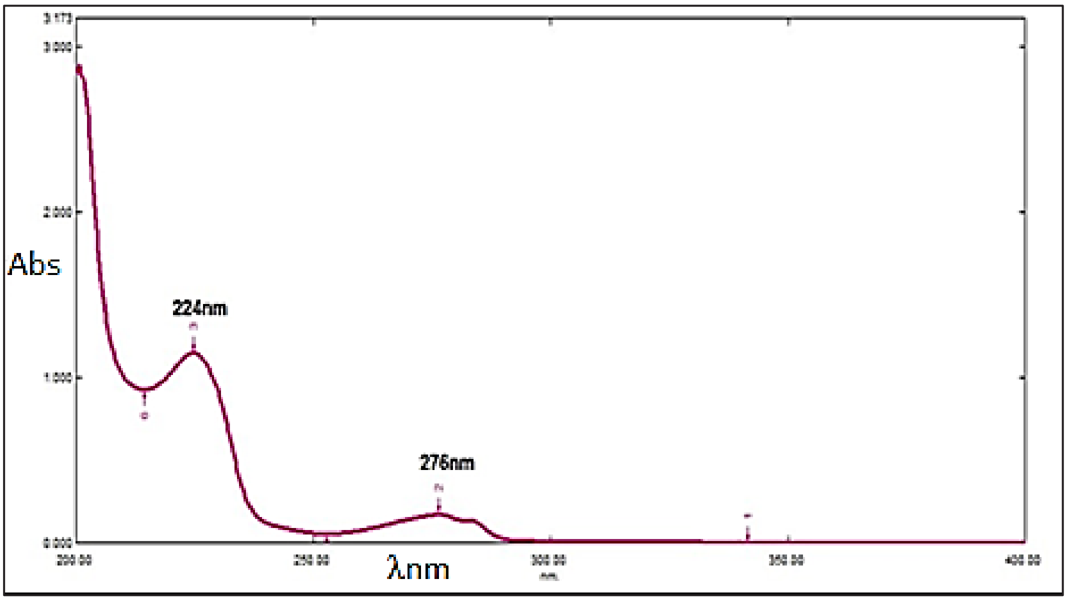

Fig. 3. The spectrum of Glyphosate.

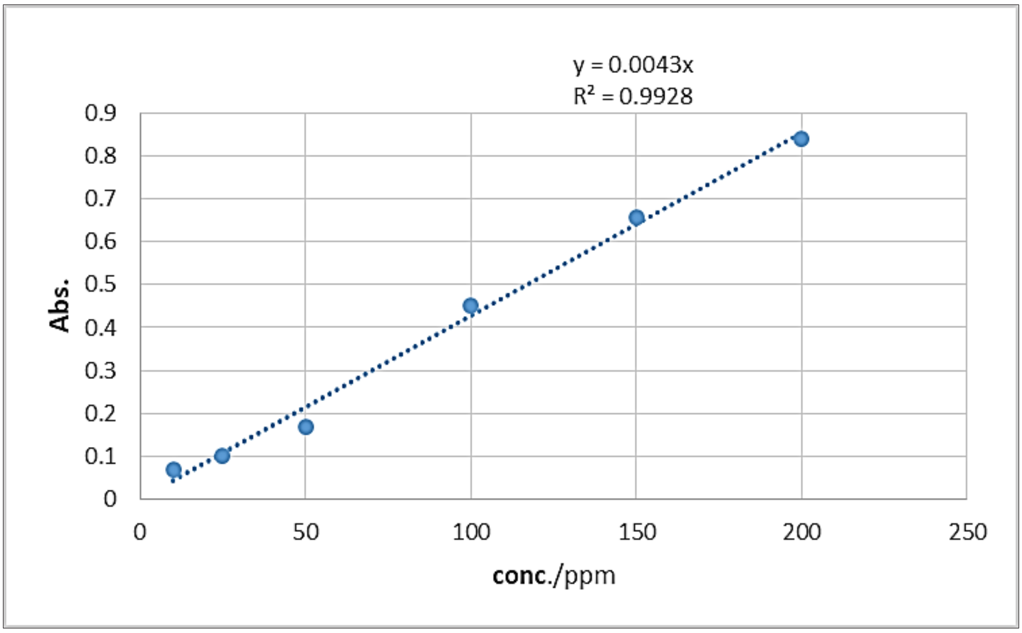

Fig. 4. Calibration curve of the Glyphosate.

Synthesis of MgO nanoparticles

The MgO nanoparticles were synthesis according to the method describe by Sim et al. [19]. MgO nanoparticles were prepared using the sol-gel method by dissolving magnesium nitrate in deionized water at pH 9.45 and gradually adding ammonia under continuous stirring at 80°C. The resulting precipitate underwent intensive washing and drying, then thermally calcined at 500°C for 2 hours and 20 minutes to ensure the final nanostructure.

Instrumentation and characterization

To verify the properties of the prepared nanomaterial, several advanced analytical techniques were employed. X-ray diffraction (XRD) was employed to analyze the crystal structure and purity phase of the magnesium oxide flakes.

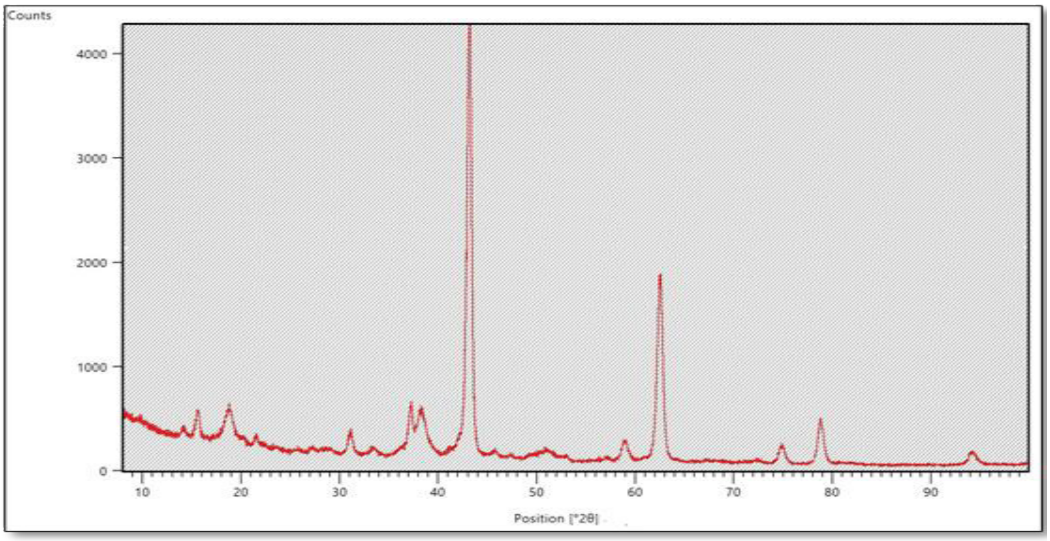

Fig. 5. X-ray diffraction (XRD) diagram of magnesium oxide nanoparticles.

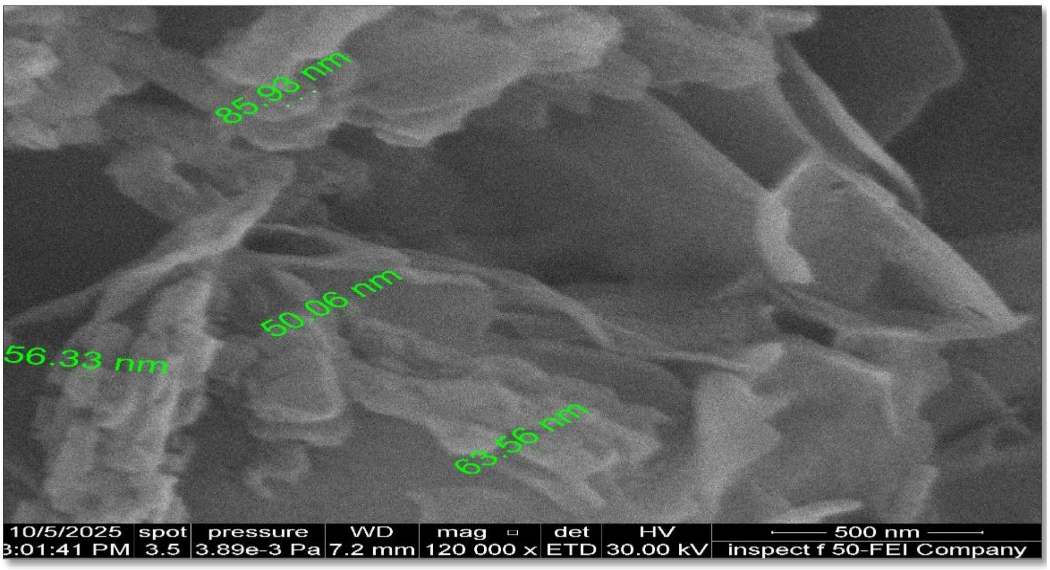

Fig. 6. Field emission scanning electron microscopy (FE-SEM) images of magnesium oxide nanoparticles at different magnifications illustrate the surface morphology.

Electron refraction (EDX) microscopy, attached to the scanning electron microscope unit, was used. This technique allows for the determination of the atomic and weight ratios of the constituent elements of the magnesium oxide flakes and ensures they are free from chemical impurities resulting from the preparation process. For a deeper understanding of particle shape and morphological distribution, field emission scanning electron microscopy (FE-SEM) was used. The concentrations of glyphosate pesticide before and after adsorption were determined using UV-Vis spectrophotometry at a specific wavelength to ensure the accuracy of the removal efficiency

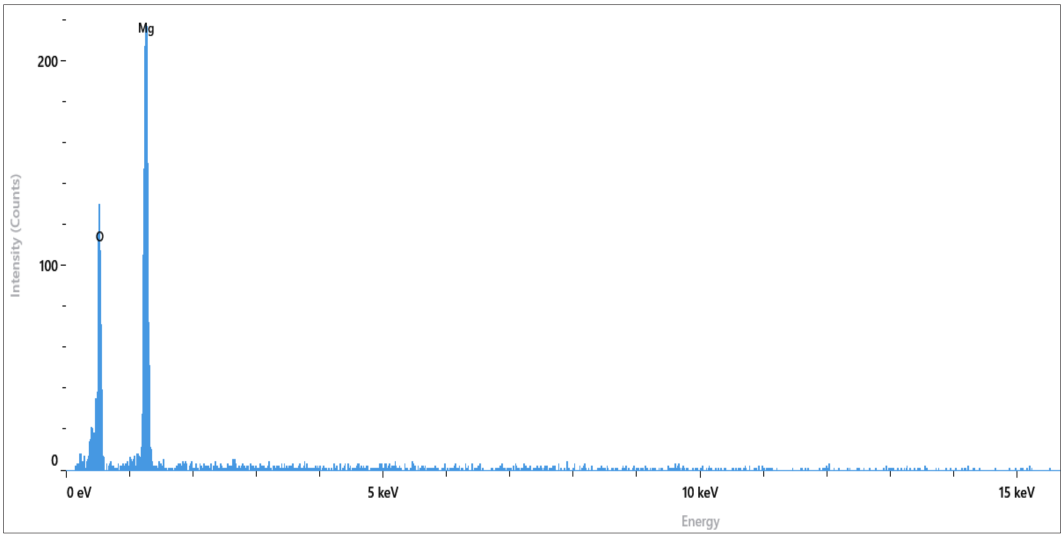

Fig. 7. Energy-dispersive X-ray spectroscopy (EDS) analysis of MgO NPs sample.

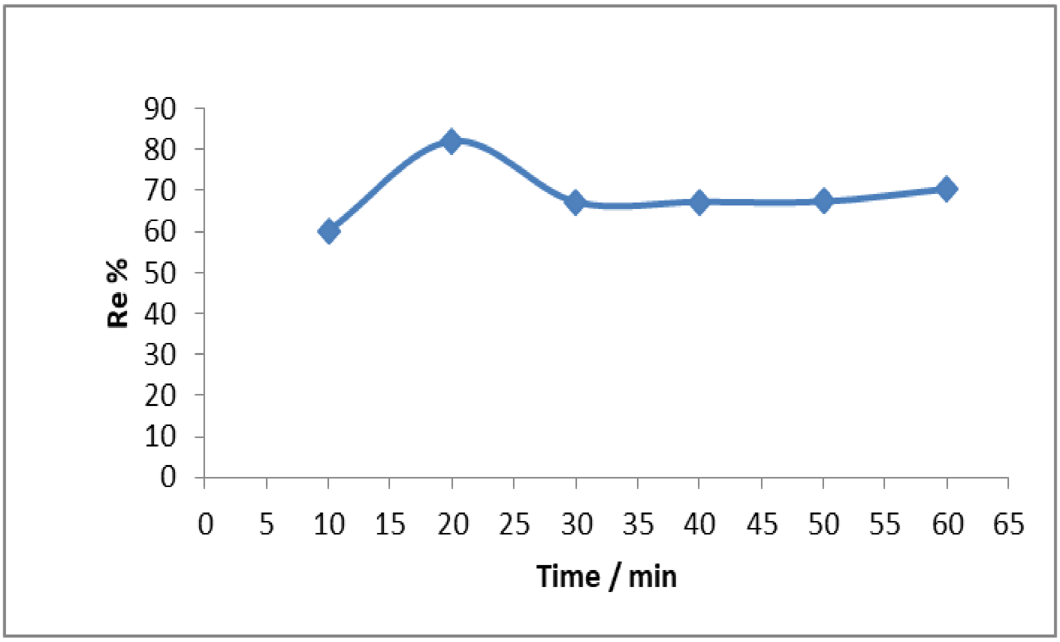

Fig. 8. The effect of time with removal percentage % (Re%).

study.

Procedure

The maximum wavelength (λmax) of Glyphosate is 224nm (Fig. 3) determined in Uv-visible spectroscopy. The calibration curve of glyphosate was determined by preparing five concentrations within the range (10 -200) mg.L^{-1} (ppm) (Fig. 4). Mix 0.1 g MgO with 10 ppm of the glyphosate solution for the procedure. Subsequently, the samples undergo shaking via a vibrator that maintains a constant temperature of 298K [20]. The sample was then filtered and within the range (298 - 318 K) the temperature effect is studied.

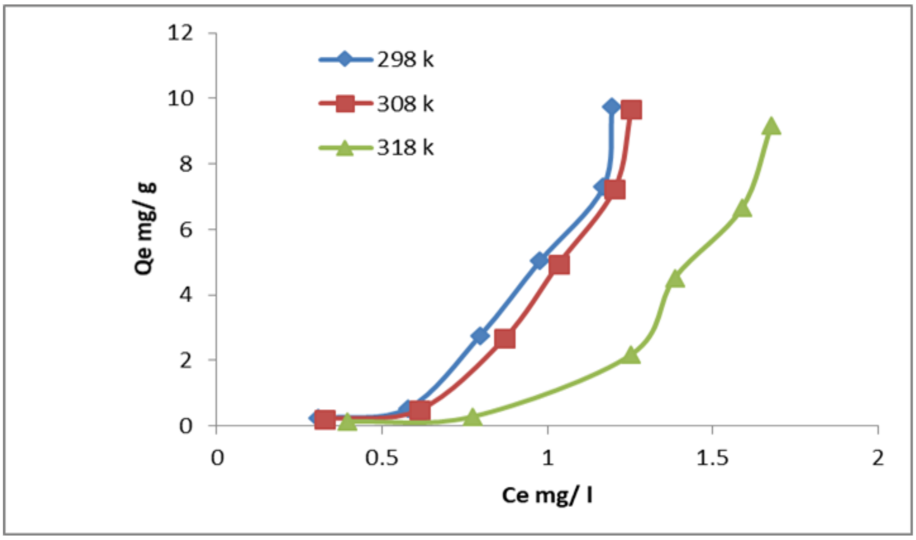

Fig. 9. The adsorption isotherms at 20miat in different temperature.

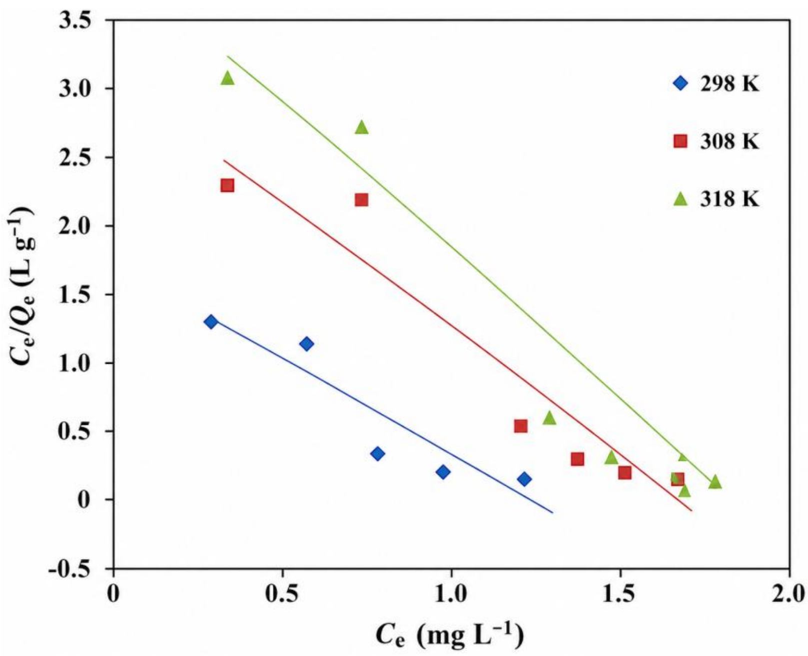

Fig. 10. Langmuir isotherm model.

RESULTS AND DISCUSSION

Characterization of MgO nanoparticles

The properties of the prepared magnesium oxide nanoparticles (MgO NPs) were characterized using a combination of advanced analytical techniques: Utilizing X-ray diffraction (XRD) and field emission scanning electron microscopy (FE-SEM). XRD results as shown in Fig. 5 revealed a diffraction pattern that clearly confirmed the crystalline structure and high purity of the nanoparticles, with peaks conforming to standard specifications. FE-SEM images Fig. 6 provided a detailed view of the surface morphology and porous aggregates. The results from these three techniques complement each other, collectively demonstrating that the prepared nanoparticle surface possesses structural properties and an effective surface area, making it an ideal surface for glyphosate adsorption and achieving high removal efficiency. To confirm the elemental composition and purity of the prepared nanoparticles, electron refraction (EDX) microscopy was performed, as shown in Fig. 7. The results showed sharp, distinct peaks belonging only to magnesium (Mg) and oxygen (O), indicating the correct chemical composition of the MgO particles. The absence of any peaks belonging to impurities or foreign elements is also evident from the diagram, which supports the results obtained from the XRD analysis.

Adsorption studies

The effect of time

The best contact time showed in (Fig. 8) the adsorption was at 20 min. due to the all adsorbate bonding on the adsorbent surface at the time due to the large number of active sites that are not occupied before that time [21].

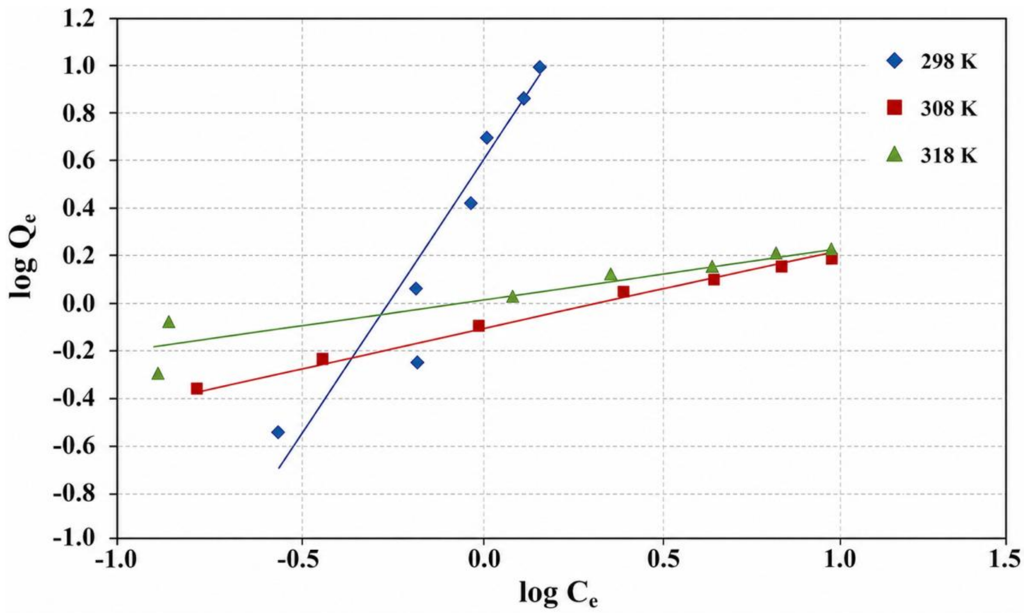

Fig. 11. Freundlich isotherm.

Table 1. The value of Freundlich and Langmuir constant.

Temp. K	Freundlich isotherm			Langmuir isotherm			
	log Kf	n	R^2	a (mg/g)	b (mg/l)	R^2	RL
298	5.072	0.3496	0.9642	-1.1032	-0.6787	0.7557	-0.2666
308	2.0749	0.3173	0.9444	-0.6179	-0.5700	0.8603	-0.3344
318	1.5573	0.3004	0.9493	-0.4906	-0.5469	0.8741	-0.3535

The adsorption isotherms

The study of adsorption isotherms gives important information about the adsorption process and its conditions. The general shape of glyphosate isotherm on the surface of MgO nanoparticles in Fig. 9 is comparable to the S1-type according to Giles classification. The S-type refers to the orientation of the active sites of the glyphosate that are inclined or vertically on the surface of the nanoparticles [22].

The Freundlich constants k_f and n which are indicated adsorption capacity and adsorption intensity in respectively, while a and b are the Langmuir constants which are represented the capacity and binding energy respectively of adsorption. The R2 values refer to the goodness of fit for Freundlich and Langmuir. As seen in Table 1 and Figs. 10 and 11 Langmuir adsorption model was fitting for the glyphosate on MgO nanoparticles [23].

The effect of temperature

The adsorption nature is confirmed by calculating the thermodynamic parameters of the adsorption process at (298, 308, and 318) K. Gibbs free energy is evaluated by the Eq. 1 [24].

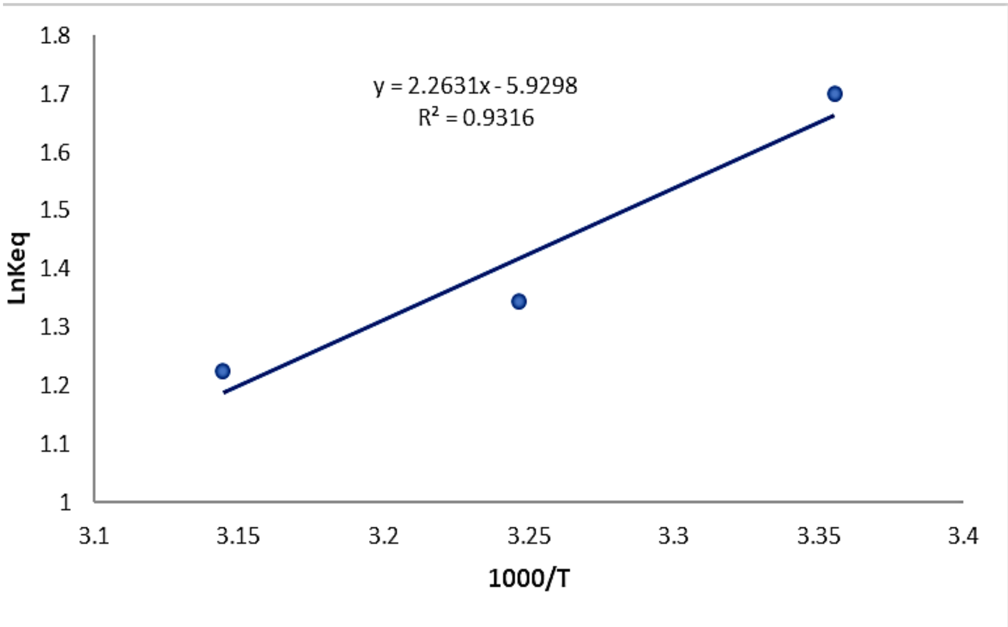

Fig. 12. The value of ln Keq against 1/T k.

Table 2. The Keq values at various temperature.

°C	TK	1000 /T K	Ln Keq
25	298	3.3557	1.7006
35	308	3.2467	1.3432
45	318	3.1446	1.2254

Table 3. The thermodynamic constants.

T(K)	ΔH(KJ. mol^{-1})	ΔG(KJ.mol^{-1})	ΔS(J.mol^{-1})
298	-4.2133	-18.8154	-49.0001
308	-3.4395	-18.8154	-49.9216
318	-3.2397	-18.8154	-48.9799

$$\Delta G = -RT \ln Keq \quad (1)$$

Where K, R, and T are equilibrium constant, the ideal gas constant and temperature respectively.

Equilibrium constant is calculated from the Eq. 2:

$$Keq = \frac{Qe\,.m}{Ce\,.v} \quad (2)$$

Qe is adsorption capacity in unit mg.g^{-1}, Ce is equilibrium concentration in unit mg.L^{-1}, m is weight of adsorbent in unit g. v is volume of adsorbant solution unit of L (Eq. 3) [25]:

$$LnK_{eq} = \frac{-R\Delta H}{T} + \frac{\Delta S}{R} \quad (3)$$

From Table 2 and Fig. 12 the enthalpy is calculated depends on Van Hoff- Arrhenius Eq. 3.

The entropy is calculated by Eq. 4:

$$\Delta G = \Delta H - T\Delta S \quad (4)$$

From the data in Table 3, the negative value of the free energy of Gibbs ΔG indicates the spontaneous nature of the adsorption process. The adsorption process is exothermic because the value of the enthalpy ΔH is negative and the value of entropy ΔS is negative, which refers to the randomness being decreased during the adsorption process [23,25, 26].

CONCLUSION

This study shows that MgO nanoparticles are useful to remove glyphosate pesticides from aqueous solutions in soil and decrease their toxicity in the environment. The adsorption process is cheap, easy, and not toxic to remove many contaminants from soil and water.

CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this manuscript.

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