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Isotherm study for adsorption of lead and cadmium ions using magnesium oxide nanoparticles

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***Abstract*---**In our study, the green method was adopted using green tea leaf extract in the preparation of nano-magnesium oxide. This method was chosen in the preparation because it reduces toxicity and produces low-cost materials that do not require high energy and are environmentally friendly. The prepared magnesium oxide was diagnosed using UV-Vis, FTIR, XRD and FESEM techniques. After diagnosis, the prepared MgO NPs was used in the adsorption of heavy metal ions, lead and Cadmium, to determine the optimum conditions for the oxide adsorption process and study the experimental data through adsorption isotherms. (Langmuir, Freundlich and Temkin) in order to clarify how the correlation occurs between the adsorbate ions and the surface of the adsorbent material to understand the mechanism of adsorption.

***Keywords*---**magnesium oxide nanopartical, adsorbtion, heavy metal, isotherm.

Introduction

One of the most important challenges facing the world is the provision of fresh water suitable for use in light of the large growth in the population and the resulting increase in human activity in the industrial, agricultural and commercial fields [1] Water pollution with heavy metals (inorganic pollutants) is extremely dangerous, as heavy metals are transmitted to humans directly through drinking water or indirectly through the food chain. The danger of heavy metals lies in their high solubility in addition to their stability and lack of decomposition, and thus accumulate inside living organisms and humans,

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resulting in many diseases that negatively affect human health because of its toxic and carcinogenic effect [2]

Heavy metals that have attracted great interest to study their removal are lead and cadmium. Considering it the most dangerous and prevalent metal in sewage water [3]. Lead exists in the form of divalent ions in highly toxic polluted water. The maximum value (Pb^{+2}) that is allowed in drinking water has been set by the World Health Organization (Pb^{+2}) of 0.01 mg / liter, as well as the US Environmental Protection Agency for drinking water USEPA. Exceeding the prescribed limit leads to harmful effects on the human body, as it causes disturbances in the nervous system, digestive system and anemia, in addition to affecting the functions of the liver, kidneys and reproductive system, and also leads to delayed growth, low intelligence and mental retardation in children under six years of age [4].

Cadmium exists in the form of divalent ions in polluted water, and the World Health Organization (WHO) has set the maximum permissible value for cadmium 0.003 mg / liter in drinking water. The US Environmental Protection Agency USEPA classified it as a group B1 carcinogen because of its carcinogenic effect on humans. It also has an effect on bone health through disturbing calcium metabolism, in addition to having a significant impact on the liver, kidneys, and lungs [5].

There are many techniques that have been used to remove or reduce the concentration of heavy metals to the permissible limit according to WHO standards. Such as chemical precipitation, membrane filtration, coagulation and flocculation, ion exchange, electrochemical treatment, solvent extraction and oxidation. In general, these methods are characterized by many disadvantages, such as their high energy consumption, high cost, difficulty in application, insufficient removal with large quantities of pollutants and the generation of new wastes such as sludge that requires disposal [6].

Therefore, adsorption is the best technique to remove heavy metals due to its ease of application and low cost, in addition to the high efficiency in removal [7]. Despite the effecetiveness of the adsorption technology, it faces the challenge of resisting mass transfer in relation to the size of the adsorbents. This problem has been overcome by using nanotechnology in the manufacture of adsorbent materials. Nano materials have proven their effectiveness in adsorption because of their advantages such as the large surface area relative to the volume and the lack of resistance to diffusion and because they have many active sites that have the ability to adsorb heavy metals even with low concentrations, the surface energy of nanoparticles increases [8].

There are many nano materials that were used in the adsorption of heavy metal ions such as carbon tubes and activated carbon , in addition to metal nanoparticles and nano metal oxides such as iron oxides, copper oxides, graphene oxide, cerium oxide, manganese oxide , aluminum oxide and many other nano metal oxides that have a high adsorption capacity to remove heavy metal ions even with low concentrations [9]. In our study, magnesium oxide was chosen to study its effectiveness in adsorption of lead and cadmium ions.

Experimental

Materials needed to conduct the pilot study

Green tea leaves were purchased from the local market in Fallujah, AL Anbar, Iraq , while [$MgSO_4.7H_2O$] was purchased with a purity of 98% produced by a company BDH in addition to salts of lead nitrate [$pb(NO_3)_2$], and cadmium nitrate tetrahydrate [$Cd(NO_3)_2.4H_2O$] used in preparing adsorption standard solutions. As well as titration solutions containing sodium hydroxide 0.1M and hydrochloric acid 0.1M.

Prepare green tea leaf extract

Green tea leaves were washed with distilled water several times to remove dust and suspended matter and left in the shade until completely dried out of the water in a place far from dust. The crushed leaves were ground and 20gm of ground tea was taken and mixed with 200 mL of deionized water in a 500 mL capacity beaker. It was placed in a water bath at a temperature of 353 K for an hour, then the filtration process was carried out after cooling it to room temperature. Then the filtrate of the green tea extract was kept in the refrigerator for use when preparing the oxide [10].

Biosynthesis of MgO NPs

MgO NPs was prepared according to what was mentioned in the publication [11] with some minor modifications, where 50 mL of magnesium sulfate heptahydrate was prepared at a concentration of 0.1 M, mixed with leaf extract in a volume ratio of 1:1 with magnetic stirring for two hours and at a temperature of 60 °C, it was observed that the color changed from yellow to brown. Evidence of the reduction process has occurred and the formation of nano-magnesium oxide [12]. Where phytochemicals absorb heat to turn into an active reducing agent, reducing and stabilizing [13]. The process of separation, washing, drying and then calcination process was carried out at a temperature of 500 ˚C for two hours to obtain MgO NPs.

Characterization of magnesium oxide nanoparticles

MgO NPs was characterized using UV-Vis spectrophotometer (UV-2601 from BIOTECH COMPANY LTD:UK) to confirm the formation of MgO NPs and Fourier-transform infrared spectrometry (SHIMADZU - 1800) to determine the functional groups of the plant extract responsible for the synthesis of nanoparticles and to determine the morphology of MgO NPs, X-ray diffraction technique was used by (SHIMADZO LabX xrd-6000) to determine the crystal structure, as well as field emission scanning electron microscopy by (MIRA III from TESCAN COMPANY) to characterize the size and shape of the prepared magnesium oxide nanoparticles.

Batch adsorption study

To study the effectiveness of the prepared magnesium oxide in the adsorption of pb ions and Cd ions from their aqueous solutions, the optimal conditions for adsorption were determined by studying five factors: pH (2,3,4,5,6,7,8 and 9), contact time (5, 10, 15, 30, 45, 60, 90 and 120) minute, temperature (25, 35, 45 and 55) °C, adsorbent dose (5, 15, 25, 35 and 45) mg, initial concentration (20, 40, 60, 80 and 100) mg/L . The pH function was modified to fit by 0.1M of HCL and 0.1M of NaOH.

Then the residual concentration of the ions in their solutions was measured by a flame atomic absorption spectrometer to calculate the removal efficiency: % R (1) , and the adsorption capacity at equilibrium: q_e (2) to use the equations [14]:

$\% R = [(C_o - C_e) / C_o] \times 100$ … (1)
$q_e = (C_o - C_e) . V (L) / m (gm)$ … (2)

C_o, C_e: the initial and final concentration of heavy metal ions in solution
m: the mass of the adsorbent substance in grams
v: volume of heavy metal ions solution

To analyze the experimental data, the adsorption isotherms were studied using the linear equations of (Langmuir (3) , Freundlich (4) and Temkin (5)) [15]:

$(C_e/q_e) = (1/K_L. q_m) + (C_e / q_m)$ … (3) (K_L, q_m) Langmuir constants
$Lnq_e = LnK_F + (1/n) Ce$ … (4) (K_F, n) Freundlich constants
$q_e = B_T LnA_t + B_T LnC_e$ …(5) (B_T , A_t) Temkin constants

C_o, C_e: the initial and final concentration of heavy metal ions in solution
q_e: the adsorption capacity at equilibrium

Results and discussion

Spectroscopic and morphological diagnosis of the prepared particles

UV - Vis Spectroscopy

It is a method used in quantitative and qualitative analysis to determine the quantity and type of particles by describing the optical properties of nanoparticles by absorbing part of the spectrum UV-Vis. By observing the color change of the solution from the pale yellow color of the extract with salt at the beginning of the reaction, it turns to brown at the end of the reaction, which indicates the formation of MgO NPs [16] and when scanning the sample with wavelengths ranging from (200-800) nm the appearance of an absorption peak at 270 nm shown in the Figure (1) , which falls within the resonance range that determines the formation of MgO NPs that is confined between (260-280) nm as stated in studies [17-18].

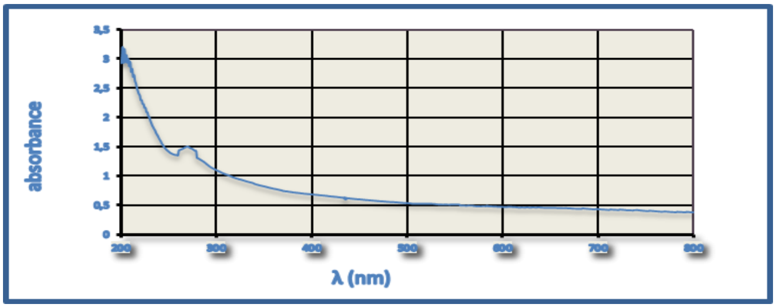


Fig.1.UV-Vis spectrum of MgO

Fourier transform infrared spectroscopy

FTIR was analyzed to determine the effective groups likely to be present on the surface of MgO NPs prepared using green tea leaf extract, by scanning the sample with waves with a wavenumber range ranging from (500-4000) cm $^{-1}$ as shown in the figure (2) It was noted that the broad absorption band at 3340 cm $^{-1}$ indicates a hydroxyl group which may belong to the phenolic or alcoholic groups or carboxylic acids of plant compounds [19-20], while the sharp absorption bands at (1739,1647,1593,1458 and 1315) cm $^{-1}$ refer to the expansion vibrations of (C = C) for alkene and (C = O) for aldehydes and ketones, and the peaks at (1647, 3745) cm $^{-1}$ can indicate (C = O , C - N) to amides [21] and NH_2 to amines respectively that confirm the presence of protein [22]. Finally, we note that the absorption peaks (586-624) cm $^{-1}$ are attributed to the vibration of the Mg-O bond stretching, which indicates the success of preparing MgO NPs using green tea leaf extract. The results that were obtained are almost similar to what was stated in studie [23] This confirms the activity of green tea extract, which contains biologically active phytochemicals that act as a reducing and stabilizing agent on the surface of MgO NPs.

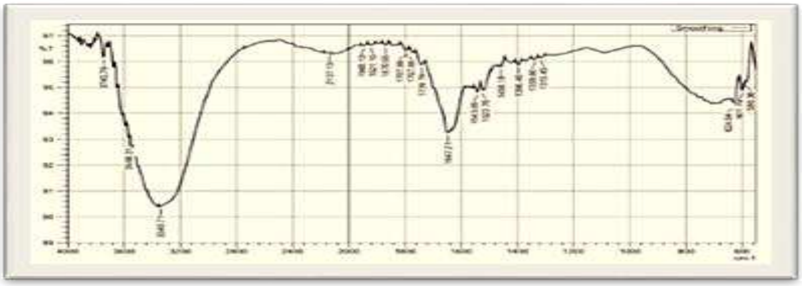


Fig.2.the diagram shows the spectroscopic analysis using FTIR of the prepared MgO

X-ray diffraction analysis

Is used to determine the crystal structure, the location of atoms in the lattice structure. Figure (3) represents the X-ray diffraction plot of MgO NPs powder prepared by green method using green tea extract after calcination. The obtained peaks at 2θ for angles °37.08 (111), °43.09 (200), °62.57 (220),

°75.03(311), °79.00 (222) are shown in the diagram. Which corresponds to the presence of MgO [24] , which is identical to what was stated in the JCPDS standard reference file for MgO NPs No. 98-007-7821 . According to these peaks, the crystal structure of MgO NPs was determined as the face center cube that corresponds to [25] It can also be noted that the X-ray diffraction peaks of the diagram Which was obtained sharp, which indicates that MgO crystallized well [26].

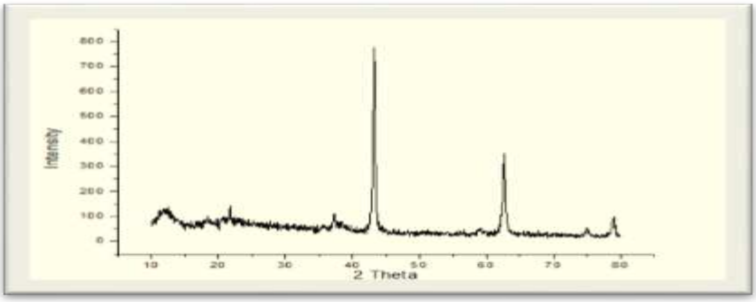


Fig.3.XRD diagram of MgO NPs prepared using green tea extract

Other characteristic peaks do not appear in the X-ray diffraction plot, which indicates a high purity of magnesium oxide nanoparticles [27].

Field emission scanning electron microscopy analysis

The field emission scanning electron microscope technique was used to determine the surface formation

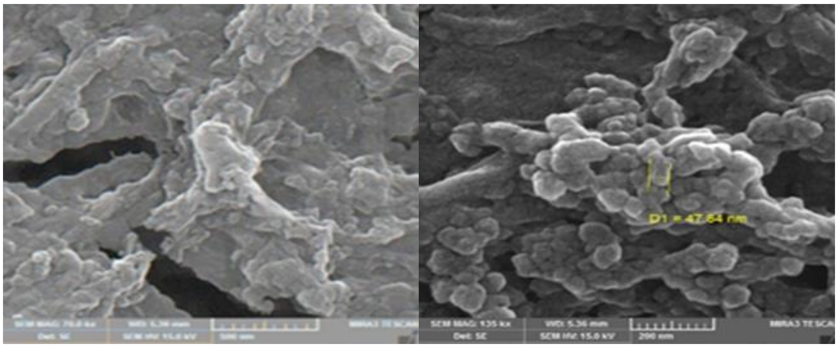


Fig.4.Shows pictures of FESEM to MgO NPs prepared using green tea extract

of nanoparticles by scanning the particles with high-energy electrons focused by lenses to show images FESEM shown in Figure (4), which revealed the spherical shape of magnesium oxide nanoparticles prepared by the green method, and this is similar to what was stated in [28] with sizes less than 100 nanometers, one of its dimensions 47.84 nm.

Adsorption study

Factors affecting adsorption

Effect of pH function

It is considered the main factor that controls the adsorption of heavy metal ions. The effect of the acidity function of the range (2-9) was studied as shown in Figure (5) where it was observed at PH (2-4) that the adsorption capacity decreased due to the association of the adsorption active sites of nanomagnesium oxide with The protons in the acidic solution crowd out with the Pb^{+2} and Cd^{+2} ions and impede their access to the active adsorption sites in the oxide due to electrostatic repulsion, which means an increase in the repulsive forces on the surface of MgO NPs for lead and cadmium ions [29].

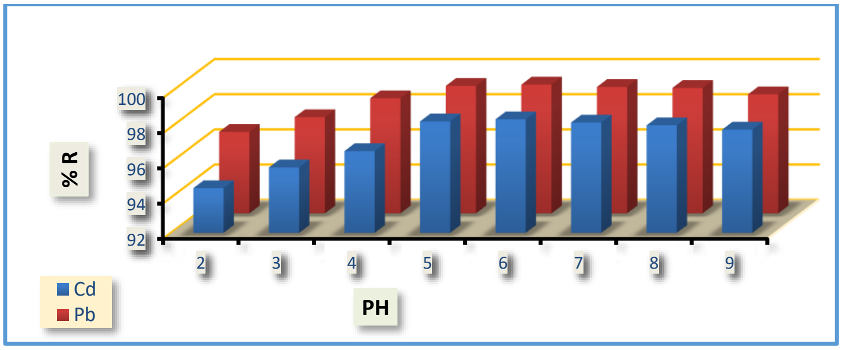
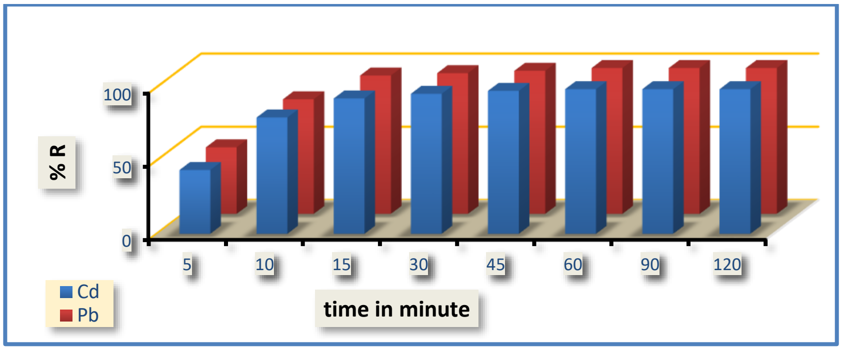


Fig.5. Effect of PH on the percentage of ions removal (Pb,Cd)

As for the PH (5-7), the increase in pH leads to a decrease in the proportion of protons in the solution, so the obstruction caused by the protons on the active sites of adsorption decreases, resulting in a decrease in the electrostatic repulsion of the wave ions of heavy metals on the surface of the adsorbent material, in addition to an increase in hydroxide ions in the solution of heavy metal ions (Pb^{+2} and Cd^{+2}). The adsorption of metal ions increases with increasing PH [30]. but at the value of PH (8-9), we notice a slight decrease in the adsorption capacity, this may be due to the formation of hydroxyl complexes of heavy metal ions (Pb^{+2} and Cd^{+2}) with the sites Active of adsorption , which leads to the reduction or blockage of some active sites. In other studies, this was explained by the deposition of heavy metal ions in the form of hydroxides [31].

Effect of contact time

The study of the effect of the contact time is important to obtain the best time at which the removal of heavy metal ions occurs (determining the equilibrium time). Increasing the contact time of all adsorbent materials leads to an increase in the adsorption efficiency until reaching the equilibrium state at which the removal value stops when conducting the adsorption process [32].

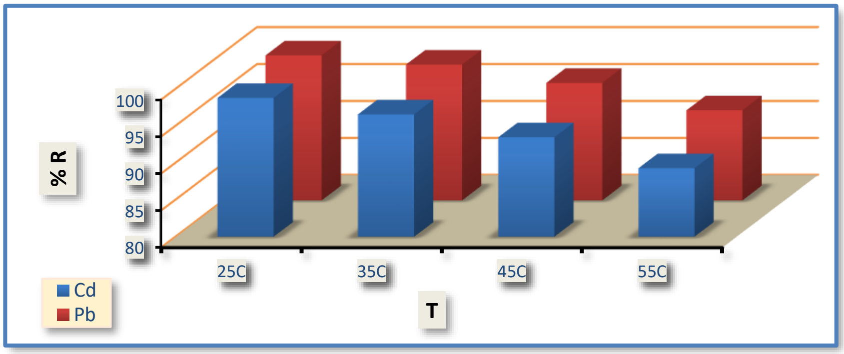


Feg.6.effect of contact time on the percentage of removal of heavy metal ions (Pb, Cd)

Note from Figure (6) that during the first 15 minutes the adsorption efficiency is fast to remove heavy metal ions (Pb^{+2} and cd^{+2}), followed by a slowdown in the removal speed until the amount of (Pb^{+2} and cd^{+2}) ions adsorbate on the surface of MgO NPs prepared using green tea extract reaches the equilibrium limit, and this can be explained because at the beginning of the adsorption process the active sites for adsorption on the surface of MgO NPs were vacant, meaning that the surface area of the MgO NPs that adsorption occurs is the largest at the beginning of the adsorption process [33-34] so the adsorption process occurs very quickly in the first 15 minutes. Then followed by a significant decrease in the number of active sites for adsorption on the oxide due to their association with the adsorbate ions of heavy metals, so we find that the efficiency of adsorption to remove heavy metal ions (Cd^{+2} and Pb^{+2}) slows down, but when the equilibrium state is reached, the adsorption process becomes independent of the contact time [35]. In other words, the adsorbate metal ions on the surface of the adsorbent impede the adsorption of the remaining ions in the solution due to the repulsive forces between them [36].

Effect of temperature

The effect of temperature upon adsorption of heavy metal ions depends on the type of adsorption process, indothermic or exothermic. The effect of temperature was studied at four degrees Celsius, starting from 25°C with an increase of 10 degrees Celsius for each study (25,35,45,55) °C according to the results that were reached and shown in the figure (7), we note The efficiency of removing heavy metal ions (lead and cadmium) on the surface of the MgO NPs prepared by the green method decreases with increase in temperature, and this indicates that the adsorption process is exothermic [37], where we notice at 25°C the largest removal rate. This can be explained by the fact that the initial increase in temperature to 25°C led to an increase in the rate of diffusion of heavy metal ions on the MgO NPs surface resulting from an increase in the strength of the electrostatic interaction between the active sites of adsorption on the surface of the adsorbent material and heavy metal ions [38].



Feg.7.effect of temperature on the percentage of removal of heavy metal ions (Pb,Cd)

As for raising the temperature, we noticed that the adsorption efficiency decreased with the increase in temperature. This can be explained by the electrostatic attraction force between the active sites on the surface of nano-magnesium oxide and heavy metal ions (Pb^{+2} and cd^{+2}). It decreases when the temperature rises. It is similar to what was stated in the study Tamjidi and esmaeili that included the adsorption of heavy metals on the surface CaO/Fe_3O_4/SDS [39].

Effect of adsorbent dose

The effect of the dose of MgO NPs adsorbent material on the adsorption process was studied at the following weights (5, 15, 25, 35 and 45) mg at pH = 6 and a temperature of 25°C for the same volume and concentration of heavy metal ions solution (25 ml, 100 mg / L), respectively, as it was observed according to the obtained results. It is shown in Figure (8) that the increase in the dose of the adsorbent material (MgO NPs) leads to an increase in the adsorption efficiency to remove heavy metal ions (pb^{+2} and cd^{+2}).

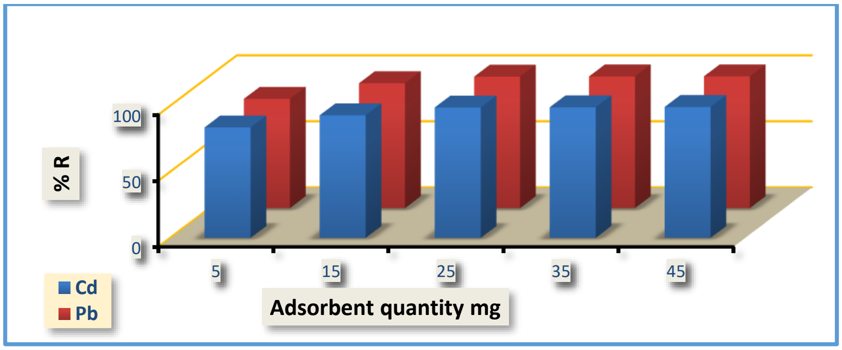


Fig.8.effect of adsorbent dose on percentage of removal of heavy metal ions (Pb,Cd)

This is due to the availability of many active sites available for adsorption by increasing amount of adsorbent while keeping the volume and consentration of the heavy metal ion solution constant, while in contrast the adsorption capacity (the amount of adsorbent material) decreases due to the fact that some adsorption active sites remain vacant or are not saturated with heavy metal ions adsorbate during the adsorption process [40] and in a similar study, the same results were obtained, as by increasing The amount of adsorbent material, the removal efficiency of heavy metal ions increased [33], and despite the increase in the adsorbent substance dose, the increase in the removal efficiency of doses (25, 35 and 45) mg was not a significant difference and this was explained by the occurrence of agglutination Agglomeration of the adsorbent material, which enhances the interaction between the adsorbent particles and reduces the number of active sites for adsorption [41].

Effect of initial concentration

The effect of the initial concentration of heavy metal ions for concentrations (20, 40, 60, 80 and 100) mg/L was studied. It was noted through the results that were reached and shown in Figure (9) that the increase in the concentration of metal ions in the solution while the amount of adsorbent remained constant (25 mg) led to a decrease in the removal efficiency For heavy metal ions (pb^{+2} and cd^{+2}) in a simple way, this can be explained by the constant number of active adsorption sites on the surface of the adsorbent material versus the increase in the number of adsorbate ions [42].

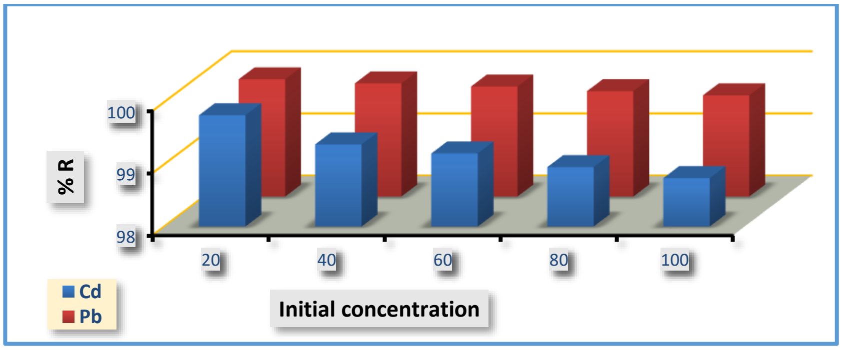


Fig.9.effect of the initial concentration of ions (Pb, Cd) on the percentage of their removal

In other words, low concentrations of heavy metal ions in the solution are adsorbate on the specific active sites of adsorption without crowding. However, with increasing concentration, many of the active sites become saturated with heavy metal ions, and thus the remaining ions crowd out the vacant active sites in the adsorbent material, thus decreasing the adsorption efficiency [43].

While the adsorption capacity is in contrast to the adsorption efficiency, where the adsorption capacity increases with an increase in the number of ions available for adsorption (increase in concentration) due to the availability of a large number of ions with a binary positive charge, which leads to an increase in electrostatic attraction between positively charged metal ions and active negative adsorption sites On the surface of the adsorbent material, the adsorption capacity decreases in low concentration because few metal ions are available for adsorption on the surface of the adsorbent material [44].

Adsorption Isotherm study

The experimental data for the isothermal adsorption of lead and cadmium ions for individual systems at temperature (298,308,318) K were studied by drawing the linear relationships of the isotherms of Langmuir, Freundlich and Temkin, through which the constants of these relationships and the described correlation coefficients were reached in table (1).

Langmuir Isotherm					Freundlich isotherm			Temkin isotherm		
Ions	TEM. K	R^2	$K_L\times10^{-3}$ (L/mg)	qm (mg/g)	R^2	n	K_F (mg/g)	R^2	At (L/g)	B_T
Cd	298	0.9021	36.21898	124.8034	0.9832	2.112058	84.20645	0.8846	40.97599	22.15582
	308	0.9394	10.5376	136.2287	0.9886	1.696721	45.38161	0.9493	6.637818	27.95784
	318	0.9616	4.933393	178.1126	0.9981	1.409537	24.59798	0.9568	2.11750	32.64744
Pb	298	0.9771	116.8389	139.2701	0.9982	1.726644	181.1921	0.9630	71.94123	28.22534
	308	0.9268	23.13244	141.7019	0.9948	1.688995	71.95638	0.9368	14.32084	28.23222
	318	0.9466	8.491412	151.9915	0.9994	1.578056	38.8019	0.9454	4.646527	29.67219

Table (1) includes constants and correlation coefficients of the Langmuir, Freundlich and temkin isotherm

We conclude through the results of applying the isothermal equations of Langmuir, Freundlich and Temkin that the best description of the adsorption of lead and cadmium ions from their aqueous solutions on the surface of MgO NPs is the Freundlich isotherm. This conclusion was reached based on the high correlation coefficient values of the Freundlich isotherms compared to the rest of the isotherms as shown in the figures (10), (11) and (12) , which indicates the multilayer coverage of Pb^{+2} and Cd^{+2} ions on the surface of its heterogeneous nature with different distribution of energy levels [45].

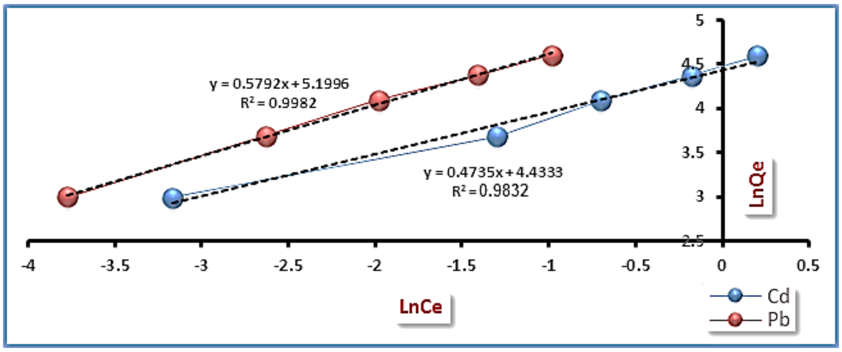


Fig.10.Show the Freundlich isotherm at 25 °C

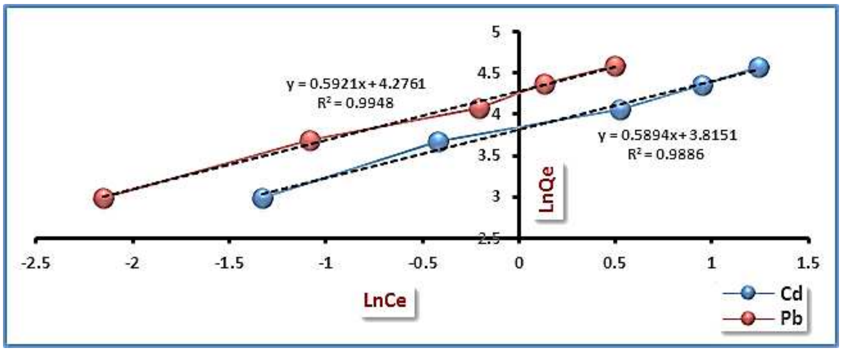


Fig.11.Show the Freundlich isotherm at 35 °C

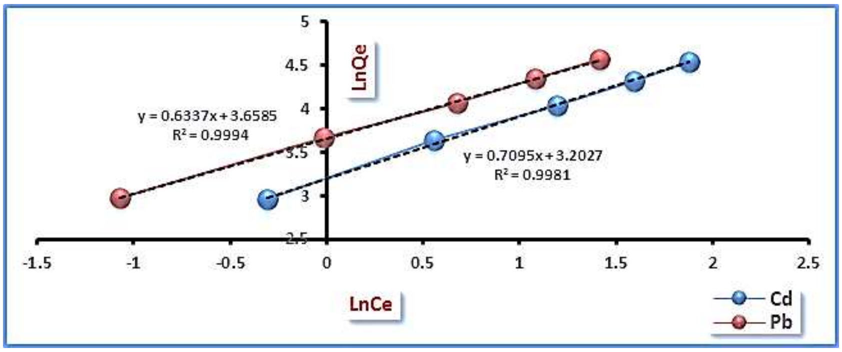


Fig.12.Show the Freundlich isotherms at 45 °C

In addition to the values of the correlation coefficient, the values of (n) were adopted, which represents one of the constants of the Freundlich isotherm, which has an important role in understanding the mechanism of the adsorption process, and it is called the heterogeneity factor expresses the intensity or strength of adsorption where its values were greater than one (n > 1), which indicates that the adsorption of ions pb and cd is a good adsorption of physical nature [46].

Conclusions

First, the prepared magnesium oxide particles were diagnosed as nano-sized and spherical in shape according to what was mentioned in the morphological diagnosis in XRD and FESEM, and then the effectiveness of the prepared MgO NPs in the adsorption prosess of Pb and Cd ions was tested to determine the optimal conditions under which adsorption occurred (PH = 6, equilibrium time 60 min, temperature 25°C, adsorbent dose 25 mg and initial concentration 100 mg/L).From the isothermal study of the experimental data of the adsorption process, it was found that the Freindlich model gave the best fit, which by analyzing the constants, it was concluded that the adsorption process is of a physical nature.

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