

## Use of nano-magnetic materials for removal of lead (II) and cadmium (ii) ions from aqueous solutions

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**Abstract** - Ferrite nanoparticles (NPs) with structure NiFe<sub>2</sub>O<sub>4</sub> were created following the sol-gel auto-combustion method using lemon juice as surfactant and fuel agent. This method is located within the green chemistry, a method of environmentally friendly and less expensive than other methods. Nanomaterial's have been diagnosed using different physical and chemical techniques: Brunauer-Emmett-Teller (BET) analysis, Field Emission-Scanning Electron Microscopy (FE-SEM), Energy Dispersive X-ray Spectroscopy (EDX), Fourier-Transform Infrared Spectroscopy (FTIR), Transmission Electron Microscopy (TEM) and X-ray diffraction (XRD). X-ray diffraction patterns confirmed the phase purity and the particle size 24.27nm. The effects of adsorbent concentration, pH, temperature, and time of contact on Lead and Cadmium ions uptake behavior were measured. The optimum time of contact to attain equilibrium is 60min for Lead (II) ion and 90 min for Cadmium (II) ion, and pH values are in the range (3-9). The results of adsorption showed a strong correlation with Freundlich model compared with the Langmuir model. The thermodynamic parameters were given, revealing endothermic reaction for  $\Delta H$ ,  $\Delta G$  was found a spontaneous process and  $\Delta S$  had positive value, increase of disorder of the process.

**Key words:** Adsorption, Pb(II), Cd(II) ions, ferrite nanoparticles, Langmuir and Freundlich models, Thermodynamic.

### Introduction

After fast growth of population and manufacture societies, the release of heavy metals into the environmental water system is one of the most important environmental concerns. Despite that most of the harmful chemical materials when they are in the atmosphere after some years become less dangerous. When pollution by heavy metal ions occurred, removing them are extremely difficult and maybe gradually built up and enter to our body across food chains (Khoshkardar and Esmaeili, 2019; Luo *et al.*, 2015). Heavy metals are one of the most important pollutants among the different types of water pollutants. They are characterized by severe toxicity even at very low concentrations and are stable in the environment, threatening living organisms and the environment in general. (Seiler *et al.*, 1988; Yadanaparthi *et al.*, 2009). There are several sources of pollution of the water environment with heavy elements, of which electronic industries, electroplating industry, coal-fired electric power stations, tanneries and other industries (Wang and Chen, 2009; Yin *et al.*, 2010).

Water pollution is one of the modern issues by dangerous metal particles like Cd(II), Pb(II) and contamination by microorganisms that causes the saturation of danger in the water and turn into a serious natural medical problem and a general seriousness.

A lot of materials are used to remove heavy metals particularly if they are able to remove the ions but relies on which is your interest in the activity and performance of the process. So many ways were established to guide the waste, such as water precipitation, Commerce Particles, float, Electrochemical oxidation drugs, adsorption, invert osmosis, dissipation, film filtration, and biosorption procedures are widely used (Chen *et al.*, 2009; Ivanov *et al.*, 2004).

The adsorption method has been recognized as a very efficient and economical method (Chai *et al.*, 2013). Since the adsorption mainly depends on the properties of the adsorbent, the selection of a suitable adsorbent is essential. There is currently much research focused on adsorbents for cadmium removal (Chen *et al.*, 2011), inclusively zeolite, activated carbon Red clay, chitosan, resins and Red clay (Liu *et al.*, 2018). However, these materials have some minor disadvantages in their applications, such as poor adsorption capacity, poor adsorption efficiency, or separation problems. In the last few years, there was an increased interest in spinel ferrites having a new row of being favorites for the purpose of water treatment (Masunga *et al.*, 2019; Kefeni *et al.*, 2017). This material has a high surface area as well as contains active places for interacting with the pollutants, thus saving a high absorption capability. Spinel ferrites have possessions super paramagnetic properties (SPM), enabling them to be quickly retrieved From the reaction combination with the use of an external magnetic field (Baig and Varma, 2013; Baig *et al.*, 2015). Spinel ferrites have been studied for their ability to remove organic compounds, nutrient salts, and toxic metals from water (Tran *et al.*, 2020).

The general chemical formula for magnetic spinel ferrites nanoparticle  $MFe_2O_4$  (where  $M = Co^{2+}, Ni^{2+}, Mn^{2+}, Zn^{2+} . . .$ ) provides great magnetic adjustability by changing its size and chemical composition as the high surface area enhances its ability to remove elements (Wang *et al.*, 2004; Yavuz *et al.*, 2006).

These compounds are prepared as powders or thin films by different methods and techniques. The sol-gel technique is considered as the most common methods (Prasad and Gajbhiye, 1998; Dehghanpour, 2016), ball milling technique (Abdel-Latif, 2012), precipitation (Shi *et al.*, 1999), Electrode spinning method (Tan *et al.*, 2009; Nejati and Zabihi, 2012), Hydrothermal method (Salavati *et al.*, 2009), the process of reverse micelles (Maaz *et al.*, 2009), Exact emulsion method (Kavas *et al.*, 2009) and the auto-combustion sol-gel method (Ismat *et al.*, 2017). In the current work,  $NiFe_2O_4$  the ferrite sample was prepared on the basis of the method of sol-el auto-combustion employ Lemonade as a surfactant and a fuel agent. The effect of lemon juice on nitrates, works in the way that lemon juice attaches the metal ions under the assumption that metal ions substitute the hydrogen from  $-COOH$  groups in the lemon juice.

When the citric acid molecules in lemon juice as chelating agent could orient in different directions, hence, the obtained particles had little opportunity for connecting together (Gholipour and Rahimi-Nasrabadi, 2019). This technology is considered environmentally friendly and cost effective against the conventional synthesis of technologies. This labor relates to one of the applications from the adsorption behavior of an aqueous solution. The susceptibility of the produced nanomaterials as adsorbents of Lead and Cadmium ions was examined on the surface of the prepared nanomaterial. The objective of this work is to find for a surface that is with a high degree of Portability for  $Pb(II)$  and  $Cd(II)$  ions adsorption, be used in the process of contamination of the aqueous solution in nature.

**Materials and Methods**

Synthesis of NiFe<sub>2</sub>O<sub>4</sub> nanoparticles:

The process for synthesis of NiFe<sub>2</sub>O<sub>4</sub> was carried out as in the following:

1. 14.54g of Fe(NO<sub>3</sub>)<sub>3</sub>·9H<sub>2</sub>O (M.W 404) was mixed with 5.26g of Ni(NO<sub>3</sub>)<sub>2</sub>·6H<sub>2</sub>O (M.W 290.69) and dissolved in 45ml of lemon juice extract.
2. Ammonia solution (NH<sub>4</sub>OH) was added in order to keep the pH of the solution at 7.0 with continuous stirring using a magnetic stirrer.
3. Continuous stirring for 30min at 80°C, the clear sol. was completely turned to a gel.
4. Oven desiccate at 80°C to get a steady weighing and grinded to fine powder, then calcined at 600°C for 5 hours in furnace under air atmosphere.

Characterization techniques:

For the purpose of diagnosis, many advanced and highly efficient technologies that are used worldwide have been used. These are represented by X-ray diffraction (XRD), transmission electron microscopy (TEM), Brunauer-Emmett-Teller (BET) to find out the surface area, scanning electron microscopy with energy dispersive X-ray spectroscopy (FESEM-EDX). For calculation of the Pb(II) the flame atomic absorption spectrophotometer (AAS) has been used.

Adsorption Studies:

The stock solution of Lead and Cadmium have been made by melting 1.59g of lead nitrate Pb(NO<sub>3</sub>)<sub>2</sub> and 2.74g of Cd(NO<sub>3</sub>)<sub>2</sub>·4H<sub>2</sub>O in 1000ml of deionized water, from this concentration several other dilute solutions were prepared. To determine the ideal pH, both HCl (0.1M) and NaOH (0.1M) were used to keep the pH range at 3-9. Perfect contact time 5-180min for adsorption of Pb(II) and Cd(II) ions was determined using batch method, where 0.05g of the adsorbent has been added to a 25ml solution with concentration of 100 mg.l<sup>-1</sup> of Pb(II) and 20 mg.l<sup>-1</sup> of Cd(II) placed in 100ml flasks. Different temperature is 10.0, 25.0, 37.5 and 50.0°C, were used for equilibration, with agitation rate of 120 rpm using an orbital shaker. The amount of ions adsorbed and removal percent of adsorbent depending on the difference between concentration of the ion before and after adsorption were obtained from equations (1) and (2) (Jain *et al.*, 2019).

$$Q_e = V(C_o - C_e)/m \dots\dots\dots(1)$$

$$\text{removal \%} = [(C_o - C_e)/C_o] \times 100\dots\dots\dots(2)$$

Where; Q<sub>e</sub> is the maximum capacity of adsorption at equilibrium (mg/g), V is the volume of solution (l), m is the weight of the adsorbent material (g), C<sub>o</sub> is the first concentration of the solution in mg.l<sup>-1</sup>, C<sub>e</sub> is the concentration of the solution in mg.l<sup>-1</sup> after adsorption. Langmuir and Freundlich isotherms were used to analyze the adsorption data.

Thermodynamic Parameters:

From the stock solution of Pb(II) different concentrations with ranges of 60-120mg.l<sup>-1</sup> and the 5-30mg.l<sup>-1</sup> of Cd(II) have been prepared, placed in flasks and subsequently 0.05g NiFe<sub>2</sub>O<sub>4</sub> was added to every concentration. The flasks were shaken at 120rpm and in the right balance time at different temperatures 10.0, 25.0, 37.5 and 50.0°C. The mixture was centrifuged and the lead ion concentration was determined. ΔG is the variations in free energy, enthalpy ΔH and entropy ΔS related to the adsorption process were calculated using equations (3-5).

$$\Delta G = -RT \ln k \dots \dots \dots (3)$$

$$K = C_{\text{solid}} / C_{\text{liquid}} \dots \dots \dots (4)$$

$$\ln K = \Delta S / R - \Delta H / RT \dots \dots (5)$$

Where  $\Delta G$  is the Gibbs free energy ( $\text{KJ.mol}^{-1}$ ),  $K$  is the equilibrium constant,  $C_{\text{solid}}$  is the concentration of solid phase at equilibrium ( $\text{mg/l}$ ),  $C_{\text{liquid}}$  is the concentration of liquid phase at equilibrium ( $\text{mg/l}$ ),  $T$  is the temperature in Kelvin and  $R$  is the constant gases ( $0.0083 \text{ KJ.K}^{-1}.\text{mol}^{-1}$ ).  $\Delta H$  ( $\text{KJ.mol}^{-1}$ ) and  $\Delta S$  ( $\text{KJ.mol}^{-1}.\text{K}^{-1}$ ) were calculated by the values of the slope and the intersection using the equation.(5) (Ge and Ma, 2015).

## Results and Discussion

### FT-IR Spectroscopy:

The FTIR spectra of the sample calcified at  $600^\circ\text{C}$  are shown in Figure (1). The infrared spectrum recorded many absorption peaks at the range  $4000\text{cm}^{-1}$  to  $400\text{cm}^{-1}$ . The peak observed at  $3441.01\text{cm}^{-1}$  may be attributed to the stretching vibration of the hydroxide(O-H) bond, which indicates the adsorption of water on ferrite (Anandan and Rajendran, 2011). The position of spinel ferrite finds in the region of  $400\text{-}600\text{cm}^{-1}$  by (Ni-O) and (Fe-O) is stretching vibration (Kareem and Rajeh, 2018).

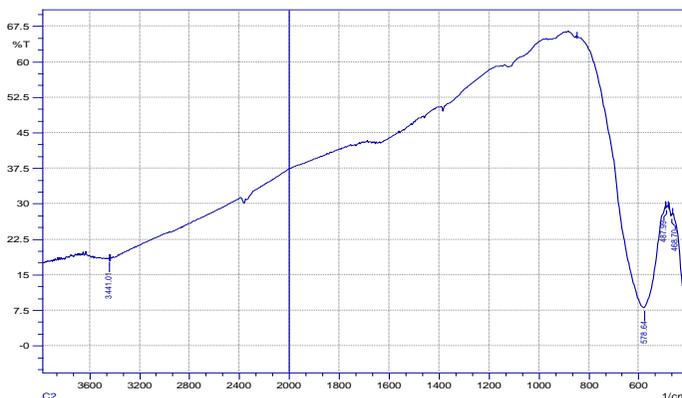


Figure 1. FT-IR spectra of  $\text{NiFe}_2\text{O}_4$ .

### X-ray Diffraction (XRD):

The results in Figure (2) showed the identification of ( $\text{NiFe}_2\text{O}_4$ ) sample prepared by using X-ray diffraction technique. The spectrum of the prepared sample exhibited crystalline and monophasic properties. The standard data (JCPDS file No: 10-325) for  $\text{NiFe}_2\text{O}_4$  (Sezgin *et al.*, 2013) has been used for comparison with the present results, and proved that both of the two study samples can be included within the spinel cubic designated in the reflecting planes (111), (220), (311), (400), (422), (511), (440) and (533) in the patterns. The size of the crystals for the X-ray spectrum and for plane (311) were calculated using the Debye-Scherrer equation:

$$\tau_{hkl} = (K*\lambda) / (\beta_{hkl}*\cos(\theta_{hkl})) \dots \dots \dots (6)$$

Where  $\tau$  is the particle size perpendicular to the natural line of (hkl) plane,  $\beta_{hkl}$  is the full width at half maximum,  $\theta_{hkl}$  is the Bragg angle of (hkl) peak,  $K$  is constant equals to 0.9 and  $\lambda$  is the wavelength of the X-ray (Cullity, 1978). The particle size of nanoparticles calculated is about 24.27nm for  $\text{NiFe}_2\text{O}_4$ .

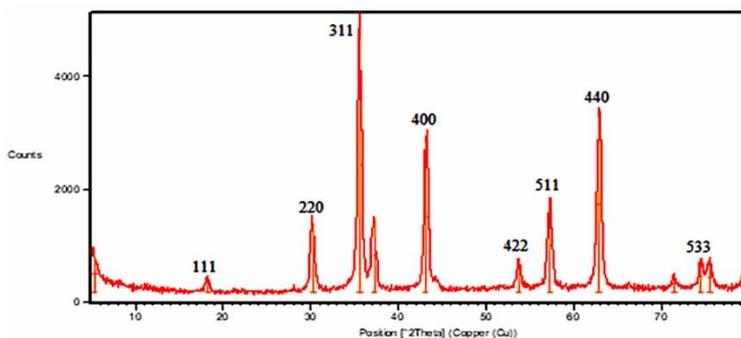


Figure 2. XRD spectrum of NiFe<sub>2</sub>O<sub>4</sub>.

#### Surface Morphology and Elemental Analysis:

Field Emission Scanning Electron Microscopy (FESEM) was used to investigate the morphology of the as-prepared nanoparticles. Figure (3) showed that the bare NiFe<sub>2</sub>O<sub>4</sub> particles were quasi-spherical with small agglomeration. On the other hand, Figure (4) represented the TEM images NiFe<sub>2</sub>O<sub>4</sub> nanoparticles, the image showed the presence of pores and voids within a network formed as a result of the deposition of gases through the reaction of combustion. This finding is typical of combustion-synthesized powders.

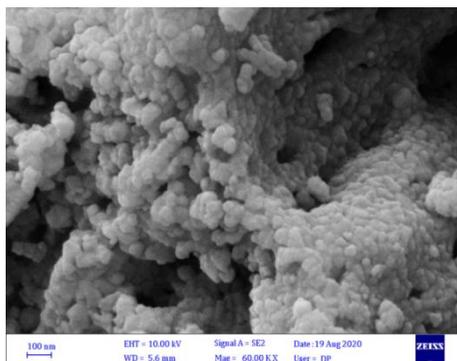


Figure 3. FESEM images of NiFe<sub>2</sub>O<sub>4</sub>.

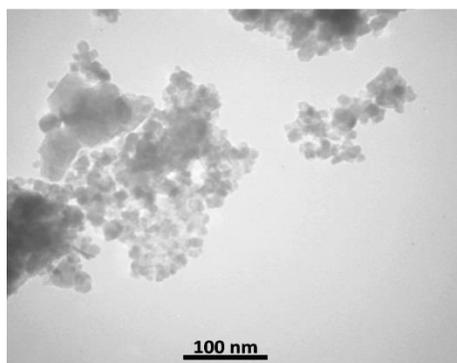


Figure 4. TEM micrograph of NiFe<sub>2</sub>O<sub>4</sub>.

The samples of the particles are roughly spherical. Compositional purity was confirmed by analysis (EDX) of various regions of each sample. In addition the use of EDX technology offers many possibilities in terms of sample components and elements' mapping. Figure (5) and as a Table (1) showed the EDX spectrum of the prepared  $\text{NiFe}_2\text{O}_4$ . Through the results, it is clear that the elements Ni, O and Fe was present in different proportions the presence of impurities are very low.

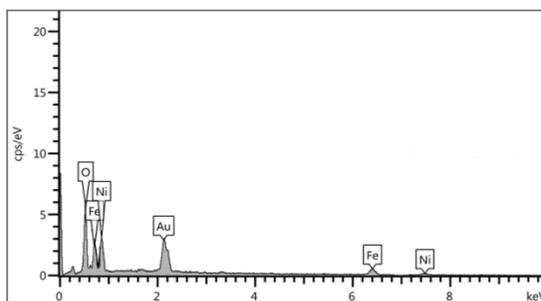


Figure 5. EDX spectra of  $\text{NiFe}_2\text{O}_4$ .

Table 1. Elements percentage calculated by EDX analysis.

Element	Weight %
Fe	42.8
Ni	23.4
O	33.3
Co	0.2
Zn	0.3
Total %	100

Brunauer-Emmett-Teller (BET):

The nitrogen adsorption-desorption isotherms of  $\text{NiFe}_2\text{O}_4$  NPs have studied using Brunauer-Emmett-Teller (BET) analysis (Fig. 6). The BET surface area of  $\text{NiFe}_2\text{O}_4$  NPs was found to be  $3.91\text{m}^2.\text{g}^{-1}$ . The higher surface area of nanoparticles  $\text{NiFe}_2\text{O}_4$  can be attributed to the reduced grain size. Figure (6) showed the size and distribution of pores in the prepared nanoparticles.

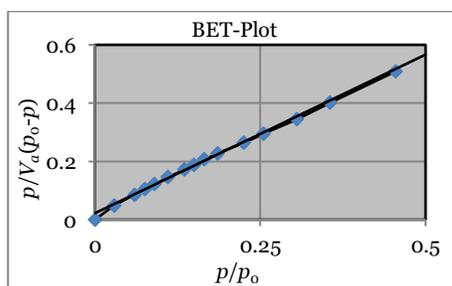


Figure 6. BET of  $\text{NiFe}_2\text{O}_4$ .

The average pore diameter of the nanoparticles  $\text{NiFe}_2\text{O}_4$  is  $7.99\text{nm}$  (Table 2). The average pore diameters were received from ferrite due to the intra-granular pores formative into the metal oxides.

Tables 2. Specific size and surface area of the examined samples.

BET plot		C	Languer plot		B	t-plot a(m <sup>2</sup> .g <sup>-1</sup> )	BJH plot		Peak (Area), R <sub>p</sub> (nm)
V <sub>m</sub> (cm <sup>3</sup> .g <sup>-1</sup> )	a (m <sup>2</sup> .g <sup>-1</sup> )		V <sub>m</sub> (cm <sup>3</sup> .g <sup>-1</sup> )	a (m <sup>2</sup> .g <sup>-1</sup> )			V <sub>m</sub> (cm <sup>3</sup> .g <sup>-1</sup> )	a (m <sup>2</sup> .g <sup>-1</sup> )	
0.90	3.91	49.97	0.90	3.94	0.89	3.22	0.06	5.38	7.99

Adsorption Studies:

Effect of Contact Time:

Different periods of time (5-180min), at fixed temperature (25°C) were used to investigate the influence of contact time on the pb(II) and Cd(II) ion adsorption. Figure (7) showed an increase in the elimination removing percentages of the metal ions by increasing the period of contacting till reaching the equilibrium. The adsorption amount was high in the beginning because there is a large adsorption surface area suitable for attaching the pb(II) and Cd(II) ions. At the end the adsorption rate is slower probably due to that the active sites become saturated and equilibrium is stabilized (El-Ashtoukhy *et al.*, 2008; Pehlivan *et al.*, 2009). The contact time has been reached at 60min for pb(II) and 90min for Cd(II). As for the percentage of items removed in the NiFe<sub>2</sub>O<sub>4</sub> nanoparticles for pb(II) and Cd(II) were found to be 61.74% and 64.30%, respectively.

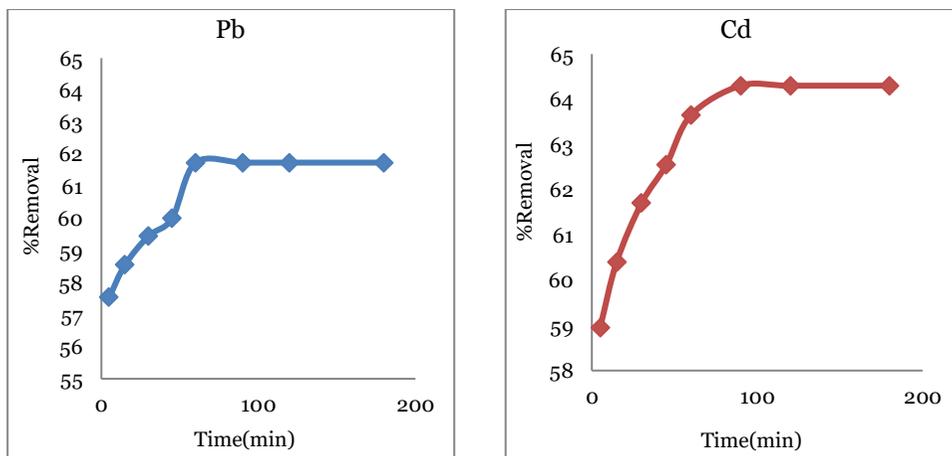


Figure 7. Effect of contact time on adsorption of pb(II) and Cd(II) ions onto NiFe<sub>2</sub>O<sub>4</sub>.

Effect of pH on the Adsorption:

Acidity is a significant aspect that affects the adsorption of heavy metals in aqueous solutions during solid-water interfaces. The accessibility in the solution of metal ions and the Link sites of the adsorbent are affected by pH (Adamson and Gast, 2001). The influence of pH on the adsorption of pb(II) and Cd(II) ion on NiFe<sub>2</sub>O<sub>4</sub> nanoparticles in various pH (3, 5, 7 and 9) were studied by using affixed concentration and time contact of 60, 90min for pb(II) and Cd(II), respectively at 25°C (Fig.8). It showed the effect of pH on the adsorption process.

The present results indicated an increase in the removal of Pb(II) and Cd(II) percentage toward the base. The active sites gain a positive charge in the presence of an acidic medium (low pH) with the presence of hydronium ions, leading to the repulsion between the positive charge metals and the adsorbent. Binding sites begin to deprotonate at higher pH values, making various functional groups accessible for binding the metal.

In general, As the pH increase, cation binding increases; however, as pH rises, the solubility of Pb(II) and Cd(II) ions decreases, resulting in an increase in the adsorption affinity towards the surfaces of NiFe<sub>2</sub>O<sub>4</sub> (Esposito *et al.*, 2002; Lund, 1994). Accordingly, the pH values of 5 and 7 for pb(II) and Cd(II), respectively were chosen to carry out the experiment to avoid uncertain results, as other mechanisms such as sedimentation plays a turn in removing the metals.

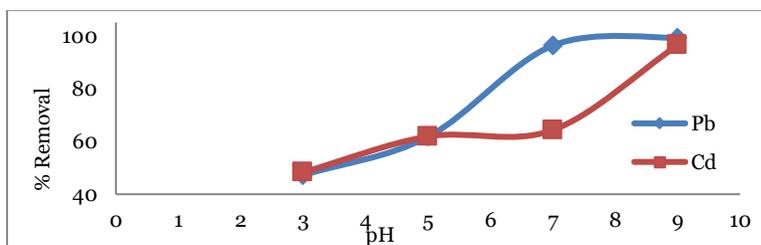


Figure 8. Effect of pH on Pb(II) and Cd(II) adsorption on NiFe<sub>2</sub>O<sub>4</sub>.

#### Adsorption Isotherm:

Various temperatures (10, 25, 37.5 and 50°C), were selected to assess the capacity of nanoparticles NiFe<sub>2</sub>O<sub>4</sub> to remove ions Pb(II) and Cd(II) from their aqueous solutions. Figure (9) showed the isothermal adsorption of Pb(II) and Cd(II) ions and the amount of adsorbent on NiFe<sub>2</sub>O<sub>4</sub> planned at occupying the balance concentration (C<sub>e</sub>).

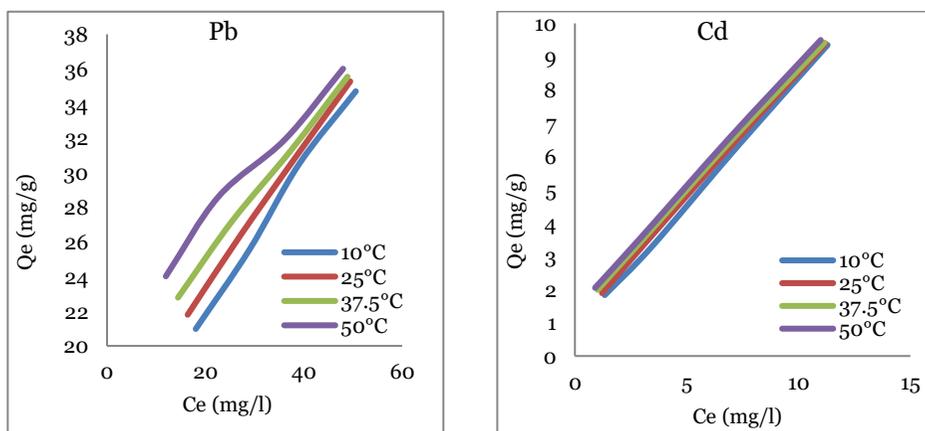


Figure 9. Adsorption isotherm of Pb(II) and Cd(II) on NiFe<sub>2</sub>O<sub>4</sub> at different temperatures.

The isothermal adsorbent of Pb(II) and Cd(II) ions form on nanoparticles NiFe<sub>2</sub>O<sub>4</sub> conforms to the Giles classification of the (S-type). The S-curve reveals the vertical or flat alignment of the adsorption, there is a strong attraction between the molecules within the adsorbent layer as the molten layer is actually adsorbed, where additional quantities are fixed (Giles *et al.*, 1960). The adsorption results were applied in practice using the Freundlich and Langmuir isotherm. The effects were followed up by using Freundlich's (eq. 7) and Langmuir's (eq. 8) (Veena and Robert, 2002), and Table (3) shows the data of isotherm adsorption.

$$\text{Log } Q_e = \text{log } K_f + 1/n \text{ log } C_e \dots\dots\dots(7)$$

Where  $K_f$  is a function of adsorption capacity and  $n$  is the adsorption intensity.

$$C_e/Q_e = 1/Q_m b + C_e/Q_m \dots\dots\dots(8)$$

Where  $Q_m$  is the maximum adsorption capacities (mg/g) and  $b$  is related to the sorption energy.

The Freundlich and Langmuir isotherms are applied to the absorbance experimental data of Pb(II) and Cd (II) ions on NiFe<sub>2</sub>O<sub>4</sub> nanoparticles from drawing log  $Q_e$  opposite log  $C_e$  and  $C_e/Q_e$  opposite  $C_e$ , respectively (Figs. 10 and 11). The results in Table (1) the R<sup>2</sup> values for the Freundlich model are closer to unity than Langmuir for adsorption process onto NiFe<sub>2</sub>O<sub>4</sub>, this proves that the application of the Freundlich model was better in characterizing the Pb(II) and Cd(II) ions adsorption on NiFe<sub>2</sub>O<sub>4</sub> nanoparticles than the Langmuir models and it is the most accurate characterization of the multi-layer adsorption method on the surface of a heterogeneous adsorbent surface (Barrow, 1988).

Table 3. Results of the application of Freundlich and Langmuir isotherms on the system studied.

Ion	Temp. (C)	Langmuir Constants			Freundlich Constants		
		Q <sub>m</sub> (mg/g)	b (l/mg)	R <sup>2</sup>	n	K <sub>f</sub>	R <sup>2</sup>
Pb(II)	10.0	55.285	0.032	0.977	2.027	4.974	0.994
	25.0	51.085	0.042	0.982	2.310	6.422	0.994
	37.5	46.531	0.061	0.986	2.791	8.661	0.992
	50.0	42.533	0.099	0.989	3.544	11.855	0.990
Cd(II)	10.0	23.474	0.054	0.758	1.309	1.397	0.991
	25.0	19.531	0.075	0.863	1.388	1.583	0.994
	37.5	16.366	0.107	0.856	1.538	1.860	0.989
	50.0	15.220	0.130	0.845	1.635	2.050	0.985

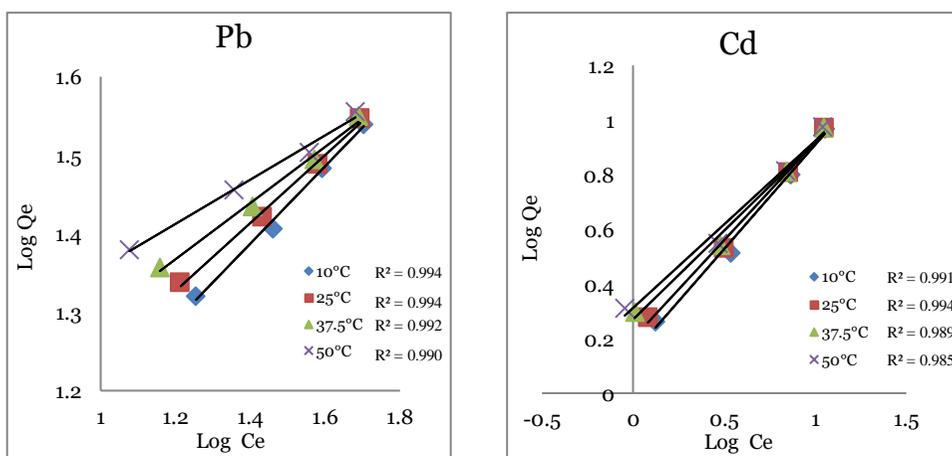


Figure 10. Linear form of Freundlich isotherms of Pb(II) and Cd(II) on NiFe<sub>2</sub>O<sub>4</sub> at different temperatures.

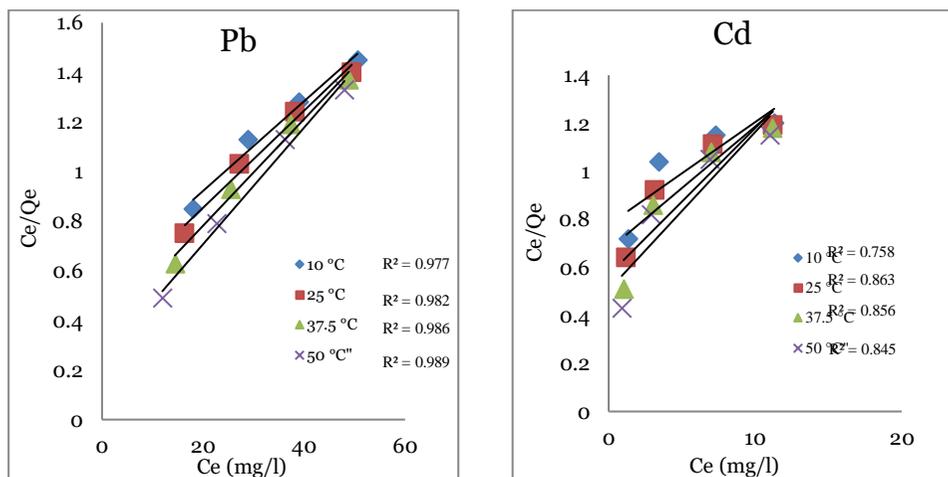


Figure 11. Linear form of Langmuir isotherms of Pb(II) and Cd(II) on NiFe<sub>2</sub>O<sub>4</sub> at different temperatures.

#### The study of thermodynamics:

The effect of temperature on Pb(II) and Cd(II) ions adsorption was investigated using various initial concentrations and temperatures ranging from (10-50°C). The parameters of thermodynamic are represented by ( $\Delta G$ ), ( $\Delta H$ ), and ( $\Delta S$ ) was used to assess the feasibility the adsorption method, as shown in Table (4).

The relationship was linear between  $\ln K$  against.  $1/T$  with the correlation coefficient ( $R^2 = 0.954 - 0.993$ ) and ( $0.923 - 0.998$ ) for Pb(II) and Cd(II) ions, respectively adsorption on of NiFe<sub>2</sub>O<sub>4</sub> nanoparticles (Fig. 12).

As seen from Table (3) represented, the enthalpy values ( $\Delta H$ ) of adsorption of ions Pb(II) and Cd(II) on nanoparticles NiFe<sub>2</sub>O<sub>4</sub>. The results indicated a positive value, which means that the adsorbent process is endothermic (Kapoor, 1994). The positive value of  $\Delta S$  during the adsorption process is expressed that there is an increase in the randomness as a result of the interference between liquid and solid (Al-Saadie and Jassim, 2010), while the value of  $\Delta G$  is negative and all temperatures had values increased with increasing temperatures, which indicates that the adsorption process occurs spontaneously (Hefne *et al.*, 2008).

Table 4. Thermodynamic functions for adsorption of Pb(II) and Cd(II) ions.

Ion	Co (mg/l)	K				- $\Delta G$				$\Delta H$	$\Delta S$
		Temperature									
		283	298	310.5	323	283	298	310.5	323		
Pb(II)	60	2.327	2.660	3.160	4.020	1.664	2.294	2.819	3.344	10.222	0.042
	80	1.767	1.943	2.136	2.522	1.082	1.487	1.824	2.162	6.559	0.027
	100	1.563	1.613	1.673	1.766	0.844	1.009	1.146	1.284	2.269	0.011
	120	1.372	1.427	1.454	1.501	0.606	0.726	0.826	0.926	1.658	0.008
Cd(II)	5	2.759	3.132	3.901	4.555	2.119	2.749	3.274	3.799	9.767	0.042
	10	1.915	2.154	2.311	2.424	1.440	1.755	2.017	2.280	4.503	0.021
	20	1.732	1.801	1.853	1.898	1.101	1.251	1.376	1.501	1.729	0.010
	30	1.654	1.676	1.688	1.729	1.196	1.301	1.388	1.476	0.785	0.007

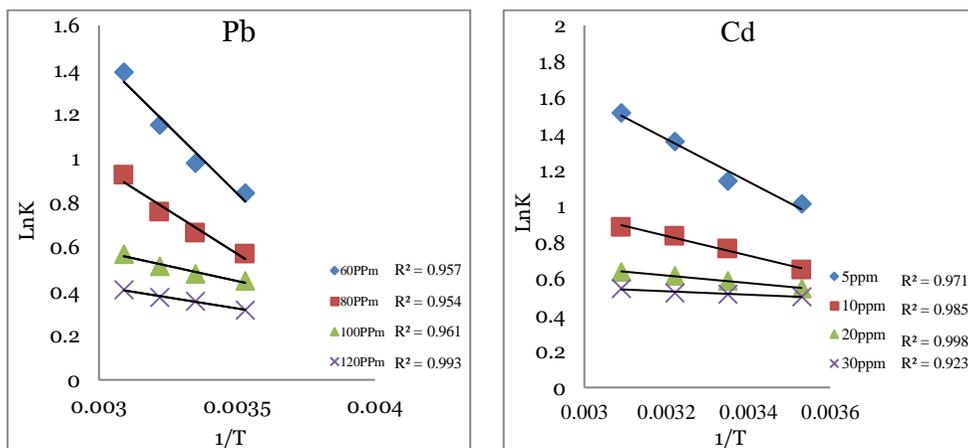


Figure 12. Plot of  $\ln K$  against  $1/T$  for adsorption of Pb(II) and Cd(II) ions on  $\text{NiFe}_2\text{O}_4$  nanoparticles.

## Conclusion

In the present study,  $\text{NiFe}_2\text{O}_4$  nanoparticles ferrites were prepared by sol-gel auto-combustion process and lemon juice, which is surfactant and a fuel agent. A single-stage spinel ferrite was obtained by sample calcining the sample at  $600^\circ\text{C}$ . The BET surface area analyses showed that the composite materials have a large surface area. The effect of different variables on the adsorption capacity, such as contact time, initial concentration of ions, the effect of pH and temperature, were studied. The contact time was at 60min for lead ion, 90min for cadmium ion. The effect of pH with range of 3-9 was studied. The study showed that the percentage of removal of lead ion is 61.47% and cadmium ions 64.30%. The isothermal adsorption was calculated using the models of Langmuir and Freundlich, as it was observed that the Freundlich model was more suitable for adsorption compared to the Langmuir model. As the adsorption reaction was automatic and endothermic according to thermodynamic properties, the prepared compound can be used as a heavy metal adsorbent and considered an important application to preserve the environment.

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## استخدام المواد المغناطيسية النانوية لإزالة أيونات الكاديوم (II) والرصاص (II) من المحاليل المائية

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**المستخلص** - حضرت جسيمات الفرايتات النانوية (NPs) ذات الصيغة  $NiFe_2O_4$  بطريقة السول - جل ذات الاحتراق التلقائي باستخدام عصير الليمون كعامل خافض للشد السطحي ووقود للتفاعل. وتقع هذه الطريقة ضمن الكيمياء الخضراء وهي طريقة صديقة للبيئة وباقل كلفة من الطرق الأخرى. شخّصت المواد النانوية باستخدام تقنيات فيزيائية وكيميائية مختلفة: جهاز تحليل المساحة السطحية Brunauer-Emmett-Teller (BET)، تقنية المجهر الإلكتروني الماسح ذات مجال الانبعاث الضوئي (FE-SEM)، أطيف الأشعة السينية المشتتة للطاقة (EDX)، مطيافية الأشعة تحت الحمراء (FTIR)، المجهر الإلكتروني النافذ (TEM) وحيود الأشعة السينية (XRD). تناول البحث أحد تطبيقات ظاهرة الامتزاز من المحاليل المائية، إذ درس امتزاز أيونات الرصاص والكاديوم على سطح المادة النانوية المحضرة، لغرض الحصول على سطح يمتلك قابلية عالية على امتزاز أيونات الرصاص والكاديوم بهدف معالجة المياه الملوثة بأيونات هذه العناصر. درس تأثير المتغيرات المختلفة على سعة الامتزاز مثل زمن الاتزان والتركيز الابتدائي للأيونات والدالة الحامضية ودرجة الحرارة، كان زمن الاتزان عند (60) دقيقة لايون الرصاص، (90) دقيقة لايون الكاديوم. كما درس تأثير الدالة الحامضية وبالمدى (3-9). أظهرت الدراسة ان النسبة المئوية للإزالة لايون الرصاص 61.47% ولايون الكاديوم 64.30%. استخدمت النتائج التي تم الحصول عليها من دراسة تأثير درجة الحرارة في تطبيق المعادلات الرياضية لايزوثيرمات لانكماير وفريندلش على البيانات العملية للامتزاز، فقد أعطى ايزوثيرم فريندلش علاقة خطية بمعاملات ارتباط جيدة أفضل من ايزوثيرم لانكماير. بينت الدراسة الترموديناميكية ان عملية الامتزاز ماصة للحرارة من قيم  $\Delta H$  الموجبة، وتلقائية من القيم السالبة لـ  $\Delta G$ ، بينما دلت القيم الموجبة لـ  $\Delta S$  على زيادة العشوائية لعملية الامتزاز.