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Removal of lead from water using polyaniline coated cobalt ferrite Nanocomposite

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ABSTRACT
Lead is one of the most toxic heavy metals in industrial effluents which can hurt the environment and human health. Conventional methods for removing this metal from wastewater are often expensive and for this, a cheap and affordable method of purification is needed. The purpose of this study; the measurement of lead removal from aqueous solutions was using cobalt ferrite Nanocomposite coated with polyaniline. In this paper, cobalt ferrite Nanocomposite coated with polyaniline is prepared in a single container method and was examined and identified using scanning electron microscopy (SEM) and Fourier transform (FTIR) and pattern (XRD) infrared spectroscopy techniques. In a discontinuous process to achieve the maximum percentage of lead removal, the effect of pH parameters, adsorbent amount, stirring time, and different temperatures were investigated. The maximum percentage of lead removal was obtained at pH = 7 and 25 ° C and 1 g of Nanocomposite. The two isotherms of Langmuir and Freundlich were also examined by drawing the Langmuir curve, the maximum adsorbent capacity under optimal conditions was 23.31 mg g^{-1}. Also, kinetic studies of the adsorption process were examined the results showed that the removal of lead by cobalt ferrite Nanocomposite coated with polyaniline follows the quasi-first-order model.
KEYWORDS: Adsorption, Heavy metals, Nano sorbents, cobalt ferrite, polyaniline, lead.

I. INTRODUCTION

Removal of toxic heavy metal ions from river water, groundwater, wastewater, especially in industrial and mineral waste effluents, has been extensively studied in recent years due to its harmful effects on human, animal, and plant health [1- 4]. Heavy metal contaminants can be found in the wastes of many industries such as metal plating [5], mining [6], tanning [7], Chlor-alkali [8], radiator production [9], smelting and alloy industries [10] as well as industries. There is battery life [11]. Furthermore, other sources for scrap metal may include: Wood processing industry [12] (arsenic-containing wastes produced by chromatid arsenate copper wood treatment), Production of inorganic pigments [13] which may produce pigments containing chromium compounds [14] and cadmium sulfide, In refining industries in the field of oil refining catalysts are contaminated with nickel, vanadium and chromium and photographic operations that may produce a film with high concentrations of silver and ferrous cyanide and pollute the environment [15].

Due to the possibility of entering large amounts of wastewater contaminated with heavy metals, industries such as Cd, Cr, Cu, Ni, As, Pb and Zn can be considered as one of the most dangerous industries in the chemical industry [16]. Also, due to their high solubility in aqueous media, heavy metals can be absorbed by living organisms. When they enter the food chain, large concentrations of heavy metals may accumulate in human and animal bodies [17]. If these metals are ingested in excess, they can lead to serious health problems. Therefore, treatment of metal-contaminated wastewater before discharge into the environment is much easier, easier, and less expensive, because the complexes created after the accumulation of contaminants and many problems for the separation and treatment of complex effluents can be prevented [18].

The use of magnetic Nanocomposites to remove heavy metals as a simple method can prevent the expansion and generation of complex effluents. One of the most interesting studies that have been done is the use of magnetic nanoparticles on polymers that have led to the formation of two-component Nanocomposite materials [19]. In this research, Due to the importance of nanotechnology and the need to use it in industry, To remove ions of heavy metals such as lead from water, using Nano sorbent cobalt ferrite Nanocomposite coated with polyaniline was prepared by single container method and its adsorption and morphology were investigated.

II. MATERIALS AND METHODS

The materials used to produce cobalt ferrite Nanocomposites coated with polyaniline all have a degree of analytical purity and have been prepared by Merck and Floccat Company.

Infrared spectra of (FT-IR) the compounds were taken by FTIR device model BOMEM MB 102 in the range of 400-4000 cm^{-1}. The XRD (X-ray scattering) spectra of the synthesized compounds were taken using the 1840 Philips XRD PW. SEM images were taken by the LEO 1455 VP Scanning Electron Microscope (SEM). The flame atomic absorption device of the Varian SpectrAA 220FS model was used to measure the absorption of 383.3 nm wavelength for lead (II). The fuel used was acetylene with an exit rate of 2 mL min^{-1} and the oxidant used was the air with an exit rate of 10 mL min^{-1}. The light source used is a hollow cathode lamp with a current of 4 mA for lead (II) with a bandwidth of 0.5 nm.

Absorbent preparation

Preparation of cobalt ferrite by combustion method

Doses of 0.873 g of cobalt nitrate (Co (NO3) 2.6H2O) (II), 2.424 g of iron nitrate (Fe (NO3) 3.9H2O) (II), and 0.9 g of the amino acid glycine (NH2CH2COOH) put in a Crucible, Then add 15 ml of distilled water, a glass mixer is used for mixing. Place the above Crucible, which contains a dark reddish-brown liquid, on a hot electric plate at a temperature of ℃ 300. After a few minutes, the reddish-brown mixture inside the Chinese plant begins to boil and bubbles form on it, then sparks are created around the container, and after the gas is ignited and ignited, a solid black powder is obtained which has a magnetic property. The resulting product is transferred to a human and after three washes with distilled water and then ethanol and separation with a magnet, its impurities are removed. The separated solid is placed in an oven at ℃ 100 ° C for 12 hours to dry completely and then into a mortar to form a soft powder [20].

Coating of cobalt ferrite (combustion) with polyaniline

First, prepare a 50 ml solution containing 0.1 M aniline (0.461 mL), 0.125 M ammonium peroxysulfate (1.42 g) in 0.5 M nitric acid (1.13 mL) and with the help of a magnetic stirrer, the contents inside the human were mixed. Then, combustion-synthesized cobalt ferrite was added to the said human and stirred evenly at room temperature for one hour. After one hour, the aniline polymerization process was completed and the resulting mixture was left for one day. The next day, the polyaniline-coated cobalt ferrite was removed with a magnet and washed with dilute hydrochloric acid (0.1 M), distilled water, and acetone to remove impurities. Then it was placed in an oven at 50 ° C for drying for 8 hours and then the morphology of nanoparticles was examined by XRD, FTIR, and SEM devices [21].

Preparation of solutions

Weigh 1.59 g of lead nitrate salt and dissolve in one liter of distilled water. Then, from the main solution with a concentration of 1000 mg / l, solutions with concentrations of 10, 50, 100, and 250 mg / l were prepared.

Check the amount of absorption; Adsorbent Nanocomposites

To evaluate the process of adsorption of lead metal ions from the solution, 1 g of adsorbent Nanocomposite was added to 5 ml of 50 mg / l solution of metal salts. After adjusting the pH of the environment and stirring at the desired times with a magnetic stirrer and placing the magnet next to the human, the solution becomes clear and the nanoparticles in the human wall are compacted and separated from the solution. The amount of lead metal in the solution was investigated using an atomic absorption spectrometer.

III. RESULTS AND DISCUSSION

Investigation of FT-IR spectrum of chloride -CoFe2O4@PANI Nanocomposite

The infrared spectrum of CoFe2O4@PANI synthetic Nanocomposite with chloride counter ion in the range of 14000-400 cm^{-1} is shown in Figure 1. Peaks associated with cobalt ferrite as well as polyaniline can be seen in the FT-IR spectra of the Nanocomposite.

The peak appearing in the 564 cm^{-1} regions in the infrared spectrum of synthetic Nanocomposites is attributed to the vibration of the Fe-O bond at the quadrilateral position of the $CoFe_2O_4$ spin structure. The rest of the peaks were observed, i.e. the peaks observed in 1574 cm^{-1} (corresponding to the C = C tensile strength of the quinoid ring), 1494 cm^{-1} (corresponding to C = C benzenoid ring tension), 1302 cm^{-1} (related to tensile vibration of C-N benzenoid ring) And 1134 cm^{-1} (corresponding to the vibrational state N = Q = N where Q represents the quinoid ring) is attributed to polyaniline. Also, the C-H outside of the polyaniline aromatic plate is the reason for the peak observed in 819 cm^{-1} [24-20].

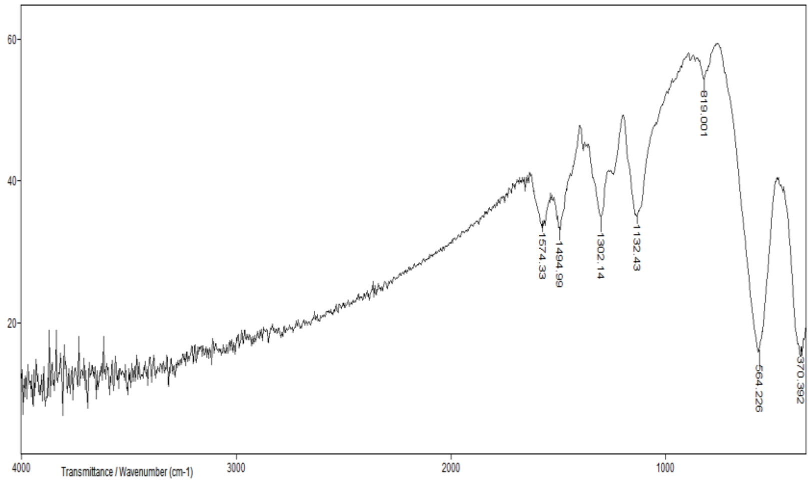

Figure 1: FT-IR spectrum of chloride -$CoFe_2O_4$@PANI Nanocomposite

Investigation of XRD pattern of chloride-CoFe2O4@PANI composite

The X-ray diffraction pattern (XRD) of the composite is shown in Figure 2. The size of the microcrystals was obtained by calculating the Debbie Scherer equation ($D = 0.9 \lambda / \beta\cos\theta$) nm 43. In the XRD pattern of a synthetic composite including cobalt ferrite coated with polyaniline-nitrate, Peaks can be seen at 62 / 745 ˚, 57/200˚, 53/625 ˚, 43/ 235 ˚, 35 /595 ˚, 30 / 210 ˚ = 2 θ angles which are related to the crystal plates (220), (311), (400), (422), respectively 511) and (440) are the cobalt ferrite cube spinel structure.

XRD pattern of chloride -$CoFe_2O_4$@PANI composite shows the characteristic peaks of cobalt ferrite according to its standard card (22-1086) JCPDS, this indicates that the ferrite reverses spinel structure is maintained during the coating process, but the peak intensities are reduced. Also, due to the amorphous nature of polyaniline, no characteristic peak related to this polymer is observed in the XRD pattern [24-20].

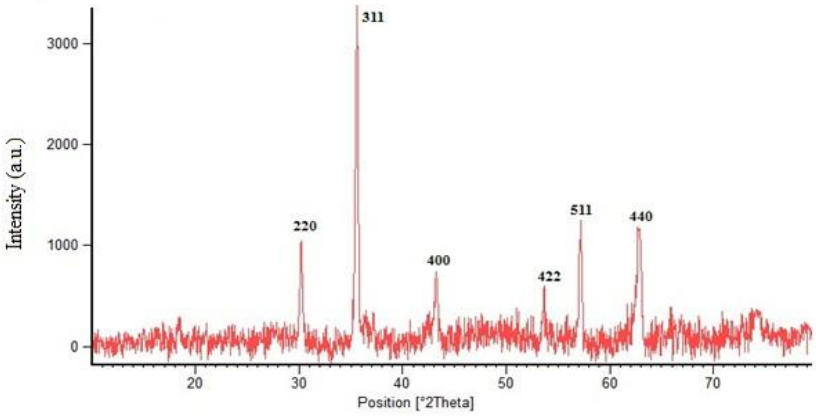

Figure 2: XRD pattern of chloride-$CoFe_2O_4$@PANI composite

Investigation of composite morphology chloride-$CoFe_2O_4$@PANI

The morphology of cobalt ferrite nanoparticles coated with polyaniline-nitrate conductive polymer was determined using scanning electron microscopy and the SEM image of this Nanocomposite is shown in Figure 3. Cobalt ferrite, in addition to having a crystalline structure, also has pores on its surface due to the release of gases from the ignition of glycine. After the polymerization process, a continuous layer of the conductive polymer is formed on the surface of cobalt ferrite which covers the mentioned pores and defects of ferrite, and also polyaniline can reduce the possibility of accumulation of cobalt ferrite nanoparticles due to magnetic properties [24-20].

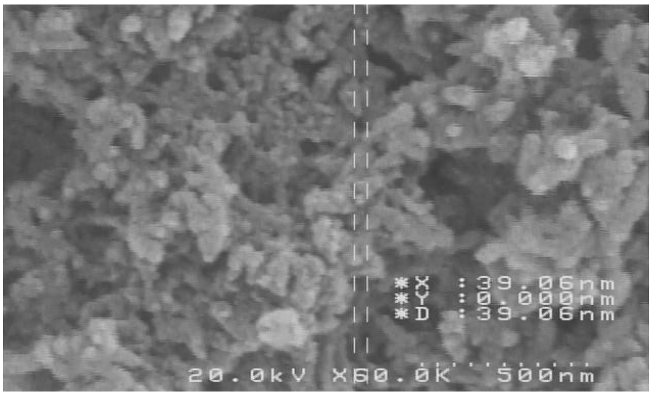

Figure 3: Composite SEM image chloride -$CoFe_2O_4$@PANI

Investigation of the effect of pH on the adsorption process of lead metal on Nanocomposite adsorbents $CoFe_2O_4$@PANI

The pH of the range between 1 and 12 was determined in the presence of 1 g of Nano sorbent in 50 ml of the desired solution and the optimal time of 120 minutes and the initial concentration of metal ions 17 mg / l. The results show that; the residual concentration of metal ions decreased with increasing pH. In other words, the absorption percentage has increased. As a result, pH changes for lead metal at pH <7 are converted to lead oxide; Therefore, it is not possible to study adsorption changes at pH above this value for the lead metal. Therefore, the best pH at which it had the highest absorption of metal ions (98%) is 7. (Figure 4).

Finally, the lead removal percentage (Removal%) was calculated using Equation 1 below.

$$\text{remove percentage} = [(C_i - C_f)/C_i] \times 100 \qquad (1)$$

Where C_i is the initial concentration of lead (before interaction with composite nanoparticles) and C_f is the final concentration of lead (after interaction with composite nanoparticles).

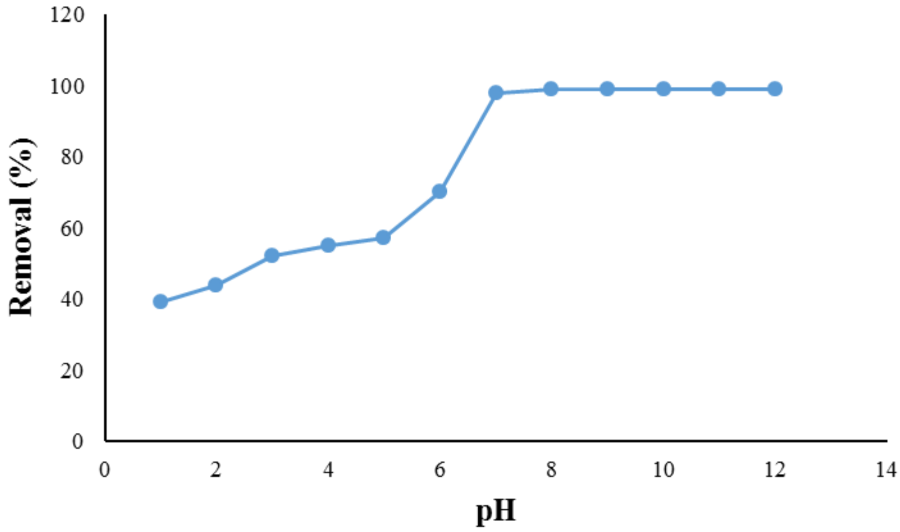

Figure 4: Effect of pH change on lead removal

Investigation of the effect of time on lead adsorption process on Nanocomposites $CoFe_2O_4$@PANI

To evaluate the effect of time on the adsorption process of lead ions from solution using $CoFe_2O_4$@PANI Nanocomposite, the adsorption value at pH is 7 and in 180 minutes in the presence of 1 g of adsorbent, the initial concentration of metal ions is 17 mg / l and the constant volume is 50 ml. Liters is done. The results obtained from the diagram in Figure 5 show that; in the mentioned conditions, the amount of absorption was constant at 120 minutes onwards, so the optimal time was 120 minutes.

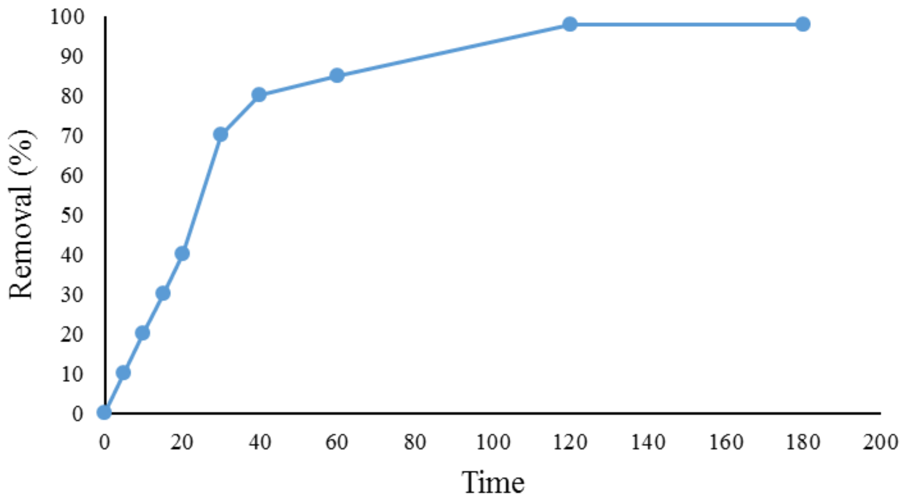

Figure 5: Effect of stirring time of adsorbent and lead solution

Investigation of the effect of $CoFe_2O_4$@PANI Nanocomposite as adsorbent

The results show that the percentage of lead removal per 0.1 g of adsorbent and more is a good value (Figure 6). Therefore, 0.1 g of adsorbent was selected as the optimal amount of adsorbent.

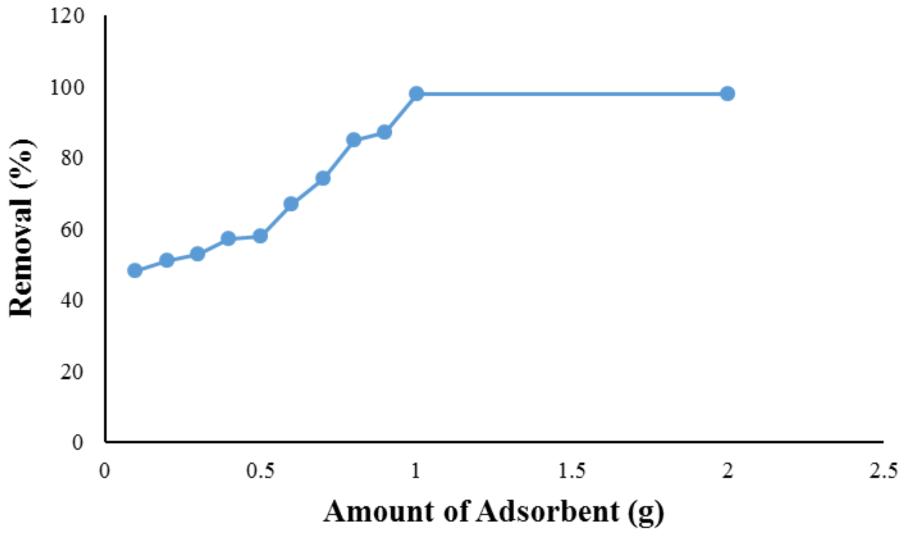

Figure 6: Effect of adsorbent amount on lead removal

Investigation of the effect of temperature on lead adsorption process on Nanocomposites $CoFe_2O_4$@PANI

To investigate the effect of temperature on the percentage of lead removal, 25 ml of 34µg mL^{-1} lead solution with pH = 7 was added to several 50 ml volumetric balloons. The resulting solutions were made up to volume by distilled water with the temperature adjusted so that the temperature of the lead solution in the balloons was fixed at 0, 15, 25, 50, 75, and 100 ° C, respectively. The prepared solutions were then added to several humans, each containing 1/0 g of the **$CoFe_2O_4$@PANI** Nanocomposite. Each of the resulting mixtures was stirred with a stirrer for 120 minutes (a water bath was used to maintain the temperature during the stirring of the solution). In the end, each human was placed on the magnet for 1 minute and after settling the Nanocomposite using a magnet, the absorption of the solution compared to the control was measured. The results of Figure (7) are shown.

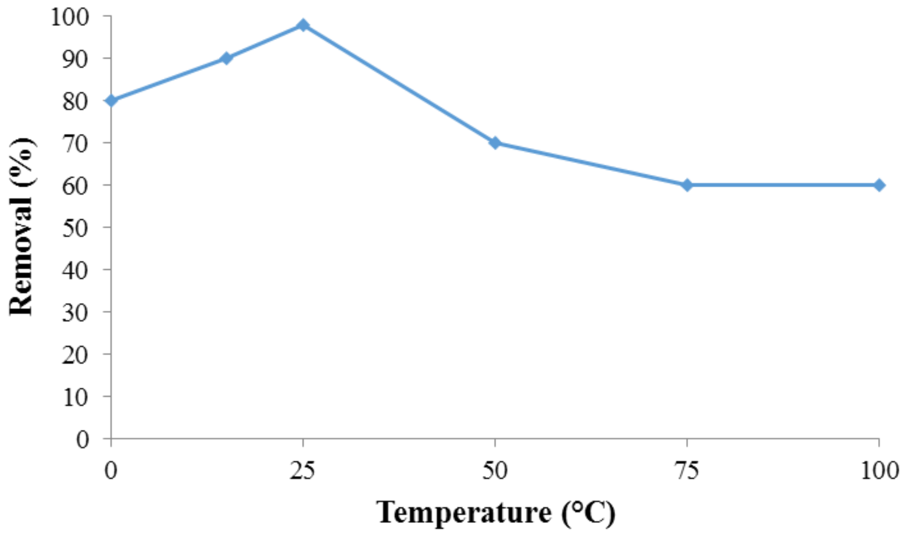

Figure (7): Investigation of the effect of temperature on removal

The amount of adsorption capacity and the percentage of Nano sorbent adsorption used

An isotherm contains information about the tendency of species molecules to adsorb on the surface, as well as a possible way of absorbing species molecules. To optimize the design of an adsorption system, it is important to have the highest correlation for the information of each system. The most common method to achieve the adsorption isotherm is to determine the concentration of the species in solution before and after the species adsorption process. Several surface adsorption isotherm equations are available. In this study, the Langmuir and Freundlich isotherms were investigated.

To obtain the adsorption capacity and adsorption percentage of Nano sorbents in the optimal conditions obtained at pH equal to 7, a time of 120 minutes and a weight of 0.1 g of adsorbent were used and the results are shown in Figure 8.

Using the Langmuir equation, K_L and q_m can be calculated from the slope and width from the origin of the linear curve c_e/q_e, respectively, in terms of c_e.

Based on this, the value of q_m equal to 23.31 mg g^{-}and the value of K_L equal to was 0.2104 L mg^{-1}. The line equation of this curve is as follows:

$$Y=0.0429\ X + 0.093 \qquad r = 0.9848$$

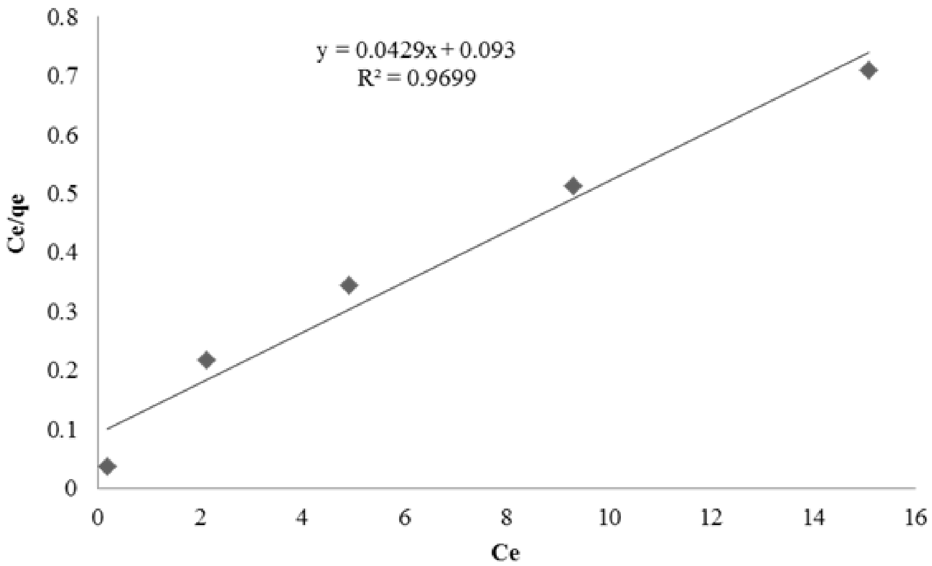

Figure 8: Langmuir curve

The Freundlich curve was also plotted (Figure 9), the equation of which is as follows:

$$Y=0.3311\ X + 0.925 \qquad r = 0.9952$$

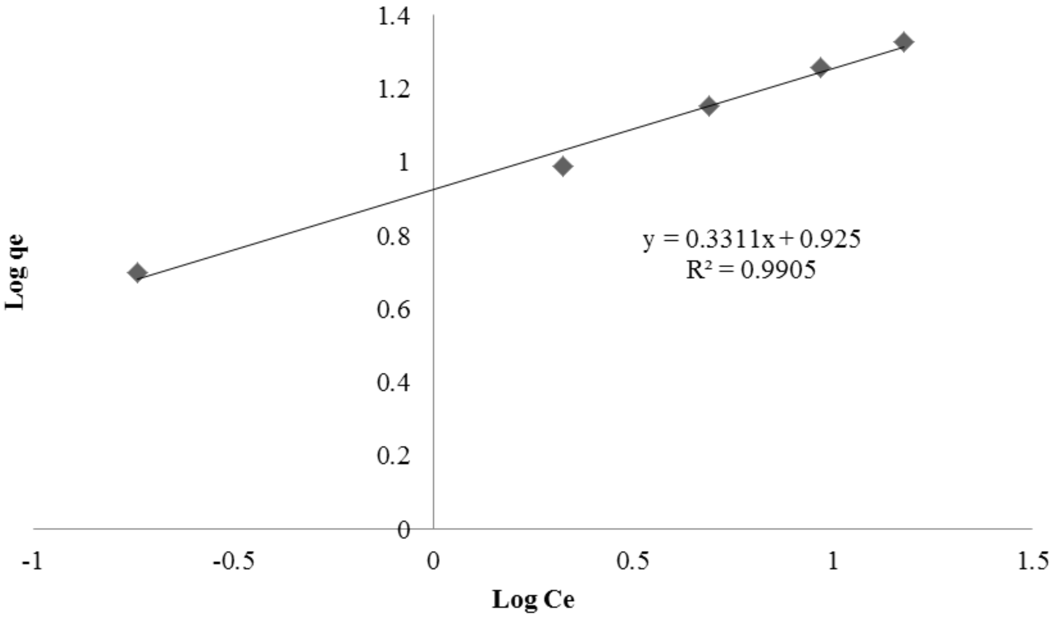

Figure 9: Freundlich curve

The smaller correlation coefficient (r) of the Langmuir isotherm than the Freundlich isotherm indicates that; this model has less correlation with results than the Freundlich model. Therefore, the adsorption of lead on $CoFe_2O_4$@PANI nanoparticles follows the Langmuir equation.

Table 1 Comparison of isothermal parameters for adsorption of metal ions on the studied adsorbent CoFe2O4 @ PANI Different concentrations of metal ions, contact time 120 minutes, adsorbent amount of one gram, rotation speed 250 rpm, temperature 25 ± 0.5.

Table 1: Comparison of isothermal parameters

Langmuir constants			**Freundlich constants**		
Qm (mg g^{-1})	**b (L mg^{-1})**	**R^2**	**k (mg g^{-1})**	**1/n**	**R^2**
23.31	0.2104	0.96	2.065	0.3311	0.9905

Kinetic studies of the adsorption process

There are several kinetic models for understanding adsorption behavior as well as for investigating the mechanism controlling the adsorption process. In this study, kinetic information was investigated using a quasi-first-order kinetic model and a quasi-second-order kinetic model.

For kinetic analysis in this study, data related to times of 30 to 270 minutes were used. It was found that the dye adsorption process on the adsorbent reaches equilibrium in 120 minutes. Then the $\log(q_e\text{-}q_t)$ curve in terms of t for the quasi-first-order equation (Figure 10) and also the t/q_t curve in terms of t for the second-order quasi-equation (Figure 11) for the lead adsorption process on the surface of $CoFe_2O_4$@PANI nanoparticles, for determination of kinetic parameters was investigated. According to the correlation coefficients of these two models, the lead adsorption process follows both quasi-first-order and quasi-second-order kinetic models; However, the quasi-first-order model is more consistent with the experimental results, which suggest a chemical adsorption mechanism.

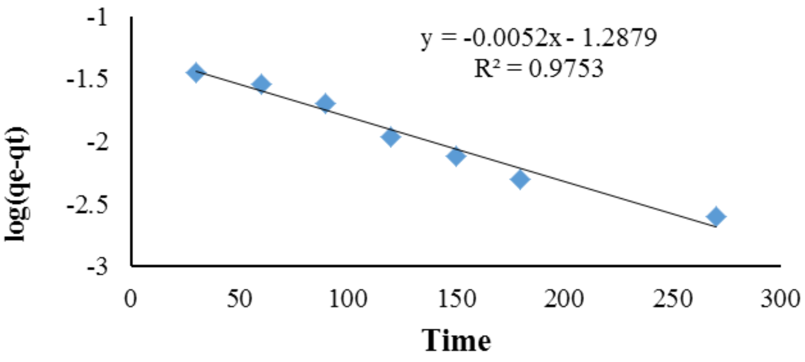

Figure 10: Investigation of quasi-first order kinetic model

The obtained line equations and correlation coefficients are as follows:

30 µg mL^{-1} → $\log(q_e\text{-}q_t) = -0.0052t - 1.2879$ r = 0.9875

Also, to investigate the quasi-second-order kinetic model, the t/q_t change graph was plotted in terms of t.

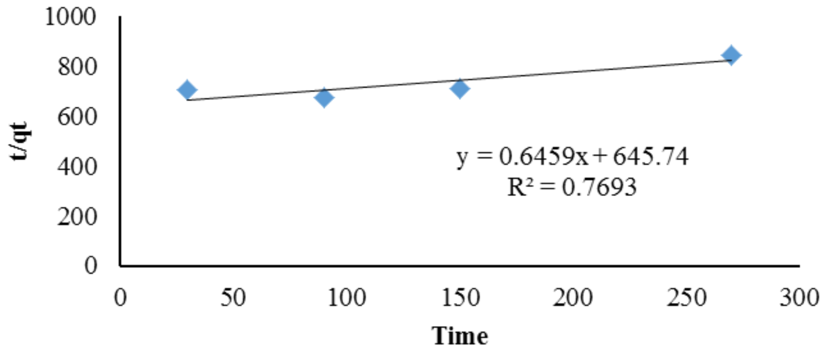

Figure 11: Investigation of a quasi-second order kinetic model

The obtained line equations and correlation coefficients are as follows:

30 µg mL^{-1} → $t/q_t = 0.6459t + 645.74$ r = 0.8770

IV. CONCLUSION

According to the studies, it can be concluded that; the remarkable structure and properties of polyaniline-coated cobalt ferrite Nanocomposite make it possible to use it as an adsorbent for the adsorption of heavy and toxic metals. Outer space and laminate of aniline polymer particles allow cobalt ferrite nanoparticles to be successfully placed in the space between its layers as a solid substrate, Due to its synergistic effect; it removes 98% of lead metal from water in 120 minutes and has a maximum lead absorption capacity of 23.31 mg g^{-1}. Also, pH changes showed that the adsorption process can show the highest adsorption in a neutral environment and ambient temperature, which indicates the ability and speed of using the adsorbent in general applications. Considering the simplicity of the preparation method, it can be concluded that; Application of cobalt ferrite Nanocomposite coated with polyaniline Increased the effective surface area of adsorption and the results obtained using the atomic absorption apparatus have also shown that; This adsorbent has a high adsorption capacity for adsorption of lead metal ions, which can be used in industry and to help prevent the entry of heavy and toxic metals into the environment. A study on the adsorption kinetics of lead showed that; it follows the quasi-first-order model, and among the Langmuir and Freundlich isotherm models, the laboratory results were consistent with the Freundlich isotherm model, and the maximum lead adsorption capacity was set at 23.31 mg g^{-1}.

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