

Heavy Metal Removal from Basrah waste water via Fe₂O₃ Nanocrystals

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Abstract: In this study, the co-precipitation method were used to synthesize α -Fe₂O₃ nanocrystals, that were then used as an adsorbent to remove Cu²⁺, Zn²⁺, Cd²⁺, and Pb²⁺ from wastewater at room temperature. The prepared sample's structural and morphological properties are assessed using XRD, BET surface area, and FESEM. The development of a pure hematite structure with an average particle size of about 48 nm is confirmed by the XRD patterns and is further corroborated by the 70 nm nanocrystals' FESEM pictures. The BET specific surface area of the nanocrystals is determined to be approximately 39.89 m² g⁻¹. Adsorption experiments are conducted for a range of solution pH values, contact times, and initial metal ion concentrations. The maximum adsorption capacities of Fe₂O₃ nanocrystals related to Cu²⁺, Zn²⁺, Cd²⁺, and Pb²⁺ are found to be 96.56 ppm for Pb²⁺ ions for a contact time 120 min. At pH 3, 6, and 9, high efficiency removal of Cu²⁺, Zn²⁺, Cd²⁺, and Pb²⁺ takes place for zinc ions of 99.79 ppm. These results clearly suggest that the synthesized Fe₂O₃ nanocrystals can be considered as potential nano-adsorbents for future environmental and health related applications.

Keywords: water treatment, heavy metal removing, Iron oxide nanoparticles; Fe₂O₃.

1. INTRODUCTION

The growing population and industrialization are closely linked to the health and environmental problems associated with water pollution. Because wastewater contaminates soil and water resources with heavy metals and other contaminants, it poses a serious risk to the environment [1]. Cadmium, lead, and copper are ranked in the top eleven hazardous pollutants [2]. Wastewater from the battery, steel, paint pigment, fuel, photo materials, engineering and manufacturing, and coating industries are the main sources of lead pollution [3,4]. The second most toxic metal is lead (Pb), which makes up 0.002% of the Earth's crust. Lead is found naturally in very small amounts, but most of the lead that is produced is used in human-made industries, cars, batteries, and other products, which pollutes the environment and affects people. The main routes for human contact with lead salts and oxides are dust, battery vents, lead paint, and contaminated food [5]. Paint pigments and Ni-Cd batteries both make extensive use of the heavy and toxic metal cadmium. It has been reported that cadmium is a potent carcinogen and hemopoietic [6]. Conventional environmental and biological processes do not effectively remove or reduce a large number of organic pollutants and heavy metals from wastewater. Common techniques for removing heavy metal ions from wastewater include adsorption, chemical precipitation, ion exchange, reverse osmosis, and electrochemical treatment. Copper can be found in nature as native copper, as well as in sulfide and oxide ores and salt minerals. It represents the second most of the non-ferrous metal that industry uses [7,8]. No review that covers the most recent reports on copper smelter-related air, soil, and water pollution has been published in the past ten years. Monitoring and testing the harmful effects of hazardous waste is still important because the quantity and variety of pollutants are too great for the environment to be able to clean itself [9]. Common techniques for removing heavy metal ions from wastewater include adsorption, chemical precipitation, ion exchange, reverse osmosis, and electrochemical treatment.[10]. Of these, adsorption is a popular

and reasonably priced method.9) Silica gel, activated alumina, molecular sieve zeolites, activated carbon, and polymeric adsorbents are commonly utilized adsorbents in this technique. These sorbents do, however, have a number of drawbacks, including their high cost and poor adsorption capacities [10]. Iron oxide nanoparticles with a large surface area have the highest number of reactive surface sites among the inorganic materials found in the environment, along with organic contaminants such as cations and anions[6]. Metal ions from solution can scatter onto the active sites of the adsorbent's surface due to iron oxide's small size[12]. Hematite, or α -Fe₂O₃, is thought to be the most environmentally safe and stable iron oxide.

In this work, the co-precipitation method were used to synthesize Fe₂O₃. Both chemical and physical methods were used to characterize the synthetic sample. As nano-adsorbents, the α -Fe₂O₃ nanocrystals were employed to extract the heavy metal ions Cu²⁺, Zn²⁺, Cd²⁺, and Pb²⁺ from water. We looked into the ideal pH, adsorption capacity, and equilibrium parameters. The present study's results unequivocally indicate that hematite nanocrystals can be an affordable and practical material for recovering heavy metal ions from wastewater.

Synthesis of Fe₂O₃ MNPs: Fe₂O₃ nanoparticles were synthesized by simple co- precipitation procedure as follows: solution A contains 10 g of FeCl₃.4H₂O was dissolved in 150 ml of pure water and left under stirred at room temperature for ten minutes, Solution B included ammonium hydroxide, this solution was added drop wise to solution A under continuous stirred for one hours and left at room temperature about thirty minutes , finally the brown powder precipitate was formed, the product was filtered, washed several times by deionized water and dried in oven at 90 °C for 6 hours

2. RESULTS AND DISCUSSIONS

2.1. Grain size analysis of Fe₃O₄ and Fe₂O₃ by Scanning electron microscopy(SEM).

The nonhomogeneous outer surface characteristics of agglomerated plates with a cubic-like shape as a result of particle magnetostatic interactions and humidity are depicted in the images of Fe₂O₃ Figure 1. In the SEM micrograph, numerous channels and grooves that provide a good adsorbent surface are visible. For Fe₂O₃ nanoparticles falling within the nanoscale range, the mean particle size within the range was calculated to be 71 nm. following the collection of grain diameter statistical data using the Image-J Log Normal function as the best fit. Tables 1 displays the results.

Table 1. Statistical results of particle size distribution for the Fe₂O₃ sample fitted with Log-Normal function.

Sample			Mean (nm)	Standard deviation	Minimum (nm)	Median (nm)	Maximum (nm)
Fe ₂ O ₃	70.958	4.195	61	71.4		81.2	

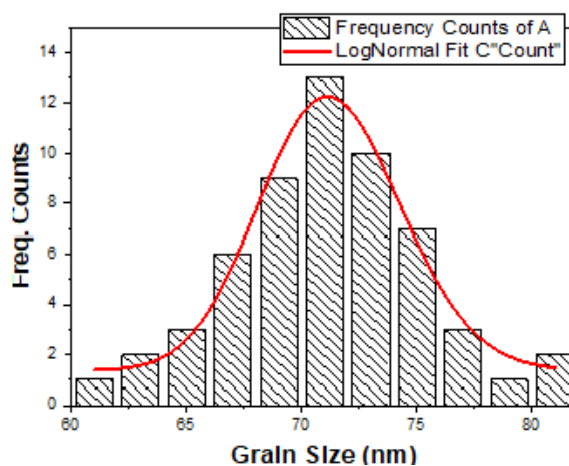
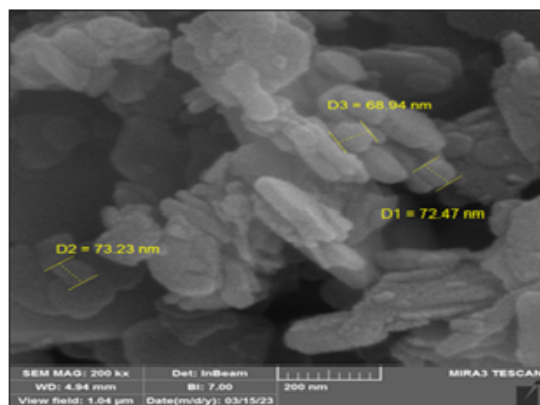


Figure 1. (Left Panel) The SEM Image and (right Panel) grain size analysis of Fe₂O₃

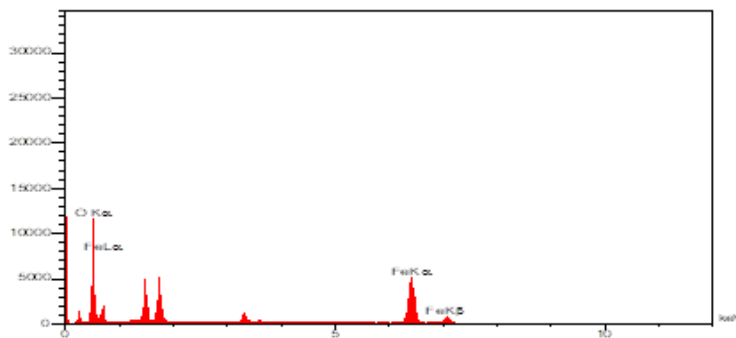


Figure 2. Energy dispersive X-ray analysis (EDX) spectrum of Fe₂O₃ nanoparticles

2.2. Energy dispersive X-ray analysis(EDX) for Fe₂O₃

Only the iron and oxygen ratio atoms, which indicate the prepared iron oxide's purity and are consistent with the prepared material's weigh ratio, were visible in the EDX result. The information was shown in Figure 2.

2.3 X-ray diffraction analysis for Fe₂O₃

Fe₂O₃ nanoparticles' XRD pattern revealed multiple peaks at the 2θ position (26.73°, 33.27°, 35.74°, 36.68°, 40.99°, 54.19°, and 62.52°), with corresponding FWHM values of approximately 0.1726, 0.3023, 0.5421, 0.1842, 0.1692, 0.3239, and 0.2835 2θ. The results are consistent with earlier research [13, 14], which validates the iron oxide Fe₂O₃ compound that was prepared. Using the Debye-Sherrer equation, the average crystal size was determined to be 48.17 nm. Fig. 3 displays the X-ray diffraction pattern.

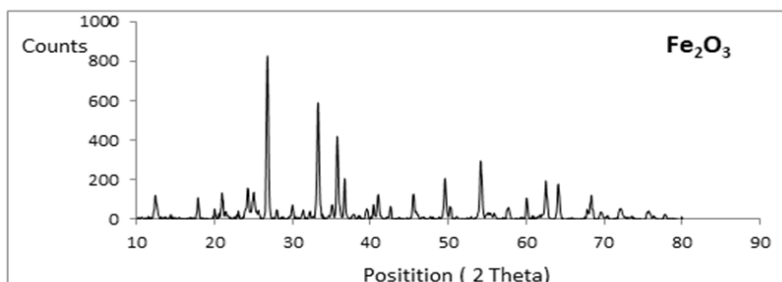


Figure 3. The X-ray diffraction pattern of Fe₂O₃ nanoparticles

2.4 Détermination of Zeta potentiel for Fe₂O₃

The aqueous solution of iron oxide (Fe₂O₃) shows instability and a negative surface charge. At -13.4 mV, the mean zeta potential was measured. Figure 4 displays the results of the zeta potential.

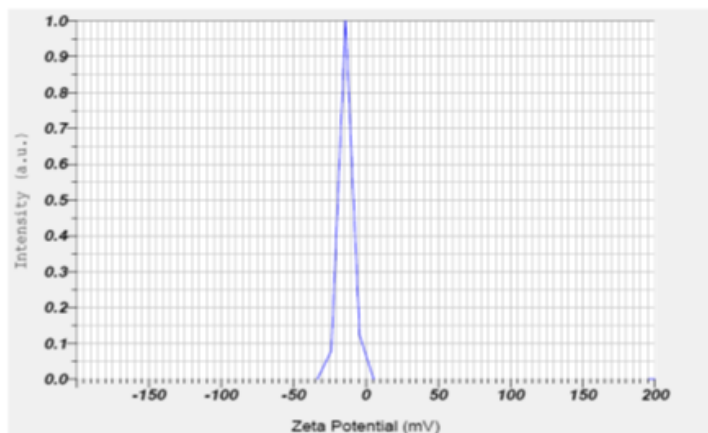


Figure 4. Zeta potential diagram of Fe₂O₃ nanoparticles

2.5 Surface area and porosity analyses

The functional groups and pore structures on an adsorbent surface essentially determine its adsorption efficiency [15]. However, there are three categories of structural pores: macro, mesoporous, and micro pores [16]. Many techniques, including Barrett Joyner Halenda (BJH), Brunauer Emmett Teller (BET), T-Plot, and Langmuir, are used to determine a material's surface value. These techniques involve the occupation of all active sites on the material's outer surface by inert gases, such as nitrogen, and a temperature of 77 K, at which the molecules of the inert gas are formed [16]. For adsorbent surfaces, the BET and BJH methods were utilized to determine the pore structure type and surface area. Fe₂O₃ nanoparticle results showed a surface area of 2.93 m²/g and an apparent pore diameter of 7.98 nm. Given its larger surface area compared to the other oxides, Fe₃O₄ might be a more effective adsorbent for eliminating heavy metal contamination from aqueous solutions. Although the iron oxide Fe₂O₃ had the lowest surface area value when compared to the other oxide adsorbents, the agglomeration process may occasionally be to blame for the surface area decline. When compared to iron and zinc oxide, titanium oxide also demonstrated a good surface area value; therefore, the chosen synthesis method was regarded to be a significant factor in the material's preparation. Table 2 presents the results of the mesoporous category, where the pore structures of all the adsorbents were found.

Table 2. Surface area and porosity analyses for nanoparticles

Nanoparticles	Surface area (m ² /g)	Pore structure type (nm)
Fe ₃ O ₄	35.89	Mesopores

2.6 Treatment processes

Toxic heavy metals, which are introduced into the environmental system through a variety of pathways and pose numerous risks to human health and ecology, particularly through the food chain, must be removed from an aqueous solution [17]. In the current study, we used simulated polluted water by using a water sampler instrument to collect three real samples from Shatt Al-Arab sub-channels, such as Al-Rebat, Al-Ashar, and Al-khora, at a depth of 15 cm on surface water. The samples that were gathered are regarded as waste water because a variety of contaminants, including home and industrial waste, are discharged into the channel without any kind of treatment. In order to achieve the highest removal ratio possible using four nano adsorbent surface Fe₂O₃ nanostructure, the samples were first contaminated by using stock solutions for four metals: lead, cadmium, copper, and zinc. The samples were then further contaminated by choosing the optimal conditions for contact time, temperature, and acidic function.

2.7 Selecting the affecting parameters on the adsorption efficiency

Numerous factors can influence the adsorption process and change the percentage of pollutants removed, either positively or negatively. This chapter examined the removal of various contaminants, including zinc, lead, cadmium, and copper, from their aqueous solutions by identifying the ideal parameters for contact time, temperature, and pH.

2.8 Effect of contact time

Under certain fixed parameters, such as concentration of approximately 10 ppm for each heavy metals solution, mixing speed of 120 rpm, volume of 10 ml of the heavy metals solution, and amount of 50 mg of nanoadsorbent under the solution's natural pH, the equilibrium time between the contaminated solution and the solid phase was studied for a duration ranging from 5min to 120 min. The following relation can be used to calculate the removal percentage of heavy metals from four nano adsorbent surfaces [18]:

$$\%R = [(C_0 - C_e) / C_0] \times 100$$

Where *C₀*, *C_e*, are in unit of mg/l, are the initial and residual concentrations of the heavy metals solution, respectively. Following the use of four elements—zinc, lead, cadmium, and copper—to contaminate three actual waste water samples, these elements were removed using four nanoadsorbent surfaces, such as iron oxides (Fe₂O₃). The findings indicate that, following the removal of four heavy metals from the zinc oxide surface, the contact time required to extract zinc ions from

the solution ranged from 15 to 30 minutes, with the AlKhora and AlAshar stations recording the shortest contact times. In contrast, the AlRebat station recorded the longest contact times, which could be explained by the presence of a naturally occurring active site on the zinc oxide surface and competition between zinc ions and other ions in the solution for occupancy of the vacant active site. Due to the obvious of salt content in this station solution, Al-Khora station showed a high removal percent of 88.3%, while Al-Rebat station showed a low removal percent of 25.2% [19]. Table 3 contains a list of the data.

In addition, a short time was recorded in the Al-Ashar station while a high removal percent was recorded in the Al-Rebat station. Iron oxide Fe₂O₃ nanoparticles were utilized as the adsorbent surface for the removal of four heavy metals. The adsorption ratio was calculated as a time function and it recorded a high ratio to remove zinc ions from aqueous solution at contact times of 30 and 60min. More so than the other stations, the Al-Rebat station has observed that lead ions have the shortest contact time and the highest removal ratio when iron oxide Fe₂O₃ nanoparticles are used as the adsorbent surface. For the purpose of adsorbing cadmium ions, a low removal percent onto Fe₂O₃ was observed.

Three polluted stations recorded the same contact time, with Al-Khora station recording a higher ratio than the other two stations. For the purpose of eliminating copper ions by iron oxide Fe₂O₃ surface, Al-Ashar and Al-Rebat stations recorded a short contact time of 30 minutes, whereas Al-Kkhora station recorded a long contact time. However, Al-Ashar station recorded a higher removal ratio than the other stations, at 92.79. Due to the presence of channels and grooves on the outside of Fe₂O₃, as well as a strong binding between the adsorbent surface and lead and copper ions, lead and copper ions were found to have the highest removal percentage onto Fe₂O₃. The contact time effect results for copper, zinc, lead, and cadmium that adsorbed onto the Fe₂O₃ surface are shown in Table 3.

Table 3. Effect of contact time for Removal of some heavy metals onto Fe₂O₃

Station	Contact time (min)	Zinc	Lead	Cadmium	Copper
		C _e ppm (%R)	C _e ppm (%R)	C _e ppm (%R)	C _e ppm (%R)
Al-Khora	5	2.3312 (76.68)	1.0547 (89.45)	9.4941 (5.05)	2.4586 (75.41)
	15	2.3124 (76.87)	0.9698 (90.30)	8.4859 (15.14)	2.2083 (77.91)
	30	2.2029 (77.97)	0.8677 (91.32)	7.7712 (22.28)	2.0941 (79.05)
	60	1.6425 (83.57)	0.8091 (91.90)	7.2015 (27.98)	1.6292 (83.70)
	120	1.3139 (86.86)	0.6511 (93.48)	7.1670 (28.33)	1.3277 (86.72)
Al-Ashar	5	1.4787 (85.21)	1.0309 (89.69)	9.0049 (9.95)	0.8918 (91.08)
	15	1.4278 (85.72)	0.7470 (92.53)	8.0315 (19.68)	0.8204 (91.79)
	30	1.3379 (86.62)	0.6875 (93.12)	7.7236 (22.76)	0.7204 (92.79)
	60	1.2126 (87.87)	0.6311 (93.68)	7.4196 (25.80)	0.6079 (93.92)
	120	1.1345	0.6311	7.3932	0.5548

		(88.65)	(93.68)	(26.06)	(94.45)
Al-Rebat	5	2.2981 (77.01)	0.4718 (95.28)	9.4491 (5.50)	3.8119 (61.88)
	15	2.1697 (78.30)	0.4014 (95.98)	9.0929 (9.07)	3.4191 (65.80)
	30	2.0427 (79.57)	0.3679 (96.32)	8.7977 (12.02)	3.0164 (69.83)
	60	1.1115 (88.88)	0.3581 (96.41)	8.5811 (14.18)	2.9945 (70.05)
	120	1.0352 (89.64)	0.3438 (96.56)	8.4833 (15.16)	2.9730 (70.27)

2.9 Effect of temperature

All stations recorded the closed values for the removal percentage of zinc ions from the ferric oxide surface; however, the percentage varied depending on the temperature, with Al-Rebat recording the highest percentage at 30 °C and Al-Ashar recording the second-highest percentage at 50 °C. An increase in the percentage of zinc ions removed from ferric oxide at a higher temperature could indicate the formation of chemical adsorption between the two. At the lowest temperature of 40 °C, Al-Rebat polluted station recorded the highest removal percent of lead ions using iron oxide nanoparticles, 97.06%. This was followed by Al-Ashar station at 40 °C, 94.74%, and Al-Khora station at 20 °C, 94.26%. An increase in the removal percentage could be caused by the salts in the collected waste water. The ions of cadmium and copper had the highest removal values in Al-Ashar station compared with all stations at low temperatures of 20 and 30 °C respectively, while Al-Rebat and Al-khora station recoded a low removal values.

For cadmium ions onto ferric oxide at 30 °C, compared to other ions. Additionally, the following is the order of the percentage of copper ions removed from all stations onto the ferric oxide surface: At low temperatures, Al-Ashar > Al-Khora > Al-Rebat, which could be caused by ion competition and the surface morphology of iron oxide. Table 4 lists every outcome of the removal percentage for copper, zinc, lead, and cadmium ions onto Fe₂O₃ nanoparticles.

Table 4. Effect of temperature for Removal of some heavy metals onto Fe₃O₄

Station	Temperature (°C)	Zinc	Lead	Cadmium	Copper
		C _e ppm (%R)	C _e ppm (%R)	C _e ppm (%R)	C _e ppm (%R)
Al-Khora	20	0.6 (94)	0.601368 (93.98)	0.912858 (90.87)	0.63756 (93.62)
	30	0.6 (94)	0.61146 (93.88)	0.91856 (90.81)	0.797274 (92.02)
	40	0.6 (94)	0.608397 (93.91)	1.164952 (88.35)	0.769503 (92.30)
	50	0.6 (94)	0.612312 (93.87)	1.065942 (89.34)	0.819336 (91.80)
Al-Ashar	20	0.6 (94)	0.604617 (93.95)	1.086479 (89.13)	0.657011 (93.42)
	30	0.6 (94)	0.60725 (93.92)	0.891647 (91.08)	0.709819 (92.90)

	40	0.6 (94)	0.604405 (93.95)	1.133094 (88.66)	0.700133 (92.99)
	50	0.6 (94)	0.607985 (93.92)	1.243881 (87.56)	0.741119 (92.58)
Al-Rebat	20	0.6 (94)	0.6 (94)	1.011232 (89.88)	0.662714 (93.37)
	30	0.6 (94)	0.604968 (93.95)	0.921504 (90.78)	0.674442 (93.25)
	40	0.6 (94)	0.603558 (93.96)	1.498761 (85.01)	0.761095 (92.38)
	50	0.6 (94)	0.604857 (93.95)	1.070433 (89.29)	0.702227 (92.97)

2.10 Effect of pH

One of the key elements influencing adsorption efficiency is the acidic function, which increases the removal of certain pollutants by causing a change in charge between the heavy metal ions and the outer surface of the adsorbent [20]. The zeta potential analyses of the four adsorbent surfaces used in this study indicate that they are all negatively charged, with the exception of the zinc oxide surface, which has a positively charged outer surface. The removal percentage was chosen using three solutions with varying pH values (3, 6, and 9).

At pH 9, the stations of Al-Rebat and Al-Khora recorded the highest values, 99.79 and 99.67%, respectively, for the removal of zinc ions from an aqueous solution using iron oxide Fe₂O₃ as an adsorbent surface. Although the pH levels at stations 3 and 6 were recorded, the removal percentage convergence across all stations fell between 82.05 and 86.16%.

The best adsorption value for removing lead ions onto Fe₂O₃ surface was found at pH level 6, and Al-Rebat had the highest removal percentage (100%)—possibly because there was less competition for lead ions' active sites on the outer adsorbent surface from salts.

The behavior of the Fe₂O₃ surface in eliminating cadmium ions is similar to that of the TiO₂ surface, where the removal percentage in the Al-Rebat station reached its highest value of 78.92% when the basic medium was increased. In addition, the ideal pH ranges for adsorbing copper ions increase as the hydrogen concentration levels decrease toward the basic medium, with Al-Rebat having the lowest hydrogen concentration level (98.43%) compared to the other stations. Table 5 lists every outcome of the removal percentage for copper, zinc, lead, and cadmium ions onto Fe₂O₃ nanoparticles.

Table 5. Effect of the pH level for Removal of some heavy metals onto Fe₂O₃

Station	pH	Zinc	Lead	Cadmium	Copper
		C _e ppm (%R)	C _e ppm (%R)	C _e ppm (%R)	C _e ppm (%R)
Al-Khora	3	1.754694 (82.45)	6.85342 (31.46)	2.368125 (76.31)	2.63263 (73.67)
	6	1.594886 (84.05)	0.534202 (94.65)	2.396226 (76.03)	2.40445 (75.95)
	9	0.032761 (99.67)	0 (100)	2.227619 (77.72)	0.173987 (98.26)
Al-Ashar	3	1.790651 (82.09)	6.495114 (35.04)	2.360096 (76.39)	6.078152 (39.21)

	6	1.518977 (84.81)	0.143322 (98.56)	2.340024 (76.59)	2.296064 (77.03)
	9	0.524171 (94.75)	0 (100)	2.275793 (77.24)	0.362236 (96.37)
Al-Rebat	3	1.746704 (82.53)	7.407166 (25.92)	2.380169 (76.19)	2.621221 (73.78)
	6	1.38314 (86.16)	0 (100)	2.327981 (76.72)	2.102111 (78.97)
	9	0.020775 (99.79)	0 (100)	2.107186 (78.92)	0.156874 (98.43)

3. CONCLUSIONS

In conclusion, Fe₂O₃ nanocrystals were successfully prepared using a straightforward co-precipitation technique. Heavy metal removal from wastewater was tested using the synthetic sample. The adsorption activities of Fe₂O₃ nanocrystals towards heavy metal ions were investigated under various experimental conditions. In the process of heavy metal ion adsorption, pH was crucial. Cu²⁺, Zn²⁺, Cd²⁺, and Pb²⁺ ion removal were studied different temperature, contact time, and at pH 3, 6, and 9. Adsorption is a physico-chemical process that significantly influences the electrostatic attractions between heavy metal ions and Fe₂O₃ nanocrystals. The findings demonstrate that as contact time and initial metal ion concentration increased, so did the capacity of heavy metal ions. The monolayer adsorption of the metal ions under study onto the surface of Fe₂O₃ nanocrystals was confirmed by the higher linear regression coefficient values of the Langmuir adsorption isotherm model, which fit the data better than the Freundlich model. The Cu²⁺, Zn²⁺, Cd²⁺, and Pb²⁺ ions showed Langmuir reasonable adsorption capacities. As demonstrated by the current study's results, Fe₂O₃ nanocrystals are effective adsorbents for removing heavy metal ions from wastewater.

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