



REMOVAL OF HEAVY COBALT ELEMENT USING IRON OXIDE NANOPARTICLES

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Abstract

In this study, nanomaterials, iron oxide nanoparticles (Fe₃O₄), iron oxide nanocoated with citric acid (CA MNP) and iron oxide inlaid with wheat shells (WH MNP) were prepared and used under conditions such as pH, equilibrium time) and were studied on heavy cobalt as a pollutant, the nanomaterial was diagnosed by XRD analysis and FTIR infrared spectroscopy. Where the results of the removal Fe₃O₄ at the equilibrium time in the 10th minute and by 97.91%, while in CA MNP in the 10th minute, it gave a removal rate of 65.52%, while at WH-MNP in the 10th minute, it gave a removal rate of 95%. As for the results of the pH function Fe₃O₄ at PH 10 with a removal rate of 70.85%, either in CA-MNP at PH 8 with a removal rate of 95.97%, and in WH-MNP at PH 2 with a removal rate of 74.54%. The results of the concentration were in Fe₃O₄ at a concentration of 30 with a removal rate of 95.6%, while CA-MNP at a concentration of 30 with a removal rate of 77.62%, and in WH-MNP at a concentration of 30 with a removal rate of 96.6%. In XRD analysis, the grain size of Fe₃O₄ was calculated at (15.29) nm, CA-MNP was nm (15.78) and WH-MNP was about nm (17.14). The FTIR analysis of Fe₃O₄ shows the appearance of beams of the O-H and HOH group, while CA-MNP indicates that citric acid envelops the manganite surface by chemical adsorption of carboxylate strite ions in the acid, while WH-MNP is the hydroxyl group that indicates the vibration of C=O.

Keywords: Iron Oxide, Heavy Cobalt, Nanoparticles.

INTRODUCTION

Pollution is the introduction of pollutants into the natural environment in which living organisms live, which damages them and is under the influence of an external or internal influence, which causes a change in the ecosystem, and these pollutants are either natural materials but exceeded the permissible limit, or materials foreign to the environment, and this change is either chemical, physical or biological, and therefore pollution can affect the air, water and soil (Shasha *et al.*, 2020). Heavy metals have negative effects on living organisms in general, heavy metals accumulate in the environment due to direct and indirect human activities The reason is the continued presence of heavy metals in the water because they are not biodegradable and toxic, and on the other hand heavy metals become toxic when they are not metabolized inside the body and accumulate in soft tissues that may be released to the body through water, air, food or absorption through the skin on contact (Selvasembian *et al.*, 2022) such as the element cobalt, although it has a wide range of industrial and technological uses, but it can be harmful to health and the environment, it can cause poisoning if inhaled or swallowed in large quantities and can cause fever and diarrhea, as well as health problems such as respiratory infection and pulmonary fibrosis (Eltit *et al.*, 2019) in addition to accumulating in soil and groundwater, It can cause environmental pollution, affecting wildlife and plants. Recently, many treatment methods have appeared in removing or reducing their danger in the environment, including techniques that depend on physical, biological and chemical

treatments, and these methods differ in their efficiency to remove pollutants, and one of these modern methods is treatment with magnetic nanomaterials, one of the most important features of nanotechnology is that it has a high magnetism if the solutions are separated using an external magnetic field without thermal energy or chemical addition and therefore can be reused again, which leads to a reduction Total cost, surface area to volume ratio is high, low toxicity and finally chemical stability and biological suitability Nowicka, B. (2022)

MATERIALS AND METHODS

Synthesis of Magnetic Nanoparticles (Fe₃O₄)

1. The chemical sedimentation method was used by preparing 1:2 of FeCl₃ and FeSO₄
2. Then add enough NH₄OH solution and stir for 30 minutes
3. Magnetic separation was used to filter Fe₃O₄ and wash it three times with distilled water (Osial *et al.*, 2018)

Preparation of biochar (spelt husks)Biochar

1. The wheat husks were collected from different farms, then washed and dried in the oven at a temperature of 105 ° C for 4 hours.
2. The dried wheat husks were ground using a grinder at a rate of (3000 rpm) for 10 minutes, then the ground wheat was sieved using a sieve (0.5) mm and then collected the part under the sieve.

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3. After that, the dried parts were washed with distilled water and dried again in an oven at a temperature of 500°C in the absence of oxygen for 6 hours.
4. Charcoal is prepared using the oven until the wheat husks turn gray.
5. The nanomaterial coated with spelt husks is then prepared by mixing (2)g of charcoal with (0.5)g of nanomaterial, in a baker containing (500)ml of deionized distilled water for 4 hours at room temperature.
6. The prepared substance is dried on filter paper and then washed 5 times with deionized distilled water.
7. Finally, the prepared material is dried in the oven at a temperature of 80°C for 6 hours.

Preparation of iron oxide nanoparticles coated with citric acid

1. Take (gm4) of iron prepared Fe₃O₄ and then add (ml200) distilled water and then exposed to ultrasound for the purpose of dispersing the precipitate for a period of (min15), first solution.
2. Take (gm5) from the surface of citric acid CA and dissolve it in (ml10) of deionized distilled water, a second solution.
3. Add the second solution to the first solution with continuous stirring at a temperature of (95°C) and continue for an hour and a half and then dry in the oven

Adsorption for heavy elements

Magnetic iron nanooxide, magnetic iron nanooxide coated with citric acid, and coated with natural material (wheat husks) were used as a surface to remove the cobalt element. Where the amount of removal for each of the above adsorbent materials was studied, as well as other factors affecting the removal process.

Preparation of the concentrations of the cobalt element

Standard solutions of cobalt ion at a concentration of (1000 ppm) were prepared by dissolving (1)g of metal salt in (1000) ml of deionized distilled water. Several concentrations of cobalt ion were prepared from the standard solution (10,20,30 ppm) and the prepared solutions were left in the water bath at 20°C for thermal equilibrium.

Determine the equilibrium time for cobalt

To determine the equilibrium time of the adsorption of the cobalt element ion on the nano-adsorbent surface consisting of iron oxide nanoparticles and coated with citric acid and spelt husks, the concentration of the element ion was chosen, which is (30)ppm and placed in a volumetric vial of (30) ml containing (0.1) gm of the adsorption surface, the volumetric vial was placed in a temperature-controlled vibrator that vibrates at a speed of 100 revolutions per minute, then the concentration of the remaining ion in the solution was found at different times by the atomic spectrometry and these times are (10,20,30,40,50,60 min), after which The amount of cobalt ion at equilibrium Q_e in units (mg/g) is calculated and according to the relation

$$Q_e = \frac{V_{sol}(C_i - C_f)}{m} \dots\dots\dots(1)$$

Effect of PH on the adsorption process of the cobalt element

The effect of the pH function of the solution on the adsorption of the cobalt element on a surface of iron oxide nanoparticles coated with the two surfaces (citric acid and spelt husks) was studied using the solution of the element in different acid media by adding drops of concentrated sulfuric acid, and the pH of the system was measured by pH-meter measuring device. The values (pH2,4,6,8,10) were taken to determine the effect of the value of the pH function at which the amount of adsorption is at the highest efficiency. Where the equilibrium time that was previously calculated was fixed, and then the solution was measured with an atomic spectrometer to find out the concentration of the element after treatment, and the amount of removal was calculated according to the previous relationship (1).

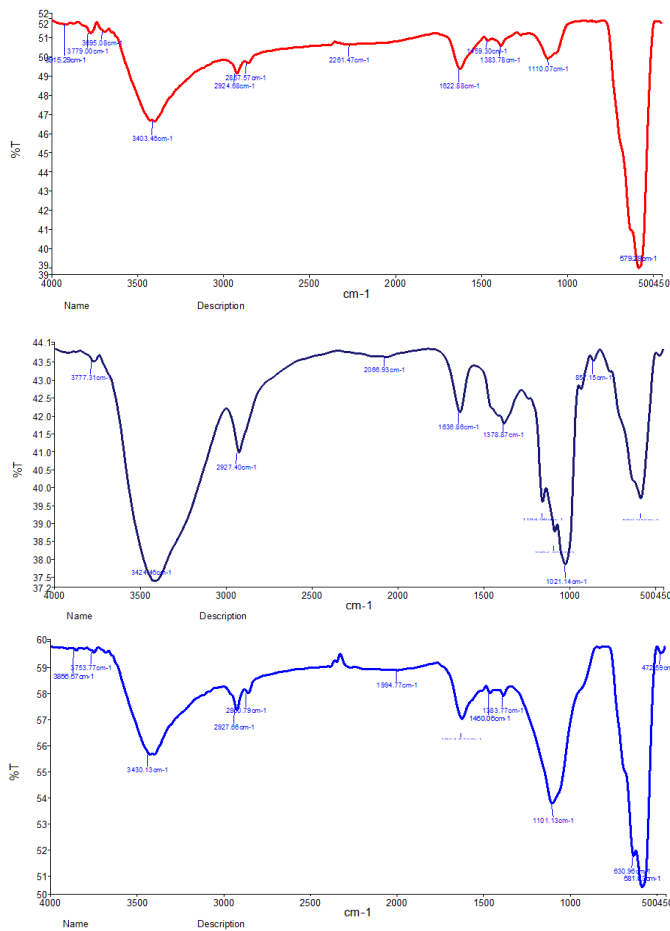
RESULTS AND DISCUSSION

Fourier Transform Infrared Analysis (FTIR)

Using FTIR spectroscopy technology to diagnose functional aggregates effective in prepared material structures, FTIR provides quantitative and qualitative analysis of organic and inorganic samples. FTIR technology is a distinctive molecular fingerprint that can be used to examine and scan samples for many different components. FTIR was diagnosed for iron oxide nanoparticles Fe₃O₄ MNP, iron oxide nanocoated with citric acid CA-MNP and iron oxide nanografted with wheat shells B.ch-MNP, as the results showed the use of:

Fe₃O₄ MNP Emergence Bundles (3915 cm⁻¹) (cm-13779) belonging to O-H alcohol groups, packages (cm-1 3695) (cm-1 3403) belonging to Aromatic active groups C-H and also to alcohol active groups O-H, packs (cm-1 2924) (cm-1 2857) return to Aldehyde groups O-H, package (cm-1 2261) return to Nitrile active groups C,N, package (cm-1 1622) return to hydrocarbon active groups C=C, package (1459 cm⁻¹) return to carboxyl acid groups O-H and package (1383 cm⁻¹) return to the homologous CO in the carboxyl group, either (cm-1 1101) due to the extension of the bond C-Fe and the bundle (cm-1 579) which belongs to the active groups C-CL hydrochloric acid These groups contribute to increasing the adsorption process of contaminants heavy element cobalt and these results are similar to what we found in a previous study in the use of iron oxide nanocoated with citric acid (Taher A. 2019). As for Figure (B-6), which represents the AC-MNP infrared spectrum, which indicates that citric acid envelops the manganite surface by means of chemical adsorption of strete carboxylate ions in acid (Zhao S., *et al.*, 2020) as the acid carboxylate group is complex with iron atoms on the surface of manganite, we notice from Figure (A-6) the appearance of beams (3777 cm⁻¹) that belong to the O-H alcohol groups (3424 cm⁻¹) they are beams belonging to the O-H bond and the bundle (cm-1 2927) belonging to aldehyde groups C-H Bundles (cm-1 2066) belong to the groups of isothosyanate N=C=S-, which is a medium bundle, a bundle (1636 cm⁻¹) belonging to the amine groups consisting of a double bond of carbon and nitrogen, bundles (1378 cm⁻¹) belonging to nitro groups NO₂, beams (cm-1 1154) belong to the C-Fe bond, bundles (cm-1 1082) (cm-1 1021) belong to the active aggregates sulfoxide S=O, packages (857 cm⁻¹) return to the active aggregates N-H and (cm-1 580) C-CL bundles return to

the active aggregates Hydrochloric acid as these aggregates contribute to an increase in the process of adsorption of contaminants heavy element cobalt and these results are close to what we found (TP R., & Philip J., 2022). In Figure C-6, which represents the infrared spectrum B.ch-MNP, we can see that the packages (cm-1 3856) (cm-1 3753) belonging to the alcohol groups O-H (3430 cm-1) belonging to the aromatic groups C-H, the packages (cm-1 2927) (cm-1 2860) belonging to the Aldehyde groups C-H, the bundles (cm-1 1994) belonging to the acid anhydride groups C=O, the package (1623 cm-1) belonging to the active groups of aromatic C=C, and the package (cm-1 1460) belonging to the active groups amides. C=O and bundle (cm-1 1383) belongs to nitro-NO2 groups, bundle (1101) belongs to the extension of the C-Fe bond, bundle (cm-1 630) (cm-1 581) belongs to the active groups alkyl halides, and package (cm-1 472) belongs to the active groups hydrochloric acid as these groups contribute to increasing the process of adsorption of contaminants heavy element cobalt



FTIR examination of prepared materials
A: MNP(Fe₃O₄) B: AC-MNP C: WH-MNP.

X-ray Diffraction (XRD)

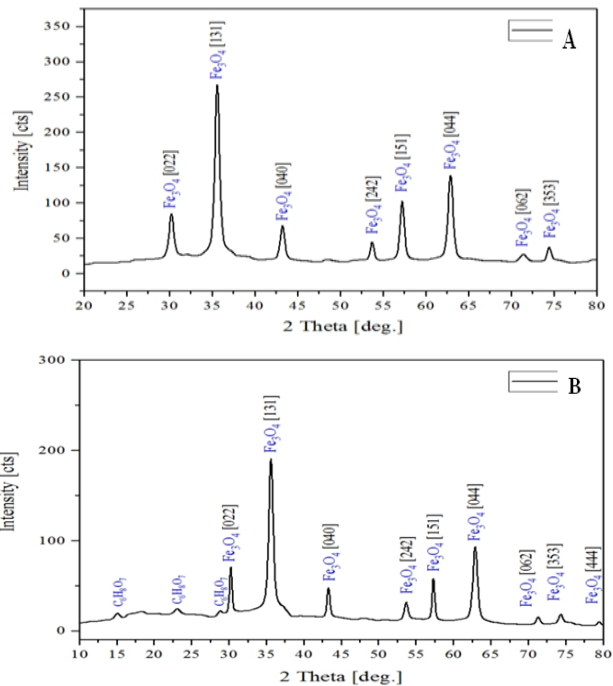
X-ray diffraction (XRD) is a common technique for studying the crystal structure, crystal size, crystalline characteristic, atomic divergence and study of X-ray diffraction of organic and inorganic crystalline samples. It has been used to study different aspects of materials at the nanometer scale, as X-ray wavelengths are short enough to provide high accuracy on small atomic scales (Sonawane *et al.*, 2016). X-ray diffraction was studied using an XRD type device (shimadzu 6000). Using a monochromatic beam of copper spectrum with a wavelength (nm 0.154606) where the granular size of the prepared

materials was estimated according to the equation (Scherrer–Debye).

$$D = \frac{d \cos\theta}{(0.9 \times \Delta)} \dots\dots\dots (1)$$

$$\Delta = 0.154606 \text{ nm} \times 0.9 = 0.1391454 \text{ \AA}^0$$

where θ : is the diffraction angle of the rays, d : the mid-peak width is estimated in (rad), λ : the wavelength of the X-rays, D : the diameter of the nanoparticles, Figure 3 represents the X-ray diffraction spectrum resulting from the examination of the prepared materials, where the results of the examination showed that the prepared magnetic material MNP(Fe₃O₄) showed diffraction with diagnostic peaks at atomic levels, which gave eight peaks at angles ($2\theta = 30.19^\circ, 35.55^\circ, 43.18^\circ, 53.67^\circ, 57.18^\circ, 62.83^\circ, 71.33^\circ, 74.37^\circ$) which corresponds to the standard card (JCPDS card (01-0880315 (Hariani P., *et al.*, 2013) at crystalline levels (022, 131, 040, 242, 151, 044, 062, 353) and when calculating the size of the granules using the Scherrer–Debye relationship it was within (15.29 nm). This corresponds to the value of the researcher (Husain S., *et al.*, 2019) who obtained a granular size of the prepared substance within (15nm). The diagnostic peaks resulting from the examination of the magnetic material coated with citric acid CA-MNP, which gave nine diagnostic peaks at angles ($2\theta = 14.53^\circ, 24.03^\circ, 29.04^\circ, 30.21^\circ, 35.56^\circ, 43.24^\circ, 53.61^\circ, 57.25^\circ, 62.82^\circ$) at the crystalline levels (022, 131, 040, 242, 151, 044, 062, 353, 444) respectively, and when calculating the size of the granules using the Scherrer–Debye relationship, it was within (nm). 15.78 This corresponds to the value of the researcher (Wei Y., *at el.*, 2012) who obtained a granular size of the prepared substance up to (15nm). The test result of wheat husks inlaid with WH-MNP showed sixteen diagnostic peaks at angles ($2\theta = 24.79^\circ, 28.55^\circ, 30.21^\circ, 35.67^\circ, 37.28^\circ, 41.40^\circ, 42.59^\circ, 43.35^\circ, 46.45^\circ, 48.76^\circ, 52.00^\circ, 53.71^\circ, 57.32^\circ, 62.87^\circ, 67.30^\circ, 70.66^\circ$). At the crystalline levels (011, 200, 022, 131, 222, 220, 021, 040, 112, 311, 222, 242, 151, 044, 244, 062) respectively, and when calculating the size of the granules using the (Scherrer–Debye relationship) it was about (17.14 nm) and this corresponds to the value of the researcher (Nasser A., 2023) who obtained a granular size of the prepared substance by (17.9nm).



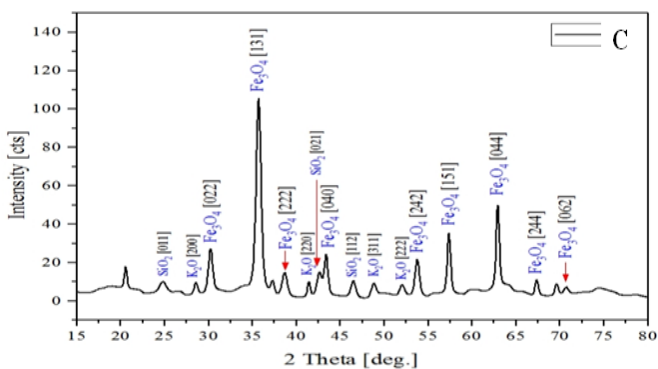


Figure 1: XRD X-ray diffraction of prepared materials.
A: MNP(Fe_3O_4) B: AC-MNP C: WH-MNP

Adsorption for heavy elements

Effect of equilibrium time

Table (8) shows the effect of shaking time affecting the adsorption process using the prepared materials adsorption materials for this element, where the following variables were fixed, namely the concentration at (30) ppm, the dose at (0.1) g, the pH function at pH = 4, and the temperature at (25) $^{\circ}\text{C}$ and different time intervals were determined (10,20,30,40,50,60) min

It was noted from Table (8) that iron oxide nanooxide A:MNP(Fe_3O_4) that the equilibrium time is min(30), which represents the appropriate time for the adsorption process. Where the highest adsorption rate was recorded at the time of min (10), where the removal rate is 97.91%, as for iron oxide coated with citric acid B:CA-MNP that the equilibrium time occurred in min(30) and continued in the same pattern until the end of the period and therefore this time is considered the right time for adsorption, as well as the table shows that the highest removal rate at min(10) with a removal rate of 65.52%, and it begins to decrease as the time increases. These results can be explained due to the arrival of the equilibrium state between the element and adsorbent materials quickly, in addition to the possibility of a state of internal agglomeration and a decrease in the number of pores in adsorption materials when time increases. Where it was noted that the adsorption is fast at the beginning, due to the availability of effective sites on the surface that are not occupied.

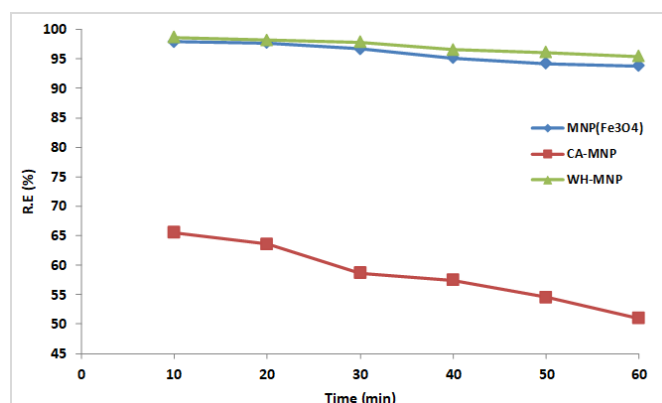
The adsorption is then slowed down until it reaches equilibrium due to the saturation of these active sites that do not allow further adsorption to occur (Zhu M., *et al.*, 2007).

While in iron oxide nanoparticles inlaid with wheat husks WH-MNP:C, the results in Table (8) showed that the appropriate equilibrium time for the adsorption process occurred at min(30), the highest adsorption rate was recorded at min(10) with a removal rate of more than 98.63%. The use of iron oxide nanoparticles coated with natural material has proven efficient in adsorption of the heavy element from the aqueous medium and is considered the best of all adsorptions. As the formation of an outer layer of natural material contributed to the binding of the particles of the heavy element in an ideal manner and with the availability of the appropriate surface area, all contributed to obtaining an ideal adsorption process, where determining the equilibrium time helps determine the time necessary to allow chemical reactions to occur sufficiently to remove contaminants and aims to achieve the

maximum effectiveness of removing contaminants and avoiding excessive consumption of nanomaterials (Bhateria and Singh, 2019). Equilibrium time when using: Iron Oxide Nano-MNP(Fe_3O_4) Citric Acid-Coated Iron Oxide Nano-Oxide (CA-MNP) Iron Oxide Nano-Grafted with Spelt Husks – (WH-MNP) for Adsorption of Cobalt

Effect of equilibrium time on removal rate (%)R.E of Cobalt (Co) using prepared materials WH-MNP, CA-MNP and mnp(Fe_3O_4)

Material used	Time	C _i Ppm	C _f Ppm	Q _e mg/g	RE %
A: MNP(Fe_3O_4)	10	30	0.62	2.938	97.91
	20	30	0.67	2.933	97.74
	30	30	0.996	2.9004	96.68
	40	30	1.455	2.8544	95.147
	50	30	1.739	2.8261	94.2
	60	30	1.852	2.8148	93.82
B: CA-MNP	10	30	10.34	1.966	65.52
	20	30	10.93	1.907	63.53
	30	30	12.4	1.76	58.66
	40	30	12.77	1.723	57.43
	50	30	13.63	1.637	54.54
	60	30	14.71	1.529	50.94
C: WH-MNP	10	30	0.4	2.96	98.63
	20	30	0.54	2.946	98.17
	30	30	0.65	2.935	97.83
	40	30	1.03	2.897	96.56
	50	30	1.18	2.882	96.06
	60	30	1.36	2.864	95.45



Effect of the pH function on the adsorption process

Table (9) shows the effect of the acid function affecting the adsorption process using the materials prepared as adsorption materials for this element, where the following variables were fixed, namely the equilibrium time is (30) min at MNP(Fe_3O_4), AC-MNP, WH-MNP, concentration at (30) ppm, dose at (0.1) g and temperature at (25) $^{\circ}\text{C}$, and different acid functions (2,4,6,8,10) pH were determined.

It was observed through Table (9) for iron oxide nanooxide A:MNP(Fe_3O_4), where the highest adsorption rate was recorded at pH = 10, where the removal rate is 70.85%, where the results showed that the adsorption process increases when the pH increases, i.e. in the base media, and this indicates an increase in the difference in charges between the adsorbent and the adsorbed, which in turn is the attraction of the cutter and static with the forces of Vanderfals (Usefi F). 2016). At iron oxide nanoparticles coated with citric acid CA-MNP:A, Table (9) shows the highest adsorption rate at pH = 8 with a removal rate of 95.97%, as the increase in adsorption to the base medium is due to the increase in the number of negative ions

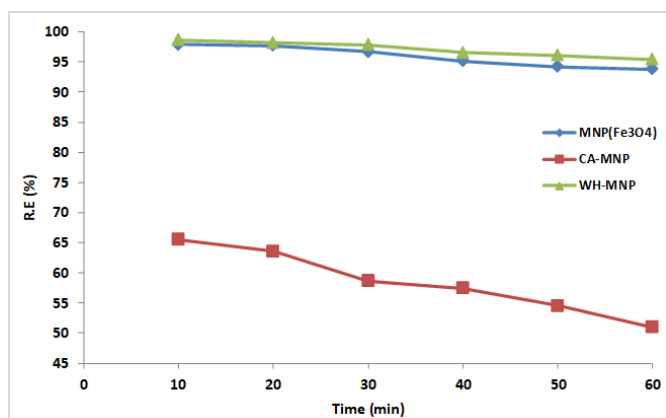
that are directed to bind to the adsorption sites on the positively charged surface and the occurrence of electrostatic attraction between them (Usefi F., 2016). While when using iron oxide coated with wheat husks WH-MNP:C

Table (9) showed that the highest removal rate was recorded at pH = 2 with a removal rate of 74.54%, as natural materials are often able to attract acid deposits due to their content of basic compounds that react with acids. In addition, they contain organic substances such as amines that contain acid-reactive groups (Nasser A., 2023).

Palh function when using: Iron Oxide Nano-MNP(Fe₃O₄) Iron Oxide Nanoparticles Coated with Citric Acid (CA-MNP). Iron Oxide Nano-Inlaid with Wheat Husks – (WH-MNP for Cobalt Adsorption

Effect of pH on the Removal Rate (%)R.E of Cobalt (Co) Using Prepared Substances WH-MNP, CA-MNP and mnp(Fe₃O₄)

Materials used	Ph	C° ppm	Cf ppm	Qe mg/g	RE %
A: MNP(Fe ₃ O ₄)	2	30	16.72	1.328	44.26
	4	30	15.001	1.499	49.99
	6	30	12.77	1.723	57.403
	8	30	9.42	2.058	68.59
B: CA-MNP	2	30	8.81	2.119	70.6
	4	30	6.53	2.347	78.21
	6	30	3.44	2.656	88.53
	8	30	1.2	2.88	95.97
C: WH-MNP	2	30	1.86	2.814	93.79
	4	30	7.63	2.237	74.54
	6	30	9.52	2.048	68.24
	8	30	9.63	2.037	67.9
	8	30	10.11	1.989	66.28
	10	30	13.38	1.662	55.37



Conclusion

Iron oxide nanoparticles were prepared by chemical deposition method, and then a compound of citric acid and a natural substance were prepared from spelt shells. The models were diagnosed with FTIR and XRD techniques, When FTIR analysis of Fe₃O₄, we notice the appearance of beams of the O-H and HOH group, while CA-MNP indicates that citric acid envelops the manganite surface by chemical adsorption of strate carboxylate ions in the acid, while WH-MNP is the hydroxyl group that indicates the vibration of C=O. Iron oxide nanooxide was prepared by chemical deposition method, then a compound of citric acid and a natural substance were prepared from wheat husks. The models were diagnosed with X-ray diffraction and infrared spectroscopy. The physical

properties of the prepared nanomaterials and composites have been studied and include magnetic properties and surface area. The surfaces prepared in this study were used as adsorbent surfaces to remove inorganic contaminants represented by the element cobalt (Co) under different conditions of acidity function and time, which include iron oxide nanoparticles and iron oxide nanoparticles coated with citric acid and ferrous oxide nanoparticles inlaid with spelt husks. The efficiency of natural materials in removing pigments was good

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