

RESEARCH PAPER

Assessment of Green-Synthesized Fe₃O₄ Nanoparticles for Wastewater Treatment

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ABSTRACT

Wastewater treatments, appear ineffective due to the increasing pollutants of many types of pollutions. Therefore, the need for environmentally friendly and effective treatment approaches has increased. Green manufacturing methods for nanomaterials have been used, which is an inexpensive and environmentally friendly method for manufacturing nanoparticles. Using cinnamon extract as a natural reducing agent, iron oxide nanoparticles (C-Fe₃O₄) were synthesized. The experiment was conducted in October 2024 to February 2025 in the Ecological Sciences Laboratories - College of Science - University of Kufa. UV-Vis spectroscopy, X-ray diffraction (XRD), and field-emission scanning electron microscopy (FE-SEM) techniques characterized the synthesized nanoparticles. The mean diameter of the spherical C-Fe₃O₄ iron oxide nanoparticles was 16.5 nm. XRD confirmed their crystalline nature, and UV-Vis spectroscopy revealed a pronounced absorption at 280 nm. The results of the C-Fe₃O₄, plant extract, and the mixture between the C-Fe₃O₄ and the extract in a ratio of 1:1, in the variables (T) temperature, pH, conductivity (EC), total dissolved solid (TDS), and total organic carbon (TOC) at different times (one hour, 12 hours, and 24 hours) were revealed. hour) at different concentrations of 0.5 mg/L and 1 mg/L. It was found that the best removal percentage was in the C-Fe₃O₄, which was (10.45%, 76.43%, 77.9%, and 95.8%) for the variables (pH, EC, TDS, and TOC), respectively, after 24 hours at a concentration of one mg/L. The conclude that it is possible to treat wastewater by manufacturing an easy-to-prepare and low-cost nanomaterial.

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INTRODUCTION

Wastewater is increasing at an alarming rate, due to various reasons, the most important of which is the discharge of waste directly into water sources, and household, mechanical and agricultural waste directly into water bodies. Most of these wastes

are rich in phosphorus and carbon, which causes a phenomenon known as over-nutrients. Over-nutrients occur due to the excessive proximity of nutrients within the aquatic body, which helps in the spread of the eutrophication phenomenon [1-3]. Removal of major toxins such as suspended

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solids, bio-oxygenase depletion (BOD), additives (organic and inorganic) and coliforms is the most objective goal of wastewater purification. This requires more advanced treatment technologies because water pollutants have increased, water resources have decreased in the world and the need for water has increased [4]. Various adsorption mechanisms such as electrostatic interaction, surface complexation, and ion exchange govern pollutant removal using Fe_3O_4 nanoparticles [5]. Green-synthesized magnetic nanoparticles have demonstrated efficient removal of heavy metals and organic contaminants [6,7]. Oil, mill water, hospital wastewater and household sewage are all pollutants that have been shown to be able to be removed using nano-treatment [8]. Nanomaterials have proven their ability to break down fats, remove dyes and precipitate suspended particles due to their high reactivity and unique properties [9]. The working principle of nanocoagulants is to form large aggregates (clusters) of small particles. These particles are often removed by simple filtration or sedimentation. This preparation will reduce and break up the natural matter and turbidity in the water [10]. Coagulation goes through three stages including clot formation, particle destabilization, and finally particle aggregation. Natural coagulants aggregate particles and aggregates by adsorption onto their surfaces, followed by charge neutralization or particle locking. There are four coagulation mechanisms that occur within the particle aggregation process: double layer compression; net agglomeration; adsorption and charge neutralization; and adsorption and particle attraction [11]. Various nano metal oxides have been used in water treatment, including nano aluminum and nano iron [12]. However, nanomaterials, being new in use, remain dangerous if they are not tested. Therefore, I am turning to using nanomaterials manufactured in a green way that are safe for the environment [13,14].

Environmentally friendly nanomaterials can be manufactured from natural sources such as bacteria and fungi, but the most important safe source for green nanotechnology is plants, which have proven their efficiency in creating effective and low-cost water treatments, and the resulting sludge is low in biodegradability [14-17]. The aim of the present study is to green synthesis of $\text{C-Fe}_3\text{O}_4$ from (*Cinnamomum spp.* plants) as a natural nano-coagulant in wastewater treatment.

MATERIALS AND METHODS

Treatment of plant

Cinnamomum spp. were collected and attended according to the method of [18].

Materials created by plants

After being purchased and sanctified with distilled water, dried cinnamon bark was allowed to air dry in a shaded area. After drying, it was ground into a fine powder and kept for later use in sterile plastic containers.

Making an aqueous extract of the plant Cinnamomum spp

In accordance with the procedure outlined by Irshaid and Mansi [11], 50 grams of powdered cinnamon bark were combined with deionized water in a 1:10 ratio and heated for 30 minutes at 70°C while being constantly stirred. After filtering, the extract was kept at 4°C .

Synthesis of iron oxide nanoparticles

Iron salts ($\text{FeCl}_2 \cdot 4\text{H}_2\text{O}$ and $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$) were dissolved in distilled water in a 1:2 molar ratio and heated to 70°C . The cinnamon extract was added dropwise under stirring. Sodium hydroxide was added to reach pH 9–11, and the mixture was stirred for 1–2 hours. The resulting black precipitate was centrifuged, washed with water and ethanol, and dried in an oven [19].

These biosynthesized nanoparticles were later characterized using standard techniques such as UV–Vis spectroscopy for optical confirmation, and XRD for determining crystalline structure. Microscopy tools like FE-SEM were employed to visualize morphology and size distribution [20].

Collecting water samples

Samples of treated water were collected from the wastewater in Barakia in Najaf Governorate in Iraq, in 4 iterations from the main sedimentation tank using different types of clean containers.

Water Sample Measurements

Temperature, pH, and electrical conductivity were measured using a multispectral spectrometer, while turbidity was measured using a turbidimeter. All the above-mentioned instruments were calibrated in the laboratory before measurements began.

Total dissolved solids, chemical oxygen demand (COD), and total organic carbon were measured

according to the methods described in the American Public Health Association (APHA) [21].

To evaluate the efficiency of natural NPs, a jar test was conducted on water samples whose initial properties. The pre-prepared NPs at room temperature underwent lime agitation to ensure homogeneity before use.

The methodology included the following steps:

Volume and Rapid Mixing: One liter of water sample was taken, the nanoflake was added, and the mixture was treated with a rapid mix at 150 rpm for one minute to provide initial mixing.

Slow Mixing and Settling: The mixing speed was reduced to 50 rpm and continued for 20 minutes to induce flocculation, followed by a static clarification and settling phase lasting 15 minutes.

To optimize performance, varying

concentrations of the nanoflake, ranging from 0.5 to 1 ppm, were tested to determine the optimal dosage based on the lowest residual contaminant level. The study also included measuring the effect of pH across a wide range (3 to 10) to determine the most effective pH in the coagulation process.

Statistical analysis

The data obtained underwent one-way analysis of variance (ANOVA), followed by the least significant difference (LSD) test to determine the substantial differences between the means.

RESULTS AND DISCUSSION

Detection of active components in *Cinnamomum spp.* plants

Table 1 presents the phytochemical screening

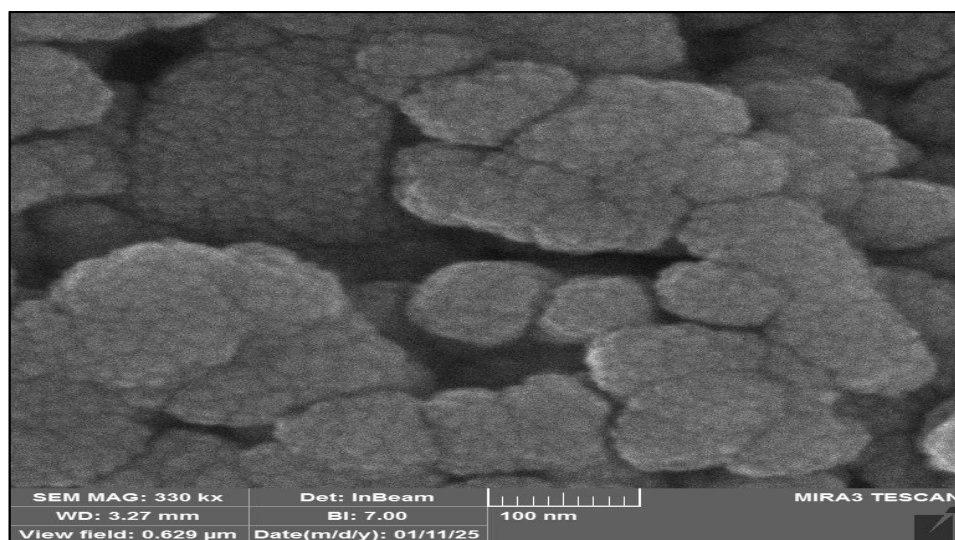


Fig. 1. The synthesized C- Fe_3O_4 NPs (FE-SEM) micrograph picture.

Table 1. Detection of active components in *Cinnamomum spp.* plants.

N	Tests	Test results	<i>U. pilulifera</i>
1.	Glycosides Test		-
2.	Terpenes	layer that is reddish-brown	+
3.	Trpenoid	Purple and red ring	+
4.	Tannins Test	Test for ferric chloride in gelatinous precipitates	+
		Lead Acetate Test (Color of bluish-green)	+
5.	Resins Test		-
6.	Flavonoids Test	a dark yellow hue Hydroxide of potassium	+
		Yellow color sulfuric acid test	+
7.	Saponins Test		-
8.	Alkaloids Test	(Dragendroff reagent) Precipitate with an orange hue	+
		Mayer reagent (Turbidity)	+
9.	Phenolate Test	Color: bluish-green	+
10.	Fuocoumarins Test	The color yellow	+

results of the aqueous extract of *Cinnamomum spp.*, revealing the presence of several bioactive compounds. Positive results were obtained for terpenes, tannins, terpenoids, flavonoids, phenols, coumarins, and alkaloids. In contrast, the extract tested negative for glycosides, resins, and saponins.

Identification of C- Fe_3O_4 NPs

The morphological properties of the nanoparticles produced using *Cinnamomum spp.* (FE-SEM) was used to examine and it reveals

the of spherical structure with nonuniform distribution. The surface morphology showed rough textures, likely due to the presence of bio-organic compounds from the plant extract on the nanoparticle surface. as in the Fig. 1. Based on SEM analysis and particle size estimation using **ImageJ software**, the nanoparticles exhibited a size distribution ranging from 13 to 20 nm, with an average diameter of approximately 16.5 ± 3 nm. The size was estimated by measuring over 50 individual particles using the scale bar provided in the SEM image Fig. 2. The clear lattice fringes

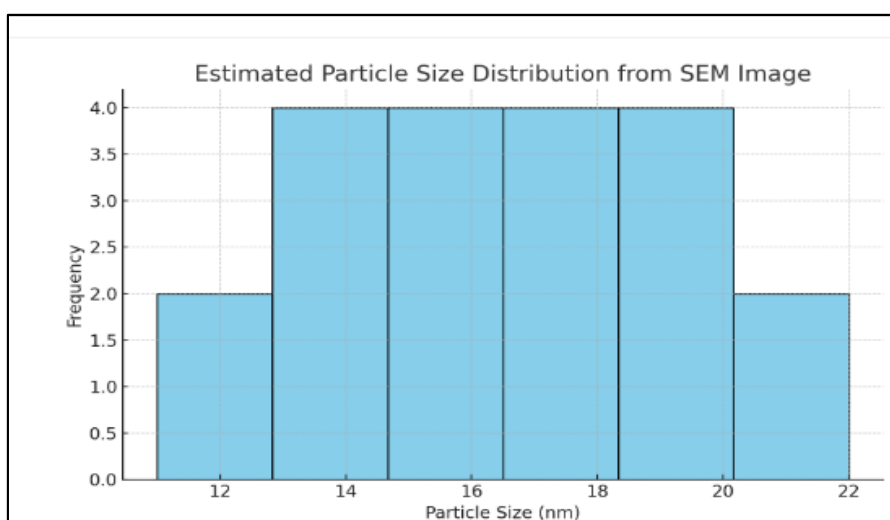


Fig. 2. The synthesized C- Fe_3O_4 NPs (FE-SEM) average size.

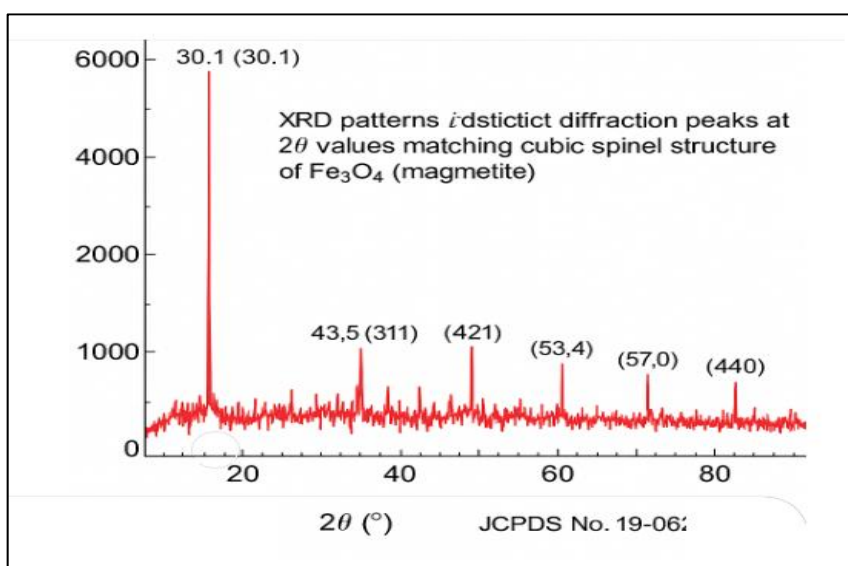


Fig. 3. XRD pattern of C- Fe_3O_4 NPs.

indicated their crystalline nature as in Fig. 3. XRD patterns displayed distinct diffraction peaks at 2θ values matching the cubic spinel structure of Fe_3O_4 . XRD patterns displayed distinct diffraction peaks at 2θ values matching the cubic spinel structure of Fe_3O_4 . XRD patterns displayed distinct diffraction peaks at 2θ values of 30.1° , 35.5° , 43.1° , 53.4° , 57.0° , and 62.6° , which correspond to the (220), (311), (400), (422), (511), and (440) planes of the cubic spinel structure of Fe_3O_4 , respectively. These peaks were identified based on comparison with the standard JCPDS card No. 19-0629. UV-visible spectra of *Cinnamomum spp.* extracts and C- Fe_3O_4 NPs are shown in Fig. 4. UV-Vis spectroscopy confirmed the formation of C- Fe_3O_4 NPs by the presence of a characteristic absorption band around 280 nm, which is typically associated with Fe^{3+} - O^{2-} charge transfer transitions, as reported in earlier studies [17,18].

Experience treating organic and inorganic pollutants

The results of the tests for treating organic and inorganic pollutants showed that an equilibrium state was reached after 24 hours of treatment, after which the absorption was constant. When equilibrium was achieved, the clear solution was reanalyzed to determine remaining contaminants and removal rates.

The current study focused on the possibility

of using C- Fe_3O_4 nanoparticles prepared from the aqueous extract of the *Cinnamomum spp* plant in treating polluted water, and its presence in wastewater was detected. The results demonstrated the possibility of removing these pollutants by exposing manufactured C- Fe_3O_4 nanoparticles at different concentrations and for different periods of time.

The data presented in Tables 2 and 3 demonstrate that pollutant removal efficiency increased progressively with time and concentration. At a concentration of 0.5 mg/L (Tables 2 and 3, moderate improvements were observed after 24 hours, particularly in EC and TOC. The nano-treatment achieved the highest EC removal (66.1%) and TOC reduction (46.8%) compared to the plant extract and the nano-extract mixture.

At the higher concentration (1 mg/L) (Table 3), removal efficiencies improved significantly. After 24 hours, the nano-treatment exhibited superior performance, achieving 86.9% TOC removal, 68.5% EC reduction, and 66.8% TDS removal. The nano-extract mixture also showed substantial efficiency (78.6% TOC removal), whereas the plant extract alone demonstrated comparatively lower but consistent removal rates.

The increase in efficiency with time indicates enhanced adsorption and surface interaction processes. Furthermore, the higher concentration

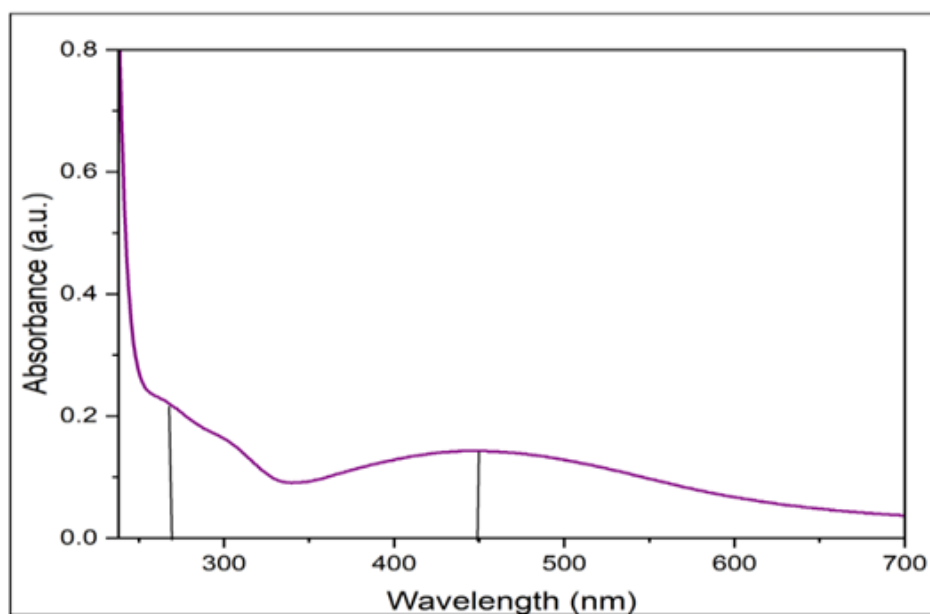


Fig. 4. UV of *Cinnamomum spp.* extracts before and after the formation of C- Fe_3O_4 NPs.

(1 mg/L) promoted greater pollutant binding capacity, particularly for organic carbon removal. The slight variations in pH across treatments remained within acceptable environmental ranges, indicating treatment stability.

Overall, C-Fe₃O₄ nanoparticles synthesized via plant extract exhibited the highest remediation capability, particularly for TOC removal after 24 hours at 1 mg/L. Many studies have been conducted to determine the effectiveness of low-cost adsorbents such as peat, ash and bentonites in removing organic materials from wastewater by adsorption method. Studies showed that an

equilibrium time of 5 and 16 hours was needed for adsorption of organic matter on peat, fly ash and bentonite, respectively. Phenol adsorption on peat and bentonite has been well studied. Using a standard solution with a concentration of 1 mg/L, it was found that peat, ash, and bentonite adsorbed 46.1%, 41.6%, and 42.5% of phenol, respectively [22]. Activated carbon (in granular and powder form) is also one of the most widely used adsorbents. It has a good ability to adsorb many organic and inorganic pollutants, although it has many disadvantages.

Activated carbon is very expensive and

Table 2. Variation in pollutants at 0.5 mg/L of C-Fe₃O₄, plant extract, and nano-extract mixture (1:1).

After 1 hour								
Parameter	Before	NANO	EXT	MIX	Removal %	NANO	EXT	MIX
pH	6.20	6.05	6.45	6.75	0.3		0.4	0.6
EC (μS/cm)	3050	3470	3535	3395	45.8		43.2	44.1
TDS (mg/L)	1598	1665	1735	1695	44.2		18.5	13.4
TOC (mg/L)	10.07	9.48	10.01	9.98	1.3		0.6	0.4
After 12 hours								
Parameter	Before	NANO	EXT	MIX	Removal %	NANO	EXT	MIX
pH	6.20	6.55	6.72	6.88	0.6		0.7	0.5
EC	3050	3725	3465	3435	34.6		33.1	24.2
TDS	1598	1765	1725	1715	14.2		13.5	11.8
TOC	10.07	9.02	9.20	9.28	0.8		0.5	0.5
After 24 hours								
Parameter	Before	NANO	EXT	MIX	Removal %	NANO	EXT	MIX
pH	6.20	6.05	6.95	6.98	1.5		1.7	1.3
EC	3050	3720	3455	3440	66.1		35.2	35.5
TDS	1598	1765	1725	1715	30.4		24.5	29.8
TOC	10.07	7.82	9.45	9.55	46.8		44.1	45.2

Table 3. Variation in pollutants at 1 mg/L.

After 1 hour								
Parameter	Before	NANO	EXT	MIX	Removal %	NANO	EXT	MIX
pH	6.20	6.05	6.68	6.82	0.4		0.4	0.3
EC	3050	3015	3045	3048	44.3		24.1	24.0
TDS	1598	1365	1315	1345	46.2		33.4	34.1
TOC	10.07	8.85	10.55	9.35	56.4		1.2	33.1
After 12 hours								
Parameter	Before	NANO	EXT	MIX	Removal %	NANO	EXT	MIX
pH	6.20	7.95	7.90	6.10	13.2		11.1	9.8
EC	3050	3310	3440	3340	57.3		35.2	26.4
TDS	1598	1660	1715	1645	68.4		57.2	46.3
TOC	10.07	9.40	9.72	9.50	57.6		38.4	37.5
After 24 hours								
Parameter	Before	NANO	EXT	MIX	Removal %	NANO	EXT	MIX
pH	6.20	7.05	7.28	6.48	12.1		7.2	9.4
EC	3050	3310	3440	3340	68.5		55.6	54.3
TDS	1598	1660	1715	1645	66.8		46.5	47.4
TOC	10.07	4.55	7.75	7.25	86.9		69.2	78.6

reactivation produces additional effluents and losses

Large: 10-15% of the absorbent material. Also, previous studies used aquatic plants such as flower Nile algae or others have demonstrated high potential in treating inorganic pollutants [23].

This opened the way for many researchers to find cheaper alternatives such as bottom ash, fly ash, fly ash-wallastonite, [24] lignite, peat, soil, wallastonite, wallastonite-china clay (1:1) to be used as alternatives that have proven their efficiency in removing many pollutants. The Lepironia plant was also studied to remove PAHs from wastewater using the batch flow system. The results showed that the removal potential reached 79.9%. Also, the use of the tropical plant and Eleocharis ochrostachys in treating oil pollutants, PAHs, both proved an effective treatment rate that reached 71.91 % respectively [25]. Overall, plant-mediated synthesis of Fe₃O₄ nanoparticles represents a promising strategy for sustainable wastewater treatment applications [26,27].

CONCLUSION

The physicochemical parameters of wastewater (pH, EC, TDS, and TOC) were effectively reduced using C-Fe₃O₄ nanoparticles, plant extract, and their 1:1 mixture. Removal efficiency increased with both exposure time and concentration. The best performance was achieved using nano C-Fe₃O₄ at 1 mg/L after 24 hours, where removal percentages reached: pH: 12.1%; EC: 68.5%; TDS: 66.8%; TOC: 86.9%. These findings confirm the high efficiency of plant-mediated C-Fe₃O₄ nanoparticles in treating organic and inorganic pollutants, outperforming the plant extract alone and the combined nano extract mixture. The results support the applicability of green-synthesized nanoparticles as an effective and environmentally compatible wastewater treatment strategy.

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CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this manuscript.

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