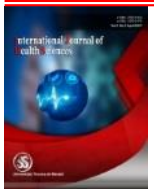

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Comparative Study Towards the Synthesis of α-Fe2O3 Nanoparticles Using a Different Precursor Via ECO-Friendly Method

Batool Sarhan Mansour [a], **Abdulqadier Hussien Al Khazraji** [b]

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Corresponding Author [a]

Keywords

Eco-friendly method;
Eucalyptus extracts;
methyl orange;
nanoparticles;

Abstract

The nano oxides were prepared in an environmentally friendly manner (by treatment with aqueous eucalyptus leaves (from trees in Diyala governorate, Iraq) extracts using different sources of iron, $FeSO_4.7H_2O$ and $FeCl_2.4H2O$ as the nanoscale iron oxide. The $Fe(OH)_2$ nanoparticles were obtained by slow addition of sodium hydroxide solution to eucalyptus extract. Then, to obtain α-Fe_2O_3, calcination of $Fe(OH)_2$ nanoparticles was carried out at 550 °C. These oxides and nanocomposites were diagnosed by FTIR, XRD, FESEM, and DLS techniques. Brunauer-Emmett-Teller (BET) was used to determine the surface area of the nanomaterials. X-ray diffraction (XRD) examination showed that the size of α-Fe_2O_3 nanoparticles was 48 nm, characterized using several techniques, including XRD, AFM, FT-IR, and FESEM. These nanocomposites were used to study the adsorption of methyl orange dye from their aqueous solutions. The effects of equilibrium time, surface area weight, and temperature on the adsorption process were investigated. The results showed that the optimal equilibrium time was 40 min for the α-Fe2O3/paraffine nanocomposite and 60 min for the Fe2O3/AC nanocomposite. The optimal weight for dye removal was 0.25 g for the α -Fe2O3/paraffine nanocomposite. The optimal pH for removing methyl orange dye was 5 for all nano composites.

Contents

[a] Department of Chemistry, College of Education for Pure Science, University of Diyala, Iraq
[b] Department of Chemistry, College of Education for Pure Science, University of Diyala, Iraq

1 Introduction

With the growing development of the chemical, pharmaceutical, and agricultural industries, many chemical compounds, such as pesticides, steroid hormones, antibiotics, and dyes, can reach the aquatic environment (Tkaczyk et al., 2020; Aslam et al., 2004). Previously, synthetic organic dyes were underestimated as pollutants due to their very low concentrations in the aquatic ecosystem (ng/l to µg/l). William Perkin discovered the first synthetic organic dye, moffin, in 1865. This discovery revolutionized the dyeing industry, and organic dye production began on a global scale at the beginning of the 20th century (AL-Niaimi & Jwameer, 2021; Abdussalam-Mohammed, 2020). Green methods are considered one of the easiest, fastest, cheapest, and most environmentally safe approaches, utilizing plant extracts to extract nanoparticles. This review examines the latest research on the green method for producing metallic nanoparticles using plant extracts (Babu & Antony, 2019; Soltys et al., 2021). It includes a diverse range of studies that utilized various plant parts (roots, stems, leaves, fruits, peels, and seeds) to produce nanoparticles of several metals, most notably silver (Ag) and gold (Au), as well as oxides of other metals such as zinc oxide (ZnO), copper oxide (CuO), iron oxide (Fe3O4), and other metal oxides (Singh et al., 2018; Mutlag. 2014). The advancement of eco-friendly technology in material production is crucial to the development of its uses. Currently, a variety of nanoparticles with different chemical compositions, sizes, and morphologies have been created using environmentally friendly techniques, and their use has been investigated in numerous cutting-edge technical domains (Khan et al., 2019; Manisalidis et al., 2020). The regenerative qualities of the plant extracts, the environmentally friendly aqueous medium, and the moderate reaction conditions make the process superior to alternative hazardous procedures. In recent years, several plant extracts and products have drawn attention due to their energy efficiency, low cost, and benign behavior during the production of metallic nanoparticles (Crawford et al., 2017). The creation of chemical compounds that reduce or eliminate the usage and manufacture of harmful materials is referred to as "green" or "sustainable" chemistry (Anastas & Eghbali, 2010; Valavanidis et al., 2009). Additionally, a reaction is referred to as "green" when it contains three components: a solvent, a catalyst, and an energy consumer (Asif, 2021; Thirumamagal et al., 2022). As in the case of hematite (α-Fe2O3), the stable form of iron oxide nanoparticles is contained in rhombohedral crystal units and plays a significant role in research (Abusalem et al., 2019). The most appealing method is chemical co-precipitation, which yields high-purity, low-cost hematite crystals as powders or thin films with a short preparation time (Bumajdad et al., 2011; Wang, 2013). Other processes include polyol, sol-gel, and pyrolysis. Our study aim synthesis of α-Fe_2O_3 nanoparticles using a friendly, environmentally friendly method. Preparation of binary composite with paraffin and activated carbon (α-Fe_2O_3/ paraffin and α-Fe_2O_3/ activated carbon). Finally, using the obtained metal oxide nanoparticles and their nanocomposite to remove methyl orange dye from its aqueous solutions.

2 Materials and Methods

The chemicals used in this study were aluminum sulfate hexadecahydrate from CDH, and ferrous chloride and ferrous sulfate from Alpha Chemika.

Instruments and Apparatus

The concentration of methyl orange dye was measured using a Shimadzu (Japan 1700) spectrometer located at the University of Diyala/College of Education for Pure Sciences, based on a Shimadzu spectrometer from Japan. The pH of the aqueous solution was calculated using a pH meter (7110 wt., Germany). A vibrating bath

(BS-11, Korea) was used to control the temperature. X-ray diffraction (XRD-6000, Shimadzu, Japan) was used to classify all the various substances. A bromide-only infrared disk spectrometer (IRPRESTIGE21, Shimadzu) was also used. The Zetasizer Nano ZS sample size distribution studies were performed using a DLS Malvern device. (SSABET) was used to measure pore size and specific surface area using a TriStar 3000 device from Micromeritics Inc.

Synthesis of α-Fe2O3 nanoparticles using eucalyptus leaves extract

Eucalyptus leaves from trees in the Diyala governorate were used to make the plant extract. The leaves were thoroughly cleaned with both ordinary and distilled water. After two days of shade drying, the leaves were chopped and thoroughly powdered. Five grams were then added to 100 milliliters of distilled water and heated to 70 degrees Celsius for thirty minutes while stirring. To remove any leftover living elements, the extract is allowed to cool, filtered, and placed in test tubes. It is then centrifuged for five minutes at 1500 rpm, collected in a vial, and stored at 25 oC. A magnetic stirrer was used to thoroughly mix 50 milliliters of distilled water with 0.3 grams of salt. Ten milliliters of the eucalyptus extract are placed in a burette and added to the mixture drop by drop at a temperature of twenty-five degrees Celsius. After raising the temperature to 80 oC, we progressively added a 0.1M sodium hydroxide solution until the solution turned basic and a precipitate developed. Pollutants are eliminated by washing the sediment with ethanol and water. The precipitate is then dried for three hours at 60 oC to produce nano iron hydroxide powder. This powder is subsequently torched for five hours at 600 oC to produce iron oxide nanoparticles (Jwameer et al., 2025).

Preparation of α-Fe2O3 /AC Nanocomposite

(0.3 g) of nano iron oxide from iron sulfate was taken in 20 mL of ethanol and placed in the ultrasound machine for 30 min and then (0.3 g) of activated charcoal was taken and dispersion in 20 mL of ethanol and placed in the ultrasonic device for a period 30 min at a temperature of 50 ºC The nano-oxide suspension is gradually added to the activated charcoal and placed in the ultrasound machine for 2hrs and left to dry.

Determining the maximum wavelength and preparing the methyl orange dye

A 1000 ppm solution of methyl orange (M.O.) was prepared by dissolving 1 g of the dye in 1000 mL of deionized water. Solutions of varying concentrations were then prepared by taking the appropriate volume and diluting it with distilled water. To determine the wavelength at which maximum absorption (λmax) occurs, a UV-Vis spectrometer in the 200–800 nm range was used to record the absorption spectrum with a 1 cm cross-sectional quartz cell. The λmax was determined to be 465 nm, as described above.

3 Results and Discussions

3.1 X-ray Diffraction

To verify the crystalline nature of the produced nanoparticles, X-ray diffraction (XRD) characterisation was carried out on the α-Fe2O3/paraffine and α-Fe2O3/Ac compounds, depending on purity and crystallization. The data for the three strongest peaks are displayed in Figures (1,2). The ferrate sulfate had the greatest α-Fe2O3 peaks at (24.3, 33.3, 35.8). The ferrate chloride produced the strongest α-Fe2O3 peaks at 24.3, 31.9, and 33.4.

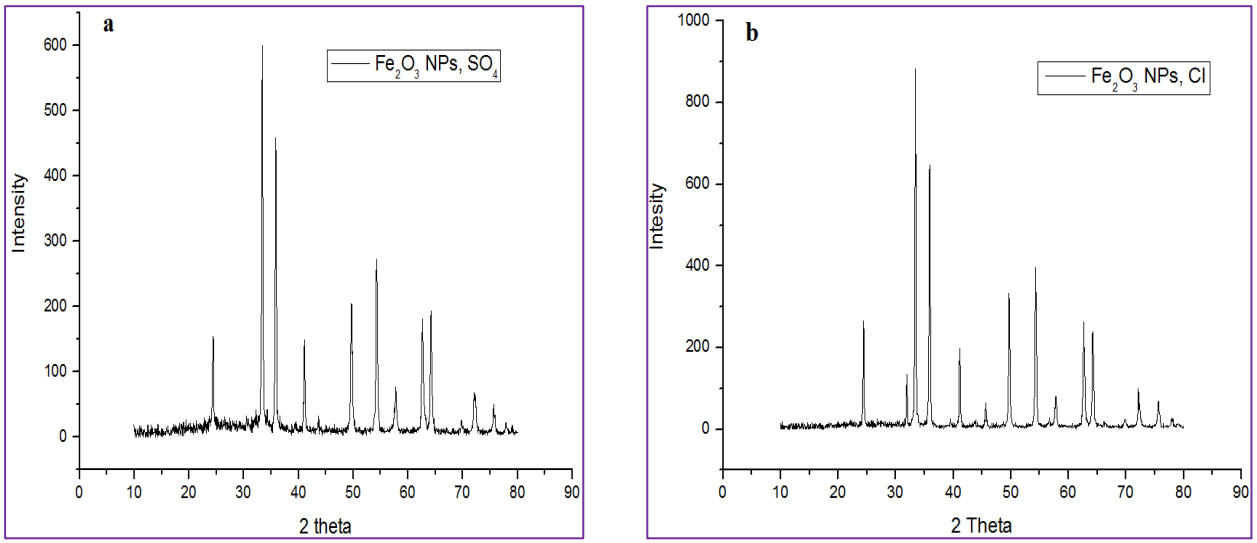

Figure 1. XRD pattern of (a) α-Fe2O3 NPs from FeSO4 precursor, (b) α-Fe2O3 NPs from FeCl2 precursor

3.2 FESEM image

Scanning electron microscopy (SEM) was used to characterize these oxides and composites, as it is useful in determining the morphology of nanoparticles. The results showed that these composites are nanoscale in size, based on their morphology. Field-emission scanning electron microscopy (FEM) images show that the nanoparticles tend to be mostly spherical, with slight variations in shape. High-resolution images clearly reveal the reduced surface characteristics, as well as the distribution of nanoparticles across the entire surface. Compared to other morphologies, these distinctive surface characteristics enable the nanoparticles to possess more active sites.

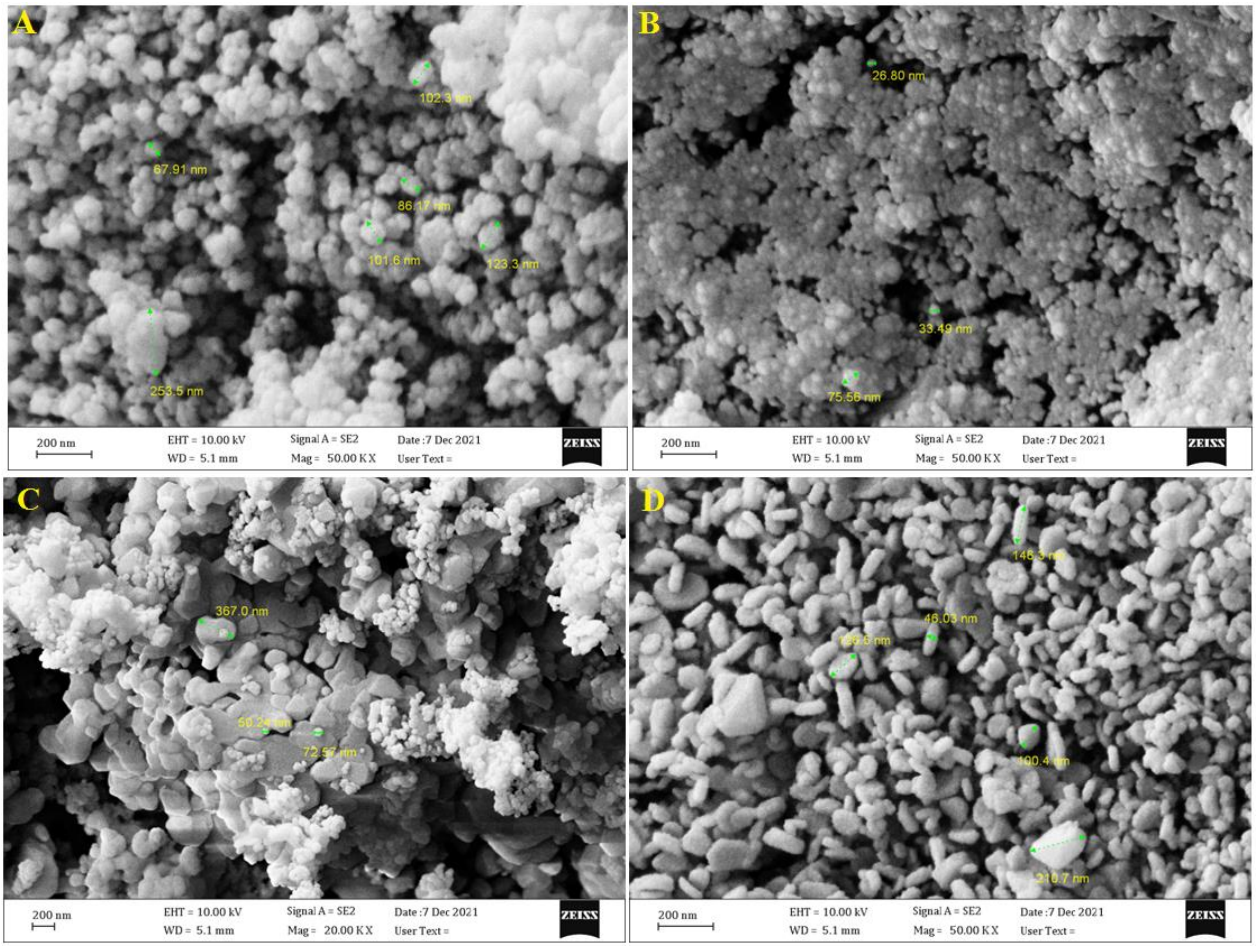

Figure 2. FESEM images of (A) Fe (OH)2 NPs from FeSO4, (B) Fe (OH)2 NPs from FeCl2, (C) α-Fe2O3 NPs from FeSO4, and (D) α-Fe2O3 NPs from FeCl2 precursor

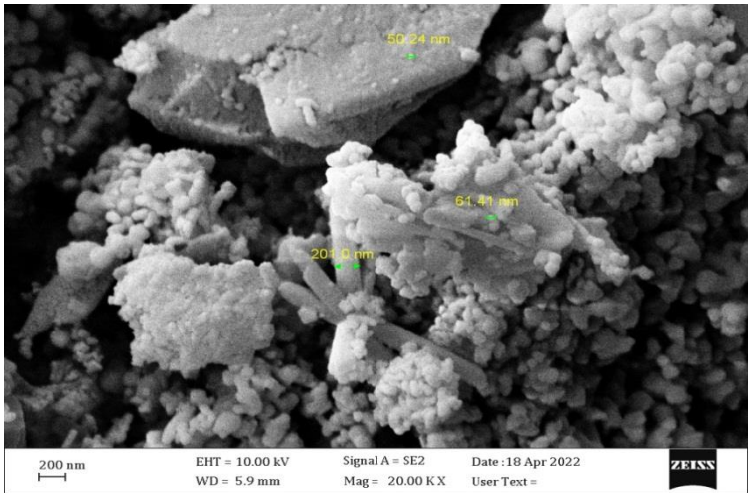

Figure 3. FESEM image of the α-Fe2O3 /AC nanocomposite produced from Fe (SO4)2 and activated charcoal

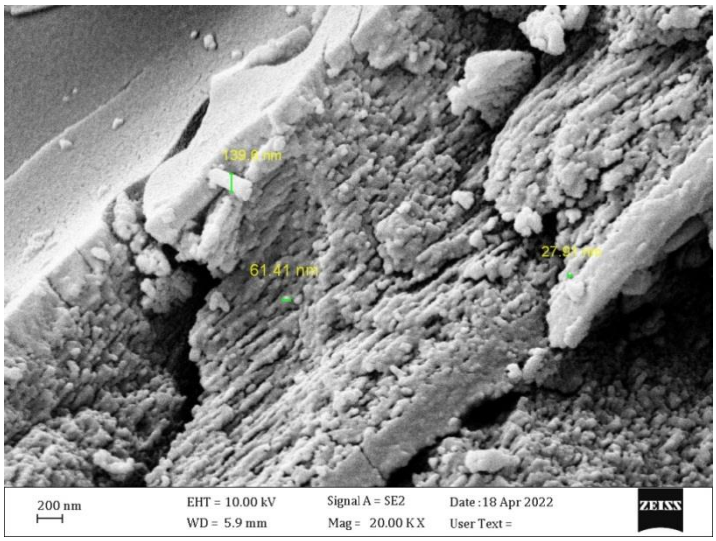

Figure 4. FESEM image of the γ -Al2O3 NPs/ activated charcoal nanocomposite produced from Al (SO4)3 with activated charcoal

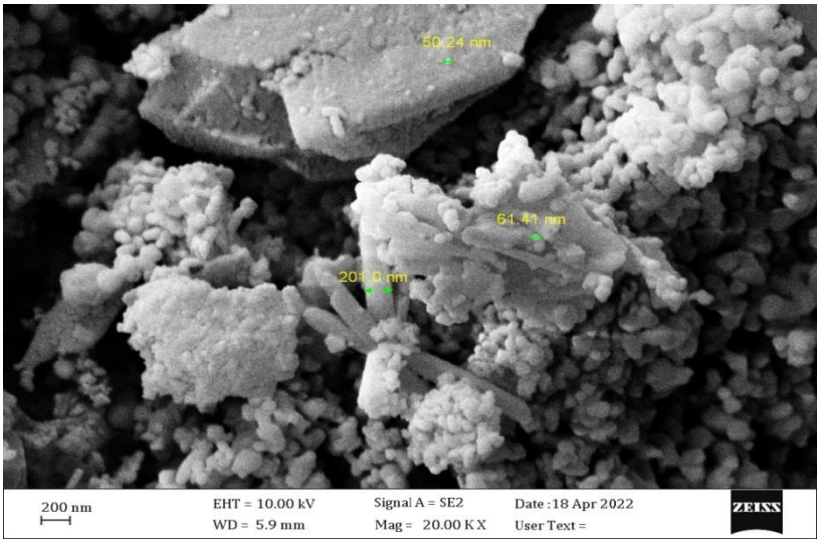

Figure5. FESEM image of the α-Fe2O3 /AC nanocomposite produced from Fe (SO4)2 and activated charcoal

The Surface Area (BET)

The surface area of nano-oxides were (α-Fe2O3 NPs from FeSO4 precursor, α-Fe2O3 NPs from FeCl2 precursor, γ-Al2O3 NPs from Al (SO4)3 precursor γ-Al2O3 NPs from AlCl3 precursor), measured using BET and shear method) (Branuuer - Emmette - Teller), which includes the use of liquid nitrogen at a temperature of (77 K). The (BET) theory is currently used as a standard equation for isothermic adsorption analysis to obtain a specific surface area for solids. The surface area of nano-oxide (α-Fe2O3) NPs from FeSO4 precursor) is (10.616m2/g) and nano-oxide (α-Fe2O3) NPs from FeCl2 precursor) is (3.3469 m2/g)

3.3 Adsorption of Methyl Orange Dye

Optimum Conditions

The effect of contact time, adsorbent quantity, and temperature on methyl orange dye adsorption was studied using iron oxide and aluminum oxide nanoparticles that were prepared from plant sources, in addition to metal oxide nanoparticles and the binary composite prepared from Fe2O3 nanoparticles/ Activated carbon or paraffin. The initial concentration of methyl orange dyes before carrying out the adsorption process by UV-visible was 50 mg/L.

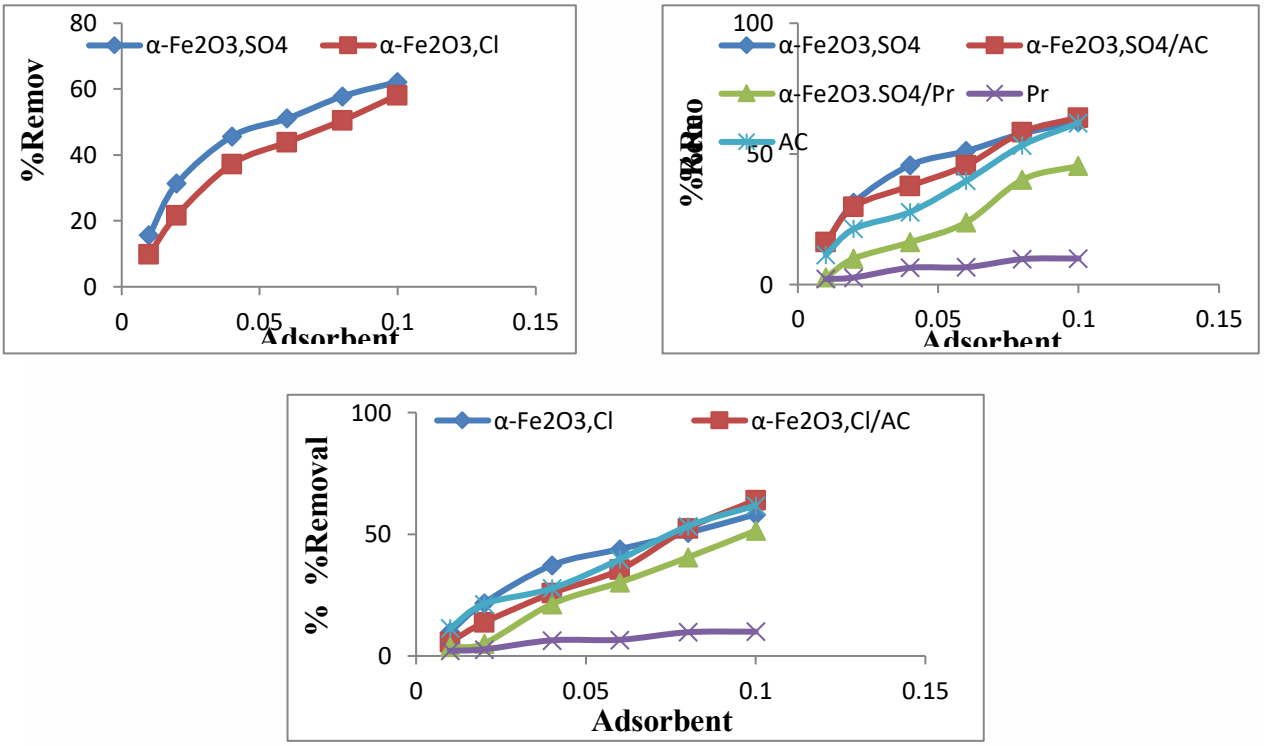

Figure 6. The effect of nano adsorbent dosage on the % removed of methyl orange dye by α- Fe2O3NPs and their composite

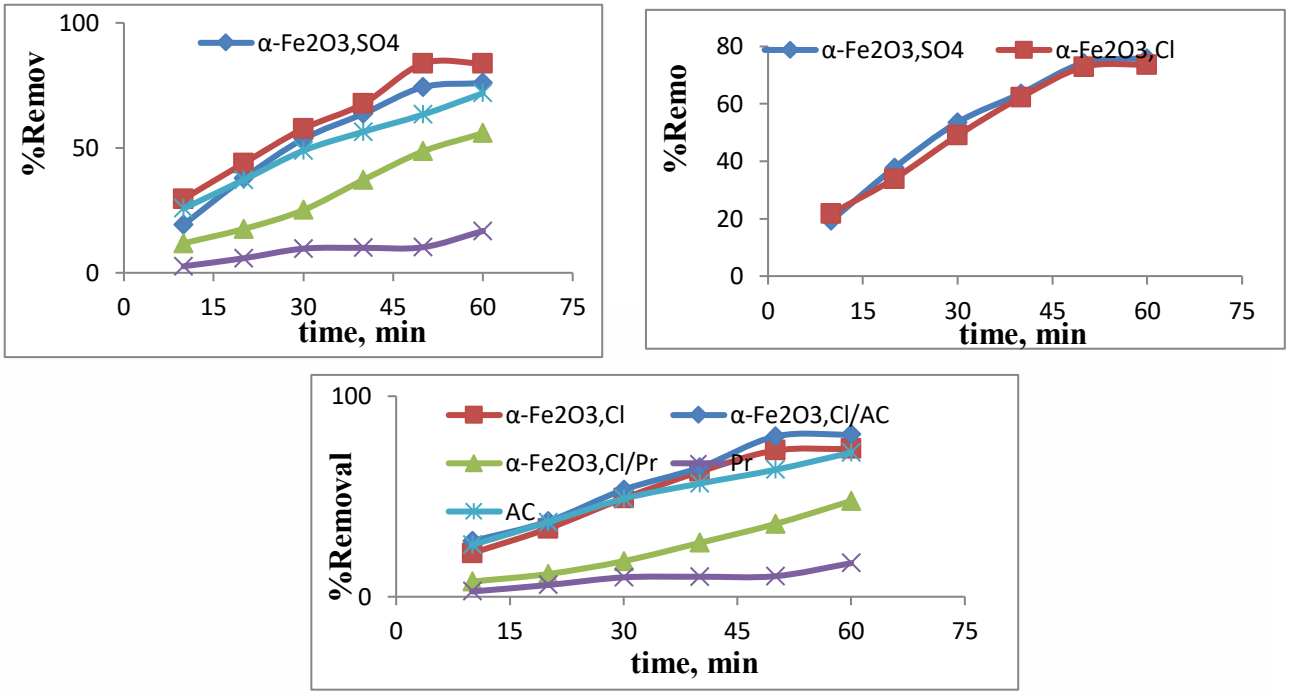

Figure 7. The effect of contact time on the % removed of methyl orange dye by α- Fe_2O_3NPs and their composite

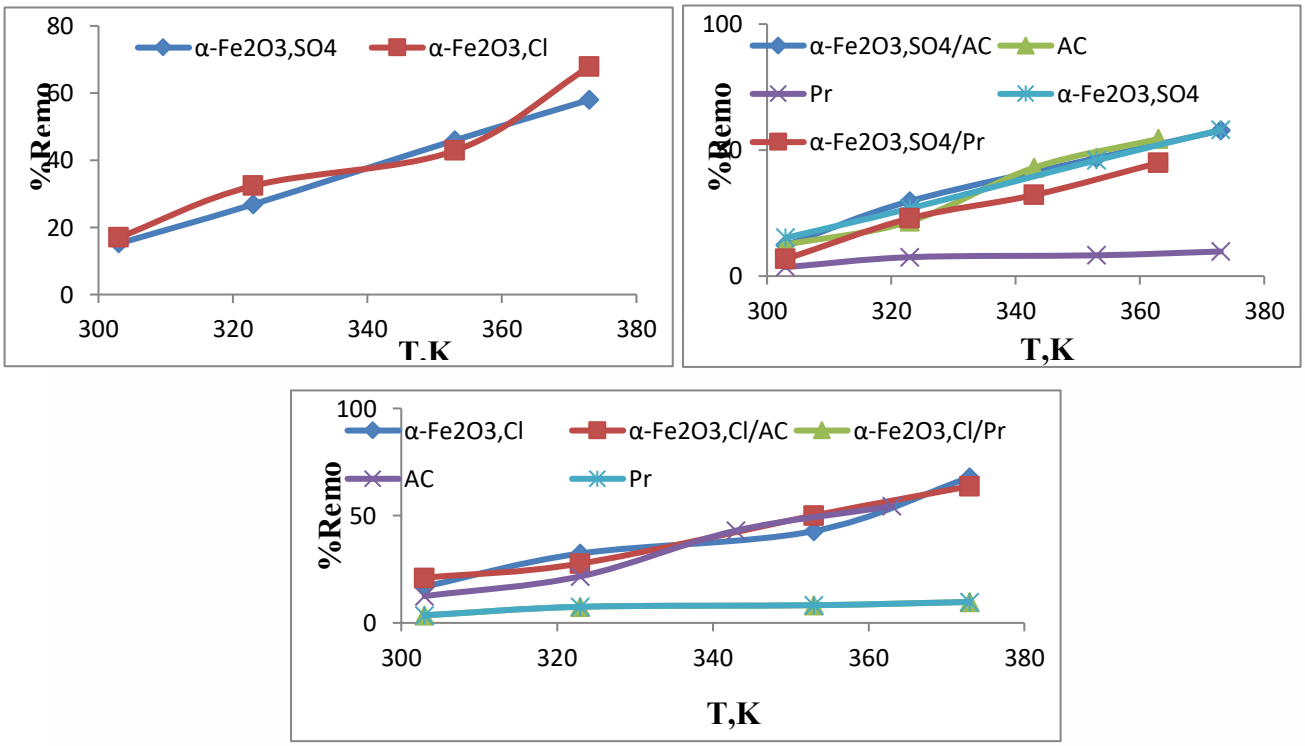

Figure 8. The effect of temperature on the % removed of methyl orange dye by α-Fe2O3NPs and their composite

4 Conclusion

The percentage removal of methyl orange reaches equilibrium in contact time (60) min, and at adsorbent weight (0.1) g. The highest percentage of removal for nano oxides and nanocomposites was for Fe2O3, SO4 (75.22%), Fe_2O_3, Cl (73.68%), Fe_2O_3, SO4/AC (83.76%), and Fe_2O_3, Cl/AC (80.84%). Fe_2O_3, SO_4/Pr (51.84 %), Fe_2O_3, Cl/Pr (47.76 %). In general, eco-friendly methods were used to manufacture α-Fe2O3 NPs, and their characteristics were studied. The effective synthesis of α-Fe2O3 NPs was shown by FTIR, XRD, DLS, FESEM, and BET techniques. According to the XRD technique, the average size of α-Fe2O3 nanoparticles was around 30.50 nm for FeSO4.7H2O and 42.30 nm for FeCl2.4H2O precursor. The functional group is identified using the FTIR method. The α-Fe2O3 NPs' strong band was detected below 600 cm-1. Different particle sizes with oval and spherical shapes and varying average diameters are shown by FESEM analysis; the spherical nanoparticles are part of the Fe (OH)2 and α-Fe2O3 nanoparticles from sulfate precursor, with average diameters of 122 nm and 163 nm, respectively. In contrast, the average diameters of Fe (OH)2 and α-Fe2O3 nanoparticles made with an oval shape from a chloride precursor are 44.6 nm and 125.5 nm, respectively. The average nanoparticle size was determined to be 691 nm for and 587 and 125 nm for the two precursors whose sizes ranged from 537 nm to 2690 nm, according to the DLS approach. Additionally, it was discovered that the sulfate and chloride precursor had an average nanoparticle size of 227 nm. The surface area of α-Fe2O3 nanoparticles using the BET approach is 10.616 m2/g for the sulfate precursor and 3.3469 m2/g for the chloride precursor.

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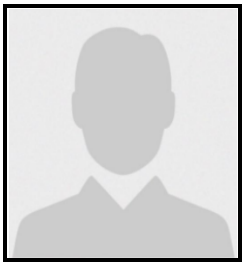
Biography of Authors

Batool Sarhan Mansour
Department of Chemistry, College of Education for Pure Science, University of Diyala, Iraq.
Email: batoolsarhan45@gmail.com



Abdulqadier Hussien Al Khazraji
Department of Chemistry, College of Education for Pure Science, University of Diyala, Iraq.
Email: abdulqadier.niama@uodiyala.edu.iq