



Synthesis of Iron Oxide Nanoparticles from Scrapped Waste Materials for Efficient Dye Removal to Purify Industrial Waste Water

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In this investigation, we used a circular economy approach by recycling metal scrap from industry as a component in creating nanoparticles. For producing iron oxide NPs we used electrochemical method with scrapped rusted iron nails as electrodes. In this technique, we used 50mM ferrous sulphate solution as an electrolyte and a direct current power supply. Different methods were used to characterise the synthesised iron oxide NPs, including ultraviolet-visible (UV-visible) spectroscopy, Fourier transform infrared (FTIR) spectroscopy, X-ray diffraction (XRD), and transmission electron microscopy (TEM). The results demonstrated that the synthesised iron oxide NPs have an average crystallite size of 7.54 nm. Iron oxide NPs has potential to adsorb Congo red dye hence prepared NPs have been utilised for the adsorption of Congo red dye.

Keywords: Nanoparticles; Waste materials; TEM; UV-Vis; Dye removal

1 Introduction

Azo dyes are synthetic colourants that are widely utilised in textile, printing, and cosmetics industries. They are known for their bright colours and ease of use, but they can release poisonous aromatic amines that can harm the environment and human health¹⁻². Unfortunately, the production and use of Congo red and other azo dyes have raised environmental concerns due to their major contribution as a water pollutant. Congo red pollution is largely caused by the discharge of wastewater from factories that utilise this dye³. Severe contamination and negative consequences on aquatic ecosystems can result from the discharge of untreated or partially treated wastewater containing Congo red into water bodies⁴. Efforts are being made to regulate their use, develop safer alternatives, and promote sustainable practices to minimize their environmental impact. For this reason we adopted a circular economy⁵ strategy by repurposing industrial metallic waste as a raw material for making nanoparticles. The elimination of Congo Red and other dyes from wastewater is just one example of the promising applications of nanoparticles⁶. Various types of nanoparticles (NPs), including metal-based NPs, metal oxides, carbon-based NPs, and hybrid NPs, have been investigated for their dye removal capabilities⁷. NPs are used as nanoadsorbents due to

their unique characteristics, such as increased surface area, reduced production costs, greater efficiency, magnetic character, etc⁸. Here, we suggest using iron oxide NPs (IONPs) created by iron nails to get rid of Congo red in water. Standard methods such as transmission electron microscopy (TEM), X-ray diffraction (XRD), and Fourier transform infrared spectroscopy (FTIR), were used to characterize the nanoadsorbent IONPs and UV-Visible spectroscopy was used to do dye adsorption studies.

2 Materials and Methods

All the chemicals used in this study were of analytical grade; we bought $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ (>99% purity, CDH) and Congo red (Thomas baker) from separate companies and collected rusty iron nails from scrap metal. Electrochemistry was used to create iron oxide nanoparticles. As an electrolyte, we utilised 50 mM $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ in a volume of 100 mL. In this case, the cathode and anode were two iron nails. We applied a pulsed DC voltage of 12 V at room temperature for 45 minutes. Obtained IONPs were washed three times with distilled water and centrifuged at 6000 rpm for five minutes to get rid of any extra impurity. Magnets were used to separate the NPs, and then they were cleaned twice in distilled water. The synthesized IONPs were dried in an 80 °C laboratory drying oven overnight before being ground into a powder using a mortar and pestle. The setup is illustrated in Fig. 1

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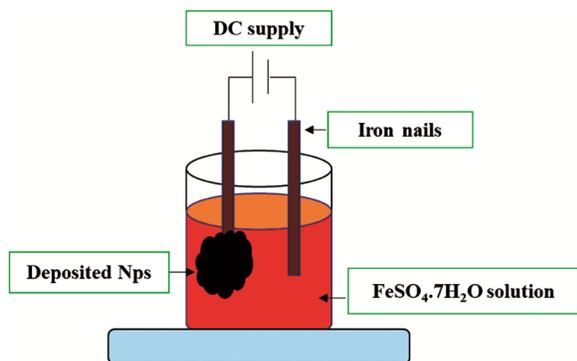


Fig. 1 — Electrochemical setup for the preparation of iron oxide nanoparticles.

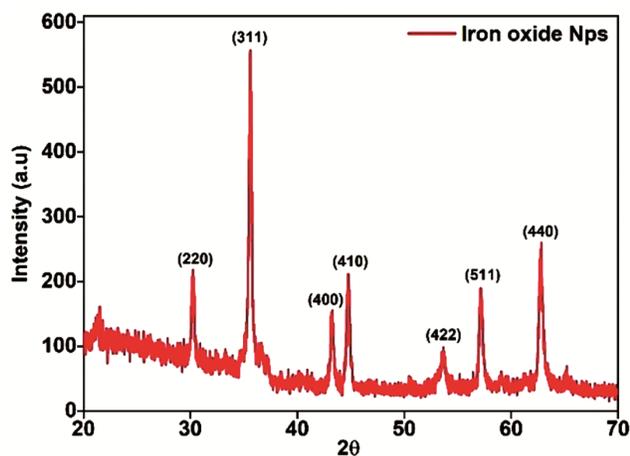


Fig. 2 — XRD plot of synthesized iron oxide nanoparticles.

3 Results and Discussions

3.1 X-ray diffraction

The powder X-ray diffraction (PXRD) pattern analysis of produced NPs is shown in Fig. 2, in the synthesised NPs, Iron oxide is in cubic phase. Iron oxide exhibits space group $P4_32(212)$, with primitive. The characteristic planes (220), (311), (400), (410), (422), (511) and (440) of X-ray pattern at $2\theta = 30.230^\circ$, 35.597° , 43.329° , 44.541° , 53.717° , 57.179° , and 62.892° respectively, confirms the formation of Fe_2O_3 (Maghemite) NPs which corresponds with JCPDS file No. 89-5892. This analysis confirms that IONPs were successfully formed via the electrochemical method.

3.2 TEM

Transmission electron microscopy (TEM) measurements of the NPs diameter were taken using a JEOL, Japan-made TEM model number JEM-2100F/HR. Fig. 3(a) displays the nanoparticle morphologies at 25,000x magnification from the TEM analysis of the grid and b) shows the TEM histogram

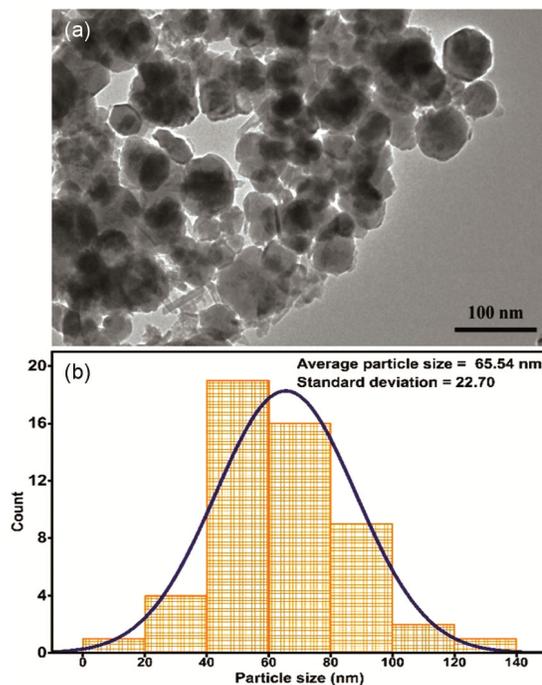


Fig. 3 — (a) TEM image of prepared iron oxide nanoparticles at 25000x magnification and 100nm scale bar (b) TEM histogram distribution plot of Fe_2O_3 NPs.

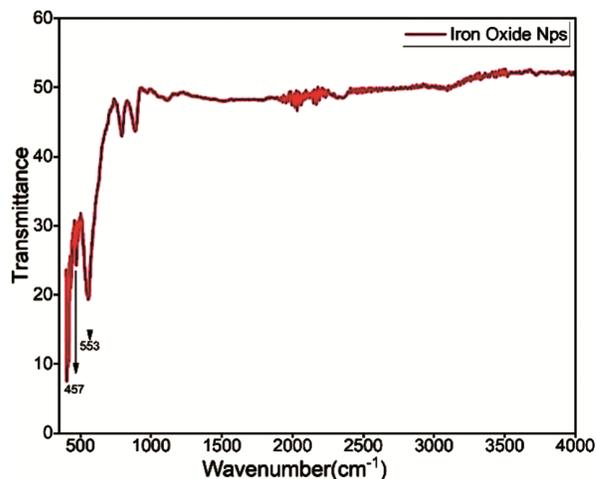


Fig. 4 — FTIR data of prepared iron oxide nanoparticles.

distribution plot of Fe_2O_3 NPs. IONPs have variable shapes such as hexagonal, spherical and rod like as disclosed by TEM images.

3.3 FTIR

Different functional groups in the produced Iron oxide nanoparticles were detected using Fouriertransform infrared spectroscopy (FTIR) analysis. FTIR analysis of produced nanoparticles is shown in Fig. 4. The bands at 553 and 457 cm^{-1} are caused by Fe-O stretching vibration modes in Fe_2O_3 ⁹.

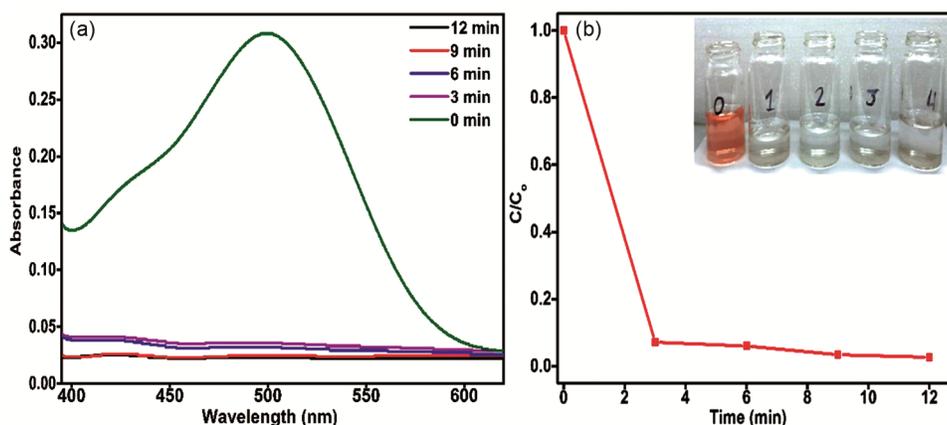


Fig. 5 — (a) Absorbance versus wavelength curve of Congo red dye with iron oxide nanoparticles at different time interval (b) c/c_0 versus time graph for determining dye removal efficiency.

3.4 Adsorption studies

The UV-Visible studies have been done for the determination of adsorption efficiency of prepared nanoparticles. We used 100mg of nanoparticles in our experiment to remove Congo red dye at a concentration of $20\mu\text{M}$. In order to determine the adsorption or removal efficiency (η), the Eq.1 has been used.:

$$\eta = [(C_0 - C_t)/C_0] \times 100 = [(A_0 - A_t)/A_0] \times 100 \quad \dots(1)$$

C_0 - Concentration at time 0

C_t - Concentration at time t

A_0 - Absorbance at time 0

A_t - Absorbance at time t

The produced analysis data is shown in Fig. 5(a,b).

4 Conclusion

This research introduces a circular economy approach by recycling metal scrap from industry as a component in creating nanoparticles and an easy-to-use electrochemical approach for creating iron oxide nanoparticles. Congo red dye was specifically adsorbed by IONPs from aqueous solution. By XRD analysis of IONPs the presence of Maghemite phase was established and Debye-Scherrer¹⁰ equation was used for analysing average crystallite size of NPs which is found to be 7.54 nm. The TEM data analysis showed that average

particle size of IONPs was observed to be 65.54 nm. UV-visible analysis shows that prepared NPs have very good adsorption efficiency. It shows 97.3% dye removal efficiency in 12 min.

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