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Iron Oxide Nanoparticle Synthesis and Characterization

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***Abstract*—— A green synthesis route was employed to produce iron oxide nanoparticles (NPs), utilizing ethanolic extracts from Phoenix dactylifera L. leaves, a natural source of polyphenols, and an aqueous 0.04 M $FeCl_3$ solution as the iron precursor. The synthesized mixture underwent annealing at 500 °C for 3 hours. The obtained nanoparticles were characterized using XRD, SEM, UV-Vis, and FTIR. XRD analysis indicated the formation of maghemite (γ-Fe_2O_3), hematite (α-Fe_2O_3), and β-Fe_2O_3 phases. SEM imaging confirmed their spherical shape, UV-Vis spectroscopy determined a band gap of 2.148 eV, and FTIR spectra confirmed the presence of Fe-O bonds.Keywords— Nickel oxide, Thin films, Spray pyrolysis, Optical gap energy, Monocrystalline structure.**

Keywords—Fe_2O_3 NPs, Phoenix dactylifera L., XRD

I. INTRODUCTION

Iron oxide nanoparticles (NPs) have garnered significant attention in various fields due to their unique physical and chemical properties, including superparamagnetism, biocompatibility, and catalytic activity. Traditional methods for synthesizing these nanoparticles often involve harsh chemicals and energy-intensive processes, raising environmental concerns. As a result, there is a growing interest in developing eco-friendly and sustainable "green" synthesis routes. This study explores such a method, investigating the physical properties of Fe_2O_3 NPs green-synthesized by reacting Phoenix dactylifera L. leaf ethanolic extract, a natural source of reducing and capping agents, with a 0.04 M aqueous iron chloride $FeCl_3$ solution. The synthesized material underwent annealing at 500°C for 3 hours to enhance its crystallinity and properties. The structural and optical characteristics of the resulting nanoparticles were comprehensively analyzed using X-ray diffraction (XRD), ultraviolet-visible (UV-Vis) spectroscopy, scanning electron microscopy (SEM), and Fourier-transform infrared spectroscopy (FTIR).

II. EXPERIMENTAL DETAILS

A. Plant Material Preparation and Extraction

In El-Oued province, Algeria, palm leaves Phoenix dactylifera L were harvested from local farms. These leaves were meticulously washed with tap and distilled water to eliminate impurities, cut into medium-sized segments, and subsequently air-dried at room temperature over 10 days. Following drying, the leaves were ground into a powder. Extraction involved stirring 50 g of this powder in 300 ml of ethanol within a 1000 ml glass jar for 24 hours at room temperature. The filtered liquid ethanolic leaves extract (L.E.L.E) was used for nanoparticle synthesis.

B. Synthesis of Iron Oxide Nanoparticles

Iron oxide nanoparticles were synthesized by reacting 40 ml of L.E.L.E with 80 ml of a 0.04 M $FeCl_3$ aqueous solution, continuously stirred in a 70°C water bath for 1

hour. A green to dark brown color change indicated nanoparticle formation. The resulting blend was then dried, washed with distilled water, and centrifuged. After drying at 90–100°C, the product was annealed at 500°C for 3 hours to obtain the final Fe_2O_3 NPs.

C. Characterization of Iron Oxide Nanoparticles

The optical band gap of the synthesized nanoparticles was estimated using a Shimadzu Model 1800 UV-Vis spectrophotometer (200–900 nm). XRD analysis was conducted on a RigakuMiniflex 600 diffractometer with Cu-Kα radiation (λ = 1.5406 Å) over a 2θ range of 10° to 90°. FTIR spectra were recorded using a Shimadzu IR-Infinity 1 within the 400–4000 cm^{-1} range. Surface morphology was analyzed using a TESCAN VEGA 3 SEM.

III.RESULTSAND DISCUSSION

A. Structural Propertie

The presence of maghemite (γ-Fe_2O_3), hematite (α-Fe_2O_3), and β-Fe_2O_3 as the primary iron oxide phases in the samples was confirmed by XRD analysis. The identification of their respective lattice plane peaks validated the formation of these distinct phases following the 500°C, 3-hour annealing process.

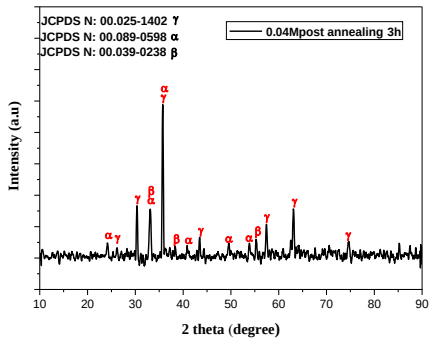

Fig. 1*X-ray diffraction patterns (γ: γ-Fe_2O_3, α: α-Fe_2O_3 and β: β-Fe_2O_3).*

B. Surface Morphology

Scanning electron microscopy (SEM) revealed that the synthesized iron oxide nanoparticles exhibited a spherical shape.

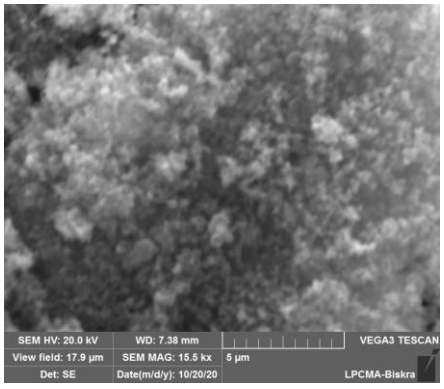

Fig. 2*: SEM micrograph of 0.04M iron oxide nanoparticle powder***.**

C. Optical Properties

The iron oxide nanoparticles exhibited a band gap of 2.148 eV. as determined by UV-Vis spectroscopy. This finding aligns well with band gap values reported in the literature for comparable iron oxide nanoparticles.

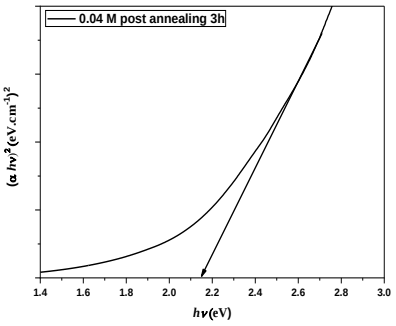

Fig. 3*Band gap (Eg) estimation for the 0.04M iron oxide nanoparticle powder derived from UV-Vis spectroscopy data using Tauc's relation.*

D. FTIR Analysis

FTIR spectra confirmed the presence of Fe-O bonds at characteristic wavenumbers absorption bonds are observed at 562, 636, and 695.31cm^{-1}, which are associated with the formation of the maghemite phase. Additionally, bonds at 448, 476, and 569 cm^{-1} indicate the emergence of the hematite phase . The presence of bonds at 528 cm-1, which are associated with the beta phase.

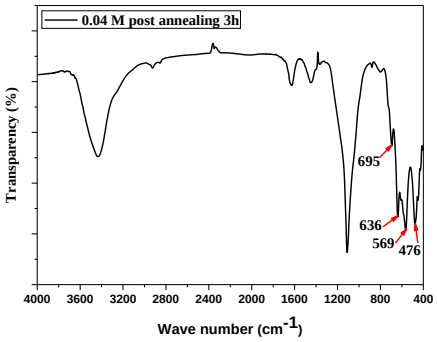

Fig. 4*FTIR spectrum of iron oxide NPs powdes for 3 hour post annealing at 500°C.*

IV. CONCLUSIONS

In conclusion, this study successfully demonstrated a facile and environmentally friendly green synthesis route for Fe_2O_3 nanoparticles utilizing Phoenix dactylifera L. ethanolic extract as a bioreductant and stabilizing agent with $FeCl_3$ as the precursor. The resulting nanoparticles, characterized by XRD as a mixture of maghemite, hematite, and β-Fe_2O_3 phases after annealing, exhibited a spherical morphology as confirmed by SEM and a band gap of 2.148 eV as determined by UV-Vis spectroscopy. The abundance of Fe-O bonds, validated by FTIR, further confirms the successful formation of the iron oxide material. These

synthesized nanoparticles, with their specific phase composition and optical properties, hold potential for applications in various fields such as catalysis, drug delivery, and environmental remediation. Further studies could explore the optimization of the synthesis parameters to achieve phase-pure iron oxide nanoparticles with tailored properties for specific applications.

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