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BIO SYNTHESIS AND STRUCTURAL CHARACTERIZATION OF IRON OXIDE NANOPARTICLES USING ALBIZIA AMARA LEAF EXTRACT

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A B S T R A C T

Iron oxide has attracted a great deal of attention among specialists because of their multivalent oxidation states. The iron oxide nanoparticles have been synthesized by adding Albizia Amara leaf extract into the aqueous solution of ferric chloride. The phytoconstituents of A. Amara leaf extract serve a dual role as reducing, capping and stabilizing agent during the synthesis. GS-Iron Oxide nanoparticles were characterized by UV-vis absorption Spectroscopy, Fourier Transform Infrared Spectroscopy (FT-IR), X-ray Diffraction (XRD) and Transmission Electron Microscope (TEM) and Scanning Electron Microscope (SEM). The existence of the Fe_2O_3 nanoparticles was revealed by UV-vis spectroscopy. The FTIR spectra of leaf extract and synthesized Fe_2O_3 nanoparticles identifies the functional groups of the active components. The formation of Fe_2O_3 nanoparticles has been confirmed by X-ray diffraction and average crystallite size for assign peaks were 37.91 nm. GS- iron oxide nanoparticles serve as potent antibacterial agent in an eco-friendly way by securing naturally biome as nanoparticles usually through target delivery. Thus, A. Amara mediated iron oxide nanoparticles can act as an alternative antimicrobial agent to the antibiotics.

Keywords: *Albizia Amara* leaves, Extract, iron oxide nanoparticles, Characterization.

Introduction

Nanoparticles are submicron moieties with diameters range starting from 1-100 nm made up of organic or inorganic materials having novel properties as compared to a large number of materials [1]. The nanotechnology process depends on synthesis, manipulation, and use of materials that are of Nano scale size. In the new era, nanoparticles take more attention due to their unique size-dependent properties and applications [2]. Metal nanoparticles gain great attention due to their wide range of applications in the fields of electronics, optoelectronics, antibacterial activity, and medical applications such as therapy, diagnosis, and drug delivery [3-4]. The development of adequate techniques for synthesizing metal nanoparticles has become a major focus of researchers. Metallic nanomaterial such as silver, gold, zinc and iron are used in various fields because of their broad applications; among these nanoparticles, iron nanoparticle is preferred for the following reasons: cost effective, antimicrobial activity, high reactivity, smaller size; therefore, it gives high surface-area-to-volume ratio, which allows interact with different chemical species and also efficient in binding metal ions [5]. There are a large number of methods (physical, chemical, and biological) to synthesize various types of nanomaterial. When synthesized by chemical and physical methods, these nanoparticles lose their reactivity due to aggregation magnetism, and dispersibility upon air exposure. Chemical synthesis methods involve toxic chemicals, the formation of hazardous by-products, and contamination from chemical precursors [6]. Therefore, there is growing interest in developing clean, simple, inexpensive, eco-friendly methods for the synthesis of nanoparticles. Bacteria, fungi, algae, and plant extracts can be used in modern alternatives for the production of metal/metal oxide nanoparticles. Plant mediated synthesis of nanoparticles is a revolutionary technique that has wide range of applications in agriculture, food industry, medicine and environmental remediation. The plant related parts such as leaves, stems, roots, shoots, flowers, barks, seeds and their metabolites have been successfully used for the efficient biosynthesis of nanoparticles. Plant extracts usually contain sugars, terpenoids, polyphenols, alkaloids, phenolic acids, and protein, which are responsible for reducing and stabilizing metal nanoparticles [7]. In the past, green synthesis of Fe_2O_3 nanostructures using different plant extracts such as Lagenaria siceraria, Hordeum vulgare and Rumex acetosa plants, peel extract of plantain and Tridax procumbens leaf extract. Therefore, in this study, we have made an attempt on the synthesis of Fe_2O_3 nanoparticles using extract of Albizia Amara leaves.

2. EXPERIMENTAL METHODS

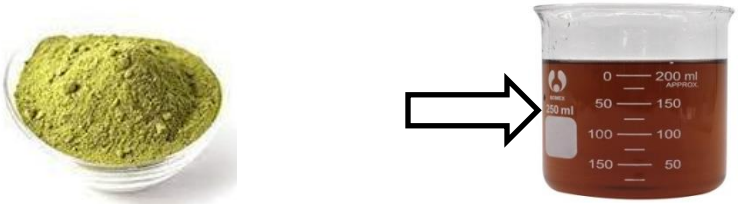
2.1. MATERIALS

All chemicals used were of analytical reagent grade without any further purification in addition to deionised water, Ferric Chloride ($FeCl_3.6H_2O$), Sodium hydroxide (NaOH), hydrochloric acid (HCl), ethanol (C_2H_5OH) and Albizia Amara Leaves.

2.2. Preparation of green synthesized Iron oxide nanoparticles

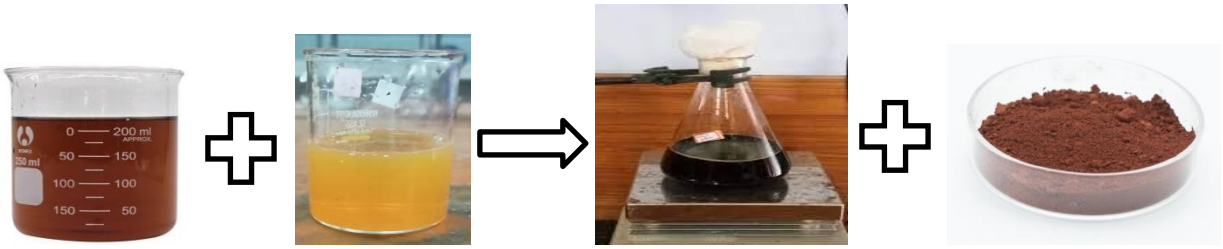
2.2.1. Preparation of Albizia Amara leaf Extract,

Albizia Amara leaves (AAL) were collected from Kallakurichi (DT), Kottaiyur (village) in Tamilnadu. The clean and fresh sources are dried in a shaded place at room temperature for 10 to 15 days and then the leaves were pulverized using commercial blender. The fine powdered was stored at room temperature for further use. In a 250 ml of conical flask 10g of leaf powder were taken and to this 100 ml of double distilled water is added and it is heated at 80°C for 1 hour. Then the solution was filtered using Whatman filter paper and kept aside for further process. The obtained extract in pale brown color and adjusted to the pH at 11 by adding 0.1M of sodium hydroxide solution.



Albizia Amara leaf powder Albizia Amara leaf extract

2.2.2. Preparation of Iron oxide nanoparticles.



Albizia Amara extract 0.1M $FeCl_3.6H_2O$ GS-Fe_2O_3NP Formation GS-Fe_2O_3NP powder

In a 250 ml conical flask, 50 ml of *Albizia Amara* leaves extract was taken and to this 100 ml of 0.1 M $FeCl_3.6H_2O$ solution is added slowly at room temperature under static conditions. The colour change of the reaction was observed and the time taken for the changes was noted. The solution colour changes immediately from pale brownish to reddish brown indicating the formation of iron oxide nanoparticles (Fe_2O_3NPs). Further the solution is centrifuged and precipitated is extracted and dried in electrical oven for 24 hours at 100°C. The dried sample kept in muffle furnace for 4 hours at 500°C. The green synthesized Fe_2O_3NPs is formed at uniform particle size and stored for further characterization and uses [8]

3. CHARACTERIZATION OF ADSORBENT

3.1. UV-Visible spectrophotometer analysis

Synthesized Fe_2O_3 nanoparticles were subjected to UV-Vis spectroscopy analysis, which confirms the formation of nanoparticles in the initial stage. The Fe_2O_3 nanoparticles synthesized were subjected to scan UV-Vis spectrophotometer in the range 190 nm - 1100 nm using Elico SL210 UV VIS Spectrophotometer.

3.2. FT-IR Spectroscopic analysis

The plant extract and green synthesized Fe_2O_3 nanoparticles were characterized by FT-IR spectrometer. The spectroscopic technique is based on the analysis of peaks at certain wave numbers. FT-IR data indicates the presence of functional groups in the plant extract and synthesized nanoparticles. The FT-IR analysis carried out in the frequency range of 4000 - 400 cm^{-1} using Perkin Elmer instrument.

3.3. X-ray diffraction analysis (XRD)

X-ray diffractometer (lakjdf) was used to study the average particle size and crystalline nature of the synthesized adsorbents. The diffraction pattern was obtained by using FeKα radiation with wavelength of λ=1.541A^o. The scanning was done in 2θ value range of 4^o to 80^o at 0.02 min^{-1} and one second time constant.

3.4. Scanning Electron Microscopic (SEM)

The SEM analysis provide the details about surface morphology, porosity and particle size distribution of the adsorbents. The surface morphology of the synthesized Fe_2O_3 nanoparticles was recorded using Hitachi instrument.

3.5. Transmission Electron Microscope (TEM)

TEM is regarded as the best among other electron microscopy techniques for the determination of particle size and morphological identities of Fe_2O_3NPs and other metal nanoparticles.

4. RESULTS AND DISCUSSIONS

4.1. Characterisation study of green synthesized copper oxide nanoparticles.

4.1.1. Ultraviolet –Visible (UV-vis) Spectroscopy.

The type of nanoparticles that has been targeted for synthesis from selected plant can be identified by two methods, one with the visual observation of colour change pattern and another one with UV–Vis analysis. In the present study, iron oxide nanoparticles synthesized from leaves extract showed a colour change pattern from brown to reddish brown in colour at the time of synthesis.

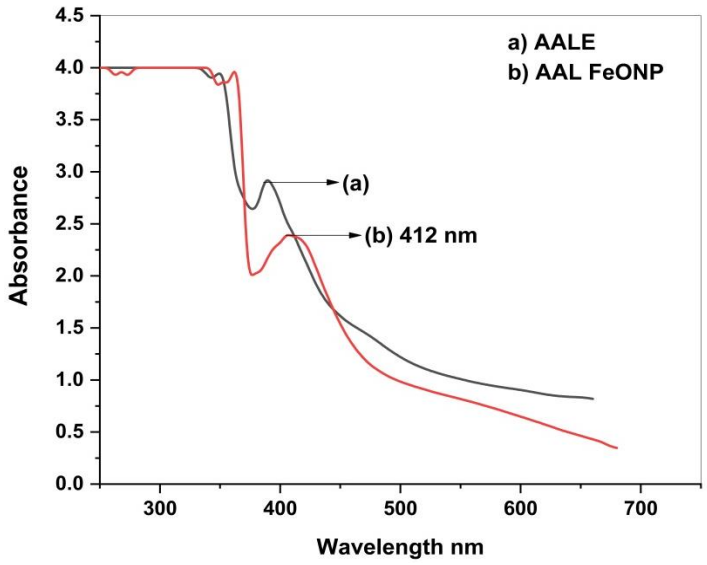


Fig 2 shows UV-Vis spectrum green synthesized iron oxide nanoparticles.

As can be seen from Figure 2, the absorption peaks for Albizia amara leaf extracts are around 370 to 390 nm, which corresponds to the existence of several natural compounds in the extracts [9]. These peaks are vanished after reacting with an iron salt, indicating that the extract compounds acted as reducing and capping agents to synthesize the iron oxide nanoparticles. Furthermore, and in accordance with the results of the present study, the surface plasmon band for iron oxide nanoparticles at wavelengths of 412 nm indicate the formation of iron oxide nanoparticles were reported by previous studies (Figure 2) [10].

4.1.2. Fourier Transform Infrared (FT-IR) Spectroscopy.

The FT - IR analysis were performed on the Albizia amara leaf extract and the synthesized iron oxide nanoparticles to identify a possible change in functional group bonds during the reduction process and presented in Figure 3.

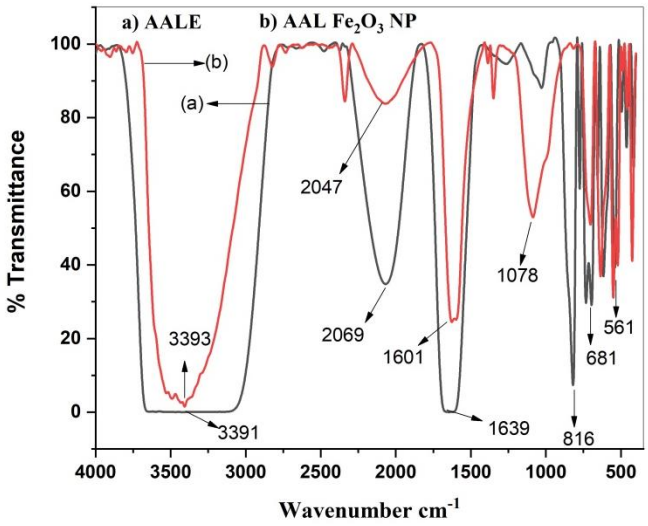


Fig 3a & 3b shows FTIR spectrum of Albizia Amara leaves extract and Iron oxide nanoparticles.

FTIR spectra of the leaf extract are presented in Fig. 3a. The absorption peaks at 3391 and 1639 cm−1 refect the O–H and N–H stretching vibration in the phenolic compound and protein in the fruit extract. The bands at 2069 cm−1 are assigned to C–H stretching in carbohydrates. The bands at 816 cm−1 is caused by the (C=O) NH2 stretching [11].FTIR spectra of synthesized iron oxide nanoparticles using extract of C. mas is shown in Fig. 3 b.The band with higher intensity assigned to the - OH groups indicates water soluble polyphenol compounds that have capped the surface of the prepared iron oxide nanoparticles. The band at 2047 cm - 1 may be due to C≡N stretching from unreacted impurities or due to CO2 in the sample compartment. The band at 3393 cm - 1 and 2800 cm - 1 corresponds to the - OH bond stretching and denotes the aqueous phase, with an increase in the absorption band, indicating the ferrous sulphate reduction. The existing findings agreed well with the reported values [12]

4.1.3. X - ray diffraction (XRD) analysis.

XRD is an analytical tool used to distinguish crystalline phase and cell dimensions of synthesized nanoparticles. Fig (6) Shows the appearance of diffraction pattern at 2θ= 35.12 º, 36.63 º, 40.64 º, 49.97 º, 57.08 º and 65.49 º with corresponding lattice plane values at (104), (110), (113), (024), (116) and (300) respectively. The intense and sharp peaks undoubtedly revealed that iron oxide nanoparticles formed by the reduction method using Albizia amara leaf extract were crystalline in nature. The crystalline size was calculated with the help of Scherrer's formula, which is given as [13]

$D = 0.9\lambda \beta \cos \theta$, (1)

Where D is the crystallite size, β the full-width at halfmaximum (FWHM) of the most intense diffraction peak in radians, θ the diffraction angle and λ the wavelength of X-ray radiation. A sharp peak at 2θ =35.12 and 36.63 with the diffraction of the (104)

and (110) plane indicates that confirmation of Fe_2O_3NPs. The average crystallite size in the samples of Fe_2O_3NPs is below 37.91nm.

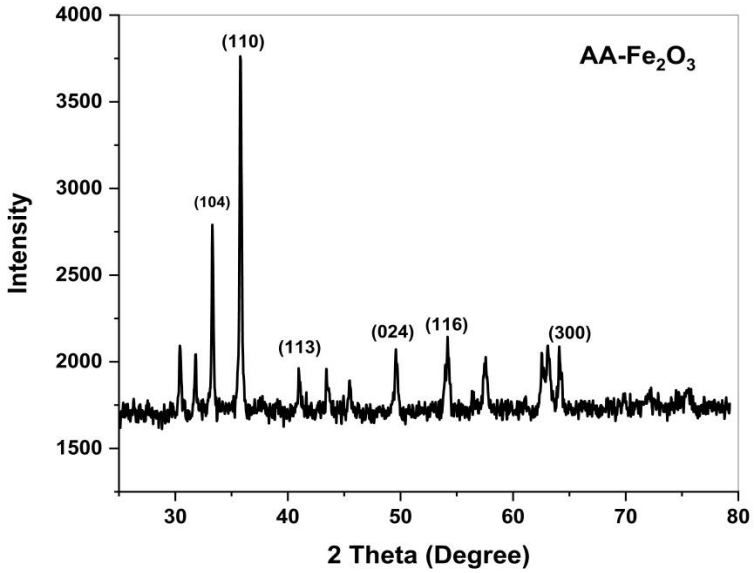


Fig 4 shows XRD pattern spectrum of Green synthesized iron oxide nanoparticles.

4.1.4. Scanning Electron Microscope (SEM)

The morphology of the prepared nanoparticles was examined using scanning electron microscopy. Figure 5 shows the FESEM image of the synthesized Fe_2O_3 nanoparticles consisted of nano-sized particles with a nearly spherical shape. It was observed that the nanoparticles formed were agglomerated with the particles and appears to adhere to each other, forming aggregate of particles, which result in irregular arrangements. The particles sizes measured about 9 to 12 nm, hence supporting the TEM result.

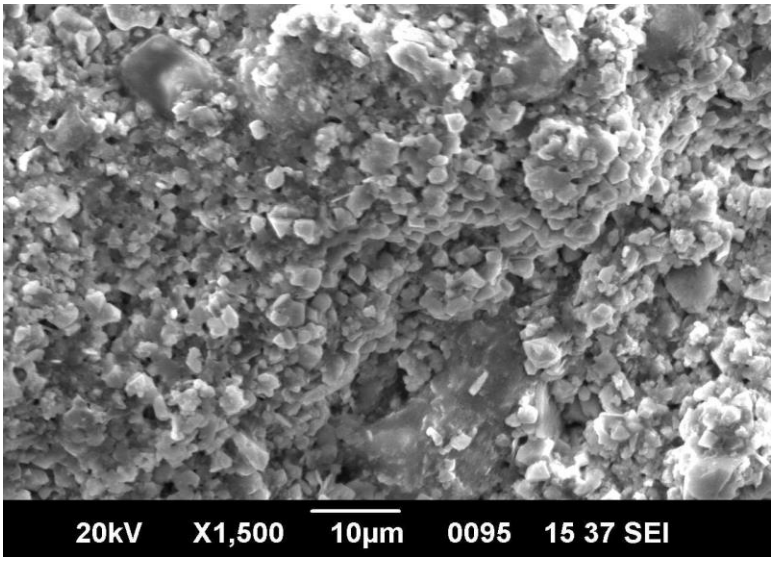


Fig 6 shows SEM image of green mediated iron oxide nanoparticles

4.1.5. Transmission Electron Microscope (TEM)

The TEM photograph of the synthesized IONPs is depicted in Fig. 7. The average particle size was found 8.03±8.99 nm for 206 individual selections. As it is seen, the nanoparticles are almost spherical like and partly as a hexagonal shape. The average size of synthesized nanoparticles shows some difference with the size of particles found by Scherer's equation in the XRD spectra. It may be due to the wide range size distribution of the nanoparticles, given the SD value calculated from the TEM analysis [15].

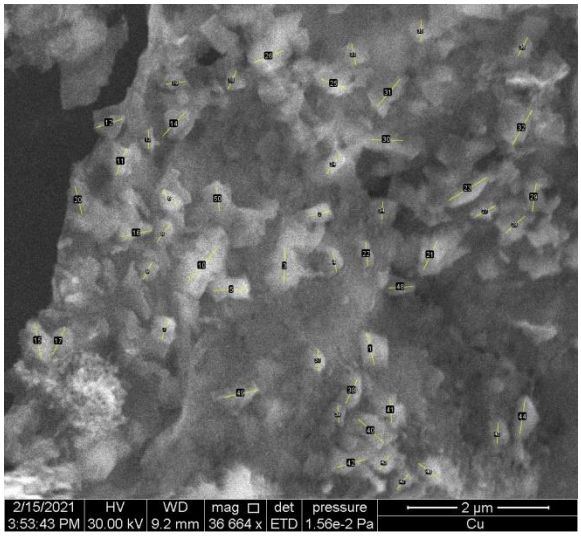


Fig 7 shows TEM image of synthesized iron oxide nanoparticles.

5. Conclusion.

In this present study, we reported the successful use of Albizia Amara as a one-pot green method for the synthesis of iron oxide nanoparticles. The color change, observed instantaneously suggested that the formation of black colored solution indicated the formation of iron oxide nanoparticles. The rapid reduction process proved the efficiency of Albizia Amara extract as reducing and stabilizing agents. The XRD pattern showed the cubic crystal structure of iron oxide nanoparticles without any impurities. FTIR showed that the interactions that existed between Albizia Amara and iron oxide nanoparticles. TEM showed the formation of $Fe_2O_3$with an average size of 9–12 nm with irregular shape. Green routes for nanoparticle synthesis are of great interest because they are ecofriendly, inexpensive, simple and rapid. They also have a wide range of applications, such as in nanomedicine, catalysis, and optoelectronics.

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