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Green Synthesis and Characterization of Iron Oxide Nanoparticles using Artocarpus Heterophyllus Leaves Extract

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Abstract:

In the present study, an ecofreindly benign way of synthesis of Iron-Oxide (Fe_2O_3) nanoparticles (IONPs) using Artocarpus Heterophyllus (Jack Fruit Leaves) as stabilizing agent. The nanoparticles formation was observed by color change from orangish-brown solution to black precipitate. The phase purity of IONPs was observed by using powder-XRD technique, the formation of nanoparticles was further confirmed from UV spectroscopic technique and the absorption peak is observed at 411nm. The presence of organic and inorganic compounds in synthesized nanoparticles was identified using FTIR analysis. The average size of the synthesized nanoparticles was estimated using XRD patterns and SEM analysis.

Keywords: Artocarpus Heterophyllus, Ferrous Chloride Solution, Iron Oxide Nano Particles, Characterization Techniques.

Introduction:

The rapid advancements in nanotechnology research and development across the globe are producing notable breakthroughs that will likely shape the 21st-century technological environment. One important result of this explosion is the synthesis of novel materials at the nanoscale, especially nanoparticles. Because of the special qualities that these particles whose sizes are fewer than 100 nanometers display, which vary according to size, nanocrystalline materials are especially interesting to material scientists. Their unique physicochemical qualities result in unmatched electrical, mechanical, optical, and imaging properties that are highly desirable in a variety of applications spanning the commercial, ecological, and medicinal domains [1-4]. Throughout the years, a wide variety of nanoparticles have been dubbed with abandon, including nanospheres, nanorods, nanochips, nanostars, nanoflowers, nano reefs, nanowhiskers, nanofibres, and nanoboxes [5]. Depending on their surrounding environment during creation or their inherent crystal habit, nanoparticles can take on a variety of morphologies. These considerations include sculpting holes in solid materials, creating emulsion droplets and micelles during precursor preparation, and inhibiting crystal formation by covering certain faces with chemicals [6]. Iron oxide nanoparticles are the focus of current research in the field of nanomaterials. Iron oxide nanoparticles are essential to daily life and are important in a wide range of applications. Their research is essential to material science because they exhibit features that vary with size from bulk materials. Iron oxide nanoparticles are very sought-after for applications in the commercial, ecological, and medical fields due to their distinctive electrical, mechanical, and optical properties. The investigation of Iron Oxide Nanoparticles stands out as a cornerstone in the age of various nanoparticles, providing insights and applications that greatly promote continuing technological breakthroughs [7-8].

Iron oxide nanoparticles, or IONPs, have become highly adaptable nanomaterials with applications in environmental remediation, biomedicine, and catalysis, among other domains [9]. Because of their low environmental effect and eco-friendliness, green approaches for synthesizing IONPs have garnered a lot of attention, the use of plant extracts stands out as a viable and sustainable method among the several green synthesis pathways investigated [10-11]. Iron oxide nanoparticles possess super magnetic properties, making it an intriguing metal oxide with numerous applications in cutting-edge technologies. These include reversing magnetic resonance imaging images tissue restoration, detoxification of living fluids, delivering drugs within living systems, removing toxic heavy metal ions from wastewater and numerous other uses. Iron oxide nanoparticles must have homogeneous qualities because all of these applications call for high magnetization values, small particle sizes, and a scale distribution within a specific range [12-14].

Greenly produced IONPs have the potential to be effective in eliminating organic contaminants from aqueous solutions due to their adsorption characteristics, because of their high surface area and reactivity nature. The nanoparticles can effectively adsorb dyes and other pollutants. Moreover, the application of plant-mediated synthesis techniques is consistent with green chemistry concepts, which prioritize environmental responsibility and sustainability [15-16]. Among iron oxides, magnetite (Fe_3O_4) and maghemite (γ-Fe_2O_3) have been the most investigated. Natural sources of iron oxide nanoparticles (IONPs) include air pollution and volcanic eruptions [17]. Magnesite (Fe_3O_4) and maghemite (γ-Fe_2O_3) can be produced chemically or released by industries and transportation. Magnetic properties are crucial for optimizing the application potential of super-paramagnetic IONPs [18]. IONPs are cost-effective, easily synthesized, versatile and their magnetic properties are significantly influenced by surface area. Various methods, including hydrothermal, sol-gel, thermal decomposition, and co-precipitation, can be used to prepare nano iron oxides [19-20].

The tree known as Artocarpus heterophyllus is frequently seen in South East Asia and is sometimes found in home gardens on Pacific islands. In particular, it is well-known for its amazing, hardy wood, which becomes orange or reddish-brown with age. Numerous plant parts, such as the fruits, bark, roots, and leaves, are said to have therapeutic qualities. The leaves of the jackfruit plant (Artocarpus heterophyllus), which are high in antioxidants and phytochemicals, have attracted attention as a possible source for the green synthesis of IONPs [21]. The bioactive substances found in jackfruit leaves are great stabilizers and reducers, making it easier to synthesize nanoparticles in a way that is both economical and safe for the environment. Furthermore, jackfruit leaves are a plentiful and sustainable resource for the synthesis of nanoparticles [22]. In this study, extract from Jack fruit leaves is used as a reducing agent to create nanoparticles with a huge surface area in a straightforward, economically viable, and eco- friendly manner.

Materials and Methodology:

i) Materials:

1. Artocarpus Heterophyllus (Jack Fruit) leaves
2. Ferric Chloride anhydrous crystalline ($FeCl_3$, SDFCL, 98.1%)
3. Distilled Water
4. Whatman No.1 and 0.45 Micron Mixed Eaters of Cellulose, Millipore Filter Papers.

ii) Methodology:

Artocarpus Heterophyllus Leaves Extract Preparation:

Initially, the Artocarpus Heterophyllus (Jack Fruit) leaves were cleaned with raw water followed by distilled water and theleaves are sun dried for 2 to 3 days. The dried leaves are crushed and sieved for uniform molecules, then 10 grams of leaves powder was taken and mixed with 100 ml of distilled water, further the solution is boiled and stirred for 60 min at 60°C. Finally, the leaves extract was obtained by filtration through whatmann-1 filter papers using vacuum filter apparatus.

Figure-1: Artocarpus Heterophyllus (Jack Fruit) Leaves.

Synthesis of Iron-Oxide Nanoparticles using Artocarpus Heterophyllus Leaves Extract:

The 100ml of 0.01M ferric chloride ($FeCl_3$) solution and 100ml of leaves extract is added to mix them in proportion of 1:1 ratio, and then the resulting mixture is stirred for 60min at 70°C. During the above process add 0.1M NaOH solution drop by drop to maintain the pH of 11. The Synthesis of Nanoparticles was observed with a color change from orangish-brown solution to black precipitate. The precipitated solution is centrifuged at 6000rpm for 30min, followed by washing of powder with acetone and distilled water thrice by filtering the nanoparticles using 0.45 micron mixed eaters of cellulose, Millipore filter paper and the particles are settled on the paper and the settled powder was air dried in hot air oven at 90°C for 2hrs. Finally, the resulting powder was heated at 800°C for 4 hours. The obtained Iron-Oxide nanoparticles powder further characterized using UV-spectroscopy, XRD, FTIR and SEM analysis.

Results and Discussions:

Characterization of Iron-Oxide Nanoparticles

A) UV -Visible Spectroscopy Analysis:

Figure-2 shows the UV-visible absorption spectrum of Iron Oxide nanoparticles that were synthesized using jack fruit leaves at 1:1 ratio of ferric chloride ($FeCl_3$) solution and leaves extract. The presence of absorbance peak in the range of 400 – 425 nm indicates the formation of iron oxide nanoparticles [23].

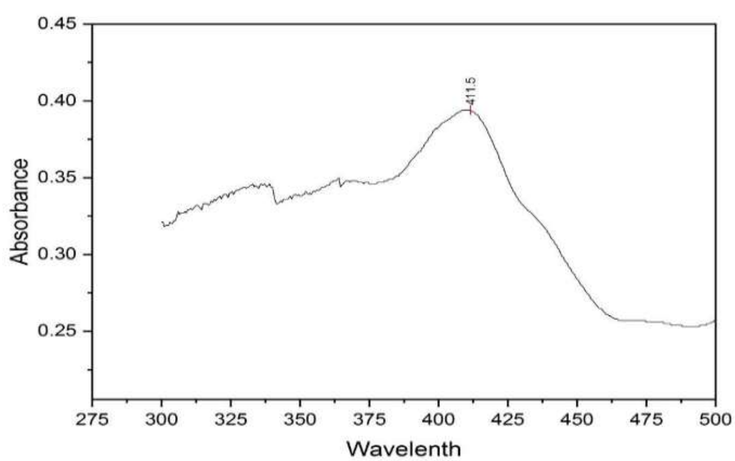

Figure-2: UV-Visible absorption spectrum of green synthesized Iron-Oxide nanoparticles

B) X-Ray Diffraction Analysis:

The phase purity and crystalline nature of the Ag nanoparticles was further confirmed from the powder XRD technique.Figure-3shows the XRD patterns of the green synthesized Iron Oxide nanoparticles using jack fruit leaves (Artocarpus Heterophyllus) extract. The observed XRD patterns establish the single-phase nature of the Fe_2O_3 nanoparticles and no impurity peaks were identified.

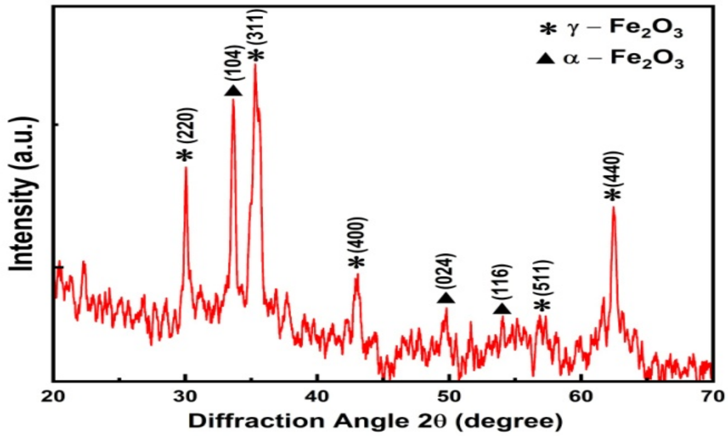

Figure-3: X-Ray Diffraction Analysis of green synthesized Iron-Oxide nanoparticles

Six peaks at 2θ values of 30.0737, 33.6583, 35.4403, 43.0806, 49.6763 and 62.4579 degreescorresponding to (220), (104), (311), (400), (024), and (440) planes of Fe_2O_3 are observed and compared with the standard powder diffraction pattern of Joint Committee on Powder Diffraction Standards (JCPDS # 39-1346, #33-0664). The proliferation of α-Fe_2O_3 crystals and promotes the growth of γ-Fe_2O_3 phase. Other authors have reported the same observations in the literature [24, 25].

XRD – Particle Size Calculation:

In the present study, considering the peak at degrees, average particle size has been calculated by using Debye-Scherrer formula [26, 27].

$$D = \frac{0.9\lambda}{\beta Cos\theta}$$

Where 'λ' is the wave length of X-Ray (0.1541862 nm), 'β' is FWHM (Full width at half maximum), 'θ' is the diffraction angle and 'D' is particle diameter size. The calculated particle size details are given below in the Table: 1

Table-1: The grain size of $\mathbf{Fe_2O_3}$ Nano Powder.

The Intense Peak Position '2θ' (Degrees)	**The Value of 'θ' in (Radians)**	**hkl**	**FWHM of Intense Peak 'β' (Degrees)**	**FWHM of Intense Peak 'β' (Radians)**	**Crystallite Size of Fe_2O_3NPs 'D' (nm)**	**Average Crystallite Size of Fe_2O_3 NPs (nm)**
30.0737	0.2624	(220)	0.7083	0.0124	12.1303	**10.0532**
33.6583	0.2937	(104)	0.7527	0.0131	11.5172	
35.4403	0.3093	(311)	0.7185	0.0125	12.1229	
43.0806	0.3759	(400)	1.9722	0.0344	4.5231	
49.6763	0.4335	(024)	0.8392	0.0146	10.8948	
62.4579	0.5450	(440)	1.0627	0.0185	9.1309	

C) Particle size analysis using SEM(Scanning Electron Microscopy) analysis:

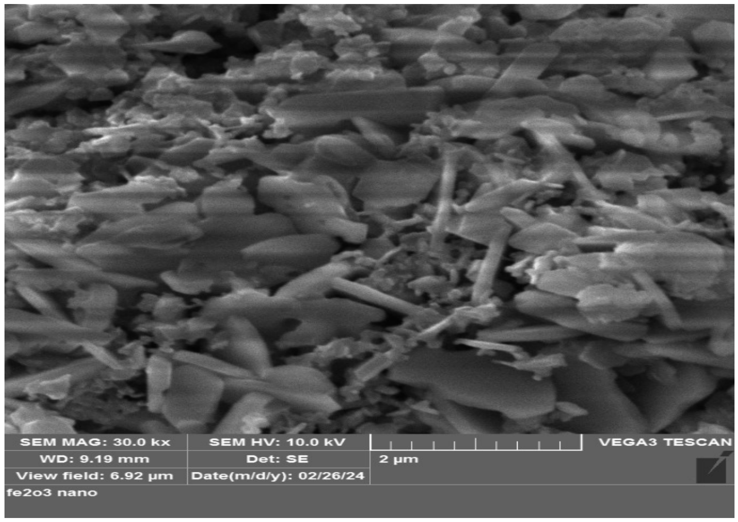

Figure-4: SEM image of green synthesized Iron-Oxide nanoparticles.

The physical structure of Fe_2O_3-NPs as shown in the figure-4 and it is quite tedious to confirm the particle size from the recorded SEM micrometers because of the highly agglomerated rod-like as well as plate like NPs with uniform distribution. The grain sizes of the samples estimated from the SEM picture is larger than that obtained from XRD data. This means that, the SEM picture indicates the size of polycrystalline particles. The SEM images were blurred as the instrument present at the research center was unable to take images of particles below 100nm.

The Fe_2O_3 nanoparticles elemental composition was further confirmed by the EDX spectrum, as shown in figure-5, which demonstrates the existence of Fe, and O peaks and the absence of any further impurity peaks confirms the produced samples' excellent purity and is in line with the XRD results.

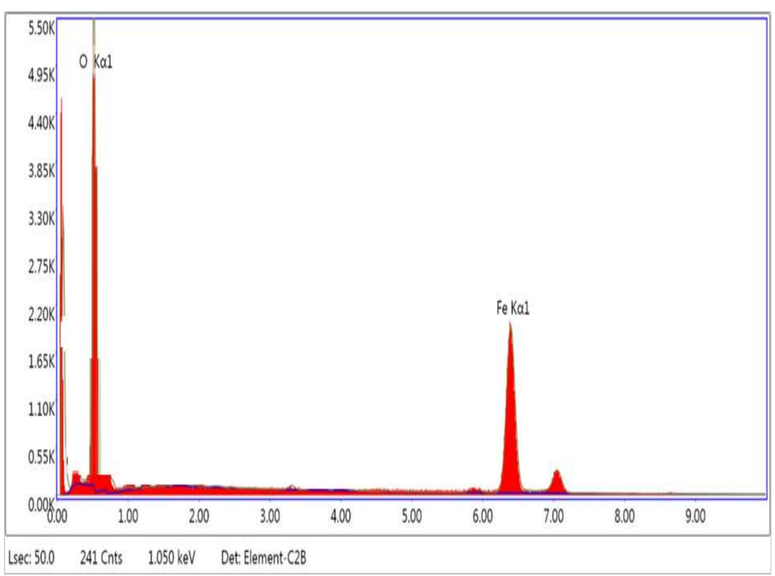

Figure-5: EDX spectrum of INOPs

D) FTIR analysis:

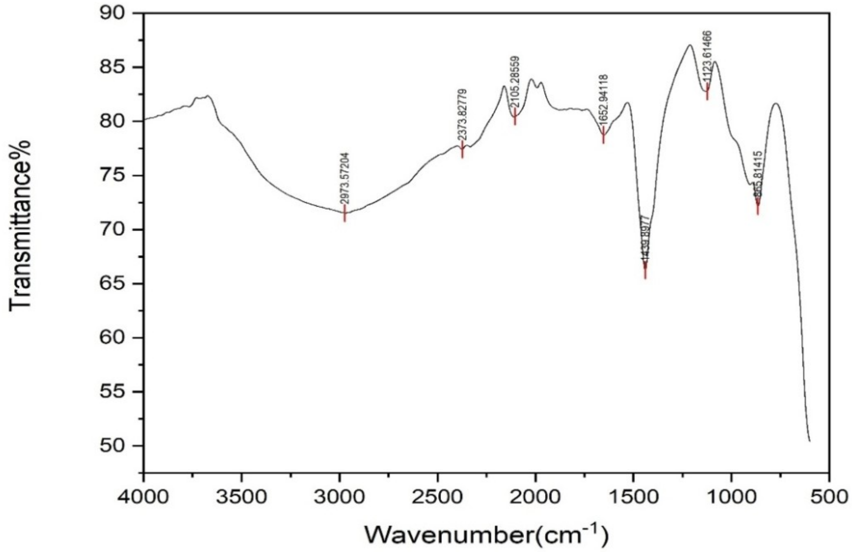

Figure-6: FTIR spectra of synthesized Iron-Oxide nanoparticles

With the help of FTIR measurements one can get the information around the local molecular environment of the organic molecules on the nanoparticles surface. These measurements were executed to detect the probable biomolecules which are accountable for capping and efficient stabilization of the metal nanoparticles synthesized by jack fruit leaves extract. Figure-6 shows the FTIR spectra of Fe_2O_3 NPs synthesized using leaf extract of different concentrations. And it is ascribed to functional groups (=C–H, C=O, N–O, C–O, C–N) present in the compound. The spectroscopic studies confirm the presence of amides, phenols, nitrogen, and aromatic compounds that has a strong binding affinity with Fe and thus play a significant role in reducing and capping ferrous ions. The spectrum reveals characteristic peaks at 2973.5 cm^{-1} stretching vibrations of C–H band, 1652.9 cm^{-1} stretching to N=O, 1123.6 cm^{-1} stretching to O–C, 865.8 cm^{-1} stretching to Fe–O stretches of Fe_2O_3 [28].

Conclusion:

Fe_2O_3 nanoparticles were successfully synthesized by using Artocarpus Heterophyllus extract and ferric chloride aqueous solution. The presence of phytochemicals in Artocarpus Heterophyllus was responsible for the formation of IONPs and it was confirmed with UV-Visible spectra. The crystalline nature of IONPs was evident from sharp peaks obtained from the X-ray diffraction. The functional groups present in the synthesized nanoparticles were identified using FTIR analysis. Further the synthesized nanoparticles are subjected to SEM analysis. The average crystallite size of IONPs was identified in the range of 10 to 11nm determined from XRD patterns. This echo friendly synthesis method of Fe_2O_3 nanoparticles may provide a quick, cheap and suitable alternative method.

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