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Green Chemical Technology to Synthesis and Characterization of Iron Nanoparticles from Extract of Marigold Flower

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Abstract

Green synthesis methods for producing nanoparticles have been explored due to the increasing demand for environmentally friendly and sustainable approaches in nanotechnology. In this work, Tageteserecta (Marigold) flower extract is used to synthesize and characterize iron nanoparticles (FeNPs), It serves as a stabilizing and reducing agent naturally. Traditional chemical methods often involve toxic reagents and energy intensive processes, which pose health and environmental risks. In contrast, the phytochemicals found in Marigold, such as flavonoids, phenols, and terpenoids, allow the reduction of Fe3+ ions to Fe^0 nanoparticles under mild conditions. The synthesis was conducted by reacting an aqueous solution of ferric chloride (FeCl3) with Marigold extract, optimizing parameters like temperature (60–80°C), time (2–4 hours), pH, and feed ratio (1:1 to 1:3).A number of characterization methods such as UV-Vis spectroscopy (absorption peak around 280–320 nm), FTIR (identifying functional groups responsible for reduction and capping),SEM/Edax (revealing spherical morphology and size) and were used to confirm the formation of FeNPs. The resulting nanoparticles ranged in size and stability from 20 to 50 nm, were impacted by the synthesis circumstances. This green synthesis approach demonstrates an efficient, low-cost, and ecologically benign route for iron nanoparticle manufacturing, contributing to sustainable nanotechnology practices and the valorization of floral biomass waste.

Keywords: Iron Nanoparticles, Mari gold flower, UV-Vis, FT-IR, Scanning Electron Microscopy, Edax.

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1. INTRODUCTION

Nanotechnology, particularly the creation of nanoparticles (NPs), has emerged as a transformational field due to the unique physicochemical features of nanoparticles at the nanoscale. These qualities include high surface area, reactivity, and quantum effects, which make nanoparticles beneficial in multiple applications spanning varied sectors, such as medicine, environmental remediation, electronics, and catalysis [1]. Because of their magnetic characteristics, biocompatibility, and catalytic activity, iron nanoparticles (FeNPs), and more specifically iron oxide nanoparticles (FeO_4), have drawn a lot of interest among different kinds of nanoparticles. Because of these characteristics, iron oxide nanoparticles are useful in fields like medicine delivery, environmental remediation, water treatment, and magnetic resonance imaging (MRI)[2-3].However, the traditional techniques for creating these nanoparticles frequently call for dangerous solvents and poisonous chemicals, which raise questions over their safety and sustainability in the environment [4]. The green synthesis of nanoparticles, which uses natural resources like plant extracts, is becoming more and more popular because it is environmentally friendly and avoids the costly reagents, high energy input, and hazardous chemicals that are used in conventional methods. In recent years, there has been a shift toward more sustainable, ecologically friendly methods of nanoparticle synthesis [5]. Bioactive substances like polyphenols, flavonoids, and alkaloids, which operate as reducing agents during the production of nanoparticles, are abundant in plants. These substances stabilize the nanoparticles, preventing aggregation and guaranteeing their dispersion, in addition to reducing metal ions to nanoparticles [6]. Because of its abundant phytochemical content and antioxidant qualities, marigold (*Tageteserecta*), a popular ornamental and medicinal plant, has become a desirable candidate for green synthesis in this context [7-8].

The ability of marigold flowers to produce stable nanoparticles has been documented in several studies focusing on the synthesis of silver, gold, and iron nanoparticles, and their use in nanoparticle synthesis is

particularly interesting because they are a natural source of bioactive compounds, such as carotenoids, flavonoids, and polyphenols, which have shown potential in reducing metal ions to nanoparticles [9-10].The synthesis of iron nanoparticles from Marigold extracts offers a promising green alternative to conventional methods of synthesis, with potential applications in areas such as drug delivery systems, water purification, and antimicrobial therapy. These compounds from the Marigold extract not only help in the reduction process but may also contribute to the stability, improved biocompatibility, and antioxidant activity of the nanoparticles [11-12].

The green production of nanoparticles using plant extracts has several advantages, including being easy, affordable, and environmentally friendly, especially Marigold flowers. The technique commonly uses plant extracts as reducing agents to decrease metal salts, like ferric chloride, to form iron nanoparticles, which is beneficial because it does not require high-energy inputs or dangerous chemicals, making it safer and more sustainable [13]. The polyphenols in the Marigold extract may help stabilize the nanoparticles, avoid agglomeration, and improve their functional characteristics in addition to lowering metal ions [14]. The resultant iron nanoparticles have demonstrated exceptional stability and dispersibility, both of which are essential for their successful use in biomedical and environmental domains [15].

Despite the increased interest in the green manufacturing of nanoparticles, the exact mechanisms underpinning the reduction and stabilization of metal ions by plant extracts remains little known. Optimizing the manufacture of iron nanoparticles with the appropriate characteristics requires the discovery of particular bioactive components in marigold extract that aid in the reduction process. Furthermore, It may be possible to control the stability, size, and shape of the generated nanoparticles by understanding how these materials interact with the metal ions throughout the nanoparticle creation process [16]. To learn more about the characteristics of Marigold mediated iron nanoparticles, a number of investigations have used characterization techniques, scanning electron microscopy (SEM), Edax, Fourier transform infrared spectroscopy (FTIR), and UV-visible spectroscopy[17].These methods provide useful details regarding the size, shape, crystalline structure, and chemical composition of the nanoparticles all of which are essential for their efficient application.

Apart from their synthesis and characterization, Marigold mediated iron nanoparticles, which have a wide range of antimicrobial activity, are of great interest for their potential uses in medicine as a substitute for traditional antibiotics[18].Because iron oxide nanoparticles may absorb organic and heavy metal pollutants, they are also used in water treatment, providing a long term solution to environmental contamination[19].Additionally, iron nanoparticles' magnetic characteristics allow for targeted medication administration, in which an external magnetic field can guide the particles to particular bodily areas[20].

2. MATERIALS AND METHODS

2.1 Preparation of Marigold Flower Extract:

Following thorough cleaning, 100 milliliters of distilled water were mixed with 300 grams of fresh marigold flowers. To get a transparent extract of marigold flowers, the mixture was filtered through a fine mesh screen after being allowed to cool.Using a Soxhlet apparatus, the mixture was left for 20 minutes to extract the bioactive components.

2.2. Synthesis of Ferric Chloride Nanoparticles:

Ferric chloride ($FeCl_3$) was dissolved in 300 milliliters of distilled water to create a 0.1 M solution. The ferric chloride solution was gradually mixed with 300 milliliters of marigold flower extract while being constantly stirred. After an hour of heating the reaction mixture to 60°C, when the hue changed from pale yellow to dark brown, it indicated that nanoparticles had formed. After cooling the resultant suspension, nanoparticles were extracted using centrifugation for 15 minutes at 10,000 rpm. After being repeatedly cleaned with distilled water to get rid of any contaminants, the nanoparticles were allowed to dry at room temperature (1.78).

2.3. Characterization of Nanoparticle

2.3.i). UV-VisSpectroscopy

An essential analytical method for characterizing iron nanoparticles (FeNPs) is UV-Visible (UV-Vis) spectroscopy, which uses Surface Plasmon Resonance (SPR) to reveal information on the particles' formation, stability, and optical characteristics[21].Depending on the size and oxidation state of the nanoparticles, the characteristic absorption peak that results from the interaction of FeNPs' conduction electrons with incident light is usually seen between 300 and 500 nm.[22].A red shift (towards higher wavelengths) suggests larger or aggregated nanoparticles, while a blue shift (towards lower wavelengths) indicates smaller nanoparticles [23]. The absorbance intensity and nanoparticle concentration are connected; a higher intensity indicates a higher concentration of FeNPs in solution [24]. A monochromator, a sample holder (usually a quartz cuvette), a light source (deuterium and tungsten-halogen lamps), and a detector that measures light absorption in the 200–800 nm range make up the UV-Vis spectrophotometer [25]. Prior to scanning the nanoparticle solution, a blank reference (such as distilled water or plant extract alone) is utilized for baseline correction in order to examine FeNPs[26]. UV-Vis spectroscopy is a quick, nondestructive, and crucial method in nanomaterials research since the existence of an SPR peak verifies the creation of FeNPs and enables real-time monitoring of their synthesis [27].

2.3.ii). FTIR Analysis

Fourier Transform Infrared (FT-IR) spectroscopy is widely used in the green synthesis of iron nanoparticles (FeNPs) to identify functional groups that are involved in the reduction and stabilization process. The existence of biomolecules

from plant extracts is confirmed by FT-IR spectra, which usually display peaks around 3400 cm^{-1} (O-H stretching from hydroxyl groups), 1600 cm^{-1}(C=O stretching from carbonyl groups), and 1000–1400 cm^{-1} (C-O stretching from proteins or polysaccharides). Around 500–600 cm^{-1}, the distinctive FeO bond emerges, signifying the development of nanoparticles. Research like that conducted by have shown that FTIR is an essential technique for assessing biologically produced FeNPs, guaranteeing their stability and functionality for a range of applications.[28-29].

2.3.iii).Energy Dispersive X-ray Analysis, or EDAX

A sophisticated technique for determining a material's elemental composition at the microscopic level is energy dispersive X-ray analysis, or EDAX [30]. It is commonly used in conjunction with scanning electron microscopy (SEM) or transmission electron microscopy (TEM) to provide structural and compositional information on nanoparticles [31].The fundamental principle of EDAX is that unique X-rays are created when high-energy electrons interact with atoms in a sample, allowing for precise elemental identification[32].EDAX is crucial for verifying the presence of iron (Fe) and detecting additional elements like oxygen (O) and carbon (C), which could suggest oxidation or the presence of plant-derived capping agents,using marigold extract in the environmentally friendly synthesis of iron nanoparticles (FeNPs)[33]. The EDAX spectrum provides a visual representation of the identified elements, which shows peaks that correspond to their distinctive X-ray energies [34]. Successful nanoparticle creation is confirmed by a prominent Fe peak, while other peaks point to potential oxidation (FeO/FeO_3) or organic residue from the plant extract [35].

Because EDAX is quick, non-destructive, and extremely sensitive, it is frequently utilized in material science [36]. Surface sensitivity, difficulties detecting light components (such hydrogen and lithium), and potential peak overlaps that need careful interpretation are some of its drawbacks [37]. Notwithstanding these difficulties, EDAX is still a vital instrument for characterizing green-synthesized FeNPs, guaranteeing their purity and compositional precision for a range of uses [38].

2.3.iv). SEM Analysis (Scanning Electron Microscope)

A popular method for examining the size, shape, and structure of nanoparticles' surfaces is scanning electron microscopy, or SEM[39].SEM operates by focusing a high-energy electron beam onto the sample surface, which creates secondary and backscattered electrons, creating high-resolution pictures[40]. This method is crucial for material characterization since it offers comprehensive details regarding the distribution, aggregation, and morphology of nanoparticles[41].

SEM is essential for verifying the successful formation of iron nanoparticles (FeNPs) in the green synthesis process employing marigold extract [42]. The resulting images provide information on the synthesis efficiency by assisting in the determination of particle size, surface roughness, and dispersion patterns [43]. The existence and purity of FeNPs are ensured by the elemental composition analysis made possible by the combination of SEM and Energy Dispersive X-ray Analysis (EDAX) [44].

SEM analysis is particularly useful due to its high magnification capabilities, depth of field, and ability to offer three-dimensional pictures [45]. Nevertheless, there are drawbacks, such as the need for sample preparation (coating non-conductive materials with a conductive layer), the possibility of beam damage to delicate samples, and resolution limits in contrast to Transmission Electron Microscopy (TEM) [46].Despite these obstacles, SEM is a vital technique in nanoparticle research, delivering valuable structural and morphological insights [47].

3. RESULTS AND DISCUSSION

3.1. UV-Visible (UV-Vis)

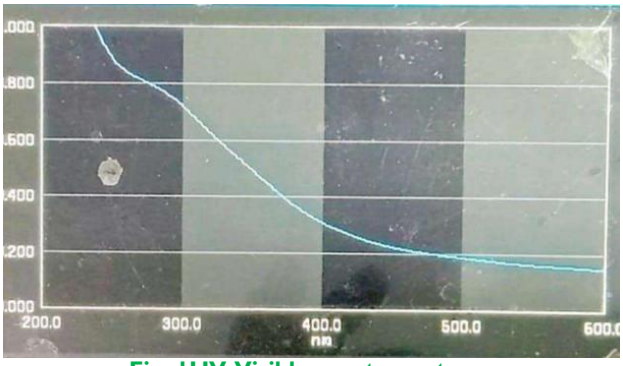

Fig: 1UV-Visible spectrometry

A UV-Visible (UV-Vis) absorption spectrum is shown by this graph, which is frequently used to examine the optical characteristics of materials. The spectrum, spanning 200-600 nm, shows strong absorption in the UV range (~200-300 nm), followed by a significant drop in absorbance between 250-400 nm, suggesting electronic transitions or an optical band gap. Beyond 500 nm, absorption levels off, indicating minimal absorption in the visible range. This suggests the material could be an inorganic or organic semiconductor, a metal oxide, or nanoparticles, with metal oxides like ZnO or TiO2 showing strong UV absorption due to their wide band gaps, and quantum dots or nanoparticles potentially displaying blue shifts or surface plasmon resonance. The band gap can be estimated using the Tauc plot method,This uses a particular equation to connect the band gap energy (Eg), photon energy (hv), and absorption coefficient (α). The band gap usually varies between 1.9 and 2.3 eV for indirect band gap materials such as iron oxide (Fe2O3, Fe3O4), where n is ½. Using the equation Eg(eV) = 1240/λonset(nm), the band gap energy is calculated from the onset wavelength (λonset). For λonset of 400 nm, the band gap is approximately 3.1 eV, and at 500 nm, it decreases to around 2.48 eV, providing insights into the material's optical properties and electronic structure.

Possible Iron Phases

Phase	Adsorption Peak Range	Band Gap (Eg in eV)
Hematite (α-Fe2O3)	~250-350 nm	2.1-2.3 eV
Magnetite (FeO4)	~200-300 nm	0.1-0.2 eV(metallic behavior)
Maghemite (γ-Fe2O)	~250-400 nm	2.0-2.2 eV

If the spectrum shows an onset around 400 nm or lower, it suggests a material with a band gap between 2.0–2.5 eV, likely hematite (α-Fe2O3) ormaghemite (γ-Fe2O3). To determine the band gap (Eg), the wavelength is first converted to energy using the equation E = hc/λ. Then, for direct band gap semiconductors, a plot of $(\alpha h\nu)^2$ versus hν is created, and the band gap is found by extrapolating the linear region to the x-axis intercept. Techniques like Energy Dispersive X-ray Spectroscopy (EDX) for elemental analysis, X-ray Diffraction (XRD) for crystalline structure, and Fourier Transform Infrared Spectroscopy (FTIR) for identifying functional groups can provide additional confirmation even though UV-Vis alone cannot directly determine elemental composition. Instruments such as the Bruker ALPHA II, which has a range of 500–3500 cm-1 and operates in transmittance mode, are commonly used for FTIR analysis.

3.2. FTIR spectrum

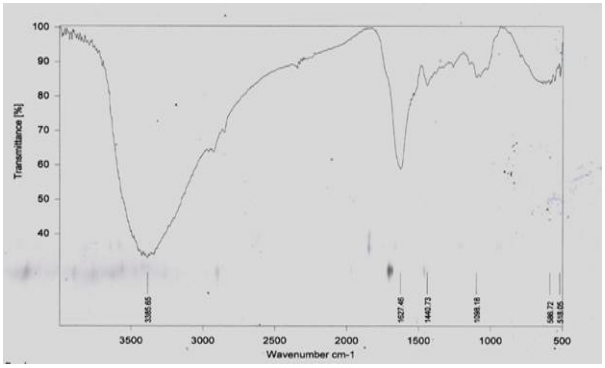

Fig: 2 FTIR spectra

The existence of particular functional groups is indicated by major absorption bands in the sample's FTIR spectrum. O-H stretching is represented by a wide absorption band at 3385.65 cm-1, indicating the presence of hydroxyl (-OH) groups in alcohols and phenols, or carboxylic acids, where a strong hydrogen bond is indicated by the broadness. C=O stretching is used to explain a prominent peak at 1627.46 cm-1, characteristic of carbonyl-containing compounds such as ketones, aldehydes, or amides.C-H bending vibrations, which are frequently seen in aliphatic or aromatic hydrocarbons, are linked to the peak at 1440.73 cm-1. Esters are present because of a significant absorption at 1098.18 cm-1 attributed to C-O stretching, ethers, or polysaccharides. Peaks at 586.72 and 518.05 cm-1 may correspond to C-X stretching, suggesting halogenated compounds or metal-oxygen bonds. The combination of broad O-H stretching, along with carbonyl and ether functionalities, implies that the sample may contain hydroxyl, carbonyl, and ether groups, pointing to a polymeric structure, ester-based compounds, or carbohydrate derivatives. The absence of peaks in the 2100–2300 cm-1 range rules out nitrile (-C≡N) or alkyne (-C≡C) groups. Comparative analysis with reference FTIR spectra suggests the sample could belong to a class of organic molecules such as natural polysaccharides, synthetic polyesters, or carboxyl-functionalized polymers. Further characterization through complementary techniques like NMR spectroscopy or mass spectrometry is recommended for more precise structural identification.

3.3. EDAX RESULT

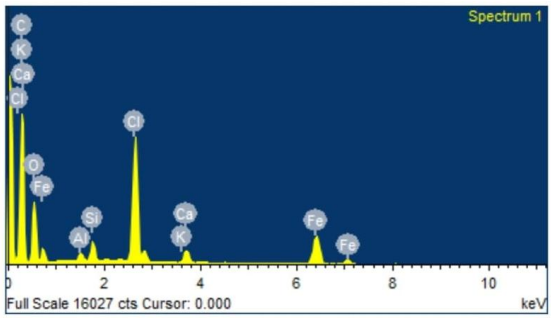

Fig: 3 Energy Dispersive Analysis X-ray

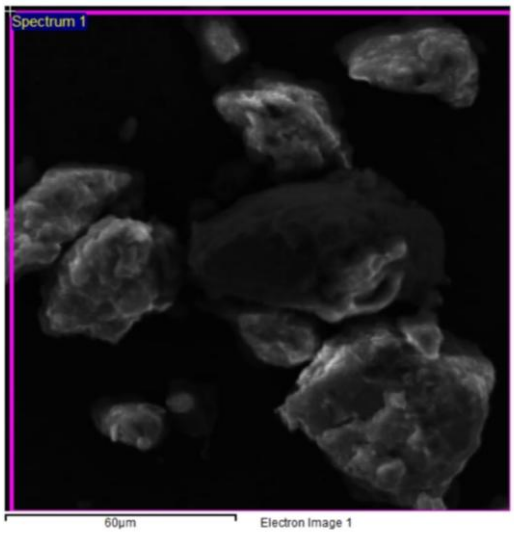

Fig: 4 SEM

The FTIR spectrum of the sample reveals peaks at 586.72 cm-1 and 518.05 cm-1, which are likely due to Metal-Oxygen (Fe-O) stretching vibrations, indicative of iron oxides such as Fe2O3 or Fe3O4, suggesting the presence of FeO bonds characteristic of iron-containing minerals. Iron (Fe), oxygen (O), silicon (Si), aluminum (Al), calcium (Ca), potassium (K), chlorine (Cl), and carbon (C) all have peaks in the EDS spectrum that indicate a complex composition, further confirming the elemental makeup. The presence of Fe and O indicates iron oxide compounds, while Si, Al, Ca, and K suggest silicates or aluminosilicates, such as clay minerals or feldspar derivatives, and chlorine may indicate surface contamination or chloridebased compounds. Carbon could arise from organic residues, sample coating, or atmospheric contamination. The SEM image provides further insight into

the sample's morphology, showing irregularly shaped, agglomerated particles with rough and porous surfaces, ranging in size from a few micrometers to tens of micrometers (~60 µm scale bar), indicating a natural mineral, industrial by product, or synthesized material likely influenced by mechanical fragmentation or chemical processing. Overall, the analysis suggests the sample is composed primarily of iron oxide compounds, with possible silicate or clay mineral content, and exhibits a heterogeneous, agglomerated structure.

4. CONCLUSION

The current study successfully proved a green synthesis approach for producing iron nanoparticles (FeNPs) by using Tageteserecta (Marigold) flower extract as a natural reducing and stabilizing agent. By avoiding the use of hazardous reagents and using less energy, this environmentally friendly technique provides a sustainable substitute for traditional chemical synthesis. Under mild conditions, the phytochemicals found in marigold, such as flavonoids, phenols, and terpenoids, were crucial in reducing Fe3+ ions to FeOnanoparticles.The tuning of synthesis parameters, including temperature, reaction time, pH, and feed ratio, had a significant impact on the size and stability of the FeNPs, which were described as spherical in shape and ranged in size from 20 to 50 nm. The effective synthesis, structure, and dispersion of the nanoparticles were validated by thorough characterization methods including UV-Vis, FTIR, Edax, and SEM. The possibility of using floral waste in the synthesis of nanomaterials is highlighted by this study, which supports waste valorization and sustainable nanotechnology.

5. AUTHORS CONTRIBUTION STATEMENT

R.Selvaraj designed whole study, S.Naveen conducted chemical analysis, E.Bharath characterized nanoparticles, R.Kabilraj designed sample collection, and R.Praveen kumar prepared part of manuscript

6. CONFLICT OF INTEREST

Conflict of interest declared none

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