

Microwave-Assisted Green Synthesis of Fe₂O₃-Tc Nanoparticles by Giloy (T. Cordifolia) Stem's Extract

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Abstract: Growing environmental concerns have driven the development of sustainable nanoparticle synthesis methods, with microwave-assisted green synthesis emerging as an efficient and eco-friendly approach. This technique combine rapid, uniform microwave heating with green chemistry principles, decreases need of excess energy, waste production, and the use of high-risk chemicals. In this study, *Tinospora cordifolia* stem extract used as a reducing and stabilizing agent, while ferric (III) chloride (FeCl₃) used as the precursor reagent for the synthesis of iron oxide nanoparticles. Bioactive phytochemicals such as phenolics and flavonoids facilitated the reduction of Fe³⁺ ions and prevented particle agglomeration. UV-Visible spectroscopy (200–800 nm) confirmed nanoparticle formation, showing a characteristic absorption peak at 341.6 nm. SEM (Scanning electron microscopy) explained the particles within the broad size range of 30–65 nm and variable surface morphology. Overall, microwave irradiation enhanced reaction kinetics and enabled rapid, controlled, and sustainable production of iron oxide nanoparticles.

Keywords: Nanoparticles, Microwave, Green Chemistry, Environmental Remediation, phytochemical, therapeutic.

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I. INTRODUCTION

Nanoparticles have attracted significant attention across many scientific and technical areas due to their exceptional physicochemical properties & broad spectrum of applications [1]. Their nanoscale dimensions impart unique optical, magnetic, catalytic, electrical, and biological characteristics that differ markedly from bulk materials [2]. These distinctive properties have enabled their use in drug delivery systems, biosensors, antimicrobial formulations, wastewater treatment, catalysis, and energy storage devices [3]. However, despite these advantages, conventional approaches to nanoparticle synthesis—such as chemical reduction, sol-gel processing, hydrothermal synthesis, and thermal decomposition—often require toxic solvents, hazardous reducing agents, high temperatures, and energy-intensive conditions. Such practices not only generate significant chemical waste but also raise serious concerns regarding environmental sustainability and human health safety.

In response to these limitations, researchers have increasingly adopted green synthesis strategies that emphasize sustainability, environmental compatibility, and resource efficiency [4]. Green synthesis combine within the core 12 principles of green chemistry, which advocate reduce

hazardous substances, reducing waste generation, and improving energy efficiency in chemical processes. Among the various eco-friendly techniques developed, microwave-assisted green approach has rising as a highly encouraging methodology [5]. This technique combines the environmental benefits of biological reducing agents with the operational advantages of microwave irradiation.

Microwave irradiation offers rapid, uniform, and volumetric heating of reaction mixtures through direct interaction of microwave energy with polar molecules and ions present in the solution. Far from conventional heating techniques that depend on conduction and convection, microwave (electromagnetic) heating ensures homogeneous temperature distribution, thereby accelerating nucleation and growth processes of nanoparticles [6]. These results in shorter reaction times, improved reaction kinetics, enhanced product yields, and better control over particle morphology. Furthermore, microwave-assisted methods significantly reduce energy consumption and reaction duration, contributing to a lower environmental footprint [7].

In microwave-assisted green synthesis, environmentally benign precursors and natural reducing agents are employed to mitigate environmental impact. These

green reducing agents include plant extracts, natural polymers, polysaccharides, proteins, enzymes, and other renewable biological resources [8–10]. Plant extracts are particularly advantageous because they contain various phytochemicals such as phenolics, carbohydrates, terpenoids, saponins, flavonoids and alkaloids, which can behave simultaneously as capping, reducing and stabilizing, agents. This multifunctional capability eliminates the need for additional synthetic stabilizers or surfactants, making the overall synthesis process simpler, cost-effective, and eco-friendly [11]. Leveraging the reducing capabilities of these natural agents enables nanoparticle synthesis under milder reaction conditions, thereby reducing decesses on hazardous chemicals and higher-energy consumption [5].

In this study, ferric (III) chloride (FeCl_3) was used as the reaction precursor reagent for the formation of Iron Oxide nanoparticles, while extract of *Tinospora cordifolia*'s stems served like the reducing and stabilizing agent. *Tinospora cordifolia*, a medicinal plant native to China and the Indian subcontinent, has gained substantial scientific interest due to its extensive therapeutic applications and commercial significance [3]. Over the past four decades, numerous bioactive compounds have been extracted from *Giloy*, including alkaloids, polysaccharides, steroids, diterpenoids, aliphatic compounds, and phenolic compounds. These phytoconstituents exhibit an extensive range of medicinal applications, such as hepatoprotective, immunomodulatory, antioxidant, hypoglycemic, antiinflammatory, and antimicrobial consequences [12]. The presence of such bioactive molecules enhances the reduction of Fe^{3+} ions and facilitates stability of the synthesized nanostructures through surface capping mechanisms.

Size and morphology of the *Giloy* mediated nanostructures can be effectively control by adjusting microwave variables such as reactant concentration, reaction time, pressure microwave power and temperature. Fine-tuning these parameters influences the nucleation and growth phases, thereby enabling controlled synthesis of nanoparticles with desired dimensions and structural characteristics [13].

Characterization of the prepared iron oxide nanostructures was done by the using UV–Visible spectrophotometry and scanning electron microscopy (SEM) [13]. UV–Vis spectrophotometric analysis revealed the spectral profile of the synthesized nanoparticles within the wavelength range of 200–800 nm, exhibiting a characteristic absorption peak at 341.6 nm, which confirms nanoparticle formation. SEM analysis provided detailed insights into particle morphology, surface texture, and size distribution. Additionally, Energy-Dispersive X-ray Spectroscopy (EDS) confirmed the element percentage and purity of the prepared iron oxide nanoparticles derived from ferric chloride and *T. cordifolia* stem extract. Overall, the study demonstrates an efficient, rapid, and environmentally sustainable approach for synthesizing iron oxide nanostructures with controlled properties and potential uses in biomedical and environmental fields [8].

II. METHOD AND EXPERIMENT OF MICROWAVE-ASSISTED VIA GREEN SYNTHESIS

The synthesis of nanoparticles via microwave-assisted green methods typically involves the following steps:

➤ *The Selection of Green Precursors and Reducing Agents:*

Phytochemical screening plays a crucial role in identifying bioactive constituents present in plant residual extracts, which was often accountable for their therapeutic and reducing properties in green nanoparticle synthesis [2]. In the present analysis, several important classes of phytochemicals were detected using standard qualitative tests.

- Alkaloids were identified using Mayer's reagent, which produces a cream or pale-yellow precipitate indicating their presence. Alkaloids are well known for their significant pharmacological activities, including analgesic, anti-inflammatory, and antimicrobial effects. Their nitrogen-containing structures often contribute to strong biological interactions.
- Carbohydrates were confirmed through Benedict's test, where a color change indicates the presence of reducing sugars. Carbohydrates play a vital role in energy production and metabolism and are also associated with immune-enhancing properties in medicinal plants.
- Cardiac glycosides were detected using Borntrager's test, confirming compounds that are traditionally associated with cardiovascular benefits. These compounds are known to improve cardiac output and help stabilize heart rhythm, making them important in heart-related therapies.
- Flavonoids were identified using the alkaline reagent test, which produces an intense yellow coloration. Flavonoids are powerful antioxidants and exhibit anti-inflammatory and antimicrobial activities. They are also known to scavenge free radicals and reduce oxidative stress.
- Proteins were detected through the Ninhydrin test, which gives a characteristic purple coloration. Proteins are essential for tissue repair, enzymatic activity, and immune function, and may also assist in nanoparticle stabilization.
- Resins were indicated by ferric chloride testing, demonstrating anti-inflammatory, antimicrobial, and antioxidant properties. Saponins were detected using lead acetate, and are associated with cholesterol-lowering effects, immune support, and antimicrobial activity.
- Steroids were identified through characteristic color changes with chloroform and acids, suggesting anti-inflammatory and antidiabetic potential. Finally, tannins were confirmed using ferric chloride, and are known for wound healing, immune modulation, and reduction of oxidative stress.

➤ *Preparation of Plant Residual Extract:*

The *Giloy* stems were collected from the garden of boys' hostel of R. B. S. Engineering and Technical Campus, Bichpuri, Agra, India. Collected stems of *T. Cordifolia*, wash 3-4 times to remove dirt and other contaminant particles. Then dissect the stems into small pieces about 1-2 inches

long, after cutting, dry them in an air oven over the 10 minutes and soak them in open air for 15 minutes. Repeat this phenomenon till the stems are fully dried. After drying, crush them and convert the stems into powder form. 10gms of stem powder was liquefied in a 250ml beaker containing 200 ml water. Boil it for 30 minutes or till the solution is decreases to half of its original volume. Filter them by the use of Whatman no. 1 filter paper and stored at 4°C for next uses [3-4].

➤ *Preparation of Reaction Precursor:*

Ferric chloride (FeCl_3), purchased from Merck, was selected as the reaction precursor reagent for the formation of iron oxide nanostructures. A 0.5 M solution of the iron salt was prepared using distilled water to ensure accurate concentration and homogeneity for the synthesis method. The giloy stem powder extract of *Tinospora cordifolia* was utilized as a natural reducing and stabilizing agent due to its rich phytochemical composition. For the reaction, 20 mL of the prepared 0.5 M ferric chloride stock solution was mixed thoroughly into 30 mL of the stem extract solution under continuous stirring to achieve uniform mixing. The resulting mixture was subsequently subjected to microwave irradiation to facilitate rapid reduction and nanoparticle formation [5–7].

➤ *Microwave Irradiation and Synthesis:*

Exposure to microwave irradiation causes a rapid and uniform increase in the temperature of the reaction mixture, which accelerates nucleation and promotes the subsequent growth of nanoparticles [8]. Unlike conventional heating, microwave energy directly interacts with polar molecules and ions in the solution, resulting in efficient volumetric heating and enhanced reaction kinetics. Careful adjustment of critical reaction parameters—such as microwave power, temperature and irradiation period—allows precise control over particle size, morphology, and distribution, thereby optimizing nanoparticle synthesis. In this study, the prepared iron salt solution and plant extract were mixed and continuously stirred for 20 minutes to ensure homogeneity. The resulting mixture was now employed in a microwave domestic oven and heated on 230°C till 5 minutes. A distinct color change from reddish-yellow to brownish was observed after approximately 30 minutes, indicating the successful reduction of Fe^{+3} ions and the synthesis of iron oxide nanostructures from the combined iron salt and extract solution [9].

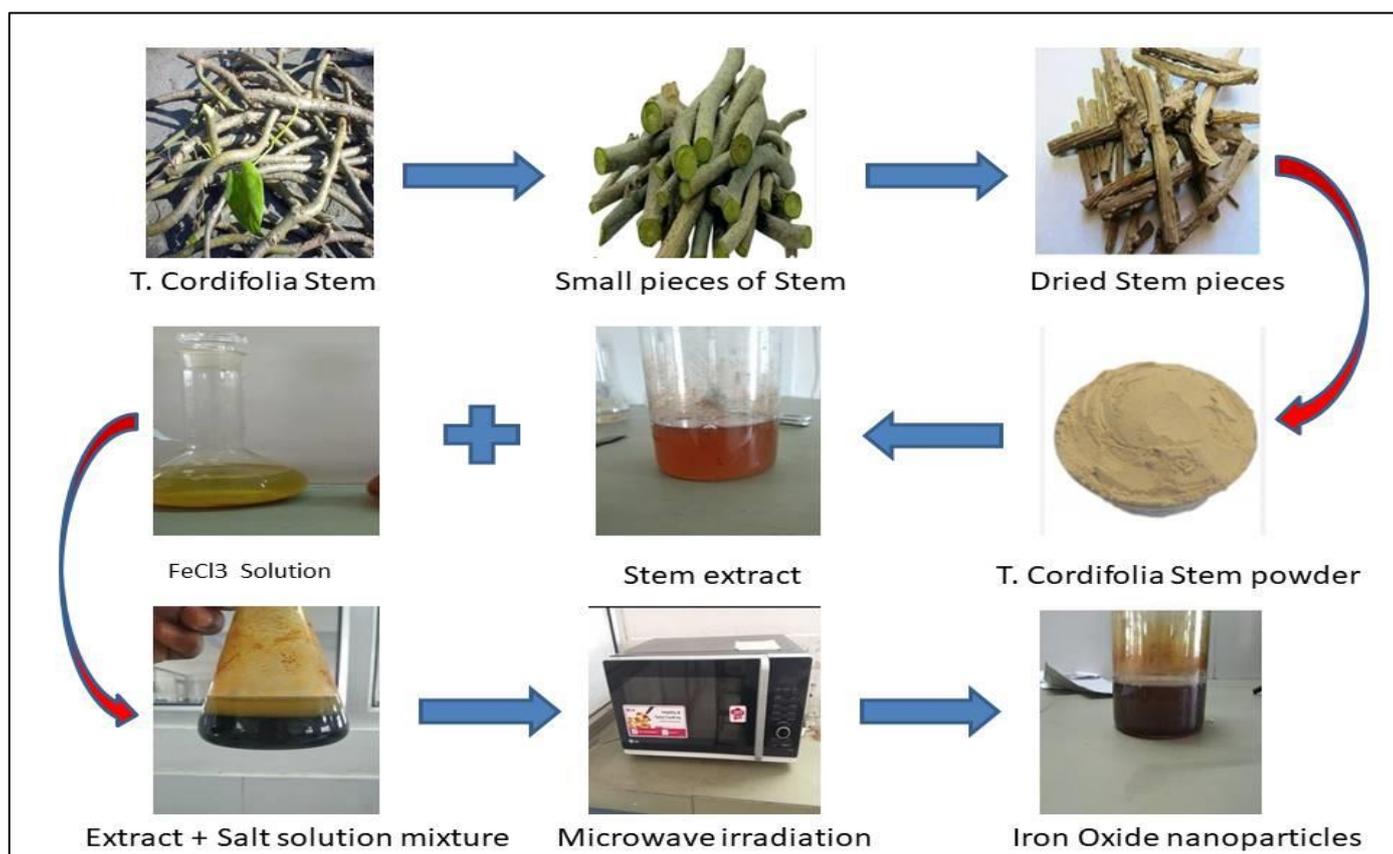


Fig1 Schematic Diagram of Iron Oxide Nanoparticles Synthesis

III. CHARACTERISTICS, RESULTS AND DISCUSSION

The synthesized iron oxide nanoparticles underwent characterization utilizing techniques including SEM and UV-Vis spectrophotometry [10]. Iron oxide nanoparticles with sizes range from 30 to 65 nm were successfully synthesized,

with an average size of approximately 30 nm, utilizing ferric (III) chloride salt solution and T. Cordifolia stem extract. UV-Vis spectrophotometry analysis revealed the formation of the synthesized iron oxide nanoparticles across the frequency range from 200nm to 800nm, with a peak observed at 341.6 nm with 0.791 Absorbance.

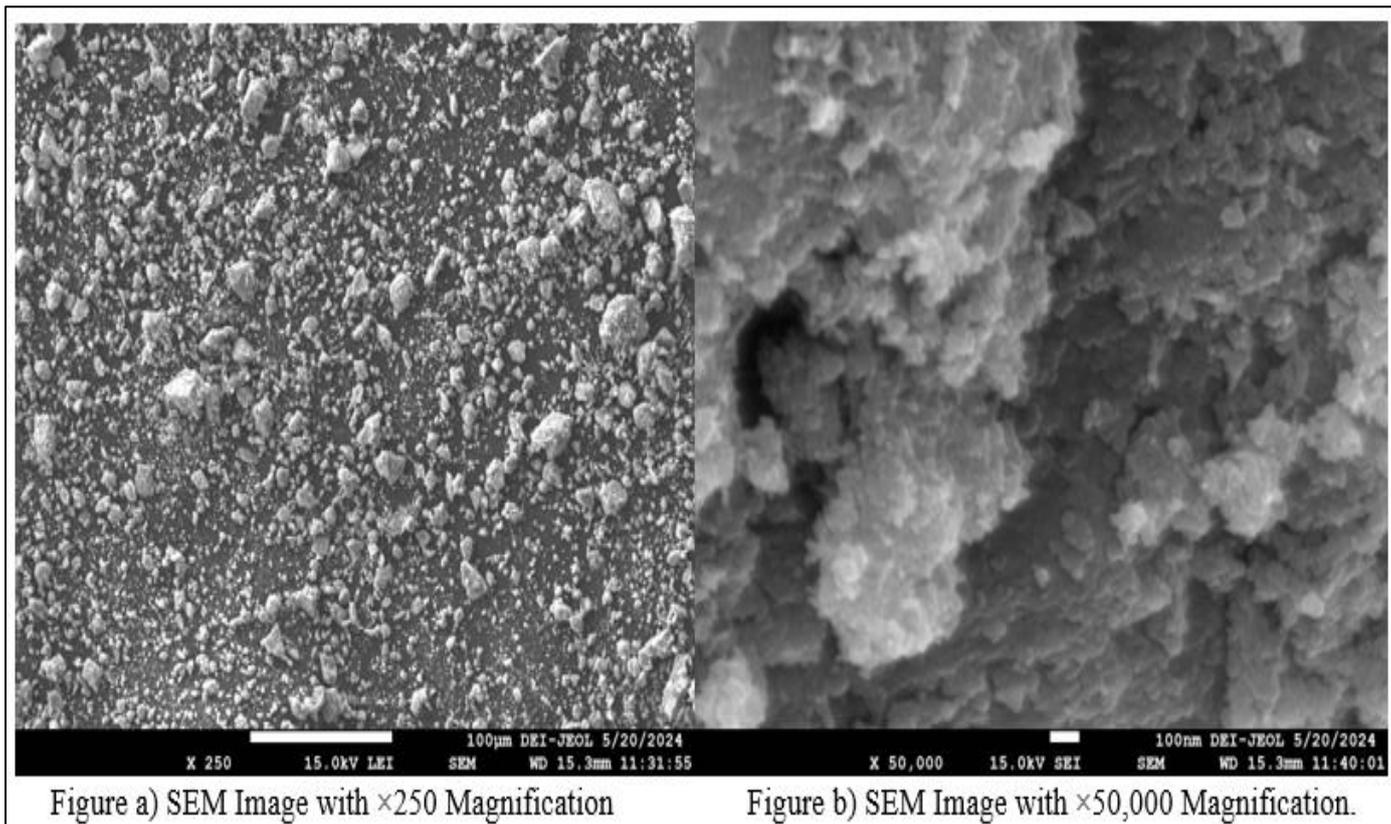


Figure 2 a) and b) Present Information Regarding the Synthesized Iron Oxide Nanoparticles at Magnifications of $\times 250$ and $\times 50,000$. Notably, these Images Suggest Incomplete Sample Grinding, as Evidenced by the Observed Large Variation in Particle Size.

SEM imaging confirmed the synthesis of Iron oxide nanoparticles, displaying a wide dimensional distribution, attributed to the use of *T. cordifolia* stem extract. Additionally, the magnetic behaviour of the synthesized iron

oxide nanoparticles was verified by a magnetic bar [11]. EDS imaging provided insight into the chemical composition and novelty of the synthesized iron oxide nanoparticles, representing a major composition of approximately 61.1%.

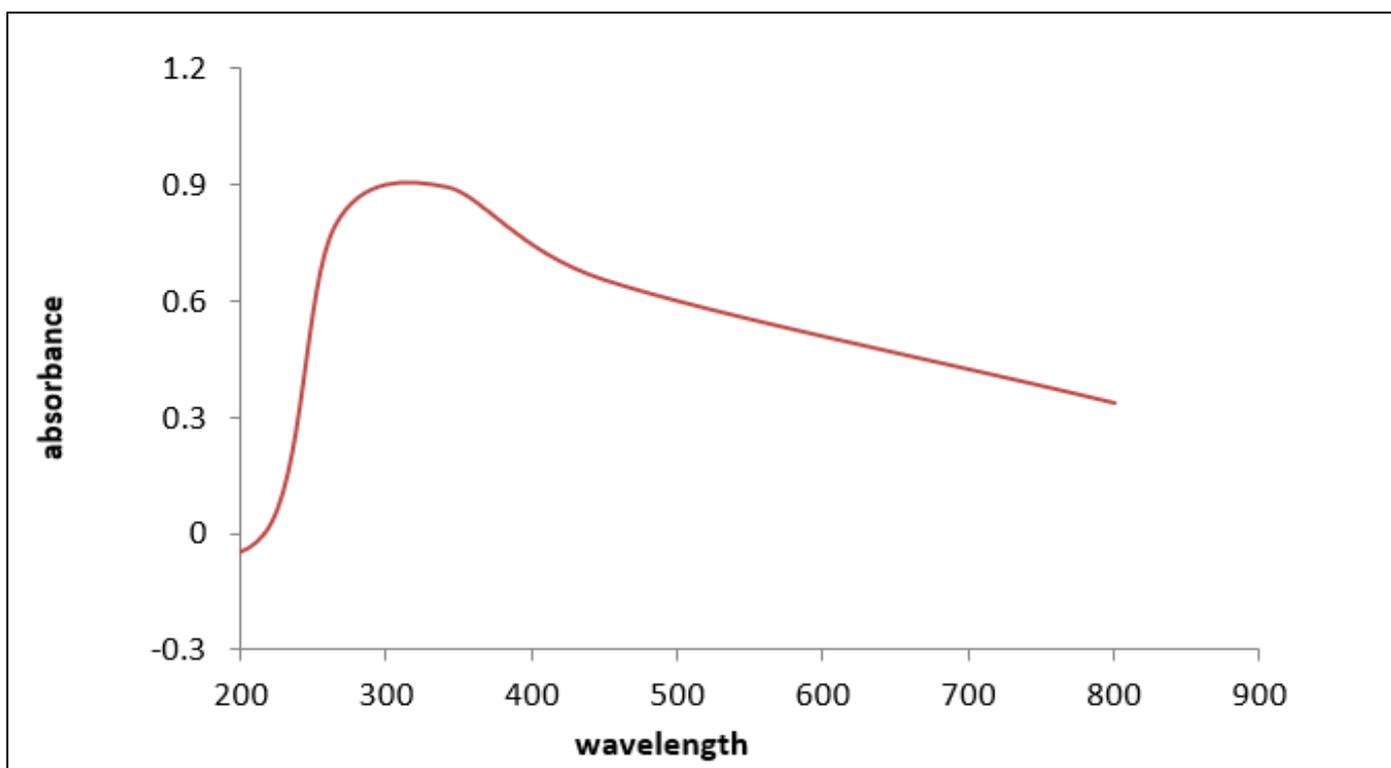


Fig 3 UV-Vis Spectral Diagram of Iron Oxide Nanoparticles

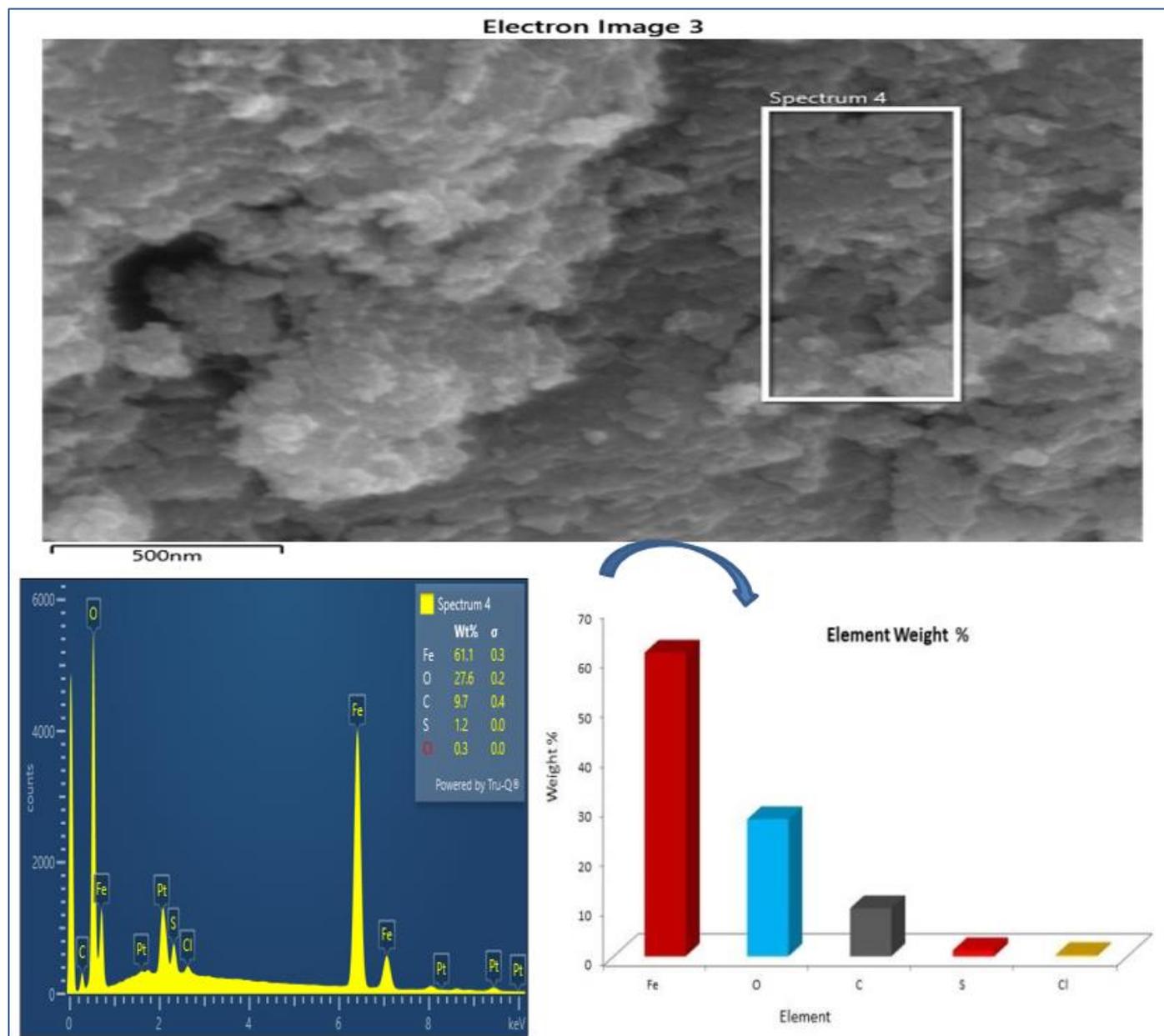


Fig 4 EDS Analysis Images of Iron Oxide Nanostructures.

Using an Ultra Violet–visible spectrophotometer (Systonics Double Beam Spectrophotometer 2203), the optical and absorbance characteristics of the prepared Iron oxide nanoparticles (NPs) were analysed. A prominent absorption peak at 341.6 nm was detected in the UV–Vis spectra of the iron oxide NPs solution (Figure C). This peak is a characteristic feature that arises because of surface plasmon resonance (SPR) occurring on the iron oxide Nanoparticles. The presence of this peak confirms the effective formation of Iron oxide nanoparticles and their unique optical properties. These outcomes get supported by previous research reports, which indicate that iron oxide NPs typically exhibit a characteristic absorption band around 300 nm [14-15]. The slight variation in the absorption peak position could be attributed to differences in synthesis methods, particle size, surface modifications, or aggregation of the nanoparticles.

The morphology of the synthesized Iron oxide nanostructures was further studied using scanning electron microscopy (SEM). SEM images (Figure a & b) revealed that the nanoparticles exhibited a predominantly spherical morphology with a particle size range of 30 to 65 nm when observed at 50,000 times magnification. The size distribution of the nanoparticles is an essential parameter as it influences their physical and chemical properties, including stability, reactivity, and interaction with biological molecules [5].

In addition to SEM analysis, elemental composition analysis was carried out using Energy Dispersive Spectroscopy (EDS) to conclude the elemental arrangement of the synthesized nanoparticles. The Energy Dispersive Spectroscopy (EDS) spectrum (Figure d) revealed the presence of key elements, including carbon, oxygen, iron, and sulfur, with weight percentages of 9.7%, 27.6%, 61.1%, and 1.2%, respectively. The substantial presence of iron in the

spectrum strongly confirms the formation of iron-based nanoparticles. Meanwhile, the significant oxygen content suggests that these nanoparticles exist as iron oxide, likely due to the oxidation of iron in the occurrence of water and air [8]. The trace occurrence of sulfur could be attributed to plant-derived compounds used in the green synthetic approach.

Furthermore, the relatively high percentage of carbon and oxygen in the EDS results suggests that the synthesized iron oxide nanoparticles are crowned with organic molecules, which include C–O functional groups such as polyphenols and flavonoids. These organic groups originate from the *Tinospora cordifolia* stem extract, which was employed as a reducing and stabilizing agent during nanoparticle formation [12]. The presence of these bioactive compounds plays a critical role in preventing nanoparticle aggregation and enhancing their constancy in solution. Additionally, these organic capping agents can impart biofunctional properties to the nanoparticles, making them appropriate candidate for applications in biomedical, environmental, and catalytic domains.

The microwave-assisted green synthesis of iron oxide nanoparticles by *T. cordifolia* extract offers many

compensations, including eco-friendliness, economic, and the impending for biomedical applications due to the biocompatibility of the phytochemical-derived capping agents. Compared to conservative chemical synthesis approaches, which often require dangerous reducing agents and stabilizers, the selection of plant extracts gives a sustainable and non-toxic substitute. The phytochemicals present in the plant extract not only assist the reduction of Iron ions to Iron oxide nanoparticles but moreover provide an additional layer of functionalization that may be beneficial for targeted drug delivery, antimicrobial activity, and antioxidant properties [9].

➤ *Applications of Nanoparticles Synthesized Via Microwave-Assisted Green Methods:*

Iron oxide nanoparticles offer versatile applications spanning medicine, catalysis, electronics, and environmental remediation, thanks to their exclusive attributes such as minor scale, extensive surface area and surface reactivity. Their ability to tackle a wide array of challenges in these fields positions them as key materials driving future technological innovations. Figure 2 shows the diverse applications of Iron oxide nanoparticles across different sectors [12].

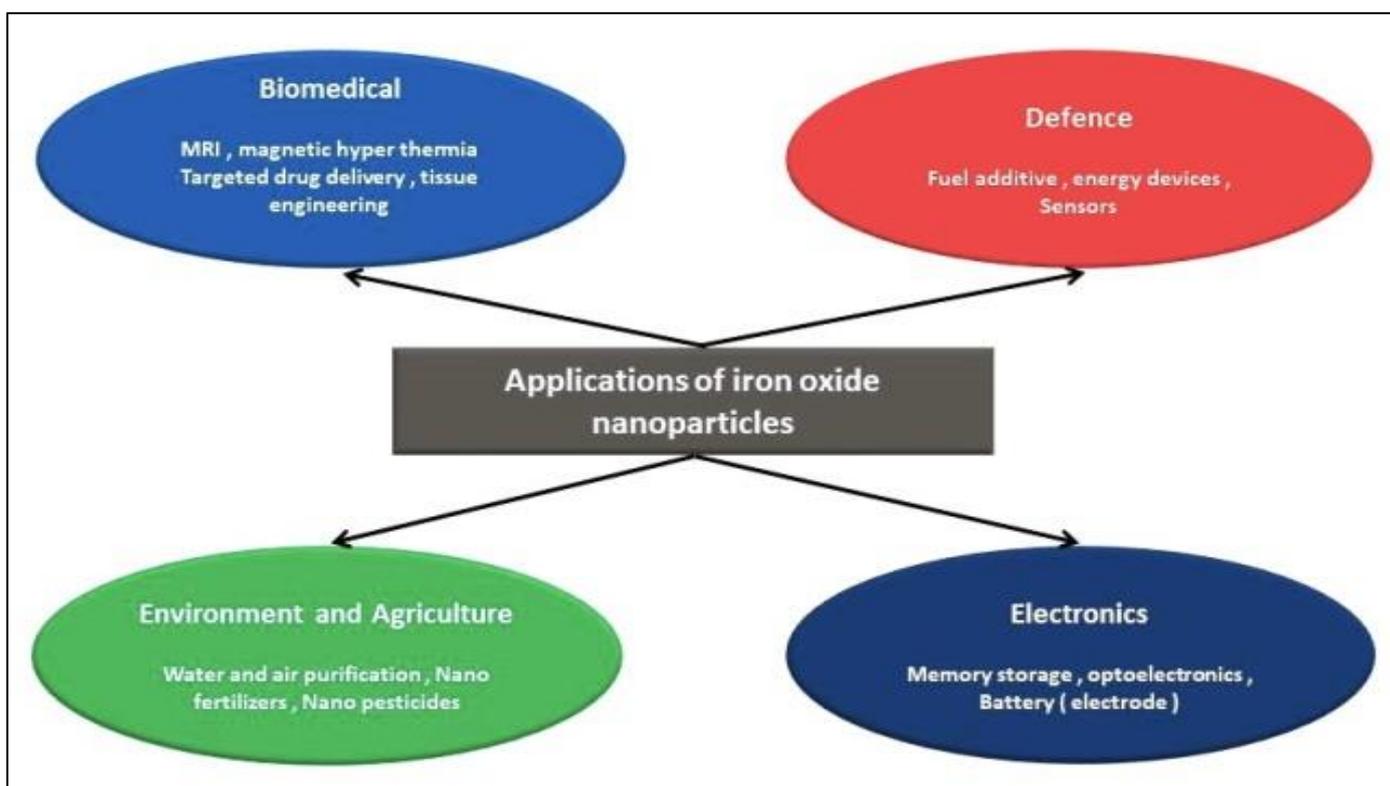


Fig 2 Schematic Diagram of Application of Iron Oxide Nanostructures

Iron oxide nanostructures have emerged as highly versatile nanomaterials with multifunctional capabilities. In the biomedical field, IONPs are extensively utilized in Magnetic Resonance Imaging (MRI) as contrast agents due to their superparamagnetic performance. They also play an important role in magnetic hyperthermia therapy, where localized heating induced by an alternating magnetic field is

used to destroy cancer cells. Furthermore, iron oxide nanostructures are widely investigated for targeted drug-delivery systems, enabling site-specific treatment with reduced side effects [9]. Their biocompatibility and surface modification flexibility also make them valuable in tissue engineering applications. In the defence sector, iron oxide nanoparticles contribute to advanced technologies such as

fuel additives, where they enhance combustion efficiency and reduce emissions. They are also used in energy storage devices and specialized sensors designed for detecting hazardous chemicals, explosives, or environmental threats [11]. Within environment and agriculture, IONPs support water and air purification through adsorption and catalytic degradation of pollutants. Their application as nano-fertilizers improves nutrient delivery efficiency, while nano-pesticides enhance crop protection with controlled release mechanisms, minimizing environmental contamination. In electronics, iron oxide nanoparticles are applied in memory storage devices due to their magnetic properties. They are also integrated into optoelectronic systems and battery electrodes, contributing to improved energy storage capacity and device performance. Collectively, these applications highlight the broad technological significance of iron oxide nanoparticles [15].

IV. FUTURE PERSPECTIVES AND CHALLENGES

Microwave-assisted green synthesis presents a highly promising strategy for the sustainable fabrication of nanoparticles, combining the environmental benefits of green chemistry with the efficiency of rapid microwave heating [8]. This approach significantly reduces reaction time, energy need, and the use of unsafe chemicals, thereby contributing a cleaner and more economical substitute to conventional synthesis techniques. However, despite its advantages, several critical challenges must be addressed before large-scale industrial implementation becomes feasible [4].

One of the primary hurdles is scalability. While microwave-assisted synthesis is highly effective at laboratory scale, translating the process to industrial-scale production remains complex due to limitations in uniform microwave penetration, reactor design, and temperature control [1]. Additionally, accurate optimization of reaction restrictions—such as microwave power, irradiation time, predecessor concentration, pH, and solvent composition—is essential to achieve consistent particle size, morphology, and reproducibility. Variations in plant extract composition can further affect reaction kinetics and nanoparticle stability [6, 14].

Another important challenge is the lack of standardized and reproducible protocols. Establishing uniform guidelines for precursor preparation, extract concentration, and characterization techniques is crucial for ensuring comparability between studies [12]. Future research should therefore focus on reactor engineering, process optimization, and protocol standardization to fully harness the potential of microwave mediated green synthesis for sustainable nanoparticle production [10].

V. CONCLUSION

Microwave-assisted green synthesis has emerged as a highly promising and sustainable approach for the generation of nanoparticles, offering a significant reduction in environmental impact compared to conventional chemical

methods. By integrating eco-friendly precursors with rapid and uniform microwave irradiation, this technique brings closely with the green chemistry's principles, including reduced energy consumption, minimal waste generation, and the elimination of toxic reagents. Such an approach contributes to the advancement of environmentally responsible nanotechnology. Using this method, Iron oxide nanostructures were successfully synthesized from ferric (III) chloride salt solution with the *T. cordifolia* stem powder extract as a reducing and stabilizing reagent. The prepared nanoparticles exhibited a size range of 30–65 nm, with an average particle size of approximately 30 nm, indicating relatively uniform nanoscale formation. The phytochemicals of the plant extract facilitated the reduction of iron ions and stabilized the nanoparticles, preventing agglomeration. UV–Visible spectroscopic analysis confirmed nanoparticle formation through the characteristic absorption profile. Scanning electron microscopy (SEM) provided detailed insight into particle morphology and size distribution, while the elemental composition and purity confirmed by the energy-dispersive X-ray spectroscopy (EDS). Continued research work and process optimization are important to expand the practical applications of microwave-assisted green synthesis across biomedical, environmental, and industrial sectors.

➤ Data Availability:

All data that has been used in this work are presented in this manuscript.

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