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Green Synthesis of Iron Oxide Nanoparticles Using *Simarouba Glauca* Leaf Extract and Application in Textile Effluent Treatment

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Abstract: In this study the iron oxide nanoparticles were synthesized by using with “*Simarouba glauca*” leaf extract, used for the treatment of textile effluent. The ability of Iron oxide NPs act as photo catalysts in dye degradation of organic pollutants. The crystalline nature and purity of synthesized nanoparticles were characterized by XRD, SEM, FTIR and UV-Visible spectrophotometer. The XRD pattern of the distinct peak of Fe₂O₃ was found at 33.47 (2θ). The SEM images demonstrated that the size of the nanoparticles was the range from 70-122nm. FTIR spectrum of Fe₂O₃ showed the presence of polyphenol shows its highest frequency at 447.49cm⁻¹. UV-Visible spectrum of the absorption peak of 352 nm demonstrated the iron oxide nanoparticles. The photocatalytic activity of the iron oxide nanoparticles synthesized using *Simarouba glauca* aqueous leaf extract was studied by the decolorization of textile effluent.

Keywords; Green synthesis, iron oxide nanoparticles, X-Ray diffraction analysis, FTIR spectra, UV-Visible spectrometer, Textile effluent treatment.

I. INTRODUCTION

Nanotechnology is becoming a new area of increasing research and industrial interest since the 1980. Metallic nanoparticles are currently a hotspot of interdisciplinary research due to their inherent potential for diverse nano technological applications. Scientists are constantly trying to devise rapid and cost-effective methods for the synthesis of these nanoparticles (NPs). Microorganisms especially magnetotactic bacteria produce intracellular iron NPs in membrane bound organelles called ‘magnetosomes’. These magnetosomes help the bacteria in responding to an external magnetic field. Research on the magneto- some biosynthesis have paved a crucial way for the development of many biomedical and other potential applications During the last two decades, the biosynthesis of metal nanoparticles (silver, copper, iron, gold, platinum and palladium) has received considerable attention due to the growing need to develop environmentally sociable technologies in material synthesis. (Srivanthi M et al., 2016) [1]

Iron oxide nanoparticles have been reported for various biomedical application such as drug delivery, magnetic resonance imaging, detection, diagnosis and treatment of illness such as neurological disease, cancer, and cardiovascular disease due to their small size, biocompatibility and low toxicity. Iron oxides are chemical compounds composed of iron and oxygen. IONPs synthesis is in focus due to their characteristic features such as superparamagnetism, non-toxic nature, being biocompatible as well as biodegradable and the production of these NPs is cost effective (Beena jose and Riya Martin et al., 2020) [3]. Methods for the synthesis of FeNPs includes co-precipitation, thermal decomposition, sonochemical are the most. In addition, electrochemical and green synthesis are introduced by many researchers. Green synthesis of nanoparticle is cost effective, ecofriendly, non- toxic, large scale production can be done easily and acts as reducing and capping agent when compared to the chemical method.

Biosynthesis of metal nanoparticles extracted from different parts (mostly leaf) of the plant is the most effective process of synthesis at a very affordable cost. The synthesis of iron oxide nanoparticles using plant materials offer several benefits of eco-friendliness and compatibility for various applications as they do not use toxic chemicals for the synthesis protocol. *Simarouba glauca* has a long history in herbal medicines in many countries. *Simarouba glauca* is commonly known as Laxmitaru or Paradise tree belonging to family *Simaroubaceae*. The leaves and bark of *Simarouba* have long been used as a natural medicine in tropics. *Simarouba* bark has been used as effective treatment for dysentery and malaria. Bark and leaf of *Simarouba* contain triterpenes useful in curing amoebiasis, diarrhea and malaria. It is also known to possess the medicinal properties such as analgesic, antimicrobial, antiviral, astringent, emmenagogue, stomachic, tonic vermifuge (Joshi et al., 2002) [4].

Green technology plays a vital role in the economy and environment. The development of green technology helps us to get rid of the mitigation of environmental degradation. Introduction of plant extracts instead of hazardous chemicals made it eco-friendly and cost-effective (*Sadia saif et al., 2016*) [5]. The iron oxide nanoparticles were synthesized by using with *Simarouba glauca* leaf extract, used for the degradation of methylene blue and textile effluent. The textile industry emits a wide variety of pollutants from all stages in the processing of fibers, fabrics and garment production. The main environmental concern in the textile industry is about the amount of water discharged and the chemical load it carries. Various techniques have been employed for the treatment of organic molecules in dye water. The approach includes chemical, physical, biological, and their combinations. The ability of Iron oxide NPs as photo catalysts and it is effective as a photo catalyst in dye degradation of organic pollutants. In this study, a lower number of catalysts will be used to degrade a large-scale synthetic dye. Iron oxide nanoparticles from *Simarouba glauca* are expected to have high potential applications. The synthesized nanoparticles were characterized by UV-Visible spectrophotometer and the dispersity and morphology were studied by Scanning electron microscopy. The crystalline nature and purity of synthesized nanoparticles were revealed by X-ray diffraction (XRD) technique. FTIR spectrum was examined to identify the effective functional molecules responsible for the reduction and stabilization of nanoparticles synthesized by *Simarouba glauca* aqueous leaf extract. The UV-Visible spectrum of synthesized nanoparticles was studied. The synthesized nanoparticles showed significant catalytic activity in the photo degradation of methylene blue and textile effluent, hence used as a promising candidate for the purification of wastewater contaminated with dyes.

II. MATERIALS AND METHODS

A. Collection And Preparation Of *Simarouba Glauca* Leaf Extract

Leaves of *Simarouba glauca* was collected from Alathur, Palakkad district in Kerala. Healthy and disease-free plants were selected for obtaining plant material. The plant leaves of *Simarouba glauca* was shade dried (10 days) and powdered using mechanical blender. 20g of powdered leaves were dissolved in 200 ml of distilled water and allow to stand for overnight refrigeration at 4° C. The mixture was then boiled for 10 minutes and cooled at room temperature and filtered through using Whatman No.1 filter paper (*Amutha et al., 2018*) [2]

B. Synthesis Of Iron Oxide Nanoparticle Using Leaf Extract Of *Simarouba Glauca*

- 1) **Chemicals:** Ferrous chloride (FeCl_2), Ferric chloride (FeCl_3), and Sodium hydroxide (NaOH), were used for the synthesis of iron NP's
- 2) **Green Synthesis of Iron Oxide Nanoparticles Using Leaf Extract Of *Simarouba GLAUCA*:** The preparation of nanoparticles was carried out with the help of a magnetic stirrer. 50 ml of 0.1M Ferrous chloride (FeCl_2) solution was mixed with 100 ml of 0.1M Ferric chloride (FeCl_3) solution in a 250 ml of Erlenmeyer flask. The flask was stirred for 10 minutes with a magnetic stirrer at 80°C until the color turns yellow. 50 ml of plant extract was then added and the flask was again stirred for 5 minutes with a magnetic stirrer at 80°C until it turns the color yellow to black. 10 ml aqueous solution of sodium hydroxide (NaOH), was then added with the constant stirring at the rate of 3 ml per minute to get uniform precipitation (*Amutha et al., 2018*) [3]. All the synthesized nanoparticle precipitation was settled under the bottom of the centrifuging tube. The synthesized nanoparticles were then poured into a Petri dish using a sterile white paper and dried at 80°C under a hot air oven for 3 hours to obtain the Iron oxide nanoparticles. The synthesized materials were stored at 4°C for further use.
- 3) **Characterization Of Iron Oxide Nanoparticle:** The crystalline nature of the iron oxide nanoparticles was recorded by X-Ray diffraction analysis using X-Ray Diffraction Unit (XRD) Pan Analytical, X-Pert pro, operated at 45Kv. The morphology of the prepared nanoparticles was analyzed by Scanning Electron Microscopy (TESCAN -MIRA3 XMU) run at a voltage of 5.0 KV. FTIR spectra of synthesized sample was recorded using FTIR spectrometer (SHIMADZU-Miracle 10) in the range number of 3750-500 cm^{-1} using KBr pellet method. The surface Plasmon resonances of synthesized nanoparticles were studied by a UV-Visible spectrometer (JASCO V-650) in the range of 250-700 nm. The textile dye effluent degradation absorbance was recorded using UV-VIS spectrum at the range of 250-700 nm.

C. Treatment Of Textile Effluent Using Iron Oxide Nanoparticle

The photocatalytic activity of the iron oxide nanoparticles synthesized using *Simarouba glauca* aqueous leaf extract was studied by the decolorization of textile effluent. 10 mg of iron oxide nanoparticles was added to 100 ml of textile effluent in an Erlenmeyer flask and control was also maintained. The reaction was carried out by stirring the flask for 30 minutes to make the equilibrium. The dispersion was then kept under the sunlight. 10 ml of the suspension was taken at a 30-minute time interval to evaluate the photocatalytic degradation of the effluent (*Arifa Tahir et al., 2019*) [5].

The suspension was centrifuged for 1 minute to remove the iron oxide catalyst for measuring the absorbance under UV. The Physico-chemical parameters of Textile Dye effluent such as color, pH, electrical conductivity, and COD were estimated after the exposure to the sunlight for half an hour.

III. RESULTS

A. Preparation and Synthesis Iron Oxide Nanoparticle using Plant Extract of *Simarouba Glauca*

The powdered leaf extracts were thoroughly mixed with 200 ml distilled water and filtered using Whatman No.1 filter paper. The chemicals were added to the extract and finally the black color of iron oxide solution was obtained and by adding the NaOH results the iron oxide nanoparticle precipitation. The precipitation of iron oxide nanoparticles was air dried in hot air oven 80°C and stored in an air tight container for the further use. Fig.1 (a, b, c, d) represented the synthesis of iron oxide nanoparticles using *Simarouba glauca* leaf extract.



a. Leaf extract of *Simarouba glauca*



b. Mixture of FeCl_2 and FeCl_3



c. Iron oxide nanoparticle precipitation



d. Iron oxide nanoparticles

Fig.1 Green synthesis of iron oxide nanoparticles from *Simarouba glauca* leaf extract

B. XRD ANALYSIS

The XRD pattern of iron oxide nanoparticles showed 3 peaks at $2\theta=33.47$, 35.96 , 54.25 as shown in Fig.2. This obtained were match with results of X-ray diffraction (XRD) pattern of the Fe_2O_3 NPs. The distinct peak of Fe_2O_3 was found at 33.47 (2θ) and intense peak at $2\theta = 35.96$ was identified as polyphenols. The sharp and narrow diffraction peaks in the XRD spectrum indicated that the synthesized Iron oxide nanoparticles were pure and highly crystalline nature.

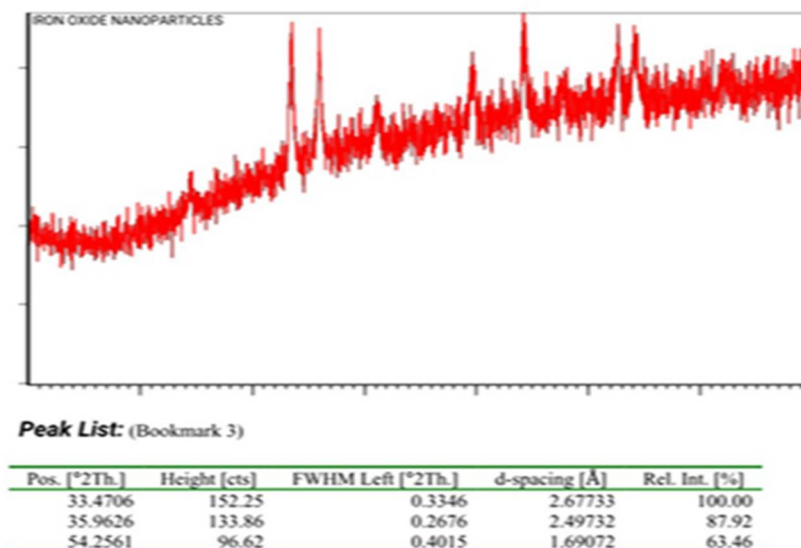


Fig.2 XRD analysis of iron oxide nanoparticles

C. Scanning Electron Microscopy

The morphological dimensions of the synthesized iron oxide nanoparticles mostly appeared to be a porous and aggregated as irregular sphere shapes with rough surfaces. The study demonstrated that the size of the nanoparticles was the range from 70-122nm. From the SEM images as shown in Fig.3 demonstrated that the synthesized products are nanoparticles, which grown in a very high density and possessed non-uniform for iron oxide nanoparticle.

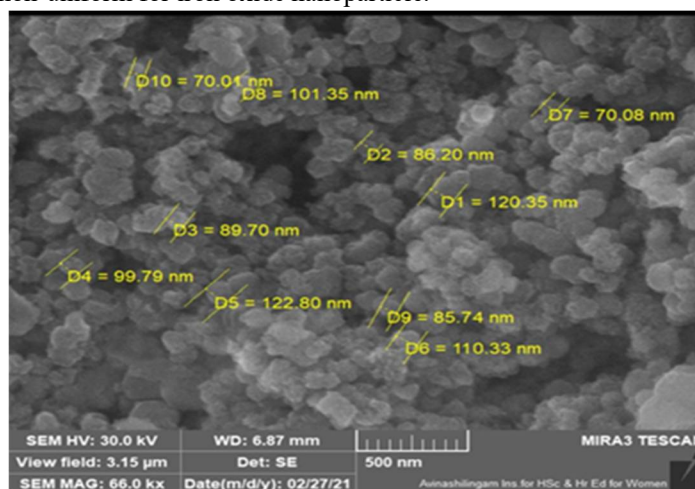


Fig.3 SEM analysis of iron oxide nanoparticles

D. FTIR Analysis

FT – IR analysis shows the potentiality and the stability of synthesized iron oxide nanoparticles. FTIR measurements were carried out to identify the possible biomolecules in the aqueous leaf extract responsible for the reduction of ions and also the capping agents responsible for the stability of the biogenic nanoparticle solution. The result of FTIR confirmed that the aqueous leaf extract of *Simarouba glauca* is having a potential in reducing and stabilizing the iron oxide nanoparticles. The FTIR analysis (400-4000 cm⁻¹) of the synthesized sample ensured the synthesis of α -Fe₂O₃ nanoparticles as well the existence of various reducing agents functional groups presents in the papaya plant leaf extract (*Shakawat et al 2020*). FTIR spectrum of iron oxide nanoparticles showed the presence of polyphenol in *Simarouba glauca* leaf extract shows its highest frequency at 447.49cm⁻¹, the frequencies at low wavenumbers 594.08 cm⁻¹ and 555.50 cm⁻¹ come from vibrations of Fe–O bonds of iron oxide. The strong peak at 447.49 cm⁻¹ in corresponds to iron oxide nanoparticle.

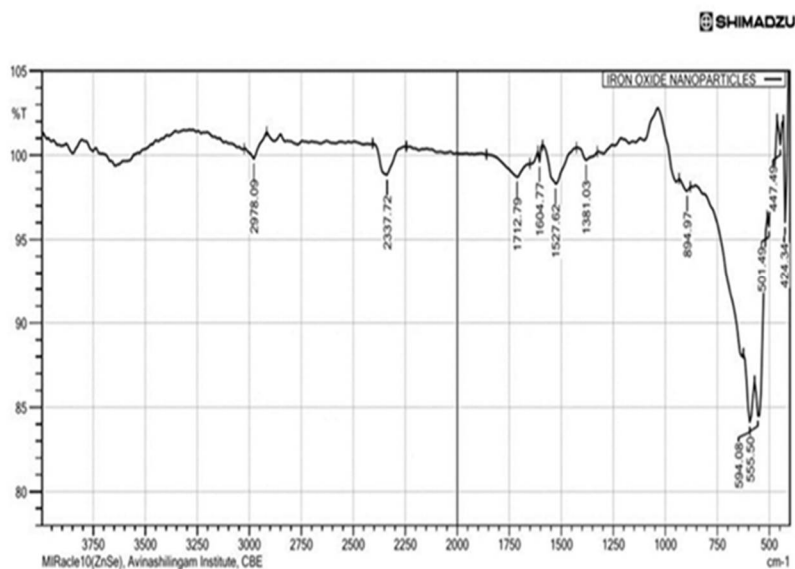


Fig.4 FTIR analysis of iron oxide nanoparticles

E. UV-Visible Spectrophotometric Analysis

The plant extracts were examined under UV-Vis spectral analysis using UV-VIS spectrum at the range of 250-700 nm and the absorption peak of 352 nm demonstrated the iron oxide nanoparticles as shown in Fig. 3. The characteristic absorption peak occurs at the wavelength ranging from 300 to 400 nm indicated the formation of iron oxide nanoparticles and the peak observed between 300-400 nm are of polyphenols.

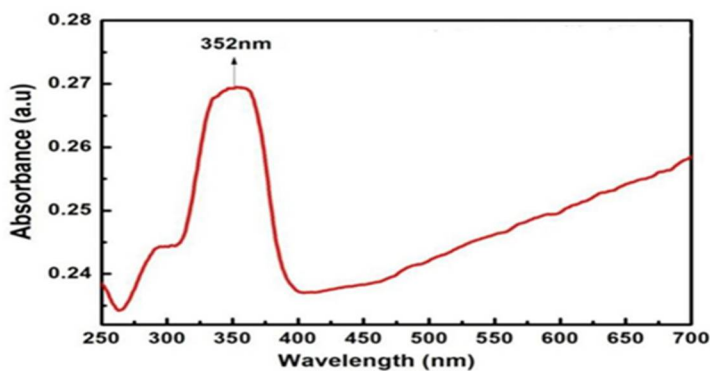


Fig.5 UV-Visible spectra of iron oxide nanoparticle

F. Treatment of Textile EFFLEUNT using iron Oxide Nanoparticl

Photo catalytic degradation was demonstrated using iron oxide nanoparticles synthesized using *Simarouba glauca* plant leaves. The Polyphenols which act as a reducing agent in the synthesis of iron oxide nanoparticles using *Simarouba glauca* leaf extract and UV spectrum demonstrated a continuous decrease in absorption and indicated that high degradation rate and better efficiency using *Simarouba glauca* leaf extract. The actual effluent from a textile industry, took longer period to decolorize because the high concentration of dye solvents which helps to prevent the transmission of sunlight from the source through the solution. The intensity peaks value between 400 to 500nm shows the reaction of binding iron nanoparticles and the decline value which is represented in Fig.5. Physio-chemical parameters of textile dye effluent before and after photo-catalytic degradation are represented in Table1. The color change of the effluent turned dark ash to complete colorless after degradation indicates the change of pH using iron nanoparticles in the solvent. The electrical conductivity and COD in the textile effluent decreased after photocatalytic activity.

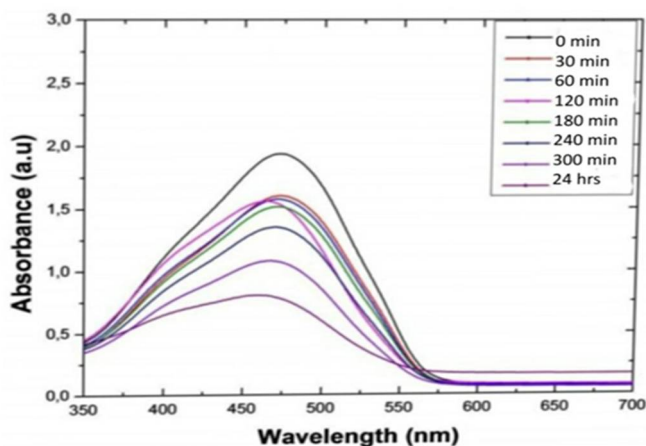


Fig.6 UV absorption spectra of dye in textile effluent



Fig.7 Degradation Textile effluent

Table.1 Physiochemical parameter of textile effluent

Parameter	Textile Effluent	Textile effluent after photocatalytic degradation
COD (mg/ml)	488	104
Color	Dark ash	Light ash (colorless)
Electrical conductivity(S/m)	712	580
pH	7.2	5.3

IV. DISCUSSION

The leaf extract of *Simarouba glauca* has potential effect to reduce ferric and ferrous ions to iron oxide nano-particles. The aqueous leaf extract of *Simarouba glauca* was added to the mixture of ferrous chloride and ferric chloride solution and the color of the reaction medium change of yellow to black indicates the reduction of iron ions to iron nanoparticles. The color change was due to the interaction between the phytochemicals and iron salts (Palash Kumar Dhar et al., 2021). The XRD pattern of iron oxide nanoparticles showed intense and sharp peaks undoubtedly revealed that Fe₂O₃ nanoparticles formed by the reduction method using *Simarouba glauca* leaf extract were crystalline in nature. The results were match with results of X-ray diffraction (XRD) pattern of the Fe₂O₃ NPs. These findings are analogous with crystalline nature of iron oxide nanoparticle synthesized from *Glycosmis mauritiana* reported by (Amutha S et al, 2018). The study demonstrated the size of the nanoparticles was in the range of 70-120 nm. It is clear from the SEM images that the synthesized products are nanoparticles, which grown in a very high density and possessed non-uniform for iron oxide nanoparticle. The plant extracts were examined under UV-Vis spectral analysis using UV-VIS spectrum at the range of 250-700 nm and the absorption peak of 352 nm demonstrated the iron oxide nanoparticles.

The surface Plasmon resonances (SPR) of iron oxide nanoparticles synthesized from the aqueous leaf extract of *Simarouba glauca* have been studied by a UV-Visible spectrometer. The absorption of visible radiations due to the excitation of SPR, imparts various colors to the nanoparticles. The FTIR spectrum of iron oxide nanoparticles show bands at 2978.09 cm⁻¹ and 2337.72 cm⁻¹ corresponding to O–H stretching and bending bands. The photo catalytic treatment of the textile effluent elucidated the Polyphenols which act as a reducing agent in the synthesis of iron oxide nanoparticles using *Simarouba glauca* leaf extract. The photocatalytic treatment converts the pollutants completely in to harmless end products like carbon dioxide and water (Iqbal M et al., 2015) [13]. When Zero-valent Iron Nano-particles incorporated into the sewage, it tends to remove BOD from 56 mg/L to 22 mg/L and COD from 405 mg/L to 85 mg/L (Turakhia B et al., 2018) [14]

V. CONCLUSION

From this result, it has been concluded that the soluble biomolecule group present in the *Simarouba glauca* leaf extract acted as capping agents, preventing the aggregation of iron oxide nanoparticles in the solution. UV spectrum has shown a continuous decrease in absorption and this indicates that high degradation rate and better efficiency were obtained by using *Simarouba glauca* leaf extract. The intensity peaks value between 400 to 500nm shows the reaction of binding iron nanoparticles and the decline value. The synthesized iron nanoparticles can be used for the removal of contaminants present in the textile dyeing industry effluent.

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