STRUCTURAL AND OPTICAL PROPERTIES OF WATER-SOLUBLE IRON NANOPARTICLES USING *MIMOSA PUDICA* LEAF EXTRACT VIA GREEN ROUTE

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The present study mainly deals with a novel green route for the synthesis of water-soluble Iron nanoparticles (FeNPs) using potent herb Mimosa pudica leaf extract (MPLE). The structural and optical properties of synthesized FeNPs were studied using X-ray diffraction (XRD), Fourier transform infrared spectroscopy (FTIR), UV-visible spectroscopy (UV-vis) and Field emission scanning electron microscopy-Energy Dispersive X-ray spectroscopy (FESEM-EDAX) analysis. The average size of the prepared FeNPs was around 30-40 nm. The Fourier transform infrared (FTIR) spectrum showed the role of Mimosa pudica leaf extract (MPLE) in the formation of Iron nanoparticles (FeNPs). The treatment of MPLE with the aqueous $FeCl_3$ led to the formation of FeNPs. During the reaction, the change in color of the reaction mixture from light green to black confirms the formation of FeNPs. The field emission scanning electron microscopy (FESEM) images showed that the FeNPs are uniform in size and shape.

Keywords: Fe, Nanoparticles, X-ray techniques, FTIR, Green route

1. Introduction

Nanoparticles research is an important aspect in the field of nanotechnology due to its numerous technological applications [1, 2]. The chemical [3-5] and physical [6, 7] methods have been widely used to synthesize nanoparticles with different shape and size. Recently, biosynthesis of metal nanoparticles using natural products is an emerging area in nanoresearch and it is a kind of bottom up approach where the main reaction will be reduction/oxidation.

With the antioxidant or reducing properties of plant extracts, they are usually responsible for the reduction of metal compounds into their respective

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nanoparticles. FeNPs exhibit improved properties as compared to the bulk material based on specific characteristics such as size distribution and morphology [8].

In recent years, size-dependent physico-chemical properties made metallic FeNPs highly potent in a wide range of applications including magnetic storage media, ferrofluids, biosensors, catalysts and environment remediation [9]. Researchers have shown that FeNPs are very effective for transformation and detoxification of a wide variety of common environmental contaminants such as chlorinated organic solvents and organochlorine pesticides [10]. Environmental applications of FeNPs have been enthusiastically accepted by many users and regulatory agencies largely due to the low cost and non-toxic in nature [11].

Several studies have shown that FeNPs are very effective for the degradation of halogenated solvents such as chlorinated methane, brominated methane, trihalomethane, chlorinated ethene, chlorinated benzene and other polychlorinated hydrocarbons in groundwater [12]. FeNPs were also shown to be effective against some pesticides, heavy metals, and dyes. Modern dyes are often intended to be able to resist the breakdown of long-term exposure to sunlight, water and other conditions which makes the treatment of the wastewater from dye and textile industries more difficult [13]. Efficient decolorization of dyes has therefore received greater attention due to physical methods. But the drawbacks of these methods are consumption of high activation energy and use of toxic chemical substances [14]. Green route for synthesizing FeNPs has emerged as an alternative to overcome the limitation of conventional methods. Various analytical techniques were used to confirm the presence of FeNPs. Few studies have reported the synthesis of FeNPs using plant extracts [15-17]. The present investigation involves a green route for the synthesis of FeNPs using the herb *Mimosa pudica* leaf extract (MPLE). Hydroxyl and phenolic groups present in MPLE are the active sites during the synthesis involving the reduction of $FeCl_3$ into Fe nanoparticles. In this work, MPLE was used as both reducing and capping agent for the formation of FeNPs *via* a facile, green and environmentally benign route.

2. Materials and methods

2.1. Materials

Fresh leaves from *Mimosa pudica* leaves were collected from Sathyabama University campus [Latitude 12.8731° N, Longitude 80.2219° E], Chennai and used as such for the study. Ferric chloride ($FeCl_3$) was purchased from Sigma-Aldrich, India. Double distilled water was used throughout the experiment.

2.2. Preparation of $FeCl_3$ and Mimosa pudica leaf extract (MPLE)

$FeCl_3$ (0.001 M) was prepared by dissolving 0.04 g of anhydrous Ferric chloride in 250 mL double distilled water in a standard conical flask. MPLE was prepared by taking 8 g of leaves and washed thoroughly with double distilled water and crushed to make it a paste using mortar and pestle by adding 80 mL double distilled water gradually. The mixture was poured in a conical flask and heated for 16 minutes at 70°C. The mixture was then filtered using Whatman's filter paper and the filtrate thus obtained was greenish brown in color.

2.3. Synthesis of MPLE assisted FeNPs

For synthesis of FeNPs, both the precursor and the reducing agent were mixed in 1:1 ratio in a clean conical flask, heated for 1 h under constant stirring condition at 50°C and changes in the color of the solution mixture was monitored. On visual perception, reduction is followed by an immediate change in color from pale green color to black color. It is well known that $FeCl_3$ exhibits bright yellowish color in distilled water. On mixing MPLE with aqueous $FeCl_3$ solution, the color of the solution changes which indicates the formation of MPLE assisted FeNPs.

2.4. Characterization of MPLE assisted FeNPs

MPLE assisted FeNPs were centrifuged at 10000 rpm for 15 min to prepare the pellet, which was washed with deionized water to remove the remaining biomass. Purified pellet was then dried and the powder was subjected to XRD. X-ray diffraction (XRD) pattern of MP assisted FeNPs was analyzed using Rigaku smart lab instrument operated at a voltage of 40 kV and a current of 30 mA with Cu Kα radiations. Fourier transform infrared (FTIR) Analysis was carried out for MPLE and FeNPs employing KBr pellet technique using Perkin Elmer 983/G detector double beam spectrophotometer to identify the possible functional groups present. The reduction of pure Fe_3^+ ions to Fe^0 was checked by measuring the UV-Vis spectrum of FeNPs. The light absorption spectrum of FeNPs was recorded using in the range of 260 to 800 nm using Shimadzu UV-vis spectrophotometer UV-1800. Field emission scanning electron microscopy-Energy dispersive X-ray analysis (FESEM-EDAX) was performed to check the surface morphology and elemental analysis of FeNPs formed using SUPRA 55-CARL ZEISS, Germany.

3. Results and discussion

Fig.1 shows the X-ray diffraction (XRD) pattern of MPLE assisted FeNPs. From the XRD pattern, it has been observed that the whole pattern was deficient in distinctive diffraction peaks, suggesting that the synthesized FeNPs are mainly

amorphous in nature. A less obvious characteristic peak of zero-valent iron (α-Fe) was observed at about 2θ of 44.8° which is in good agreement with standard data (JCPDS File No. 00-006-0696) [18]. The broad shoulder peak at 2θ = 24.6° can be assigned to organic materials adsorbed from MPLE as capping/stabilizing agent, which is consistent with the FTIR results in Fig. 1 (Supplementary data). A similar pattern for FeNPs was found and reported when synthesized using *Terminalia chebula* aqueous extract [19] and *Eucalyptus* leaf extract [17].

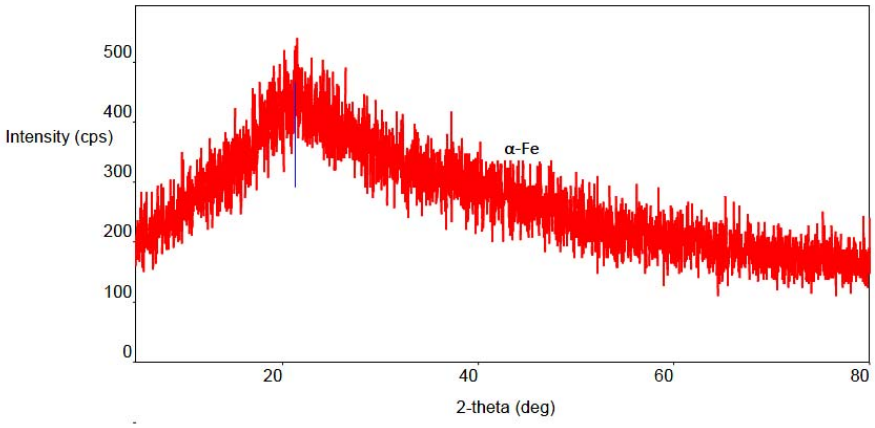


Fig. 1. XRD pattern of as-prepared *Mimosa pudica* leaf extract assisted FeNPs

The change in color of the reaction medium was noted by visual observation (Fig. 2 inset).

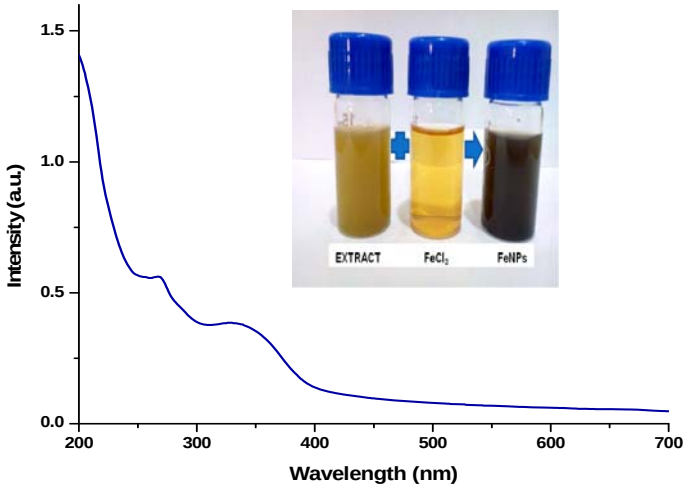


Fig. 2. UV–vis absorption spectrum of the *Mimosa pudica* leaf extract assisted FeNPs. Inset shows Colour changes during the synthesis of *Mimosa pudica* leaf extract assisted FeNPs

UV-vis spectroscopy was used to check the formation of FeNPs in aqueous solution. From UV-vis spectrum (Fig. 2), there is a broad peak at 352 nm, a peak at 265 nm and a rising absorption edge towards higher energy. The broad peak at 352 nm is due to remnants of collective oscillation of the surface electrons, i.e., surface plasmons [20].

FTIR measurements are mainly used to identify the possible biomolecules which are responsible for the reduction of the metal precursors and capping of FeNPs. Fig. 3 shows the FTIR spectra of MPLE and MPLE assisted FeNPs. The absorption bands at 3434 and 3393 cm^{-1} is attributed to O-H stretching vibration [19]. The bands at 2931 and 2918 cm^{-1} correspond to C-H and CH_2 vibration of aliphatic hydrocarbons [21]. With respect to the bands attributed to phenolic compounds, the FT-IR spectrum of MPLE shows bands at 1632 cm^{-1} which is due to C=C aromatic ring stretching vibration [22]. On the other hand, the FTIR spectrum of MPLE assisted FeNPs displays stretching vibrations at 1646 cm^{-1} for C=C and 1044 cm^{-1} for C-O-C peak [20]. Furthermore, adsorption bands at around 581 cm^{-1} correspond to the formation of FeNPs [17]. This result indicates that the hydroxyl and phenolic groups are the active sites during the synthesis and hence, the O-H & C=C groups are involved in the reduction of $FeCl_3$ into Fe nanoparticles. Wang *et al*. reported that the polyphenols have the function for the formation and stabilization of the FeNPs [17].

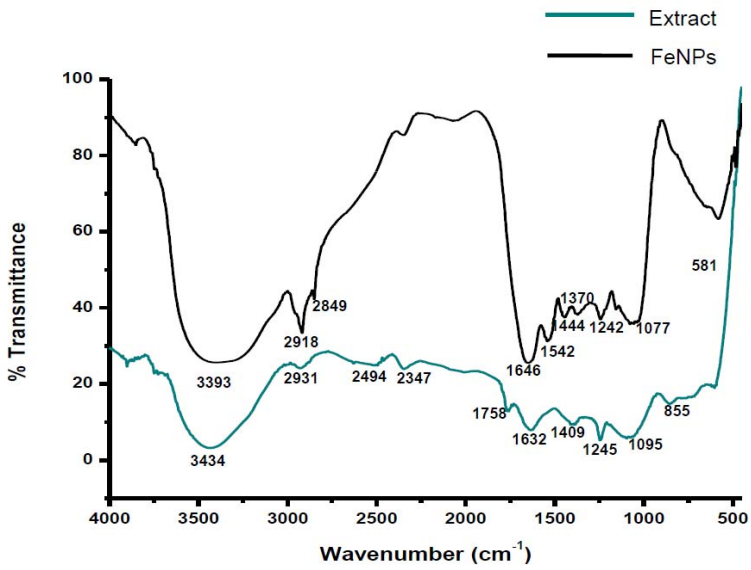
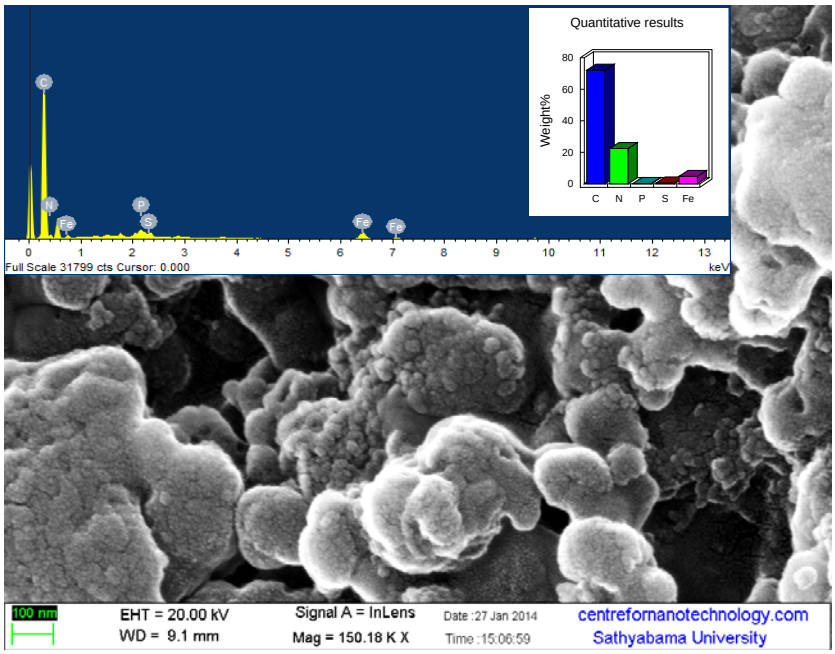


Fig. 3. FTIR spectra of *Mimosa pudica* leaf extract and *Mimosa pudica* leaf extract assisted FeNPs

FESEM micrographs of MPLE assisted FeNPs are shown in Fig. 4.



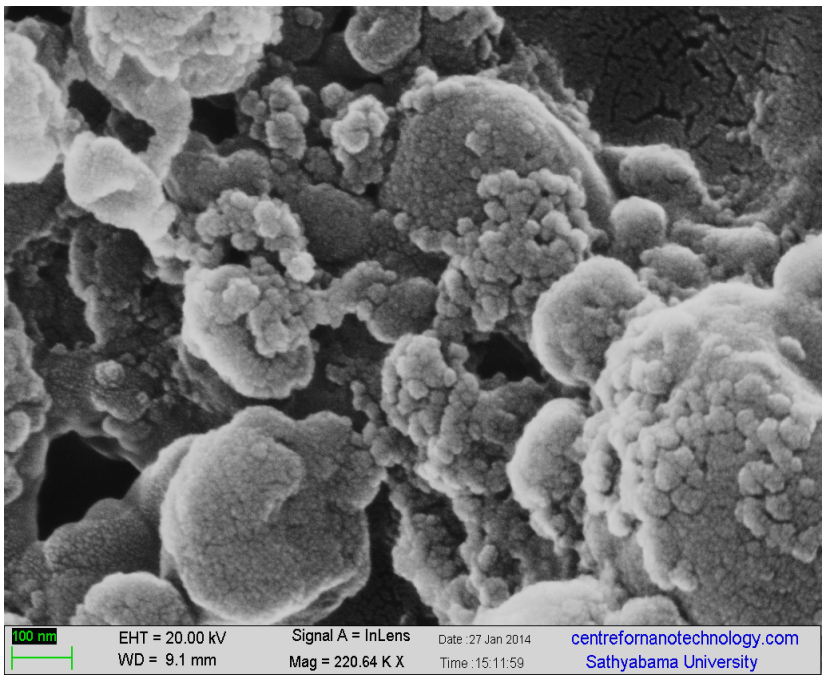


Fig. 4 (a,b) FESEM images of FeNPs synthesized using *Mimosa pudica* leaf extract and inset shows EDAX spectrum

The images clearly illustrate that the newly formed FeNPs are spherical in shape having a diameter of 30-40 nm. EDAX spectrum was used to analyze the elemental composition of MPLE assisted FeNPs. In the EDAX spectrum of nanoparticles recorded, the presence of strong signals of pure Fe was observed (Fig. 4 inset).

4. Conclusions

FeNPs were synthesized *via* a facile one-pot green synthesis route using *Mimosa pudica* leaf extract (MPLE). MPLE with high antioxidant capacity and polyphenols content acts as both reducing and capping agent for nanoparticles. XRD, UV-vis, FTIR, FESEM and EDAX analysis demonstrated that the Fe surface was capped with organic materials that originated from *Mimosa pudica* leaf extract as a capping or stabilizing agent. This method is simple, efficient and easily carried out. Biodegradable and non-toxic MPLE are environmentally friendly and these synthesis protocols can be scaled up for bioremediation applications.

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R E F E R E N C E S

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