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Green Synthesis of Iron Oxide Nanoparticles Mediated by *Actinodaphne madraspatna* Bedd Leaves

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In the present investigation, iron oxide nanoparticles were synthesized using a simple, rapid and green method using *Actinodaphne madraspatna* Bedd leaves as reducing agent. The UV-visible spectra showed strong absorption band in the visible region, which confirms the formation of iron oxide nanoparticles. TEM images showed distinct spherical shaped particles with average size of 20 nm. FT-IR spectra depicted the presence of phytomoities in *Actinodaphne madraspatna* Bedd leaves which may probably act as a reducing and capping layer and thus facilitating the formation of nanoparticles

Keywords: Green synthesis, Iron oxide nanoparticles, *Actinodaphne madraspatna* Bedd leaves, Phytomoieties

INTRODUCTION

Iron oxide nanoparticles (FeONPs) have gained wide application in materials research and biomedical fields in recent years because of ultra-fine sizes, surface-to-volume ratios and biocompability [1]. Several conventional methods have been used towards the synthesis of FeONPs, such as vacuum sputtering, aerosol, electrochemical synthesis, thermal decomposition and many other chemical methods [2,3]. Indeed, these methods involved organic solvents, energy consumption, toxic chemicals and byproducts which were potent hazard to human and the environment. In order to overcome these challenges, researchers have started focusing on eco-friendly and non-toxic protocols. In the recent decades, green synthesis of metal nanoparticles using various plants extracts received greater attention because of its (i) single step synthesis, (ii) cost effectiveness and (iii) environmentally benign solvent medium during the synthesis of nanoparticles [2,4].

Actinodaphne madraspatna Bedd, an Indian medicinal plant belonging to the family Lauraceae was widely used to cure diabetic, wound and fickle minded behavior. Saravanan *et al.* [5,6] reported that *Actinodaphne madraspatna* Bedd leaves contain tannins, saponins, alkaloids, triterpenoids, glycosides and carbohydrates. Recent studies [7,8] demonstrated that these water soluble phenolic compounds were proven to act as effective reductants and wrapped around the nanoparticles to provide excellent robustness against agglomeration. The feasibility and availability of rich phytoconstituents of *Actinodaphne madraspatna* Bedd leaves motivated us to explore their efficiency towards the synthesis of metal nanoparticles. Herein, we report a facile route for the synthesis of FeONPs with particle size of 20 nm using *Actinodaphne madraspatna* Bedd leaves extract.

EXPERIMENTAL

Anhydrous ferric chloride procured from Merck, India. *Actinodaphne madraspatna* Bedd (AMB) leaves were collected from Talakona forest, Tirupati, India.

Green synthesis of iron oxide nanoparticles (FeONPs): The freshly collected *Actinodaphne madraspatna* Bedd leaves were washed thoroughly to remove unwanted dust and impurities and dried for one week in shade and finally, the dried leaves were crushed into fine powder. 10 g of finely powdered *Actinodaphne madraspatna* Bedd leaves was soaked in 100 mL distilled water and stirred continuously for 0.5 h. The resultant extract was allowed to cool at room temperature and filtered through Whatmann filter paper.The obtained aqueous extract of *Actinodaphne madraspatna* Bedd leaves was added with 0.1 M $FeCl_3$ solution in a 2:1 ratio.The reaction mixture was stirred for 1 min in the mechanical stirrer and made to stand at room temperature for 1 h. The reaction mixture has become blackish brown in colour indicating the formation of FeONPs. The formed colloidal nanoparticles were centrifuged and a black colored pellets were obtained. Furthermore, these black pellets were washed three times with sterilized distilled water and dried at 75 ºC for 24 h until dry nanoparticles were formed. The obtained FeONPs were stored in brown sealed bottle under dry condition for further characterization.

Characterization of FeONPs: UV-vis spectrophotometer (JASCO -V 670) was used to study the formation of FeONPs. XRD patterns of the sample were obtained using Bruker D8 Advance X-ray diffractometer with CuK_{α} radiation. The morphology and average particle size of nanoparticles was obtained using TECHNAI SPIRIT G2 transmission electron microscope. Fourier transform infrared (FT-IR) SHIMADZU IR-Affinity-1 spectrometer was used to demonstrate the role of phytomoites in the nanoparticles synthesis.

RESULTS AND DISCUSSION

The UV-visible spectra of $FeCl_3$, AMB extract and synthesized FeONPs are shown in Fig.1. By the addition of *Actinodaphne madraspatna* Bedd leaves extract to iron precursor, the pale yellow coloured leaves extract changed into black instantly which confirmed the formation of FeONPs. Also, the absorbance peak of ferric chloride (iron precursor) around 300 nm disappeared and a continuous absorption was observed in the visible region from 400-600 nm and present results are in accordance with the literature [4,9]. Also the absorbance peak of *Actinodaphne madraspatna* Bedd leaves extract at 280 nm attributed to phenolic moieties [10,11] have noticeable shift in its absorbance due to probable complexation of iron with phenolic compounds. The obtained UV-visible spectral data apparently featured the formation of FeONPs *via* (i) complexation of iron salt precursor with phytomoities of *Actinodaphne madraspatna* Bedd leaves extract, (ii) instant reduction and formation of FeONPs and (iii) stabilization by capping with phenolic compounds of *Actinodaphne madraspatna* Bedd leaves extract which was in good agreement with earlier reports [4,9,12].

The XRD pattern of synthesized FeONPs is depicted in Fig. 1 as inset which reflect the diffraction peaks at $2\theta = 29°$, 40°, 50°, 66° and 74° were identified as iron oxide which are in consistent with earlier reports [3,8,11]. Additionally, the absence of peak at $2\theta = 45°$ of zero valent iron clearly confirmed the formation of FeONPs.

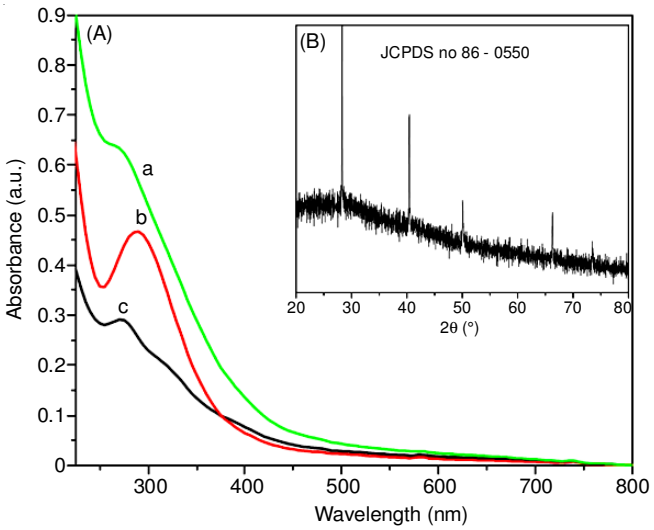

Fig. 1. (A) UV-vis spectra of (a) FeONPs (b) AMB aqueous extract and (c) Ferric chloride as metal precursor. (B) Inset XRD pattern of FeONPs

The effect of pH during nanoparticles formation is also studied and found that pH of *A. madraspatna* Bedd leaves extract and synthesized FeONPs are 5.6 and 6.8, respectively. Thus, the synthesis of FeONPs is favoured in a slightly acidic pH, whereas lower pH (highly acidic) leads to agglomeration by over nucleation and the particles were found to be unstable at higher pH [13].

The size and surface morphology of synthesized FeONPs were examined by TEM. As shown in Fig. 2, the synthesized FeONPs are nearly spherical in shape and the average particle size was found to be 20 nm (Fig. 2c). TEM images apparently revealed the appearance of thin organic layer surrounding synthesized FeONPs as capping layer and accord-ingly, the particles are well separated from each other. This clearly evidences that the phytomoities of *Actinodaphne madraspatna* Bedd leaves extract play a crucial role in the stability of FeONPs [7]. The selected area electron diffraction (Fig. 2d) pattern does not shows any prominent diffraction spots/rings

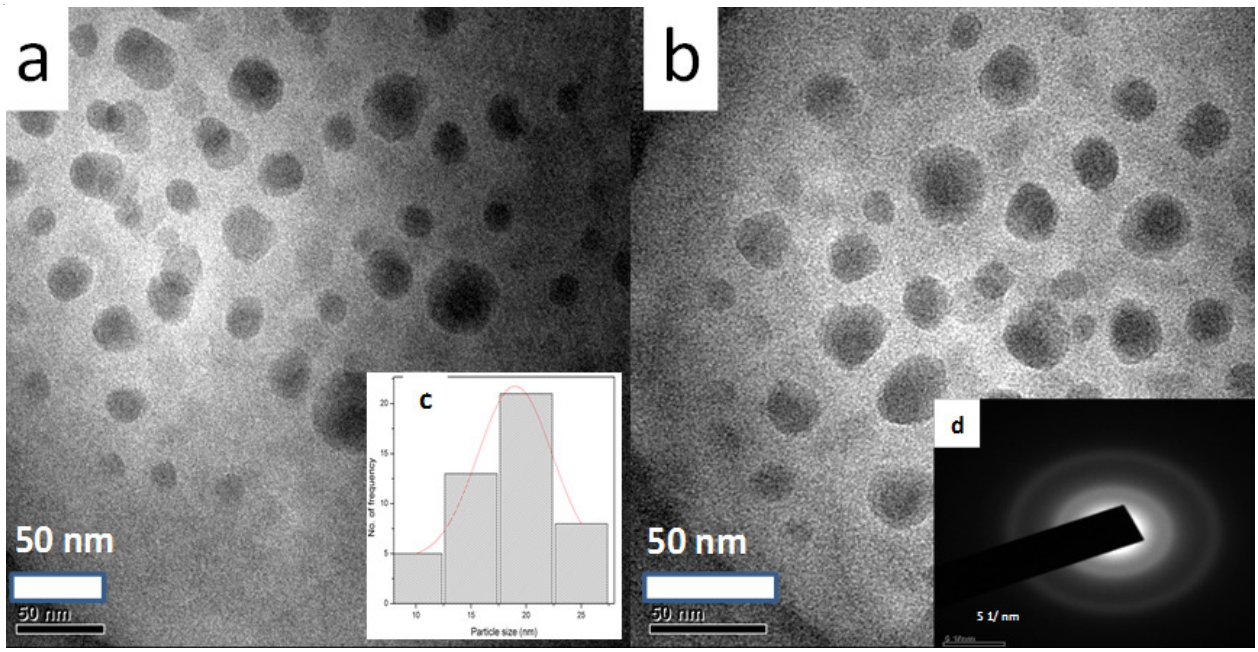

Fig. 2. (a,b) TEM images of synthesized FeONPs, (c) histogram for particle size calculation and (d) SAED pattern

which insist that synthesized FeONPs are amorphous in nature [14].

The FT-IR measurements were carried out to identify the role of phytoconstituents of *Actinodaphne madraspatna* Bedd leaves extract in the formation of FeONPs. The major significant peaks at 3422, 1645, 1404 and 1091 cm^{-1} were obtained for *Actinodaphne madraspatna* Bedd leaves extract (Fig. 3b). The strong band at 3422 cm^{-1} represents the O-H stretching vibration of hydroxyl group of phenolic compounds [2]. The intense peak at 1645 cm^{-1} is attributed to the C=C ring stretching of polyphenols [10]. The peak at 1404 and 1091 cm^{-1} were due to in-plane bending vibrations of -OH group in phenol and ester bond of tannin, respectively [15,16]. Appearance of band corresponding to these functional groups suggested that the phytoconstituents such as flavonoids, alkaloids, saponin, triterpenoids and carbohydrates were present in *Actinodaphne madraspatna* Bedd leaves extract as supported by earlier reports [5,6,17,18]. The slight shift was observed for the synthesized FeONPs (Fig. 3a), probably implied that *Actinodaphne madraspatna* Bedd leaves extracts played a dual role as capping as well as reducing agent in the green synthesis process. The stability of nanoparticles was examined using zeta potential measurement (Fig. 4). The synthesized FeONPs exhibited a zeta potential of -21.3 mV reflecting a reasonable stability, which is attributed to the phytomoities adhered on the surface of the nanoparticles. Also, no noticeable flocculation or sedimentation was found even after a week, suggesting the polyphenol capped nanoparticles without a chance for agglomeration.

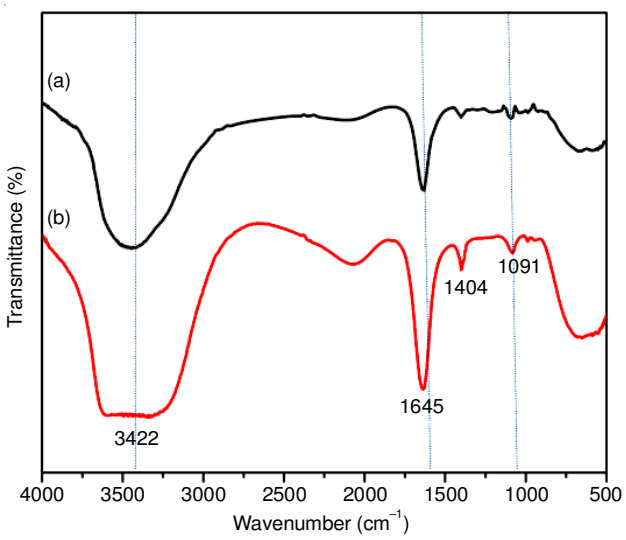

Fig. 3. FTIR spectra of (a) synthesized FeONPS and (b) dried powder of AMB leaves

Conclusion

In this study, iron oxide nanoparticles (FeONPs) were successfully synthesized *via* facile, green and eco-friendly method using *Actinodaphne madraspatna* Bedd leaves as reducing agent for the first time. The utilization of environmentally benign and abundantly available material (*Actinodaphne madraspatna* Bedd leaves) in the absence of any toxic solvent or medium was considered as an intriguing feature for this green synthesis process when compared to conventional chemical methods.

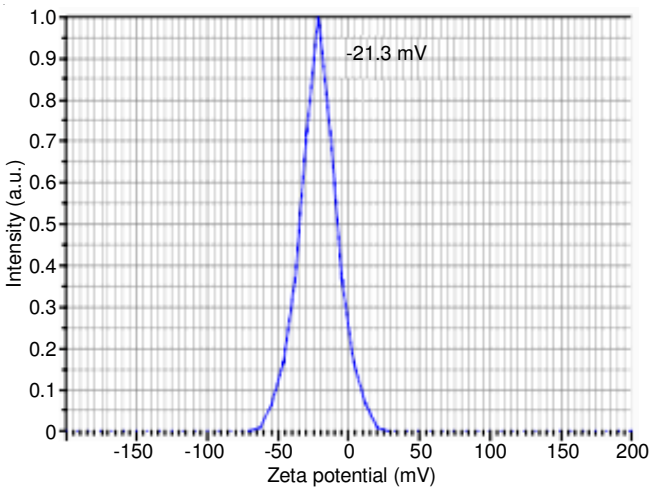

Fig. 4. Zeta potential studies of synthesized FeONPs

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