Green synthesis of iron oxide nanoparticles from *Mimosa pudica* root extract

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Abstract: An aqueous root extract of *Mimosa pudica* was used to synthesise iron oxide nanoparticles. The formation of iron oxide nanoparticles was observed on exposure of the aqueous root extract with the ferrous sulphate solution. The iron oxide nanoparticles were characterised using UV-Visible spectroscopy, Fourier transform infrared (FTIR) spectroscopy, X-ray diffraction (XRD), scanning electron microscopy (SEM), particle size analyser (PDA) and vibrating sample magnetometer (VSM). UV-Vis is a spectrum of iron oxide nanoparticles showed a sharp peak at 294 nm due to the surface plasmon resonance. FTIR spectroscopy confirmed the attachment of bioactive molecules of plant on the iron oxide nanoparticle surfaces. The phase and crystal structure were determined through XRD. The scanning electron microscopy (SEM) illustrated that the iron oxide nanoparticles were spherical in shape with 67 nm of surface volume mean diameter. Magnetisation measurements indicates that the synthesised iron oxide nanoparticles exhibited superparamagnetic behaviour at room temperature.

Keywords: iron oxide; *Mimosa pudica*; nanoparticles; superparamagnetic.

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1 Introduction

Nanotechnology deals with the creation and consumption of materials, devices and systems through the control of matter at the nanoscale (i.e., at the level of atoms, molecules and supramolecular structures) (Chiu, 2010). Nanotechnology is a reliable and environmentally friendly process for the synthesis of nanoscale particles. Nanoparticles have attracted much attention due to their unusual electronic, optical and magnetic properties (Narayanan and Sakthivel, 2010). Nano results in specific physicochemical characteristics such as high surface to volume ratio. Nanotechnology also pursues to discover, describe and manipulate those unique properties of matter at a nanoscale in order to develop new capabilities with potential applications across all fields of science, engineering, technology, and medicine. A significant progress has been made during the last decade in 'engineering on the nanoscale'. A large variety of inorganic materials and compounds have successfully been synthesised in nano level. In recent years, researchers in the field of nanotechnology have been finding that there is an expanding research in the synthesis of metal nanoparticles due to the potential applications for the development of novel technologies. Metallic nanoparticles are widely studied because of their wide applications in areas such as coatings, packaging, electronics, cosmetics and biotechnology (Jain et al., 2008).

Among the various nanoparticles (gold, silver, copper, iron, palladium, zinc and quantum dots (CdS, ZnS)), iron nanoparticles are found to have a great potential due to their unique micro configuration and properties like superparamagnetism and high

coercive force. They are used for a wide range of applications like magnetic storage media (Sun et al., 2000), ferrofluids (Miller et al., 2002), biosensors (Zhang et al., 2005), catalysts (Mahdavi et al., 2013a), separation processes and environmental remediation (Abhilash, Revati and Pandey, 2011). Specifically, magnetite (Fe_3O_4) nanoparticles have been focused recently as it exhibits unique electric and magnetic properties based upon the transfer of electrons between Fe^{2+} and Fe^{3+} in octahedral sites. Moreover, magnetite is biocompatible and they have been actively investigating for targeted cancer treatment (magnetic hyperthermia), stem cell sorting and manipulation, DNA analysis, guided drug delivery, gene therapy and magnetic resonance imaging (MRI) (Fan, Chow and Zhang, 2009).

Nanoparticles, in general, can be synthesised by two alternative approaches: the 'bottom-up' approach and the 'top-down' approach. In the bottom-up approach, the nanostructured building blocks (i.e., nanoparticles) are initially formed and then assembled into a final material using the chemical or biological procedure(s) (Sathishkumar et al., 2010). In the top-down approach, a suitable starting material is reduced in size using physical (e.g. Mechanical) or chemical means (Kalainila et al., 2014a). It is possible to obtain metallic nanoparticles with comparatively lesser defects and more homogeneous chemical composition(s) in the bottom-up approach, which is a distinct advantage (Thakkar, Mhatre and Parikh, 2010).

A variety of methods have been reported in the literature to synthesise superparamagnetic Fe_3O_4 nanoparticles, such as co-precipitation of ferrous (Fe^{2+}) and ferric (Fe^{3+}) ions (Arsalani, Fattahi and Nazarpoor, 2010), sol-gel synthesis (Lemine et al., 2012), sonochemical route (Dang et al., 2008), electrochemical synthesis (Ramimoghadam, Bagheri and Hamid, 2014), etc. However, the most common method for the production of superparamagnetic iron oxide nanoparticles is the chemical co-precipitation technique (Laurent et al., 2008).

Although chemical methods are most widely used to synthesise iron oxide nanoparticles, but the usage of chemicals involved were found to be toxic, there may be formation of hazardous by-products and there can be potential contamination from precursor chemicals (Huang et al., 2014), thereby greatly limiting their biomedical applications, particularly, in the medical field. Hence, the development of non-toxic, reliable and environmentally friendly methods for synthesis of nanoparticles is of utmost importance to expand their biomedical applications.

Over the past two decades, there has been increased emphasis on the topic of green chemistry and chemical processes. Utilisation of non-toxic chemicals, environmentally benign solvents, and renewable materials are considered to be the key issues that provide advantages towards the green synthesis strategy. One of the main concern in the preparation of superparamagnetic iron oxide nanoparticles is the choice of the reducing agent. Green synthesis of iron oxide nanoparticles is providing more advantage when compared with a chemical method that helps to overcome the concerns related to using hazardous chemicals like sodium borohydride as a reducing agent (Hoag et al., 2009).

The green synthesis of iron oxide nanoparticles using carob leaf extract as a reducing agent has been reported by Awwad and Salem (2012). Shahwan et al. (2011) synthesised iron nanoparticles using green tea leaf extracts (GT-Fe NPs), which is known to contain polyphenols that act as both reducing and capping agent. Demir, Topkaya and Baykal (2013) used maltose as the reductant and surfactant to synthesise the superparamagnetic Fe_3O_4 nanoparticles. Cai et al. (2010) used the soybean sprout (SBS) for the synthesis of superparamagnetic Fe_3O_4 nanoparticles. Lu et al. (2010) synthesised superparamagnetic

Fe_3O_4 nanoparticles using α-D-glucose as the reducing agent and gluconic acid (the oxidative product of glucose) as a stabiliser and a dispersant.

In the present work, the root extract of *Mimosa pudica* (common name: Sensitive plant) was used as the first time for synthesising superparamagnetic iron oxide nanoparticles. Aqueous Ferrous sulphate solution, after reacting with the root extract led to the formation of highly stable, crystalline iron oxide nanoparticles. The synthesised iron oxide nanoparticles were characterised using UV-Vis spectrometer, FTIR spectrometer, XRD, SEM, Particle size analyser and VSM.

2 Materials and methods

2.1 Materials

Materials with high-grade AR Ferrous Sulphate ($FeSO_4 \cdot 7H_2O$) were purchased from Sigma-Aldrich, India. Double distilled water was used throughout the experiment. Whatman no. 1 filter papers were used for filtration. All glassware used were washed well and dried using hot air oven before the experiment.

3 Plant description

Mimosa pudica (also known as the sensitive plant) contains a lot of biological compounds, which are useful for human health. The leaves of *M. pudica* (as shown in Figure 1a) contains mimosine (β-[N-(3-hydroxypyridone-4)]-α-aminopropionic acid), which is a non-protein alpha-amino acid. Roots of *M. pudica* (as shown in Figure 1b) contain tannin, ash, calcium oxalate crystals and mimosine (Ibrahim et al., 2014). Mimosine can inhibit the proliferation of a number of lung and liver cancer cells. It can also inhibit the development of bacteria and can be used as a skin bactericide. It is less soluble in methanol and ethanol, insoluble in other organic solvents, but sparingly soluble in water. It is soluble in dilute acid and base (Champanerkar et al., 2010). The structure of mimosine is given in the Figure 2.

Figure 1 (a) *Mimosa pudica* leaves and (b) *Mimosa pudica* roots (see online version for colours)

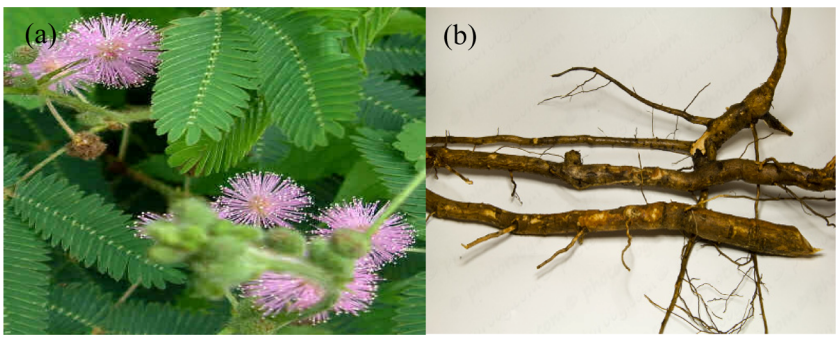
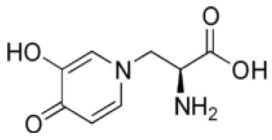


Figure 2 Structure of mimosine



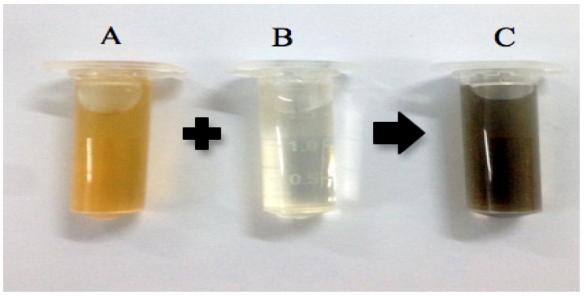
3.1 Preparation of extract

Fresh roots of *M. pudica* were collected from regions in and around Coimbatore, Tamilnadu, India. The root surface was cleaned with the help of running water, and distilled water and then chopped into small pieces. It was dried and crushed into a powdered form. Around five grams of powder was soaked in 100 ml distilled water. The root powder soaked in water was placed under vigorous magnetic stirring at a range of 80°C for 20 mins. Then it was filtered through Whatman filter paper and the collected filtrate was then stored at 4°C for further process (Saranyaadevi et al., 2014a).

3.2 Synthesis of iron oxide nanoparticles

Around three ml of the stock solution of root extract was added to 50 ml of the 20 mm Ferrous Sulphate solution. The light brown solution changed into a dark brown colour, which gave out colloids of iron oxide nanoparticles as shown in Figure 3. In order to enhance the superparamagnetic behaviour of the iron oxide nanoparticles, the pH of the solution was adjusted to nine by adding the NaOH solution in drops along with continuous stirring for 20 mins at 60°C (Faiyas et al., 2010). The colloidal solutions were then centrifuged at 6000 rpm for 15 mins. The collected precipitate from centrifugation was dried and utilised for further characterisation. The experiments were repeated thrice to access the consistency and reproducibility of nanoparticles thus produced (Rai et al., 2008).

Figure 3 Root extract of *Mimosa pudica* (A), 20 mM ferrous sulphate solution (B) and iron oxide nanoparticles (C) (see online version for colours)



3.3 Characterisation

The synthesised iron oxide nanoparticles was monitored by UV-Vis which is a spectrum of the reaction medium after diluting the solution with a range of 100–900 nm. The UV-Visible spectra were recorded on a UV-Visible Jasco V-550 spectrophotometer. The biomolecules responsible for reduction and stabilising the iron oxide nanoparticles

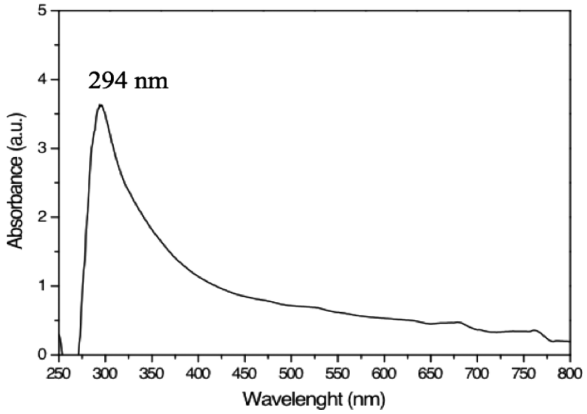
present in the root extract of *M. pudica* was analysed using FTIR spectroscopy. The root extract was dried and ground with KBr to form a pellet and analysed using FTIR instrument (Jusco 5300) with the range of 400–4000 cm^{-1}. XRD analysis was performed to identify the crystal phase and the structure of the synthesised iron oxide nanoparticles. This study was made on the powdered samples at room temperature (27°C) on a Rigaku X-ray diffractometer. The surface morphology of the iron oxide nanoparticles was studied by scanning the electron microscopy (SEM). To perform SEM characterisation, the suspensions of iron oxide nanoparticles were evaporated onto a clean glass slide. The samples were covered and left to dry completely at room temperature. SEM analysis was done using Vega3 Tescan with a resolution of 1 μm at 7 Kv. The average size of the particles, size distribution and poly dispersity index (PDI) of the synthesised iron oxide nanoparticles were determined on a Malvern zetasizernano **S-**series particle analyser using water as a dispersant. To identify the magnetic property of the synthesised iron oxide nanoparticles, the synthesised iron oxide nanoparticles were dried to a fine powder. The powdered sample was analysed on Lakeshore VSM 7410 at room temperature.

4 Results and discussions

4.1 UV-Visible spectroscopy

UV-Visible spectrum of iron oxide nanoparticles is shown in the Figure 4. Here the sharp peak at 294 nm indicates the presence of iron oxide nanoparticles. Parallel results were observed by Arokiyaraj et al. (2013). *M. pudica* root extract was shown to reduce the iron oxide nanoparticles by the indication of suitable surface plasmon resonance (SPR) with high band intensities and peaks under visible spectrum (Awwad and Salem, 2012).

Figure 4 UV-Vis spectrum of iron oxide nanoparticles



The outer electrons of the materials in their atomic state absorbs the radiant energy and undergoes transition to a higher energy level. The energy band gap of the nanomaterials will be obtained from the equation given below (Pattanayak et al., 2013). It shows that the broad absorption band at 294 nm is indicating the formation of the Fe_3O_4 nanoparticles (Figure 1).

$$E_{bg} = 1240/\lambda \text{ (eV)} \quad (1)$$

Where E_{bg} is the energy band gap and λ represents the wavelength (294 nm) of the nanoparticles. The energy gap was calculated as 4.217 eV at 294 nm. A similar type of result was previously reported by Awwad and Salem (2012) at 233 nm.

4.2 FTIR spectroscopy

FTIR analysis was performed to identify the biomolecules responsible for the reduction of iron oxide nanoparticles present in the root extract of *M. pudica.* Figure 5 shows the FTIR spectrum of the root extract. The strong broad peak at 3417 cm^{-1} is due to the stretching vibrations of O-H group attributed to water which is present in the root extract (Kumar et al., 2013). The peak at 2922 cm^{-1} is assigned to the asymmetric stretching vibrations of CH_2 in aliphatic hydrocarbon. The peak at 1415 cm^{-1} is due to the C-C groups derived from aromatic rings that are present in the root extract (Mahdavi et al., 2013b). The peak at 1074 cm^{-1} is due to the stretching vibration of C-N group in aliphatic amine. The peak at 2395 cm^{-1} is assigned for superimposed O-H stretching (Jana and Biswas, 2011). The strong band at 2354 cm^{-1} is due to C-H stretching vibration (Sepperumal and Markandan, 2014). The strong peak at 1628 cm^{-1} is due to the stretching vibration of C-C bond in the benzene ring. The above results predict the presence of mimosine (β-3-hydroxy-4 pyridone amino acid), which is a non-protein alkaloid present in the root extract of *M. pudica.*

Figure 5 FT-IR spectrum of root extract of *Mimosa pudica*

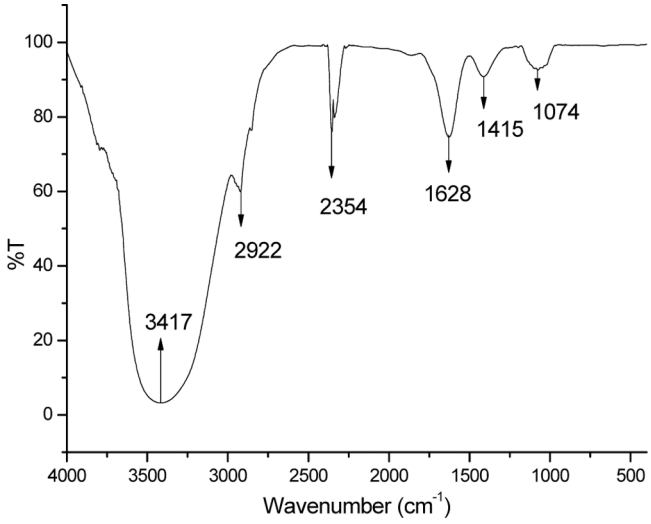
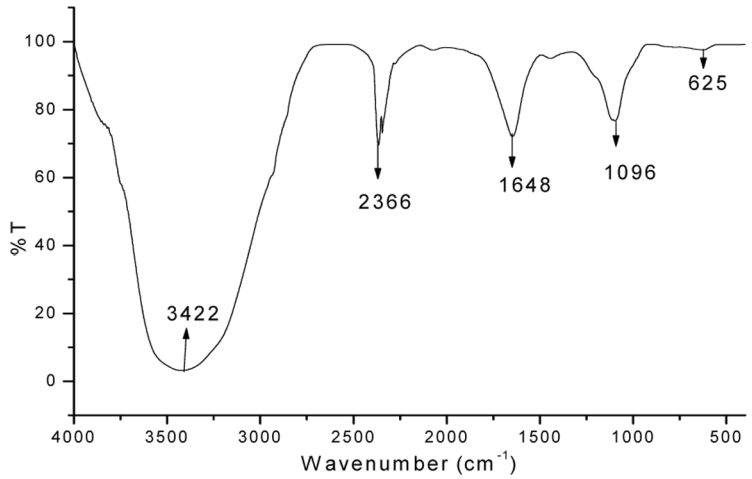


Figure 6 shows the peaks associated with iron oxide nanoparticles. The peak at 3422 cm^{-1} corresponds to the stretching vibrations of O-H. The peak at 2366 cm^{-1} corresponds to the stretching vibration of C-H bond. The strong peak at 1648 cm^{-1} corresponds to the stretching vibration of acyclic C-C. The strong peak at 1096 cm^{-1} is the characteristic IR peak for asymmetric SO_4 (Periasamy, Muruganand and Palaniswamy, 2009). When comparing Figure 5 with Figure 6, there are shift in bands from 2354 cm^{-1} to 2366 cm^{-1} and 1628 cm^{-1} to 1648 cm^{-1}, which are attributed to the binding of C-H group and C-C

group with the nanoparticles respectively (Gardea-Torresdey et al., 2002). Vibrations of Fe-O bonds of iron oxide could demonstrate the presence of iron oxide nanoparticles by the absorption band at around 625 cm^{-1} (Xua et al., 2013). FT-IR spectroscopy confirmed that the *M. pudica* root extract has the ability to act as a reducing agent and stabiliser for iron oxide nanoparticles. The FT-IR result confirmed the presence of mimosine in the root extract and its ability to reduce ferrous sulphate to iron oxide nanoparticles.

Figure 6 FT-IR spectrum of iron oxide nanoparticles



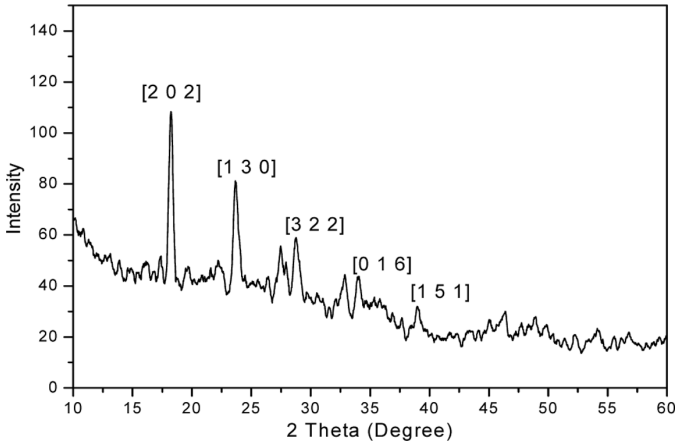
4.3 X-ray diffraction (XRD)

XRD is an effective technique to identify the phase and to confirm the crystal structure of the synthesised iron oxide nanoparticles. The X-ray diffraction patterns obtained for the iron oxide nanoparticles synthesised using the root extract of *M. pudica* is shown in Figure 7. The XRD pattern displayed five characteristic 2θ peaks at 18.2°, 23.6°, 28.7°, 33.9° and 39° marked by their indices (2 0 2), (1 3 2), (3 2 2), (0 1 6) and (1 5 1) respectively. They are quite identical to the peaks of Fe_3O_4 crystal with the orthorhombic structure. The results are in agreement with the standard XRD pattern of Fe_3O_4 (JCPDS 76-0958). Hence, from the XRD result, it was clear that iron oxide nanoparticles formed using *M. pudica* root were crystalline in nature. The average particle size of the synthesised iron oxide nanoparticles was calculated using Debye-Scherrer formula as given in Equation 2,

$$D = K\lambda/(\beta \cos \theta) \quad (2)$$

Where D is the mean diameter of nanoparticles, β represents the full width at half-maximum value of XRD diffraction lines, λ represent the wavelength of X-ray radiation source 0.15405 nm, θ is the half diffraction angle (Bragg angle) and K represent the Scherrer constant with value from 0.9 to 1 (Saranyaadevi et al., 2014b).The particle size was determined by taking the average of the sizes at the peaks and it was found to be 25.6 nm (Mandela et al., 2014).

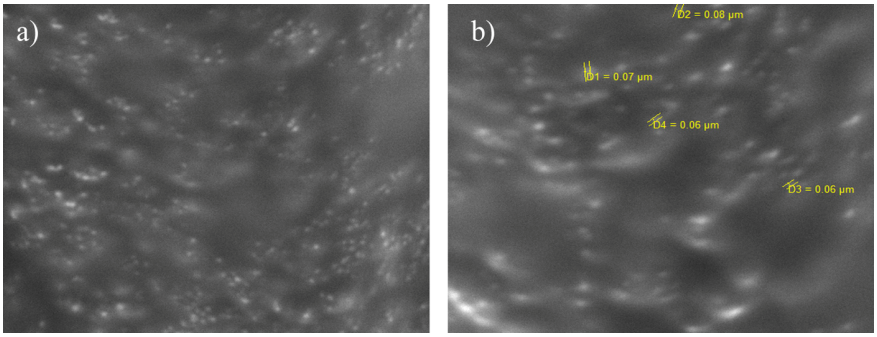
Figure 7 X-ray diffractogram of iron oxide nanoparticles



4.4 SEM analysis

The surface morphology and size of particles were determined by Scanning electron microscopy (SEM) analysis. SEM image in Figure 8(a) shows the synthesised iron oxide nanoparticles are well dispersed and roughly spherical in shape. Figure 8(b) shows that the iron oxide nanoparticles are in the size range of 60–80 nm with mean size of 67 nm. The iron oxide nanoparticles are to some extent agglomerated due to the interaction between magnetic nanoparticles (Arokiyaraj et al., 2013).

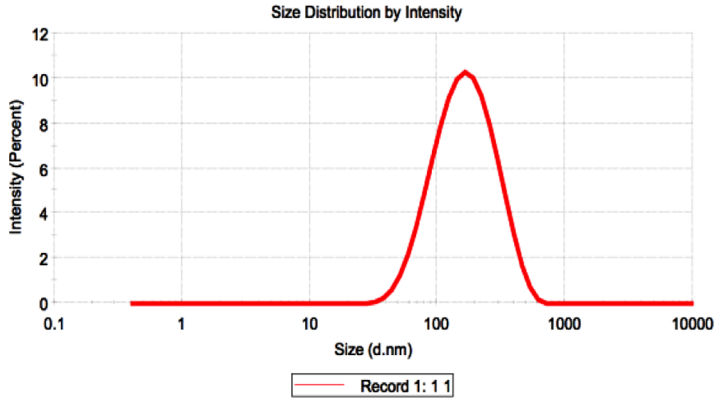
Figure 8 (a) SEM image showing well-dispersed iron oxide nanoparticles and (b) SEM image showing the measured size of synthesised iron oxide nanoparticles (see online version for colours)



4.5 Particle size analysis (PD)

The average size of the particles, size distribution and polydispersity index (PDI) of the synthesised iron oxide nanoparticles were determined by particle size analyser and the results are shown in Figure 9. It revealed that the particle size distribution of iron oxide nanoparticles ranging approximately from 32 to 600 nm with mean particle size of 147 nm and the polydispersity index was found to be 0.240. The results revealed that the distribution of iron oxide nanoparticle is more uniform with a narrow distribution range (Dwivedia et al., 2014).

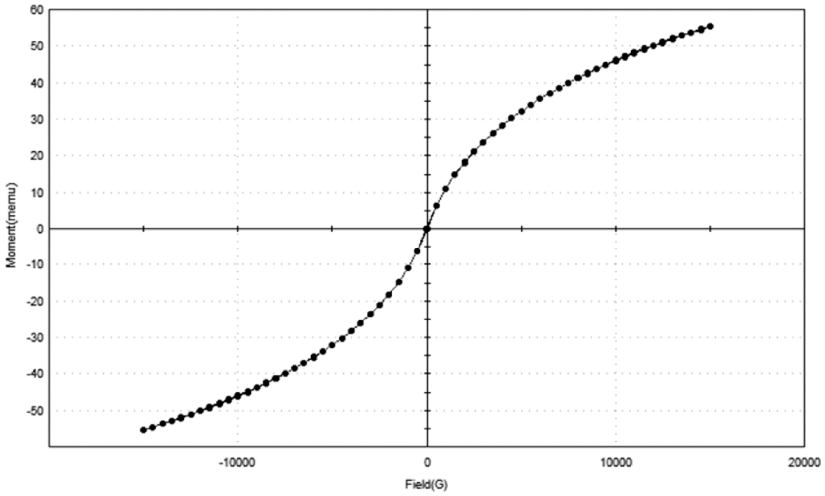
Figure 9 Particle size distribution in iron oxide nanoparticles (see online version for colours)



4.6 Vibrating sample magnetometry (VSM)

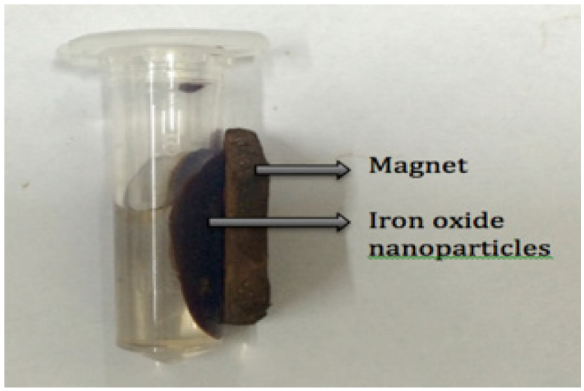
In order to study the magnetic behaviour of iron oxide nanoparticles, magnetisation measurements recorded with VSM were performed. Figure 10 shows the relative magnetisation curve as a function of magnetic field for the uncoated particles. No hysteresis curve was observed in the figure, which specifies the superparamagnetic behaviour of the particles (Darroudia et al., 2014). The saturation magnetisation value of the magnetite nanoparticles was found to be 55.402 emu/g (electromagnetic units per gram). In addition, the magnetisation decreases from the plateau value and reaches zero when the magnetic field is removed. This behaviour shows that the iron oxide nanoparticles correspond to the single-crystal domain exhibiting only one orientation of the magnetic moment and are magnetite in structure. It was found that the magnetic nanoparticles were small enough to exhibit superparamagnetic behaviour thus (Lu et al., 2010), they are of particular interest in drug targeting systems, as they do not retain any magnetism after removal of a magnetic field (Demir, Topkaya and Baykal, 2013).

Figure 10 Magnetisation curve of iron oxide nanoparticles at room temperature



The iron oxide nanoparticles exhibited a magnetic property in the presence of a magnetic field. When a magnet was placed near the glass bottle, it was observed that the iron oxide nanoparticles were attracted towards the magnet, which is shown in Figure 11. The magnet attracted the dark brown nanoparticles and when the magnetic force is removed, the nanoparticles were easily dispersed by simply shaking them. Thus, the magnetic nanoparticles can be removed or recycled in the medium with a simple magnetic device (Kumar et al., 2014).

Figure 11 Separation of iron oxide nanoparticles from root extract of *M. pudica* under an external magnetic field (see online version for colours)



5 Conclusion

In this present investigation, superparamagnetic iron oxide nanoparticles were successfully synthesised from the root extract of *M. pudica*, for the first time. Magnetic measurement confirmed that the iron oxide nanoparticles showed superparamagnetic behaviour at room temperature. Iron oxide nanoparticles were synthesised by reduction of ferrous sulphate solution using *M. pudica* root extract containing mimosine, which acts as the reducing agent. The involvement of functional group present in the biomolecules responsible for the reduction of iron oxide nanoparticles was revealed by FTIR analysis. From both the particle size analyser and SEM, it was observed that the synthesised iron oxide nanoparticles were found to be in nano size. Green synthesis of iron oxide nanoparticles using green resources is a simple, eco-friendly, pollutant-free and low-cost approach. The prepared iron oxide nanoparticles by green synthesis method can effectively be applied directly in the targeted drug delivery without any modification.

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