



Preparation of iron magnetic nanoparticles using aquatic macrophyte *Salvinia adnata* Desv and its application in dye decolorization

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Abstract

Currently, researchers are highly fascinated to the synthesis of nanoparticles, especially magnetic nanoparticles using biological materials. In the present research, synthesise of iron magnetic nanoparticles was done by using *S.adnata* Desv leaf extract as a reducing agent. Magnetic FeNP was synthesized by co-precipitation of ferric chloride and ferrous sulfate. The formation of black colored precipitate indicates the development of magnetic iron nanoparticles. The crystalline nature of the particle was analyzed by XRD and the size was 16.7 nm, functional group and capping agents were identified through FTIR and the results confirmed the synthesis of nanoparticles. The magnetic nature was authenticated by external magnetic force. The size and morphology of the particle were analyzed by SEM-EDS system. The nanoparticle was utilized for the decolorization of reactive- Blue FBS dye solution. Complete decolorization was obtained by the magnetic nanoparticles in acidic pH. This work proved the decolorizing efficiency of green synthesized magnetic nanoparticles.

Keywords: *salvinia adnata* desv, magnetic nanoparticle, decolorization, pH, temperature

1. Introduction

Nanotechnology is an emerging technique involved in the synthesis and utilization of nanostructured materials, systems and devices within nanoscale size. The fabrication of nano sized molecules was based on the morphological nature, size and constituents of chemicals with enhanced properties [1]. Currently, researchers are interested in the creation of iron based magnetic nanoparticles for various purposes in material sciences, chemistry, physics, biology because of their unique properties like higher magnetism and efficiency, low cost and less toxic to the environment [2]. Also, these nanoparticles are imported in medicine for drug delivery, nanorobotics, biosensor, remediation of environmental pollutants, potable water purification etc. [3]. Magnetic nanoparticles are synthesized by various chemical and a physical method comprising sol-gel, hydrothermal, co-precipitation, thermal decomposition, microemulsion, ultrasonication, solvothermal technique, elcetrochemical etc [4]. Nanoparticles can be fabricated by two strategies such as top-down approaches and bottom-up approaches. The nanomaterial produced from the bigger molecules as a starting material through ultrafine grinders, vaporization along with cooling of complex materials. This method is highly dependent on the surface morphology of the particle [5, 6]. Bottom-up approach is widely used for making nanomaterials by chemical and biological methods. In this, the particles are constructed by arranging tiny entities to larger nanoparticles with unique and applicable properties [7, 5]. The synthesis of nanoparticles by chemical methods are expensive and during the synthesis they produce toxic by product which contaminate the environment leading to biological risk [8].

Biogenic nanoparticles can be formed with plant extracts,

fruits, leaves, bacteria and fungi, marine algae, etc. Plant extracts act as reducing, capping and stabilizing agent for the formation of nanoparticles. These extracts are also involved in the characteristics of metallic nanoparticles [9, 10]. The uses of biological materials are eco-friendly and which diminishes the use of chemical based reducing agents. In the present research, magnetic nanoparticle was synthesized by using aquatic macrophyte *Salvinia adnata* Desv, a free floating fern, heterosporous, which form mats on the water surface with rich biomass. Dyes are the synthetic chemical compounds broadly used in textile, pharmaceuticals, food, cosmetics and paper industries for coloring the materials. The release of dye containing wastewater to the aquatic environment prevents the passage of solar light to the water and inhibits the photosynthesis process of aquatic flora, also, makes it become unfit for consumption [11]. The removal of color from the effluent is the need of the hour before discharging from the industries. So, an attempt was taken to remove color from the aqueous solution of reactive blue dye using green magnetic nanoparticles. This work has not been reported and thus we are reporting the reducing capability of *S.adnata* Desv.

2. Materials and methods

2.1 Scientific classification of *S.adnata* Desv

Domain	: Eukaryota
Kingdom	: Plantae
Phylum	: Pteridophyta
Class	: Filicopsida
Order	: Hydropteridales
Family	: Salviniaceae
Genus	: <i>Salvinia</i>
Species	: <i>adnata</i> Desv

Salvinia adnata Desv is a free floating aquatic plant widely distributed all over the world (Figure-1).



Fig 1: *S. adnata* Desv biomass in a pond

2.2 Preparation of plant extract

S.adnata Desv plant was collected from a freshwater pond and was washed thoroughly with tap water and distilled water to remove the dirt. About 5gm of fresh plant was ground with 100ml of distilled water. The content was boiled for 10 minutes and was filtered with cheese cloth. The extract was used for the fabrication of magnetic nanoparticles.

2.3 Preparation of Iron Magnetic nanoparticle

The preparation of nanoparticle was carried out by the co-precipitation of Ferric chloride and Ferrous sulfate in the required volume and stirred thoroughly under magnetic stirrer at 80°C for 10 minutes. Equal volumes of plant extract and ferric-ferrous solution (1:1 ratio) were mixed and incubated at 40-60°C for 10-20 minutes. Following which, ammonia solution was added drop wise to form the black precipitate, which indicates the synthesis of magnetic nanoparticles [12].

2.4 Magnetic nanoparticle purification

The black precipitate was washed properly with sterile deionized water to remove excess amounts of ammonia and neutralize the pH. Finally, the precipitate was rinsed with ethanol or acetone and was dried under vacuum [13, 14].

2.5 Characterization of Magnetic Nanoparticles

The purified magnetic nanoparticle was characterized by powder XRD (model-Bruker D8) which analyzes the crystalline structure and size calculation by the following Scherer's equation.

$$D_n = \frac{k\lambda}{\beta \cos\theta}$$

Where, D_n is the mean crystalline size, k is the constant value (0.9), λ refers to wavelength of radiation, β is mentioned as full width half maximum (FWHM) of 2θ scale and Bragg angle is noted as θ [15].

The functional groups of the plant involved in the capping of nanoparticle were identified by FTIR spectrometry (Model-Perkin Elmer Spectrum). The morphological characteristics of the particle and elemental analysis were made by SEM-EDX analysis.

2.6 Decolorization of Reactive dye- Blue FBS

The decolorization power of dye was studied under static condition and the pH, temperature and dosage (1-6mg) of nano particles was optimized. Different concentration of dye solutions (1-5ppm) were prepared with various pH such as 2, 4, 7, 9, 11 and the effect of pH on the dye decolorization was studied at different temperatures like 20°C, 40°C, 60°C, 80°C and 100°C on dye removal. After treatment, the magnetic nanoparticle was separated by external magnetic field for reuse. The color reduction percentage was measured by the following equation [16]. Where, A_0 is the initial absorbance of the dye solution and A_t is the absorbance after treatment.

$$\text{Color removal \%} = \frac{A_0 - A_t}{A_0} \times 100$$

2.7 Antibacterial activity of Magnetic nanoparticles

The antibacterial activity of magnetic nanoparticle was studied by Bauer-Kirby disc diffusion test. Four strains such as *E.coli*, *Bacillus subtilis*, *Staphylococcus aureus* and *Klebsiella pneumoniae* were chosen. The antibacterial activity is measured by the formation of zone of inhibition in the plate [17].

3. Result and Discussion

3.1 Synthesis of magnetic nanoparticles

The synthesis of green magnetic nanoparticle using plant extract was confirmed by the formation of black precipitate. The magnetic particle aggregation in the solution was separated by external magnetic field and it confirmed the magnetic properties of the particles. Maheswari and Sreenivasalu [18] also reported the similar view. The flavonoids such as polyphenol in the *Salvinia sp.* Mitch plant might have been facilitated the reduction and synthesis of magnetic nanoparticles [19]. The formation of magnetic nanoparticles is depicted in the figure-2.

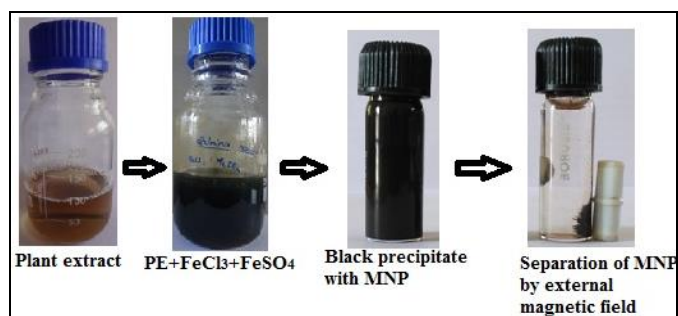


Fig 2: Synthesis of Magnetic nanoparticle using *S.adnata* Desv plant extract

3.2 Characterization of MNP

3.2.1 XRD Analysis of magnetic nanoparticle

The crystalline structure of the particle was characterized by powder XRD. The XRD pattern showed the presence of peaks with the intensity of 30.962°, 37.392°, 44.024°, 57.883° and 63.487° (Figure-3). The crystal planes of the particle were denoted by Miller indices (220), (222), (104), (115) and (440) (JCPDS File No.-01-076-1470) were corresponding for the observed intense peaks. The crystalline structure was

determined by Scherrer equation and the average size was 16.7nm. Similar results were reported by Khalil ^[20] and Keyhanian *et al.*, ^[21].

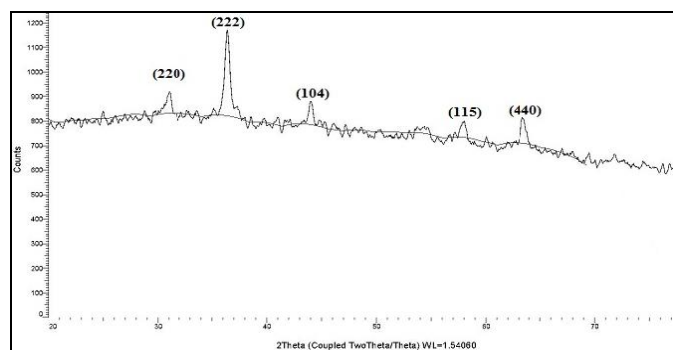


Fig 3: XRD pattern of Magnetic nanoparticle synthesized by *S.molesta*

3.2.2 FTIR analysis of Magnetic nanoparticles

The FTIR spectrum was observed from the ranges 400-4000 cm^{-1} (Figure-4). The spectrum showed the presence of peak value at 3422 cm^{-1} indicates the hydroxyl group and the spectrum in the 1640 cm^{-1} region responsible for the carbonyl group (C=O). The peak found in 1113 cm^{-1} pointed out the C-O acid anhydride stretching. The spectrum detected in the range 595 cm^{-1} was formed due to the existence of Fe-O bond. This functional group confirmed the synthesis of magnetic nanoparticle. In 2017, Ayad *et al* ^[22] also confirmed the occurrence of magnetic nanoparticles with the peak 595 cm^{-1} in their study. These spectrum peaks corresponding to the functional groups of the plant compounds which bound the particles and acts as a capping agent and stabilizing.

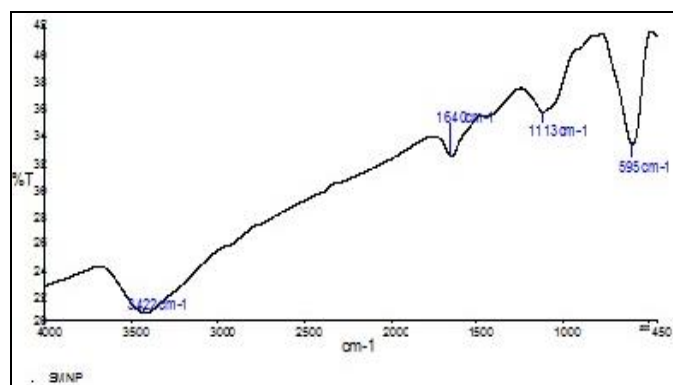


Fig 4: FTIR spectrum of Magnetic nanoparticle synthesized by *S. adnata Desv*

3.2.3 SEM- EDX analysis of Magnetic nanoparticle synthesized by *S. adnata Desv*

The SEM-EDX results are demonstrated in Figures 5 & 6. The morphological characteristic of the particle was found to be aggregated, irregular, spherical or cubical shape and the particle size were magnified in different ranges. Cubical morphology of the magnetic nanoparticle was recorded in *Camelia sinensis* by Shojaee and Shahri ^[19]. The EDX analysis illustrates the elemental factor of the synthesized particles. The spectrum showed the presence of strong peaks

of Fe and O molecules. The weight percentage of Fe was 64.34% and O was 35.66%. The purity of the particle was indicated by this elemental analysis. Hariani *et al.*, ^[23] also described the purity of magnetic nanoparticles by EDX analysis.

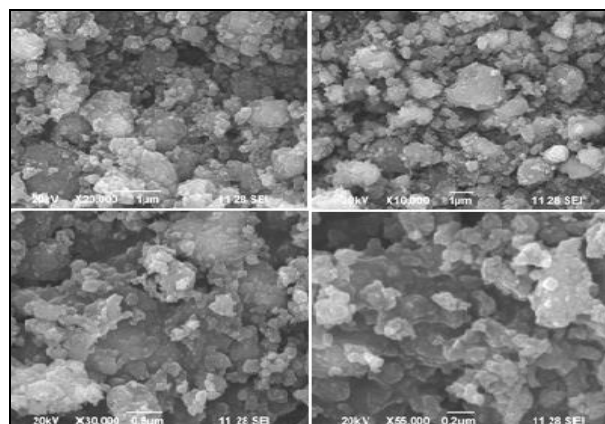


Fig 5: SEM analysis of Magnetic nanoparticle synthesized by *S. adnata Desv*

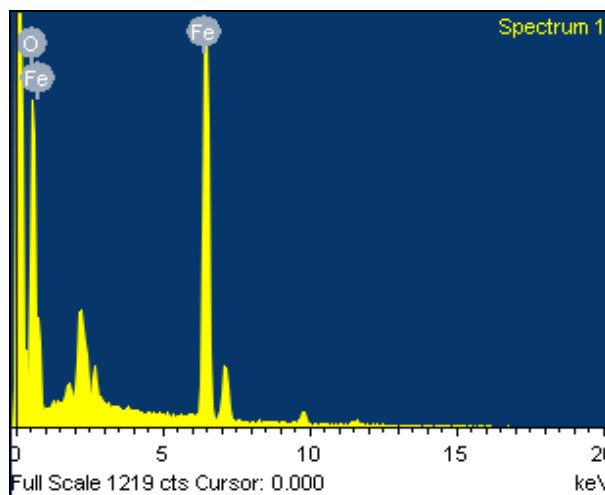


Fig 6: EDAX spectrum of Magnetic nanoparticle synthesized by *S. adnata Desv*

3.3 Decolorization of reactive- Blue FBS

The green synthesized magnetic nanoparticle was subjected to decolorize aqueous solution of reactive blue-FBS dye under optimum pH and temperature (ie) 4 and 25°C respectively. Complete color reduction was observed after 10 minutes of retention time in 5ppm concentration of aqueous dye solution (Figure-7). Among the different dosages, 5mg of magnetic nanoparticles shown complete removal than other dosages. Reactive dyes are anionic and water soluble dye used for coloring cotton, wool, silk and nylon. These molecules bind with fibers through covalent bonding with the dye reactive sites under controlled pH and temperature ^[24]. Kyzas *et al.*, ^[25] reported the decolorization of reactive black 5 by magnetic graphene oxide under standardized acidic pH at 25°C. The results agreed with the findings of Qadri *et al.*, ^[26]. They described the color reduction of acridine orange using magnetic nanoparticle at pH 4.



Fig 7: Decolorization of reactive- Blue FBS using Magnetic nanoparticles

3.4 Antibacterial activity of SMNP

The antibacterial activity of *Salvinia adnata* Desv nanoparticles was studied and the results are illustrated in the Figure - 8. The results showed that higher zone of inhibition 16mm against *Bacillus subtilis* than other selected strains. The inhibition zone for *E.coli*, *Staphylococcus aureus* and *Klebsiella pneumoniae* were found to be 12mm, 15mm and 13mm respectively. The bactericidal effect of magnetic nanoparticle brought by adsorbing on to the bacterial membrane surface and produces an internal reaction in the cytoplasm [27]. Lakshmanan *et al.*, [28] also reported the microbial reduction by magnetic nanoparticles in their analysis.

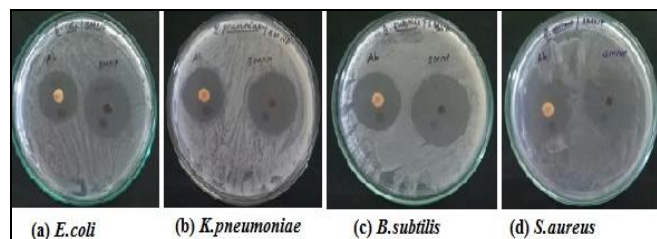


Fig 8: Antibacterial activity of SMNP

4. Conclusion

The present research confirmed the reducing and stabilizing capability of *Salvinia adnata* Desv plant extracts for the synthesis of iron magnetic nanoparticles. The characterization techniques confirmed the formation of magnetic nanoparticles. The complete decolorization of reactive blue dye was obtained at optimum pH 4, temperature 25°C and 5mg of magnetic nanoparticles. The antibacterial activity test also proved the efficiency of magnetic nanoparticles against the pathogenic bacteria. This revealed the disinfecting capability of the synthesized nanoparticles and it can be used not only for dye industrial waste water, but could be used also for the treatment of potable water. Plant based nanoparticle synthesis is an eco-friendly approach which reduces the use of toxic chemicals; it is also low cost and less toxic when compared with other metallic nanoparticles. This particle can be separated by applying an external magnetic field easily and could be reused for further treatment, which is highly appreciated.

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