Calotropis Gigantea Synthesis to Prepare Fe_3O_4 Nanopowder in Advance Food Analysis

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Abstract: ***Recently, a number of reports have been published on plant mediated NPs synthesis. The green synthesis method to prepare nanomaterial is environment-friendly, and the importance of nanomaterial is emerging with new ideas and advanced technology. This paper deals with the novel preparation method of Fe_3O_4 nanomaterial by taking Calotropis Gigantea leaves as a natural polymer. The green synthesis has been used as an environment friendly method which would not be harmful to the environment. The as-prepared nanomaterial is characterized using XRD, SEM and FTIR to study their properties which will help to give its usefulness in the field of food analysis. It is very necessary to develop various methods to characterize nanomaterials in food products.***

Keywords: Green Synthesis, Characterization, Calotropis Gigantea, Fe_3O_4, SEM, FTIR, XRD, Food analysis

1. Introduction

Nanoparticles (NPs) are at the front line in the rapid development in the nanotechnology and are subatomic particles whose diameter ranges from 1 to 100 nm. There are also certain nanoparticles whose size are several hundred nanometers and are, combinations of both inorganic and organic materials that have various novel characteristics as compared with the huge materials [1]. Fe_2O_3/Fe_3O_4 is a chemical composite that has been composed of iron and oxygen and there is 16 known Fe oxide as well as oxyhydroxides [2]. Iron oxide NPs particles have various magnetic properties. For this reason, magnetic nanoparticles (MNPs) are of great interest to researchers [3- 11].

Recently, nanoscale transitions of metal oxides (MO) such as iron oxides which include hematite, magnetite, and maghemitearegaining interest. Development of storage media, gas sensors, electronic and optical equipment, information storage, color imaging, magnetic heat cooling, bioprocess engineering, ferrofluid technology as well as adsorbents for the treatment of wastewater [12-14].

Magnetic NPWs can also be used in bioapplications, including enzymes of magnet, bacterial, virus, etc., clinical diagnosis, treatment, as well as biological labels. Choosing the substances for the fabrication of nanostructure is very decisive. For this purpose, magnetic iron oxide nanoparticles (MIONPs) have become a very important candidate [15]. However, it has been very challenging to limit the size, shape and stability of the NPWs in the desired solvent. The shapes of nanomaterial have major impacts on their properties, which also include catalysis. [16].

The method used so far for the preparation of IONPs are discharge, microemulsions, hydriothermal, Sol-Gel fabrication, chemical co-precipitation, etc. [17-19]. Mahdavi et al., has mentioned that magnetic nanopowder fabricated by green process is toxic free in comparison with the nanopartciles prepared using sodium borohydride. In current days, several studies have been carried out for the green preparation of iron-based nanopowder for the dye degradation of various textiles [20-23].

It has been observed that there is fast increase in food products containing nanomaterials, which is raising concerns over the impact of nano ranged particles (NPs) to humankind and the environment, although very less information have been gathered and published by the researchers or academicians about mineral filters in different food products.

Many studies show that the extract of this plant is successful in fabricating IONPs. This is the novel way of synthesizing iron oxide nanoparticles using ferric nitrate ($Fe(NO_3)_3$) and Calotropis Gigantea plant leave.

2. Materials and Methods

2.1 Plant Material

Calotropis Gigantea also known as the crown flower which grows to 4m. The classification of *Calotropis Gigantea* is given in the following:

Species: *Calotropis*
Habit: A large shrub.
Flowers: Large, white, lavender, not scented.

2.2 Collection and fabrication of Plant Material

Calotropis Gigantea fresh leaves free from diseases have been collected from local area of Dehradun. The leaves has been washed 2 to 3 times using tap water in order to remove the impurities. Approximately 50 gm of fresh leaves were finely cut, mixed with 100mL of distilled water and stirred.

2.3 Chemical

$Fe(NO_3)_3.9H_2O$ (Ferric Nitrate Nonahydrate salt), was procured from Merck, India and used without further purification.

2.4 Synthesis of Iron oxide Nanoparticles:

A step by step preparation method has been shown in the figure 1. For the natural synthesis of the nanopowder, the authors have used heating method which is a very available and suitable method in every laboratory. Green *Calotropis Gigantea* leaves were collected from the local region and of approximately 50 gm have been used for the preparations of nanopowder. The leaves at first, washed with distilled water in order to remove all the impurities present in the leaves.

Later on, small pieces of leaves were put into the beaker and mixed well with 100 ml of distilled water to make the homogeneous aqueous solution. In the another beaker $Fe(NO_3)_3.9H_2O$ as shown in figure 2(c), has been added which has been mixed well with 100 ml of distilled water and stirred vigorously till the salt gets mixed completely and formation of light brown colour occurs. In the next step, the two solutions have been mixed together in one beaker and heated at temperature of 80ºC for approximately 2 hours using the heating mantle.

The brownish colour precipitate settles down at the bottom of the beaker, which has been taken out and the precipitate has been dried in order to remove the moisture present in the particles. Now the as prepared mixture has been kept in the muffle furnace, and heated at different temperatures for about another 4.5 hours. After heating the precipitate in presence of oxygen using Muffle Furnace, the iron oxide nanopowder forms.

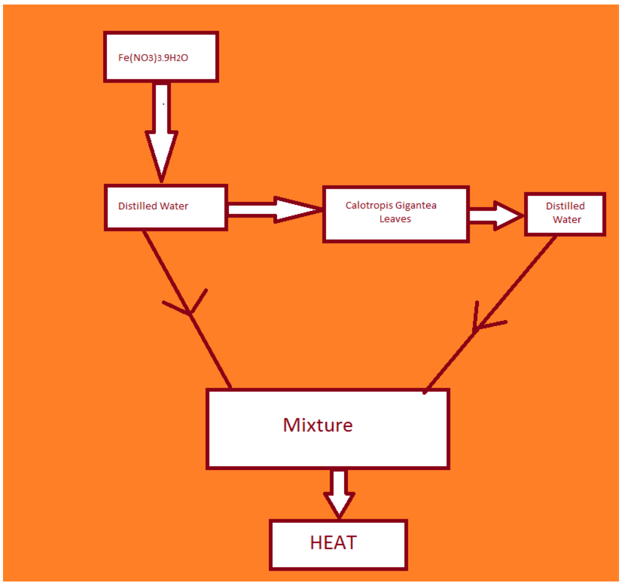

Figure 1: Step by Step preparation flowchart

Figure 1 represents the step by step flow chart for the preparation of iron oxide nanopowder using Calotropis Gigantea leaves as a natural polymer.

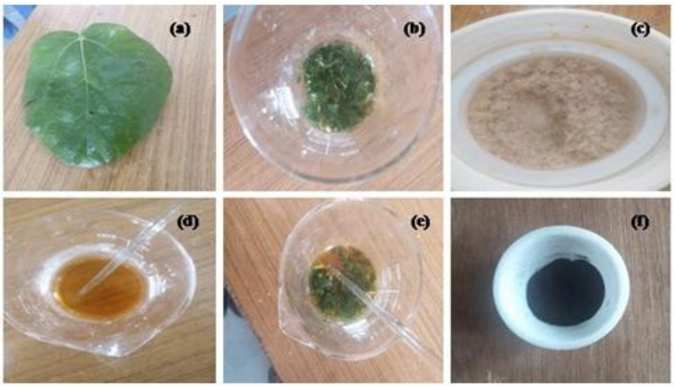

Figure 2: (a) Calotropis Gigantea leaves (Natural Polymer) (b) Mixture natural polymer with distilled water (c) Fe (NO3)3.9H2O (salt) (d) Mixture of salt with distilled water (e) Solution of salt and the natural polymer

3. Characterization

FT-IR spectrum (Thermoscientific; Nicolet summit LITE iD1) was recorded using KBr pellets in the range of 500-4000cm-1 [16]. The scanning electron microscopy (Zeiss; EVO18) was used for understanding morphological and structural features of NPs. The as prepared Fe_3O_4 nanopowder prepared via green synthesis method has been studied using SEM, FTIR and XRD.

3.1 Scanning Electron Microscopy

The substances have been subjected for analysis of SEM. Thin films of the sample has been fabricated on carbon coated grid of Cu by dropping small amount of the substance on the grid; extra solution has been then, removed with the help of blotting paper and then the film has been put on the SEM grid to dry for the result analysis purpose.

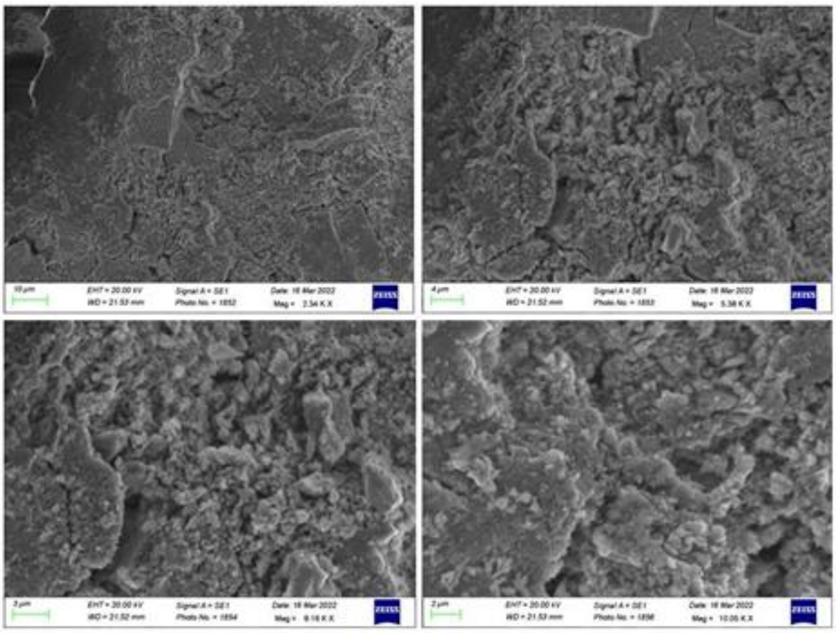

Figure 3: SEM images of Fe3O4 Nanopowder

3.2 Fourier Transform Infra-Red spectroscopy

For FTIR characterization, Fe3O4 nanopowders have been centrifuged for 30 minutes at 10000rpm. The substance has been washed three times using deionised water to remove the free particles those are not capped with the iron oxide nanoparticles. Using vacuum drier, the pellet has been dried.

FT-IR Spectral Analysis. The FTIR spectra of Fe3O4 NPs showed peaks at 3091.3, 2935.1, 2427.9, 1590.9, 1496.4, 1382.7, 1110.7, 1062.5, 559.25cm-1 (Figure 4). From those indicated peaks, the broad absorption peaks of Fe3O4 NPs observed at 3091.3cm-1 and 2935.1cm-1. The peak at 3091.3 cm-1 corresponds to the -OH bond.

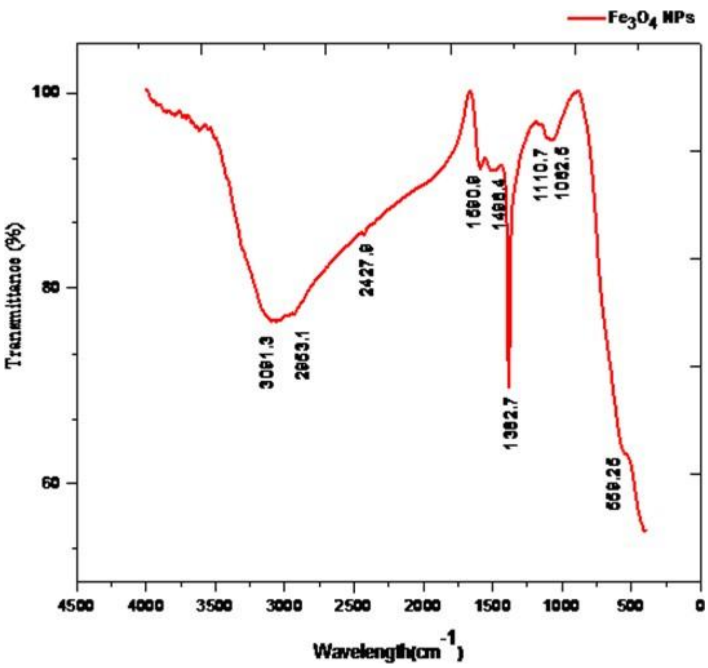

Figure 4: FTIR image of Fe3O4 nanopowder

3.3 X-Ray Diffractogram (XRD)

A thin film of Fe3O4 nanopowder has been prepared and XRD study has been carried out. The amorphous Fe3O4 nanopowder has been observed from the width of the XRD peaks.

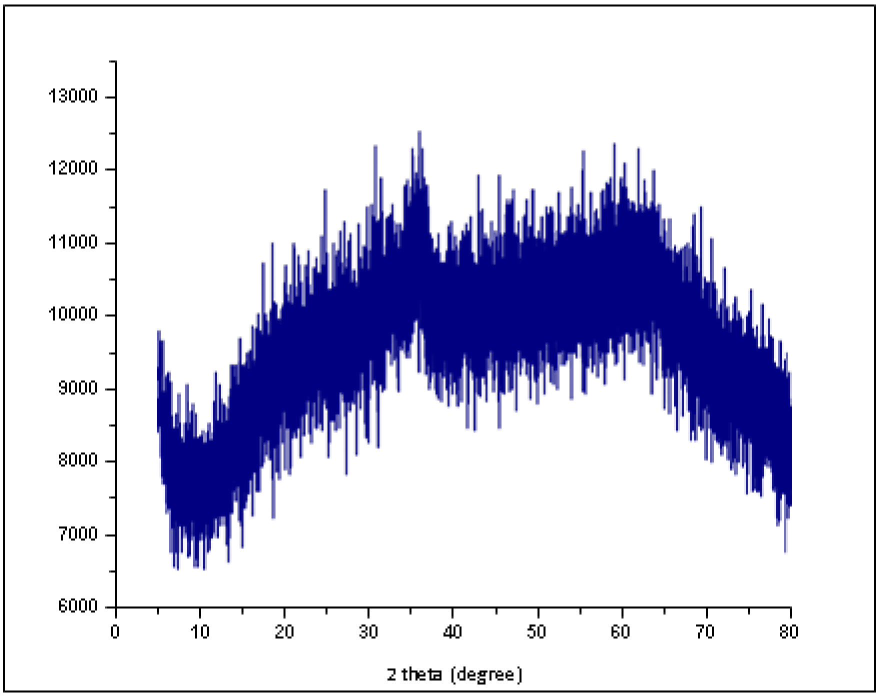

Figure 5: XRD image of Fe3O4 nanopowder

4. Results & Discussion

The formation of iron oxide nanoparticles using plant leaf extract of *Calotropis Gigantea* has been analysed by the colour change from light brown to dark brown. The analysis report of FTIR spectrum has confirmed that the bio reduction of Fe^{3+} ions is because of the reduction of the capping agents.

5. Conclusion

This paper has demonstrated that use of a natural, low cost biological reducing agent and *Calotropis Gigantea* leaves extracts can produce metal nanostructures, through efficient green nanochemistry methodology, avoiding the presence of toxic solvents and waste.

The synthesis of eco-friendly and sustainable nanoparticles by using novel green synthesis methods has been shown in this paper. The hopeful developments in the study of nanoparticle have been evaluated. In that case, the synthesis of nanoparticles with easy availability of the starting materials and the unique properties of every synthesized product will further motivate for future studies. In the present work, IONPWs were prepared using *Calotropis Gigantea* leaves and further properties has been studied using XRD, SEM and FTIR. The study can be extended in future in order to categorize its food related applications like food analysis etc.

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