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Biosynthesis Of Iron Oxide Nanoparticles Using Aquous Extract Of *Jatropha Gosspifolia* As Source Of Reducing Agent

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Abstract

In the present study, an attempt is made to synthesis iron oxide nanoparticls using the aqueous extract of a widely growing weed *Jatropha gosspifolia,* to reduce Ferric chloride into iron particles and further characterized using UV-Vis, XRD peaks and SEM images. The uv-vis spectrum showed a peak at 277nm after showing a characteristic colour change in the reaction mixture indicating the formation of iron oxide nanoparticles by the reduction of metal salts. Futher spectral characteristics from XRD showed more than 3 peaks: 2θ values of 24.5°, 36.0°, 41.2°, 50.0°, 55.4°, 55.4°, 64.1°and 72.1°.confirming the formation of iron oxide and
Closely matching with standard values of Fe3O4 given in JCPDR. The bio-factor involved in the reduction metal salts present in the select plant is also discussed.

Introduction

Nanoparticles are of great interest due to their extremely small size and large surface to volume ratio, which lead to both chemical and physical differences in their properties (e.g. mechanical properties, biological and sterical properties, catalytic activity, thermal and electrical conductivity, optical absorption and melting point) compared to bulk of the same chemical composition. (Daniel and Astruc 2004; Bogunia-Kubik and Sugisaka 2002; Zharov et al., 2005).

In recent years, the development of efficient green chemistry methods for synthesis of metal nanoparticles has become a major focus of researchers, so as to find out an eco-friendly technique for production of well-characterized nanoparticles. Green synthesis of nanoparticles makes use of environmental friendly, non-toxic and safe chemical reagents (Salam *et al.,* 2012).

It has been previously reported that amorphous nanoparticles of zerovalent iron can be synthesized (Kharissova *et al.,* 2013) using extracts of some kinds of tea (Hoag *et al.,* 2009; Shahwan *et al.,* 2011; Kuang *et al.,* 2013), coffee (Kharissova *et al.,* 2013; Hoag *et al.,* 2009). In addition, 40 to50 nm spherical amorphous iron (iron oxide) nanoparticles were synthesized using aqueous sorghum (*Sorghum* spp) bran extracts, which are known to contain high levels of freely extractable phenolic compounds. The biosynthesized iron nanoparticles were found to be nontoxic when compared with iron nanoparticles prepared using conventional NaBH4 reduction protocols (Nadagouda and Varma 2008), and were functional as demonstrated through their use in the degradation of organic contaminants in a model system (Njagi *et al.,* 2011). Although needs further improvement in order to obtain stable nanoparticles of controlled size and morphology, which would be conducive to large-scale synthesis of iron nanoparticles for environmental remediation and hazardous waste treatment applications. Valentin V. Makarov et.al., (2014) characterized the formation of iron nanoparticles using extracts of *Hordeum vulgare* and *Rumex acetosa,* and also described approaches and considerations on how to enhance the stability of plant produced iron nanoparticles. Iron nano particles have been synthesized by using aquous extract of ten plants and spices (Black tea leaves, Carom seeds, Champa leaves, Clove bud, Coffee seeds, Curry leaves, Green tea leaves, Mango leaves, Neem leaves, Rose leaves) at room temperature (Monalisa Pattanayak, and P. L. Nayak 2013). Fe3O4 nanoparticles were prepared by using *caricaya papaya* leaves extract at room temperature. The synthesized nanoparticles were characterised by using UV-Vis absorption spectroscopy, FT-IR, XRD, SEM with EDS techniques. UV-Vis absorption shows a characteristic absorption peak of iron oxide nanoparticles in the range of 190-250 nm. FT-IR measurement was carried out to identify the possible molecules like carbonyl, CH, OH band. From the XRD, it was found that the average particle size of magnetite nanoparticles was found to be 33 nm. SEM shows the plate like structure with coarsened grains and capsule like morphology and EDS showed its chemical composition. This biosynthesis approach is cost effective, eco-friendly and promising for applications in medicine Latha and Gowri 2014). Iron oxide nanoparticles (Fe3O4-NPs) were synthesized using a rapid, single step and completely green biosynthetic method by reduction of ferric chloride solution with brown seaweed (*Sargassum muticum*) water extract containing sulphated polysaccharides as a main factor which acts as reducing agent and efficient stabilizer (Mahdavi et al, 2013). In the present study, an attempt is made to synthesis iron oxide nanoparticls using the aqueous extract of a widely growing weed *Jatropha gosspifolia, to reduce* Ferric chloride into iron particles and further characterized using UV-Vis, XRD peaks and SEM images.

4. Materials and Method

Materials:

Fresh leaves of *Jatropha gosspifolia* were collected from Pondicherry university campus, Pondicherry, India.

Ferric chloride (Fecl3) was purchased from Himedia Laboratories pvt.Ltd. Mumbai, India. Distilled water was prepared in the laboratory and used throughout the experiment.

Fig.1: *Jatropha gosspifolia*

Preparation plant extract and FeCl3 solution

The plant extract was prepared by using the fallowing method given in plant extract. Fresh young leaves were collected and washed thoroughly. About 10gm of leaves were cut into small pieces and crushed with 10ml of distilled water using mortar and pestle. The fine colloidal extract was making up into 100ml with distilled water. The extract was heated at 60˚c for 30min and allows cooling. The extract was then filtered using muslin cloth. The filtrate was centrifuged at 5000rpm for 10mins. The supernatant were collected and used for further process. 0.1 mM of fecl3 solution was prepared and stored for further use.

BioSynthesis of Iron oxide Nanoparticles

The process of Iron oxide Nanoparticle, both (*Jatropha* plant extract) precursor and reducing agent $FeCl_3$ solution were mixed in sterile flasks. This mixture of plant extract and $FeCl_3$ solution allowed reacting for 2 hours at room temperature.

The reaction mixture filled with distilled water till the neck of flasks after 48 hours incubation at room temperature color changes wee appeared. The color changes Maroon into dark green and black color. At the end of 45 minutes solution, centrifuged at 500 rpm for 10mins. The sediment is washed in doubled distilled water twice by repeated centrifugation 5000rpm for 10 mins. The sediment was collected and further heated at 800˚C for 30 mins incubation. The cooled material stored for further spectral studies.

Characterisation of Iron oxide Nanoparticles:

UV-Visible spectra analysis:

For UV-visible spectrophotometer (Varian Model: 5000) analysis, 0.1mL of the sample is taken in a cavetti and was diluted to 2ml with de-ionized water and the UV-visible spectra is obtained. The dried mixture of Iron Oxide nanoparticles was collected for the determination of the formation of Fe_3O_4 nanoparticles by an Expert Pro x-ray diffractometer operated at a voltage of 40 kV and a current of 30 mA with

Cu, Kα radiation in-2 configurations.SEM Observation of iron oxide nanoparticles is also done.

The dried sample of iron oxide nanoparticles were fabricated by dropping the suspension onto clean electric Stubs and allowing water to completely evaporate. SEM images were obtained in **Hitachi, Model: S-3400N** Electron microscope (CIF, Pondicherry University).To counter check the formation of nanosized iron particles images under fluorescence light emitted by fluorophores, has been obtained.

Result

Colour change:

Synthesis of Fe_2O_3 nanoparticle takes place by the reduction of Fe ions during reaction with the *Jetrapha gossypifolia* leaves extract fallowed by color change. After 24 hours the Iron ions get reduced exhibiting in change color of the mixture from maroon color to Green color and Black color.

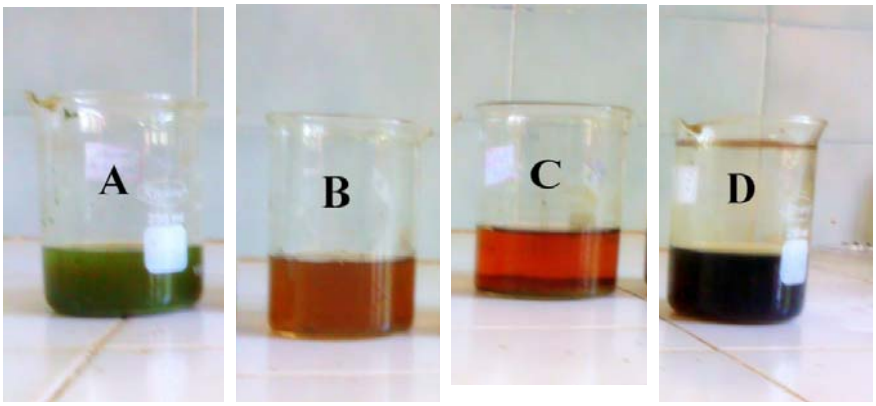
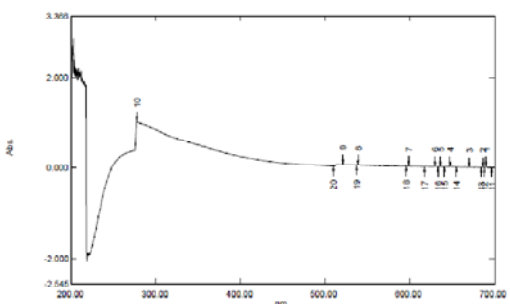


Fig 2: Reaction process A) Aqueous extract, B) 1Mm Salt Solution (Ferric Chloride), C) Mixture (Initial color), D) Mixture color after 2 hours incubation.

UV-Visible Spectroscopy:

The bioreduction of Ferric ions in aqueous solution was measured by using UV-Visible Spectroscopy in the range of 200-700 nm and peak is obtained at 277nm.



Measurement Properties	
Wavelength Range (nm.):	200.00 to 700.00
Scan Speed:	Medium
Sampling Interval:	0.5
Auto Sampling Interval:	Enabled
Scan Mode:	Auto
Instrument Properties	
Instrument Type:	UV-2400PC Series
Measuring Mode:	Absorbance
Slit Width:	0.1 nm
Light Source Change Wavelength:	360.0 nm
S/R Exchange:	Normal
Attachment Properties	
Attachment:	None
Sample Preparation Properties	
Weight:	1 mg4
Volume:	4 ml
Dilution:	
Path Length:	
Additional Information:	

No.	P/V	Wavelength	Abs.	Description
1	↑	691.00	0.036	
2	↑	686.50	0.028	
3	↑	670.50	0.032	
4	↑	648.00	0.039	
5	↑	636.50	0.038	
6	↑	630.50	0.038	
7	↑	598.50	0.055	
8	↑	539.50	0.078	
9	↑	521.50	0.089	
10	↑	277.50	1.017	
11	↓	697.50	0.003	
12	↓	689.00	0.017	
13	↓	684.50	0.013	
14	↓	656.00	0.020	
15	↓	641.50	0.026	
16	↓	633.50	0.028	
17	↓	617.50	0.030	
18	↓	596.50	0.036	
19	↓	537.50	0.066	
20	↓	510.00	0.049	

Fig 3: UV-Spectrum of Iron Oxide nanoparticles

A

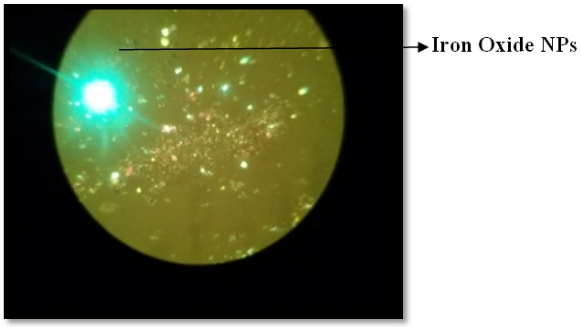


Fig 4: Fluorescent microscopic view of iron oxide nanoparticles.

SEM analysis of Iron oxide nanoparticles:

The Scanning Electron Microscopic image were obtain using Hitachi S-4500 SEM machine in CIF (Central Instrumentation Facility) at Pondicherry University. These images was used to examine the nature of morphology of Iron oxide nanoparticles with it found that Iron oxide nanopasticle and mostly spherical in shape.

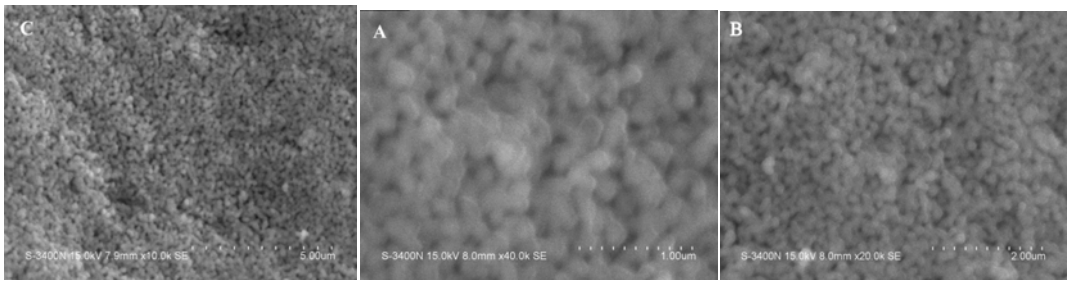


Fig 5: SEM image of Iron Oxide nanoparticles

XRD analysis of Iron oxide nanoparticles:

Crystalline structure of the nanoparticles was characterized by X-ray powder diffraction.

Strong diffraction peaks with 2θ values of 24.5θ, 33.5θ, 36.0θ, 44.2θ, 55.4θ, 62.4θ, 64.1θ, and 72.1θ was noted.

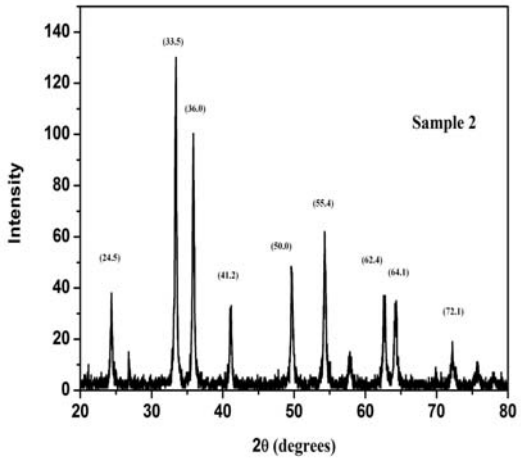


Fig 6: XRD pattern of Iron Oxide particles

Discussion

Nanomaterials have vast range of applications such as in medicine, electronics, biomedicine consumer product, groundwater treatment and energy production. Iron nanoparticles of various sizes and morphology, such as spheres, platelets, and nanorods, (Nadagouda and Varma 2008, Hoag et al., 2009; Shahwan et al., 2011; Kuang et al., 2013). These nanoparticles are with diameter ranging between 1-100nm. The two main forms are magnetite (Fe_3O_4) and its oxidized form magnetite (Fe_2O_3). Iron oxide NPs have extensive interest because of their supermagnetic properties. Iron oxide NPs have potential applications in many fields, include magnetic storage devices, analysis, sensor, magnetic resonance imaging (MRI) for medical diagnosis and therapeutics, and used for better-absorbed iron nutrient supplement. Iron oxide Nanoparticles are most preferred in medicine non-toxic to humans. Nanoparticles have specific cytotoxic mechanism for uncoated iron oxide nanoparticles. Presently iron oxide particles are synthesized using the aqueous extract of \jatropha s. in cold process (at room temperature).Change in the colour of

On comparison with the report already made with regard to colour change in the reaction solution, the colour of the Fe3+ /BS extract solution at room temperature rapidly change from yellow to dark brown indicating the formation of Fe3o4-NPs by the extract of brown sea weed *sargassum muticum* (Mahnar Mahdavi, et.al.2013 and Monalisa patttanayak and.Nayak, (2013). However on chemical reduction of iron oxide by the chemical agent present in the extract the colour change was also reported as Black Tea Leaves, Carom Seeds, Champa Leaves, Clove Bud, Coffee Seeds, Curry

Leaves, Green Tea Leaves, Mango Leaves, Neem Leaves. The plant extract was mixed in aqueous solution of the iron oxide complex. It started to change the colour due to the reduction of iron ion which may indication of formation of iron nanoparticles, the deep brown color formed during the synthesis is turned black. Knowing that after re-dissolution the brown color appears again but its depth depends on the amount of the solid Iron oxide dissolved. Therefore, the colour change is the indication of NPs formation and the intensity of colour might be depending on the concentration of the extraction other words amount of particular biochemical constituent that reduces the metal salt in to metal particles. The concentration of the tea extract determined these structures, which were found to contain hexagonal metallic iron, amorphous iron, Fe3O4 and Fe2O3 (Hoag *et al.,* 2009, Kharissova *et al.,* 2013, Shahwan *et al.,* 2011).

The characterization of iron oxide Nanopartilce under the uv-visible spectroscopy analysis shows the peak at 277nm.It indicates the presence of iron Nanoparticles and the peak in 3 samples between 277-300nm. Similar analysis made by (Monalisa Pattanayak 2013) and absorption peak at 216-265nm from various plants and spices extract (Eric Njagi *et.al.* 2010). There were small difference in the absorbtion peak range due to variation in the range and stability potential of the species. The morphology and structural analysis of iron oxide Nanoparticles from *Jatropha gossypifolia* leaves extract done using scattered Electron Microscope. The shape and size range of nanoparticles observed in the present study are also confirmety with the reports of (Monalisa Pattanayak and P.L.Nayak, 2013 and Yogamoorthi and Kalidasan., 2013., Latha and Gowri., 2014). Moreover, the synthesized powder samples /particles under fluorescent microscope are clearly seen.

Spherical characterization of iron oxide Nanoparticles from *Jatropha gossypifolia* leaves extract performed by XRD analysis.This X-ray diffraction obtained strong diffraction peaks with 2θ values of 24.5°, 36.0°, 41.2°, 50.0°, 55.4°, 55.4°, 64.1°and 72.1°.These values of XRD peak are similar to the values obtained by (Yogamoorthi and Kalidasan., 2013) and Mahnaz mahadavi et al., 2013 and (Latha & Gowri2014) using papaya leaves as reducing agents.Moreover, the 2θ values obtained in the present study closely matching with standard values of Fe3O4 given by JCPDR.The phytochemical constituents present in *Jatropha gossypifolia* reported by (Ruchi Seth and Renu Sarin, 2010) Terpenoid, Steroids, Saponin. Flavonoid and Triterpenoid together or individually, might have reduced ferric chloride into Iron oxide. However, further studies on isolation of individual compound and its reaction with ferric chloride would bring out the exact compound that is responsible for the reduction of metal salt which in turn would open up new avenue in the field of material sciences.

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