ECOFRIENDLY SYNTHESIS AND CHARACTERIZATION OF IRON OXIDE NANOPARTICLES

DR BHARAT GOVIND PAWAR, DR RAMESH ARUN BUGAD, R.D. Mahimkar, R.G. Pawar, P.S. Patil,

*Assistant Professor
Sangola College Sangola, Kadlas Road Sangola.

***Abstract*: A simple, efficient, and ecofriendly method has been developed for the exclusive synthesis of iron oxide nanoparticles using an aqueous extract of *Parthenium hysterophorusas a* weed extract act as a dropping agent. The synthesized iron oxide nanoparticles heated in muffle furnace at 600˚c for 1hours. The synthesized nanoparticles were characterized by X –ray diffraction, Scanning electron microscopy. This nanoparticles used for the study of Methylene blue (MB) dye removal with help of spectrophotometer. P^H and conductivity were also studied in aqueous solution iron oxide nanoparticles. The present study highlights the potential application of iron oxide nanoparticles can be explored for technological industries.**

***Keywords*: *Parthenium hysterophorusas*, Iron oxide, nanoparticle, Methylene blue dye**

Introduction

Parthenium hysterophorus, is a highly abundant and damaging weed, which originated in northeast of Mexico by natural hybridization between *Parthenium confertum* and *Parthenium bipinnatifidum* [1-2]. This is also known by several region definite common names such as altamisa, carrot grass, Santa Maria, bitter weed, star weed, white top, wild feverfew, gajarghas, the "scourge of India," and congress grass. *Parthenium hysterophorus* L., belonging to the family Asteraceae, is an annual ephemeral herb. Its genetic characteristics such as short-life cycle (4 to 6 weeks)[3]. It shows many bad effects on agriculture, biodiversity, and health of animals and individual beings. In man, Parthenium plant or its pollens cause health problems of asthma, fever, dermatitis, diarrhoea, bronchitis, and allergies on skin, eyes, nose, and mouth [4] Parthenium shows extraordinary adaptability over wide range of ecological conditions as well as soil types and is therefore found growing in diverse types of habitats [5].Iron oxide belongs to the most lavish minerals and that occurs with a large variation of structures, stoichiometries, and properties. Iron oxide exists in three forms in nature: magnetite (Fe_3O_4), maghemite (γ-Fe_2O_3), and hematite (α-Fe_2O_3). Hematite is the oldest known of the iron oxides and is widespread in rocks and soils. It is also known as ferric oxide, iron sesquioxide, red ochre, specularite, specular iron ore, kidney ore, or martite. Hematite is blood-red in color if finely divided, and black or grey if coarsely crystalline. It is extremely stable at ambient conditions, and often is the end product of the alteration of other iron oxides. Magnetite is also known as black iron oxide, magnetic iron ore, loadstone, ferrous ferrite, or Hercules stone. It exhibits the strongest magnetism of any transition metal oxide. Maghemite occurs in soils as a weathering product of magnetite, or as a product of heating of other iron oxides. It is metastable with respect to hematite, and forms incessant solid solutions with magnetite [6].The synthesis of magnetic iron oxide nanoparticles has drawn much attention due to the fact that the distinctiveness of nanoparticles vary appreciably with its procedure. The two major chemical formulas of iron oxide nanoparticles are Fe_3O_4 as magnetite and Fe_2O_3 as γ-Fe_2O_3 (maghemite) and α-Fe_2O_3 (hematite). Magnetite and maghemite exhibit superparamagnetic properties and high saturation magnetization, leading to their biomedical applications [7].Magnetite is transformed to maghemite by calcination in air as $4Fe_3O_4 + O_2 \rightarrow 6\gamma$-$Fe_2O_3$. Magnetite has a spinel structure with Fe3+ in all tetrahedral and Fe^{3+} and Fe^{2+} in octahedral sites, while maghemite has the same structure with cationic vacancies in one-third of octahedral sites [8].By modifying the growth conditions, the size of the iron oxide particles can be condensed to nanosize. Since iron oxide is a technology important material, a systematic study has been initiated to prepare it through green approach using ferric chloride as the precursor and *Parthenium hysterophorus*as a weed extract act as a reducing agent. Prepared nanoparticles have been analyzed to evaluate its structural properties from X-ray diffraction studies, SEM and elemental analysis. PH and conductivity also measured.

Experimental:-

2.1. Materials

The *Partheniumhysterophorus weed*used in this experiment was fresh and was collected from the college campus . Iron Chloride anhydraus (III) [Ferrous Chloride (98%) and $FeCl_2$(1%)]was purchased from Merkspecialties PVT Mumbai India. The water used in all experiments was doubly distilled.

Preparation of *Partheniumhysterophorus weed* extract

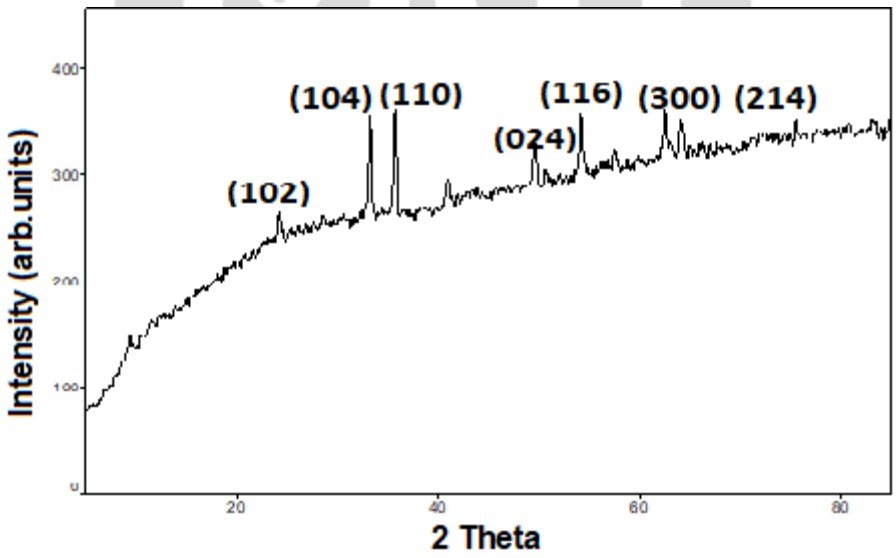
A 10.773 g of thoroughly washed *Parthenium hysterophorus leaves* was sliced finely, and crushedin mortal and pastel with help of 100 ml double distilled water for 10 min. The green extract was filtered through a filter paper. The clear filtrate was used for the synthesis of iron oxide nanoparticles for further experiments.

Fig : Actual Experimental work photographs of preparation of nanoparticle in lab.

General procedure for the preparation of the iron oxide nanoparticles

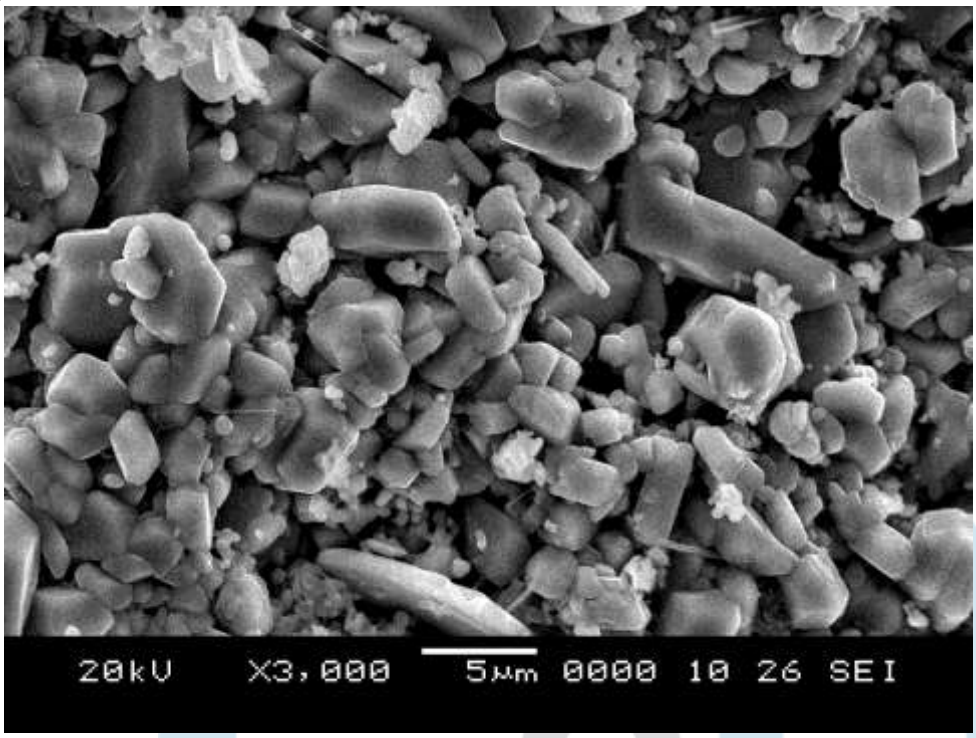
Iron oxide nanoparticles were synthesized using sol-gel method. In sol-gel method, there are two types of materials or components, "sol" and "gel". Sols are solid particles in a liquid subclass of colloids and gels are ligands contained in liquid. This method can produce highly pure and well controlled nanoparticle. This process involves formation of sols in a liquid and it is reduced to the desired product using a reducing agent. In the present study Iron Chloride anhydrate is used as the precursor, Ethanol as the solvent and *Parthenium hysterophorus* sextract as the reducing agent. In a typical reaction procedure, 100 ml of *Parthenium hysterophorus* sextract was added to 2 g of Iron Chloride anhydraus (III) already dissolved in 100 ml ethanol, under vigorous magnetic stirring for 1 h at 80 °C. During this process, the color of the reaction solution changed from yellowish translucent to a blackish color as shown in Fig. 1, indicating the formation of iron oxide nanoparticles. The resulting product, iron nanoparticle was centrifuged and washed several times distilled water, and acetone. The Purified nanoparticle powder was dried at 100 °C for 5 h and further in muffle furnace calcinatedfor 600 ^{0}C for 2 h. further characterization by XRD and with help of spectrophotometer methylene blue dye removal done.

Result and Discussion



The synthesized iron oxide nanopowders were characterized using X-ray diffraction. XRD pattern indicates that the prepared iron oxide was in α-Fe_2O_3 phase exhibiting rhombohedral structure. Observed peaks are in defined positions that shows the formation of α-Fe_2O_3 without any impurity peaks of any other phase of iron oxide, which indicates a high degree of purity of the prepared samples. The broadening of the X-ray diffraction lines reflects the nanoparticle nature of the sample. In XRD, all the

peaks are indexed and the d-values are compared with the JCPDS standards [JCPDS file no. 89-8104]. The crystalline size observed is 42 nm.



SEM analysis

The morphological extent of synthesized iron oxide nanoparticles were studied using the SEM. The study established that the size of the nanoparticles was in the range of 70-80 nm, similar phenomenons were reported in the previous studies [10]. And also exhibits the formation of irregular rod shape of iron nanoparticles as shown in the . In an another study by Kuang et al. [11][used three different tea extracts, namely, green tea , oolong tea and black tea to synthesis iron nanoparticles and the SEM image revealed the irregular spherical iron nanoparticles indicating the chain-like structure.

The concentration of MB solution before and after adsorption were estimated by measuring absorbance at 665 nm with help of spectrophotometer. 0.250 gm amount of from waste rind of Pomegranate adsorbent was placed in 50 ml flasks containing 6.25, 5.25, 4.25, 3.25, 2.25 mg/L concentration of dye solution of corresponding pH ranging from 5.5 to 6.0.

Fig:1 Diluted absorption study of methylene blue dye

Then flasks were shaken thoroughly with hand for 5 minutes, After filtration final concentration of dye solution were analyzed by spectrophotometer. The amount of equilibrium uptake of dye is calculated by using equationqe = (C0 – Ce) V/ W [9]

Qe- is the dye up taken by adsorbent mg/g, C_0- is the initial MB concentration, Ce- is the MB concentration (mg/l) after the batch adsorption process, W- is the Mass of adsorbent (gm), V is the Volume of dye solution

Absorbance of Pure dye	Absorbance of Iron Oxide + nanoparticle
1.74	1.70
1.69	1.63
1.63	1.30
1.54	1.01

From table shows that on dilution Abs value decreases it show that the Iron oxide nanoparticles degrades the MB dye. The interaction between dye molecule and adsorbent is basically a combined result of charges on dye molecules and adsorbent is basically a combined result of charges on dye molecules and the surface of the adsorbent.

Conclusion

The purpose of this work to use economic and environmental –friendly synthesis of nanoparticles.The weed *Partheniumhysterophorus is used as* as a new source of reducing agent.Thisnanoparticle is used as adsorbents for removal of methylene blue dye. In this way prepared nanoparticles can be also used for technological application.

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