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Green Synthesis of Nano Iron Oxide particles from mild steel

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Abstract : The main oxidation states of iron are Fe^{2+} (ferrous) and Fe^{3+} (ferric). When these ions interact with a base ferrous hydroxide (Green –Fe $(OH)_2$ and ferric hydroxide (brown- $Fe(OH)_3$ will be produced. Conversion of Fe^{2+} into Fe^{3+} is very easy and it can take place even by oxygen present in air or water (Aerial Oxidation). Iron hydroxides have been produced by electrochemical method. An aqueous alkaline sodium chloride solution as electrolyzed using graphite cathode and mild steel anode at room temperature. During this process a green precipitate of ferrous hydroxide was produced. During electrolysis iron is converted into Fe^{2+} which reacts with OH^- ion (NaOH) present in the solution. After sometime the solution turned brown due to formation of ferric hydroxide. This oxidation is possible by the presence of dissolved oxygen present in the solution. Moreover during electrolysis of sodium chloride solution, Cl^+ is produced which oxidizes ferrous into ferric. This ferric hydroxide has been converted into other forms of iron oxide namely α-FeO(OH), γ-FeO(OH) and finally magnetic oxide of iron (Fe_3O_4). These iron oxides have been analyzed by FTIR and SEM. The particle size of this iron oxide is in the range of Nano particles. These iron oxides will be useful in agriculture. They will increase the soil fertility various microorganisms which increase the soil fertility (Iron reducing bacteria) namely Geobacter bemidjiensis, Geobacter metallireducens, Geobacter sulfurreducens, Shewanella oneidensisMR-1 and Shewanella putrefaciens, etc., live on this iron oxides. The electron released by these bacteria is accepted by ferric iron, whereby it is reduced to ferrous. Thus this ferrous ferric iron equilibrium is very helpful for the metabolism of various microorganisms such as iron reducing bacteria. These ferrous-ferric electron transfer equilibrium can be used to convert harmful Cr^{6+} into harmless Cr^{3+}. This concept will bring revolution in tannery effluent treatment industry. This process is an electrochemical process and it can be started and stopped at any time. Hazardous chemicals are not used. The products are environmental friendly. Hence this process is considered as green approach.
Keywords: Green Synthesis, Iron oxides, Nanoparticles, Soil fertility, environmental friendly process.

Introduction

Nano particles are particles between 1 and 100 nanometers in size. In nanotechnology, a particle is defined as a small object that behaves as a whole unit with respect to its transport and properties[1]. Nano comes from the Greek word for "dwarf" and broadly speaking, the field of nanotechnology can be defined as research and technology developments at the atomic or molecular level[2]. The history of Nanotechnology can be traced back to 16th century[3]. Researchers in nanotechnology seek to understand and control some of the smallest objects known to humankind. In terms of length, one nanometer is the equivalent of about four gold atoms or one millionth of a millimeter. Nanotechnology has evolved into a multidisciplinary field, revolutionizing industries such as applied physics, mechanical, chemical, electrical and biological engineering, machine design, robotics, and medicine. Iron oxide nano particles have been widely researched for MRI (Magnetic Resonance Imaging), as they are mainly super paramagnetic. There are several types of iron oxide nano particles, namely maghemite, γ-Fe_2O_3, magnetite, Fe_3O_4, and haematite, α-Fe_2O_3, among which magnetite, Fe_3O_4, is very promising, because of its proven biocompatibility[4]. Ferric refers to iron-containing materials or compounds. In chemistry the term is reserved for iron with an oxidation_number of +3, also denoted iron (III) or Fe^{3+}. Ferrous refers to iron with oxidation number of +2, denoted iron (II) or Fe^{2+}. Iron (III) is usually the most stable form of iron in air, as illustrated by the pervasiveness of rust, an insoluble iron (III)-containing material[5]. Application of iron in low–iron soils can increase grain yield in soybean (Ghasemi *et al*, 2006). Iron compounds can use as foliar on leaves and as seed coating (Debermann, 2006). Nanotechnology can present solution to increasing the value of agricultural products and environmental problems. With using of nano-particles and nano-powders, we can produce controlled or delayed releasing fertilizers. Nano-particles have high reactivity because of more specific surface area, more density of reactive areas, or increased reactivity of these areas on the particle surfaces [6].

Iron oxide nanoparticles (IONPs) occupy a privileged position among magnetic nanomaterials with potential applications in medicine and biology. They have been widely used in preclinical experiments for imaging contrast enhancement, magnetic resonance, immunoassays, cell tracking, tissue repair, magnetic hyperthermia and drug delivery. IONPs-based medical applications are limited to the use of non-functionalized IONPs smaller than 100 nm, with overall narrow particle size distribution, so that the particles have uniform physical and chemical properties[7]. The category of iron oxide powders includes all types of synthetic iron oxides (hematite, magnetite,maghemite, etc.) and ferrite powders also, as the latter materials have as their main constituent ferric oxide(Fe_2O_3).Iron oxide powders are the most widely used of all colored inorganic pigments, used in concrete products, paints, plastics, and other media. Due to their chemical and magnetic properties, iron oxide powders also find significant commercial usage in electromagnetic components, catalysts, toners, magnetic recording media, and other applications. Magnetic Nano Particles (MNPs) are used in important biological applications, including magnetic bio separation, detection of biological entities (cell, protein, nucleic acids, enzyme, bacteria, virus,etc.), clinic diagnosis and therapy (such as magnetic resonance image), magnetic fluid hyperthermia (MFH),targeted drug delivery and biological labels. In the last decade, investigations with several types of iron oxides have been carried out in the field of MNPs, among which magnetite (Fe_3O_4) and maghemite (γ–Fe_2O_3) are the very promising reagents since their biocompatibility has already been proven[8]. Magnetic iron oxide nano particles are routinely used as contrast agents for targeting organs (liver and spleen) or lymph nodes. New developments are focused on targeting through molecular imaging and cell tracking. A challenge is the functionalization of nanoparticle surfaces. Another challenge is the synthesis of stealth nanoparticles able to circulate in the blood compartment for a prolonged time and bearing ligands able to facilitate their specific internalization in tumor cells[9,10]. The present work is undertaken to prepare the ferrous and ferric hydroxides electrochemically and characterize them by Fourier Transform Infrared Spectroscopy and Scanning Electron Microscopy and to determine the particle size.

Experimental Techniques

100 ml of an aqueous solution containing 5g of sodium carbonate and 5 g of sodium chloride was taken in an undivided cell, provided with a magnetic stirrer. Electrolysis was carried out using iron (mild steel) anode and graphite cathode for 5 minutes, with constant stirring. A potential difference of 6 volt was applied. The current density was 50mA/cm^2. A green precipitate of ferrous hydroxide was obtained. It was filtered and dried on a glass plate over night. A brown precipitate of $Fe(OH)_3$ (iron oxide) was obtained. It was characterized by FTIR spectra. This method of synthesis is considered as green synthesis, because it is an electrochemical method. The reaction can be started and stopped at any time. Hazardous chemicals are not used. Water is used as solvent. Iron waste is used as electrode (anode). Commercial sodium chloride is used. This method is an example for getting wealth from waste. The experimental set up was shown in figure 1.

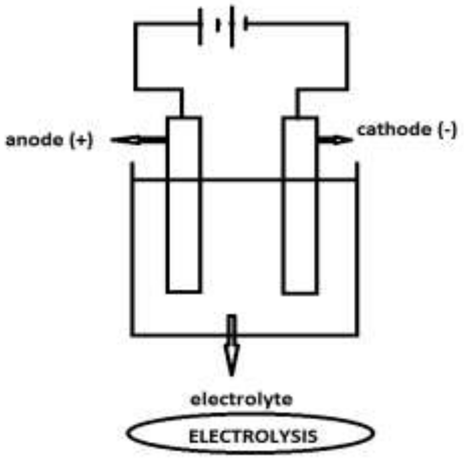

Fig.1. The experimental set up for electrolysis process.

Results and Discussion

The formations of Iron Nano Particles were characterized by using Fourier Transform Infrared Spectroscopy (FTIR) and Surface morphology was analyzed by Scanning Electron Microscopy (SEM).

Analysis of FTIR Spectrum

The FTIR Spectrum (KBr) of the ferric hydroxide (iron oxide) prepared electrochemically (first as ferrous hydroxide and next as ferric hydroxide due to aerial oxidation) is shown in Fig 2. Finally the compound would have been oxidized to various types of iron oxides. It is interesting to note that the peaks confirm the presence of ϒ- FeOOH (411,843, 1021, 1402 and 1627 cm $^{-1}$)[11]. α- FeOOH peaks appear at 411 and 645 cm $^{-1}$. The peaks due to Fe_3O_4 appear at 645, 843, 1021, 1402 and 1627 cm^{-1} [12]. The peaks at 411, 1021, 1402 and 1627 cm $^{-1}$ confirm that the iron oxides are present as nanoparticles[13].

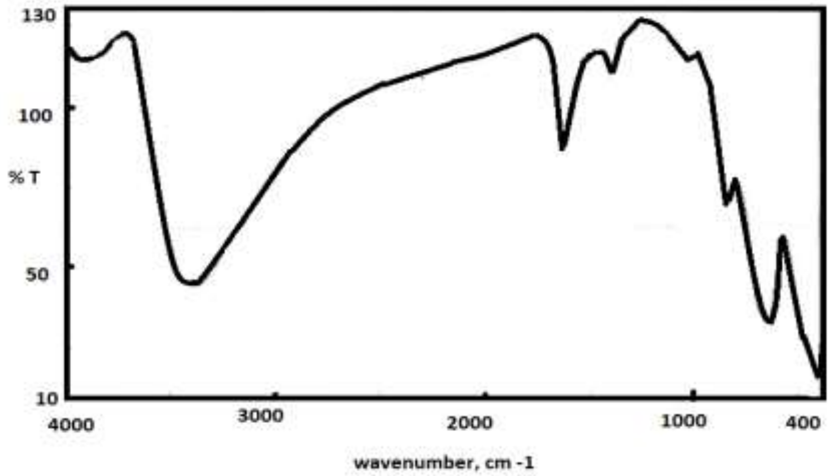

Fig 2: FTIR spectrum of iron oxide prepared electrochemically

Analysis of SEM image

The SEM image of iron oxide nanoparticles is shown in Fig 3. The particles appear as plates. The size of the particles is in the range of 50 nm. Thus SEM study confirms the formation of iron oxide nanoparticles [14].

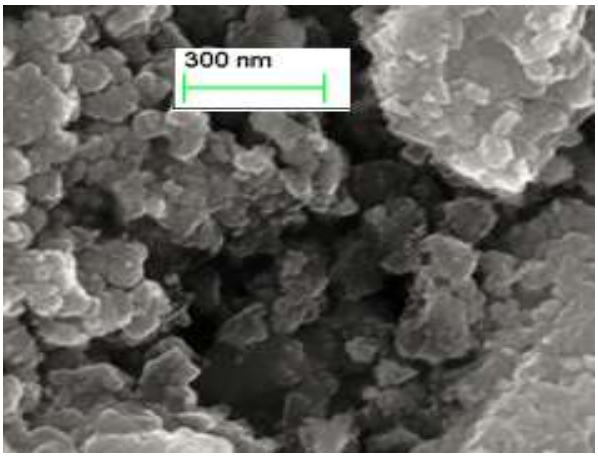

Fig 3: SEM image of iron oxide nanoparticles

Conclusion

The iron Nano particles have been synthesized from mild steel by electrochemical method. The formed iron particles were characterized by Fourier Transform Spectroscopy (FTIR). The particles size was analyzed by Scanning Electron Microscopy (SEM). The particle size found to be 50 nm. This confirms that the iron nano particles were formed by electrochemical method.

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