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Synthesis and Characterisation of Nanostructured Ferric Oxide

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Abstract

Since iron oxides have diverse physical and structural properties, it contributes to natural processing and its uses are uncountable. The structural and size dependent activities are the main characteristics which are to be defined for assessing its role. The different phases of iron oxide such as hematite, maghemite and magnetite are summarized. The article includes the synthesis and characterization of nano-ferric oxide which are at the core of nanotechnology due to their specific properties and applications. The characterization is carried out by x-ray diffraction, FE-SEM and UV/Visible spectroscopy. The aim of the characterization techniques is to define the structural, optical and morphological parameters. The structural analysis of the nanoparticle is identified by powder X-ray diffraction and the optical and topographical parameters are stated by FE-SEM and UV/Visible spectroscopy correspondingly. The preparation of ferric oxides from ferricyanide and ferro cyanide is represented where the results are comparatively discussed with the help of flowchart.

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INTRODUCTION

Modern livelihood is based on compact devices and instruments and hencethere is a need to innovate things ofNano size and structure. The atomic, molecular and functional studies represent nanotechnology. Which comprises aerosols, nanostructured metals, polymers, amplifiers, robotics, 3D networking and so on. The list is endless. Some experts classify these in terms of generations. Nano science deals with the tendency to control and work with atoms and molecules. Nanoparticles are the building blocks for chemical and electronic devices.These can be synthesized chemically. The optical properties of the nanoparticles can be modified without changing its chemical composition so that the actual characteristics of the material are not altered. Charging of nanoparticles is confined to its size and dielectric properties of the medium rather than any other quantum representations. Chemical approaches are the effective methods to produce nano-materials. In such cases, mixing of toxic chemicals has to be prevented and hence proper filtering techniques have to be accomplished. In certain cases, thermal decomposition is carried out in the synthesis which proved to be an effective method in the case of nanoparticles.

Among many other characterization techniques, X-ray diffraction, FE-SEM and UV/Visible spectroscopy are found to be more user-friendly and are hence illustrated in this paper. X-ray diffraction is used to gather information regarding the structure of ferric oxides and is applied to identify and characterize the nanostructured ferric oxides by its diffraction pattern.UV/Visible spectroscopy measures the thickness and properties of the iron particles.Hematite, maghemite, wustite and magnetite are the commonly found iron oxides. FE-SEM defines the optical parameters such as refractive index, extinction and absorption coefficients and band gap analysis.

The applications of hematite, maghemite, wustite and magnetite are realized with reference to electro-chromic structures, magnetic studies, solar technology and medical services accordingly.

RELATED WORKS

(Shavel, et.al.2007) The article presents the preparation method of ferric oxide by ironoleate decomposition at extreme temperatures. High resolution transmission electron microscopy, Fourier transform filtering analysis and x-ray photo electron spectroscopy are the methods used to characterize iron oxide. It is given that at room temperature, the nanostructured nanoparticles are super-paramagnetic in nature. (Deepti Mishra, et.al.2016) Alkalinesolvo thermal method is used to synthesis ferric oxide by using certain chemicals like sodium hydroxide and ammonium bromide.

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Its characterization includes XRD, FE-SEM, etc. Each of the characterization methods constitutes certain features of iron oxide. For example, XRD indicates the phase of iron oxide, where the size of the particle is represented by FE-SEM. (Sapana Guru, et.al.2016) Iron oxide synthesis by glycols is provided. Different methods to characterize nano-ferric oxide particle are also given. (Nazrul Islam, et.al.2013) Ferric oxide is synthesizedby using sono-chemical method using the effect of ultrasound. (GarciaCerda,et.al.2007) It includes the synthesis of magnetic iron oxide nanoparticles using sulfonated styrene-divinylbenzene copolymer. The characterization process includes X-ray diffraction, vibrating sample magnetometry and transmission electron microscopy. (HongdongCai, et.al.2013) Iron oxide is synthesized by hydrothermal synthesis using polyethylene mine. The resultant product is functionalized by various chemicals to determine its characteristics and functions. (Mohamed Rafi, et.al.2015) Its characterization methods includedx-ray diffractometer, and high resolution scanning electron microscopy. Crystalline nature, size of nanoparticles, magnetic behavior and coercivity properties are determined. (Bo-Bae Cho, et.al.2015) Iron oxides are synthesized and characterized for positron emission tomography and magnetic resonance imaging. This is used not only in the fields of technical research, but also as a significant medium in detecting and treating various diseases. (Daschner, et.al.2014) Hydrothermal process is used to synthesis iron oxide at specific temperatures and sizes. For the characterization purpose, Raman spectroscopic method is implemented. The experiment also includes differential centrifugal sedimentation process to determine the size of the nanoparticles distributed. (Aixiang, et.al.2012) defines the hydrothermal treatment. The synthesis of alpha ferric oxide is dealt with amphiphilicdiblock copolymers. The morphological factors of α-ferric oxide are determined. (HassanKarami, 2010) Solid state chemical reaction is the technique used in this paper to determine the average size of Nano- ferric oxide and the various parameters for deciding temperature and iron concentrations are discussed. It is suggested that there is decrease in size of the particles with respect to increase in milling time. The concentration also varies with temperature. (RonSnovskia, et.al.2014) It uses thermal decomposition and stabilization methods to analyze the geographical shapes of Nano iron oxides. Hydrogen reduction technique is used to determine the elemental structure of iron oxide. The characteristic of the particle depends upon the annealing time and the composition mixture. (Arturo Martinez, et.al.2009) The properties and applications of ferrous and ferric oxides are given. The synthesis of nano ferric oxides includes co-precipitation, polyol process, hydro thermal synthesis, etc. The size distribution is obtained by several approaches. (Ajay & Gupta, 2005) The size of the Nano ferric particles depends on the surface coatings and the geometric structure. It is said that the magnetic nanoparticles can be injected to the body via external magnetic field.

SYNTHESIS OF FERRIC OXIDE(Fe_2O_3)

Synthesis is an important process to execute the chemical reactions which in-turn improves the efficiency of the sample used. The main challenges in synthesis of ferric oxide include the determination of controlled size and compositionof the particles.

a-Structure of ferric oxide

Ferric oxide is known as hematite which is an ideal source of iron. It is available in many phases and the generally available ones are α and γ ferric oxide.β-phased ferric oxide rarely occurs. Some of the characteristics of alpha, gamma and beta phased ferric oxide are provided below.

Alpha phase

α-ferric oxide (hematite) is the common form which is obtained naturally. It has antiferromagnetic property below 260K. Its preparation methods include thermal decomposition and precipitation process.

Gamma phase

γ-phase(maghemite)is metastable and turns into alpha phase at high temperature. Its preparation strategies include thermal dehydration, oxidation of magnetite or thermal decomposition.

Beta phase

β-phase is metastable at certain temperatures and can be converted to alpha phase. It can be manufactured by thermal transformation.

b- Nanostructured ferric oxide

α-ferric oxide exhibits rhombohedral composition and certain other phases are orthorhombic in nature. α-ferric oxide and wustite are antiferromagnetic where γ-phased ferric oxide is ferromagnetic. Maghemite and hematite donot conduct electricity where magnetite and wustite exhibits electronic transitions.

c- Preparation of ferric oxide

One gram of potassium ferric cyanide is added to 100ml of heavy water and 30mg of cetrimonium bromide which is stirred for half an hour. It is sonicated thus to form cells, molecules and membranes by intermolecular interactions. The resulted mixture is sealed in a Teflon-lined autoclave where the room temperature is increased to 150°C in 20 minutes. This condition is maintainedfor 24 hours. The mixture is then sedimented and filtered using redistilled water by bringing back to room temperature. The process is followed by drying an oven at 80°C for 2-3 hours.

Reaction with ferricand Ferro cyanide

$$K_3[Fe(C_n)6] + CTAB + D_2O \;\; Fe_2O_3$$

(1 g) (30 mg) (100ml) (0.6351 g)

When ferricyanide reacts withcetrimonium bromide in the presence of heavy water, 0.6351 gram offerric oxide is obtained.

Ferric oxide can be synthesised by another method using a gram of ferro cyanide and heavy water. The process is same as the above and the reaction is given by

$$K_4[Fe(C_n)6] + D_2O \longrightarrow Fe_2O_3$$

(2 g) (100ml) (0.65 g)

In the absence of cetrimonium bromide, when the same reaction is proceeded, 0.65 gram of ferric oxide is obtained.

Hydrothermal synthesis

Hydrothermal synthesis includes the formation of crystal growth under high temperature and pressure. Crystal is allowed to grow in a steel pressure vessel which consists of nutrients and moisture content.The diagramatic representation of hydrothermal synthesis (Fig-1), where the reaction takes place over different temperatures. The process benefits for phosphopeptide enrichment.

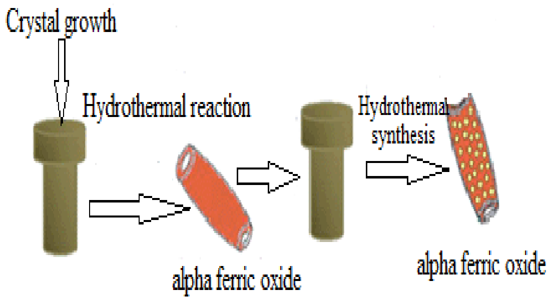

Fig-1: Hydrothermal synthesis

The synthesis process includes the reaction of ferri cyanide and ferro cyanide withheavy water, followed by sonification and hydrothermal processing. The obtained particles are filtered and dried using specific apparatus and characterised as provided. The reaction of ferri cyanide requires cetrimonium bromide along with the usage of heavy water. The flow chart for the above process is given in (Fig-2). Ferri cyanide and ferro cyanide are commonly available as potassium-ferri and ferro cyanides.

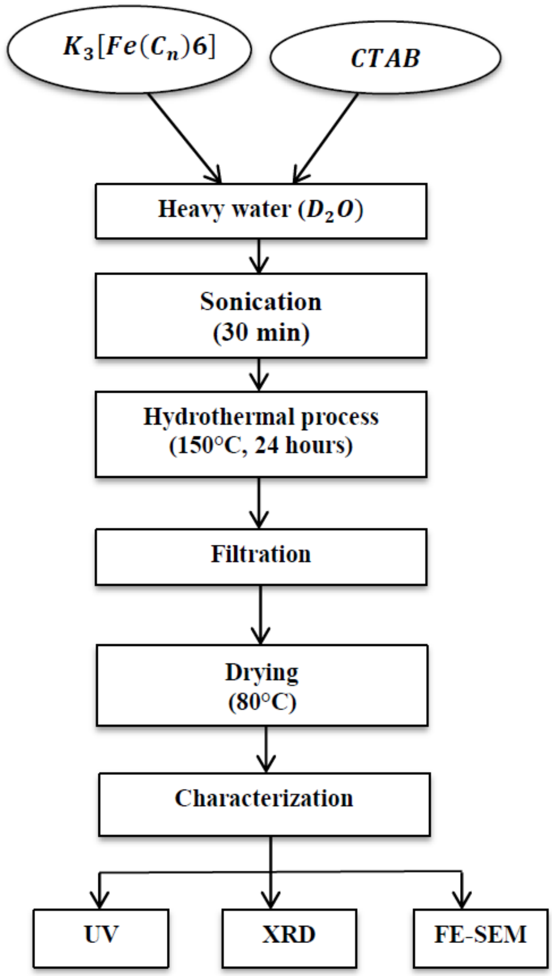

Fig-2: Synthesis and characterization of Fe_2O_3

CHARACTERIZATION OF FERRIC OXIDE

Ferric oxide which is obtained by the above process is characterized by several techniques such as X-ray diffraction, energy dispersive X-ray spectroscopy, X-ray photo electron spectroscopy fourier transform infrared spectroscopy, fourier transform filtering, Field emission scanning electron microscopy,high resolution transmission electron microscopy or thermal gravimetric analysis. For this field of study only three methods including X-ray diffraction, FE-SEM and UV/Visible spectroscopy are applied to define the characteristics of ferric oxide.

X-ray diffraction

When an X-ray beam incidents with the nanostructured ferric oxide material, the rays get scattered from atoms within the nano- material. Since this is of crystalline in nature, the scattered X-rays undergo constructive and destructive interference. The diffraction principle is based on Bragg's law. The directions and intensities of scattering vary with size and shape of the molecules and the arrangement of ferric oxide in the crystal respectively. The phase and structure of the ferric oxide is evaluated using X-ray diffraction (Fig-3). XRD provides phase determination. In many cases, the most occurring phase is found to be magnetite or maghemite.

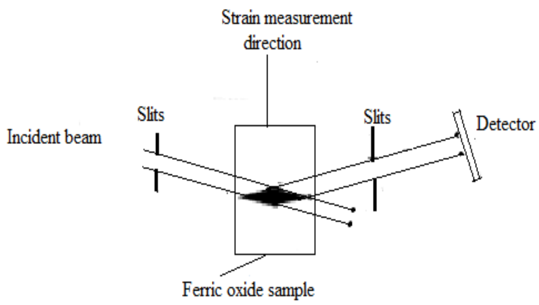

Fig-3: XRD

FE-SEM

Since the article presentsthe characterization of nanostructured ferric oxide particles, these structures require minute classification or analysis and hence to determine such topographical and elemental information at greater magnifications, field emission scanning electron microscopy is employed. The magnification provided by the FE-SEM can be compared with the conventional SEM. The usage of FE-SEM is several times better in providing resolution and clarity of the atoms of the material.

FE-SEM provides an electron gun of cathode to probe beams at different electron energy to aim better resolution and clearer magnification. The method characterizes distribution of size and shape of the material where it would range in nano-meter and spherical shape correspondingly. The basic diagram is depicted in (Fig-4).

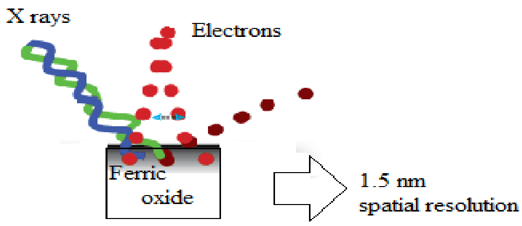

Fig-4: FE-SEM

UV/Visible spectroscopy

The device (Fig-5) measures the intensity of light passing through ferric oxide in which wavelengths of the light can be determined. This can also be used to regulate the kinetics and equilibrium constant of the sample using absorbance spectrum.

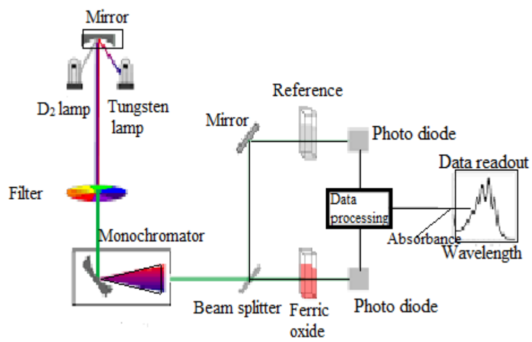

Fig-5: UV/V spectroscopy

The graph (Fig-6) comprises the optical properties of iron oxide as a result of UV/V spectroscopy.These properties are significant to determine electro-chromic and photochemical devices. Electro-chromic property can be generally analyzed using chemical vapor method which is not included in this section. Quantitative determination of the nanoparticle is performed.

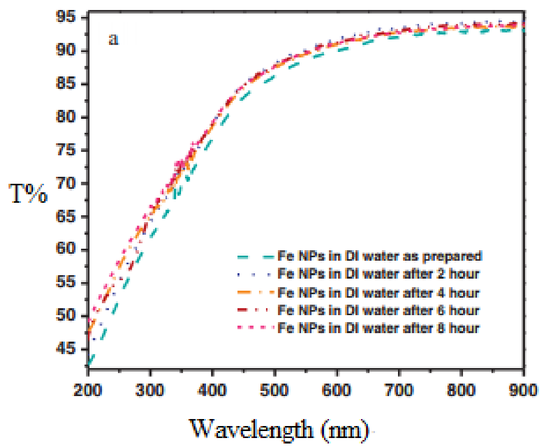

Fig-6: Optical characterization

APPLICATIONS

The uses of nanomaterial are as follows:

- Detects and diagnoses diseases using sensor test chips of nanowires
- Lowers consumption of drugs leading to adverse effects
- Benefits in chemical catalysis
- In air and water treatment
- Increases in energy efficiency and in renewable energy system
- In semiconductors and optoelectronic devices
- Provides lighter products in aircrafts
- In food production, textiles and sensor technologies

CONCLUSION

The synthesis and characterization of ferric oxide is explained. The synthesis is performed hydrothermally where the characterization is based on three techniques. The structural analysis of ferric oxide is done using X-ray diffractometer. In-order to study its optical and morphological characteristics, UV/V spectrometer and field emission electron microscopy are employed. Ferric oxides can also be subjected to several other novel technologies to define its characteristics.

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