

XRD study of iron oxide nano powders prepared using aloe-vera extract

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Abstract

Iron oxide nanopowders (IONPs), Fe₂O₃, are synthesized by using Aloe-Vera leaf extract via green synthesis method. The nanopowder of IONPs prepared through this environment friendly method is found to be very effective in various applications such as electrical and optical sensors, MRI, memory devices, photocatalysis etc. The as prepared nanopowders were characterized using X-ray diffraction (XRD) for crystallite size and crystal symmetry. The hexagonal shape with the R3C space group is confirmed by XRD study, for for as prepared Fe₂O₃ nanopowders. The average crystallite size of the as synthesized Fe₂O₃ nanopowders, as calculated from XRD, is 30 nm.

Keywords: IONP, XRD, green synthesis, calcination.

1. INTRODUCTION

Iron oxide nanopowders (IONPs) have been intensively studied in the recent years for its wide range of applications in controlled drug delivery, MRI, electrical and optical sensors, memory devices, catalysis and as biological separator [1]. Iron oxide nanoparticles have gained much attention due to their unique properties, like superparamagnetism, surface-to-volume ratio, greater surface area, and easy separation methodology. In the fields of life sciences, iron oxides have achieved great potential. Further by special surface coating with organic or inorganic molecules, including surfactants, drugs, proteins, starches, enzymes, antibodies, nucleotides, nonionic detergents and polyelectrolytes, nontoxic conduct and biocompatible applications of magnetic nanoparticles can be enriched [2].

Recently, iron oxide nanoparticles have achieved its great importance because of its applications in the information storage, colour imaging, magnetic refrigeration, medical diagnosis and treatment of diabetes, and as ferrofluids [3-8]. Magnetic nanoparticles (MNPs) of Fe₃O₄ are able to target cancerous cells and have potential use in cancer therapeutics [9]. Due to its low cost and toxicity, high heat stability and availability, Fe₂O₃ is widely used in different industries [10-13]. Numerous combination strategies have been developed to obtain the ideal particles and chemical synthesis is the most preferable route. Chemical methods like coprecipitation, hydrothermal method, sol-gel process, microemulsions, polyol method etc. have been successfully used to synthesize MNPs [5-6, 11-13]. During nanoparticles synthesis, the shape and average particle size can be altered by the application of a magnetic field [14]. By high resolution transmission electron microscopy (HRTEM), the regular shape of Fe₃O₄ nanoparticles has been

reported by Alatorre et al. [14]. Three precursors $[(\text{FeCl}_3, 6\text{H}_2\text{O}), (\text{Fe}(\text{C}_5\text{H}_7\text{O}_2)_3), (\text{Fe}(\text{NO}_3)_3 \text{ and } (\text{H}_2\text{O})]$ can be used in a precipitation method in order to prepare $\alpha\text{-Fe}_2\text{O}_3$ nanoparticles. The size and magnetic parameters of $\alpha\text{-Fe}_2\text{O}_3$ can be examined by varying the nature of the study of precursors on crystalline phase. The TEM and SEM inferred the size of $\alpha\text{-Fe}_2\text{O}_3$ nanocrystals in between 21 and 38 nm by Lassoued et al. [15]. Due to remarkable alternative properties compared to molecular antibiotics, the use of nanoparticles against bacterial growth is increasing day by day. In nano technological developments and biological applications, the use of iron oxide nanoparticles has proven one of the most important transition metals oxide-based remedy because of the enriched biocompatibility of iron. The structure of the synthesized NPs for protective effect against harmful bacteria activity was investigated [16]. Iron oxides with low polydispersity can be prepared through a simple polyol synthesis in high temperature and pressure. The polyol process produces nanoparticles with a narrow particle size distribution in a simple, reproducible and cost effective way [17]. The IONPs can be synthesized using green synthesis phenomena to create ecofriendly behavior. Further, IONPs can be characterized using XRD, Fourier transform infrared spectrum (FTIR), Energy dispersive x-ray spectroscopy (EDX) etc. Since magnetism is associate to the formation well defined crystalline structures, XRD analyses were proposed in order to obtain if the structures could be observed for the hybrid materials.

This paper explores the XRD study of Fe_2O_3 nanopowders prepared with a simple soft chemical method with green synthesis route.

2. EXPERIMENTALS

Aqueous solution of pure ferric nitrate - $\text{Fe}(\text{NO}_3)_2$ and aloe-vera gel was used. The synthesis of Fe_2O_3 precursor solution was carried out from the starting materials (99.99% pure) of $\text{Fe}(\text{NO}_3)_2$. At first 20 mg of pure $\text{Fe}(\text{NO}_3)_2$ has been taken, 100 ml of aloe-vera gel is extracted from aloe vera leaf and to it 250 ml of distilled water is mixed. In the second step, the pure $\text{Fe}(\text{NO}_3)_2$ solution is mixed and stirred with the as prepared mixture of aloe vera gel using magnetic stirrer for 2 hours. The precursor mass was dried in a furnace at 100°C and then the black precursor was calcined in a tubular furnace at different temperatures. The process is summarized in Table 1.

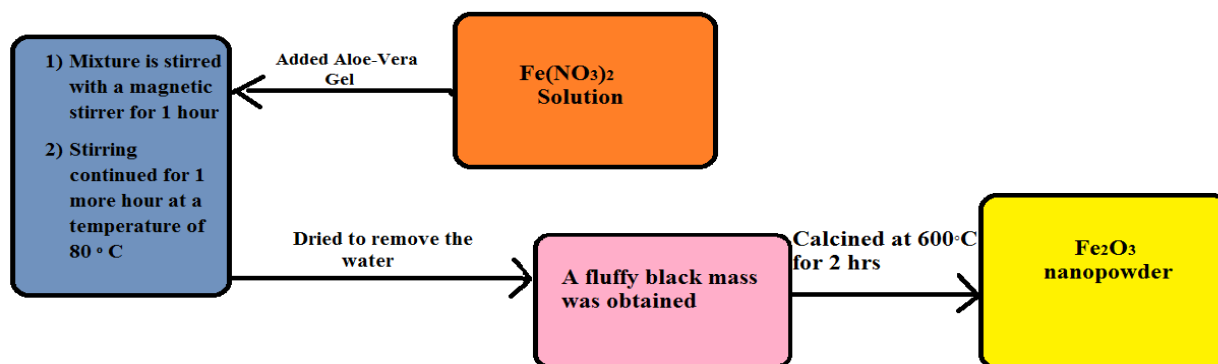


Figure 1. Reaction scheme for the preparation of Fe_2O_3 nanopowders.

The as prepared nanopowders was characterized using XRD. X-ray diffraction of the specimen was recorded with PW 1710 X-ray diffractometer using $0.15418 \text{ nm CuK}\alpha$ radiation. Average crystallite size D was calculated from widths $\Delta 2\theta_{1/2}$ in the characteristic peaks with the Debye-Scherrer formula.

3. RESULTS & DISCUSSION

The as obtained fluffy black mass which was obtained after calcinations at 600°C for 2 hrs was studied with XRD. The XRD diffractogram of Fe_2O_3 nanopowders prepared at 600°C temperature is shown in Fig. 2. The synthesized Fe_2O_3 samples at 2θ ranging from 20° to 120° have been shown and the XRD diagram indicates the synthesized nanoparticles have amorphous parts. In addition, the survey states that the IONPs synthesized by aloe-vera gel are mostly amorphous in nature [18], [19]. A number of diffraction peaks at $2\theta=30.121^\circ$, 38.453° , 54.256° , 59.127° were existing and corresponding very well with (110),(121),(220) and (120) planes respectively of synthesized IONPs.

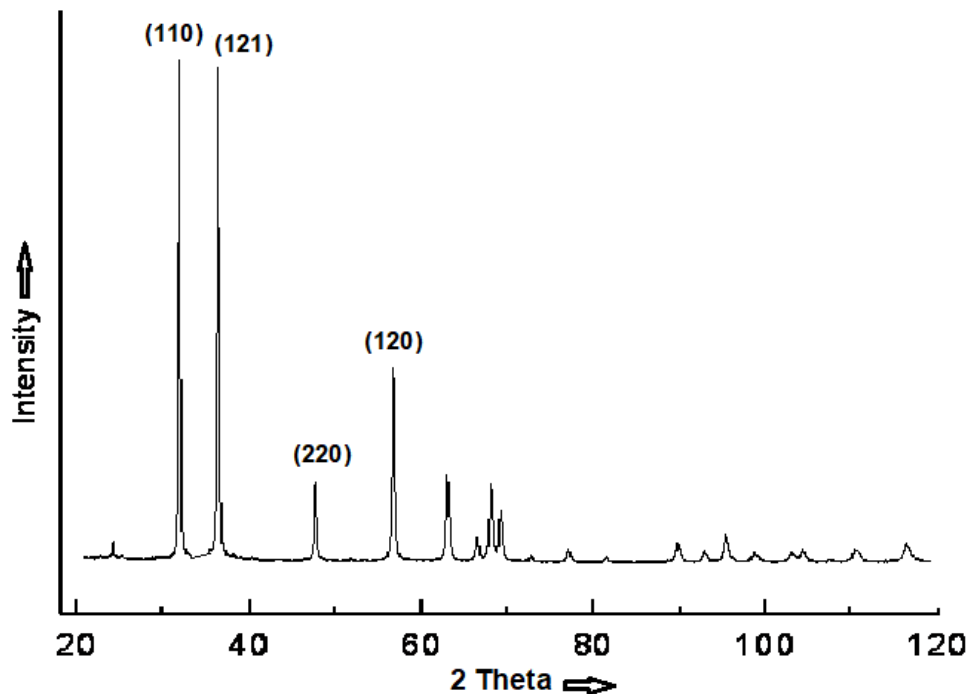


Figure 2. XRD pattern of Fe_2O_3 nanopowders

The crystallite size of iron oxide nanopowders was calculated using Scherrer's formula based on XRD data as:

$$D = \frac{0.9 * \lambda}{\beta_{1/2} \cos 2\theta}$$

Where D is the crystallite size, 0.9 is a constant shape factor, λ is the X-ray wavelength, $\beta_{1/2}$ is the full integral width at half maximum (FWHM) and 2θ is Bragg's angle. In this method the selected peaks were fitted in a computer program. A representative peak is shown in Figure 3.

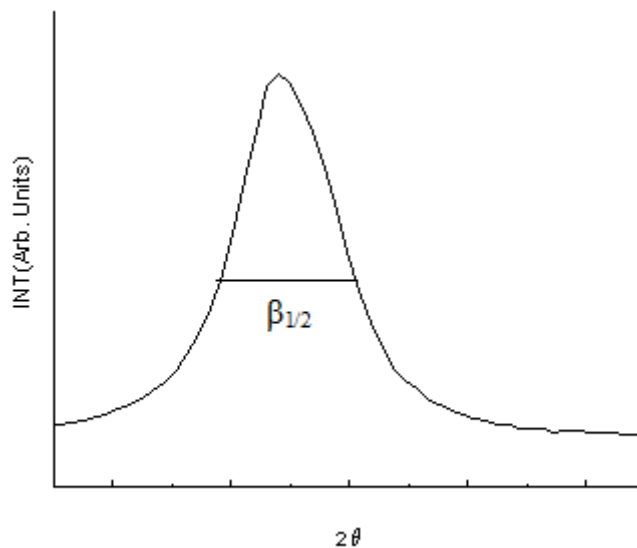


Figure 3. A representative XRD peak for the calculation of D value

With this program the value of $\sin\theta$ and β (FWHM) were calculated for each peak. D values were obtained from the intercept of the plot of β vs. $\sin\theta$ of the prominent peaks, using the relation $\beta = 1/D + 4e \cdot \sin\theta / \lambda$ [where: β (degrees) = FWHM; dimensionless, D (nm) = crystallite size; dimension = L, e = microstrain of the sample; dimensionless, 2θ (degree) = diffraction angle; dimensionless, λ (nm) = wavelength; dimension = L)]. The calculated D values by both the methods are nearly same. The average crystallite size of the as prepared Fe_2O_3 nanopowders, as measured from XRD, is 30 nm.

4. CONCLUSION

IONPs were prepared using environment friendly and cost effective green synthesis route where aloe-vera leaf extract was used as a binder. According to the characterization studies, the IONPs were synthesized successfully and the prepared nanopowders have irregular morphology. Therefore, green synthesized Fe_2O_3 nanopowder could be an excellent photo-catalyst for decolourisation and removal of toxic dyes from aqueous solutions. In addition the characterization has been carried out by XRD in order to study the crystallite size as well as crystal symmetry. A hexagonal shape with the R3C space group is measured by XRD study with an average crystallite size of the as synthesized Fe_2O_3 as 30 nm. Different diffraction peaks has

been observed at different angles. The prepared IONPs can be characterized using different tools for different properties which can be further utilized for various applications. Thus, further research can be extended for different properties and applications.

CONFLICT OF INTEREST

The authors declare that they have no conflict of interest.

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