

Synthesis and FTIR Characterization of Iron Oxide Nanoparticles

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ABSTRACT: Iron oxide nanoparticles have been prepared by using auto-combustion method. The synthesized iron oxide nanoparticles are characterized by Fourier transform infrared (FTIR) spectroscopy. Spectral data reveals that synthesised material is α -Fe₂O₃ crystalline in nature.

KEYWORDS: Iron oxide, FTIR, Nanoparticles, Auto – combustion, Calcination.

I. INTRODUCTION

In the last two decades, nanotechnology has attracted great interest in several areas of research for the development of nanoscale materials which can be obtained through different techniques, such as physical or chemical methods. Due to their size (diameters ranging from 1 to 100 nm) nanoparticles present specific and properties that are different from those they present on the macroscopic scale thus enabling unique applications. The change in properties is due to two main effects: (1) surface effects or size reduction effect (when particle size is reduced, a greater proportion of atoms are found at the surface); (2) quantum confinement-modification in electronic structure.

It is known that the ratio between the number of surface atoms and the number of massive atoms increases significantly with particle size reduction. As the surface atoms have less coordination in relation to massive atoms, nanostructured materials exhibit significantly different physical, chemical, optical, mechanical, electrical and also magnetic properties. The excess of energy of surface atoms contributes for many of the extraordinary characteristics of nanoparticles [1]. This way, nanoscale materials present great potential for applications in several technological areas such as nanoelectronics and computer technology, medicine, aeronautics and space exploration, biotechnology and agriculture.

In recent years, nanoscale transition of metallic oxides, such as iron oxide, including hematite, magnetite, and maghemite, has been attracting growing interest as they exhibit unique electrical, optical and magnetic properties for numerous applications such as production of inorganic pigments, magnetic storage media, development of gas sensors as well as electronic and optical devices, information storage, color imaging, magneto caloric refrigeration, bioprocessing, ferrofluid technology and wastewater treatment adsorbents [2, 3].

Iron oxide also plays an important role in aerospace industry, as it has been incorporated in composite propellant to improve its burning rate. Because the combustion mechanism of propellants is highly dependent on decomposition of ammonium perchlorate (NH₄ClO₄ or AP), nowadays there are many studies concerning the catalytic effect of nano-Fe₂O₃ on thermal decomposition of ammonium perchlorate.

The most widely synthesized metal oxide nanoparticles are that of iron oxide due to their wide spread applications not only in different fields of science and technology but also in biomedicines [4 - 6]. Most of applications critically depend on the size of and shape of the nanoparticles. Therefore the synthesis of well defined size and their shapes play an important role in their applications. Iron oxide nanoparticles of diameter ranging 1 to 100 nanometres find applications in the modern science due to their very fine magnetic properties.

The common methods employed for the synthesis of iron oxide nanoparticles are co-precipitation, auto combustion, micro emulsion, sol-gel technique and electrochemical synthesis etc [7 - 9].

In the present study, an attempt has been made to synthesise iron oxide nanoparticles using auto combustion method.

International Journal of Innovative Research in Science, Engineering and Technology

(A High Impact Factor, Monthly, Peer Reviewed Journal)

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Vol. 8, Issue 3, March 2019

II. MATERIALS AND METHOD

All materials were purchased from Merck Chemical Company. Equimolar concentrated Ammonium ferrous sulphate [FeSO₄(NH₄)₂SO₄] and anhydrous Oxalic acid were mixed with constant stirring in a magnetic stirrer at 270 to 280 rpm. A homogenous yellow precipitate obtained was left for 2 hours then filtered through filter paper (Wattmann 42). The Ferrous oxalate yellow powder was dried in an oven at 100 °C for 2 hours, then weighed and ground with PEG - 400 in the ratio of 1:5 in an agate mortar and burnt in an alumina crucible at around 600 °C. It started self burning. After that the sample was cooled in a desiccator for an hour and washed with deionised water followed by acetone. The sample was subjected to FTIR characterization.

For FTIR study, a small quantity of prepared powder sample was mixed with Potassium Bromide (KBr) in the ratio of 1:4 and pressed in a stainless steel die to produce thin KBr wafer, containing a relatively high concentration of the sample in IR transparent KBr matrix.

FTIR spectrum was recorded in Fourier Transform Infrared spectrophotometer (Shimadzu FTIR - 8400S) (Fig.1.) in the range of 4000 cm⁻¹ to 400 cm⁻¹. The sample was scanned at 10 scans with resolution of 4 cm⁻¹.

The double peak becomes increasingly separated, or split, as crystallinity increases. *Splitting function* or *crystallinity index* of iron oxide is calculated by summing the heights of the peaks (A & B) and dividing this value by the height (C) of the trough between them [10]. (Weiner and Bar-Yosef, 1990). Then,

$$\text{Splitting function or crystallinity index, CI} = (A + B)/C$$

$$\text{For iron oxide, Crystallinity (\%)} = C_{544, 469} / (A_{543} + B_{468})$$

Further the percent crystallinity is also measured as a peak splitting function calculated as a ratio of the area above and below the double peaks (A&B). If the area covered by the double peak is A₂ and that of between the two peaks is A₁, then Splitting function or percent crystallinity = A₁ / A₂ × 100

III. RESULTS AND DISCUSSION

Fourier Transform Infrared Spectroscopy (FTIR) analysis provides information about the chemical bonds and the molecular structure of the material. To examine composition and crystalline nature of the synthesized α-Fe₂O₃ nanoparticles, FTIR measurement is carried out at room temperature in the range of 400-4000 cm⁻¹.

Fig. 1. exhibits the typical FTIR spectrum of synthesized iron oxide nanoparticles. Absorption bands are observed from the obtained FTIR spectrum at 465 cm⁻¹, 551 cm⁻¹, 1628 cm⁻¹ and 3450 cm⁻¹.

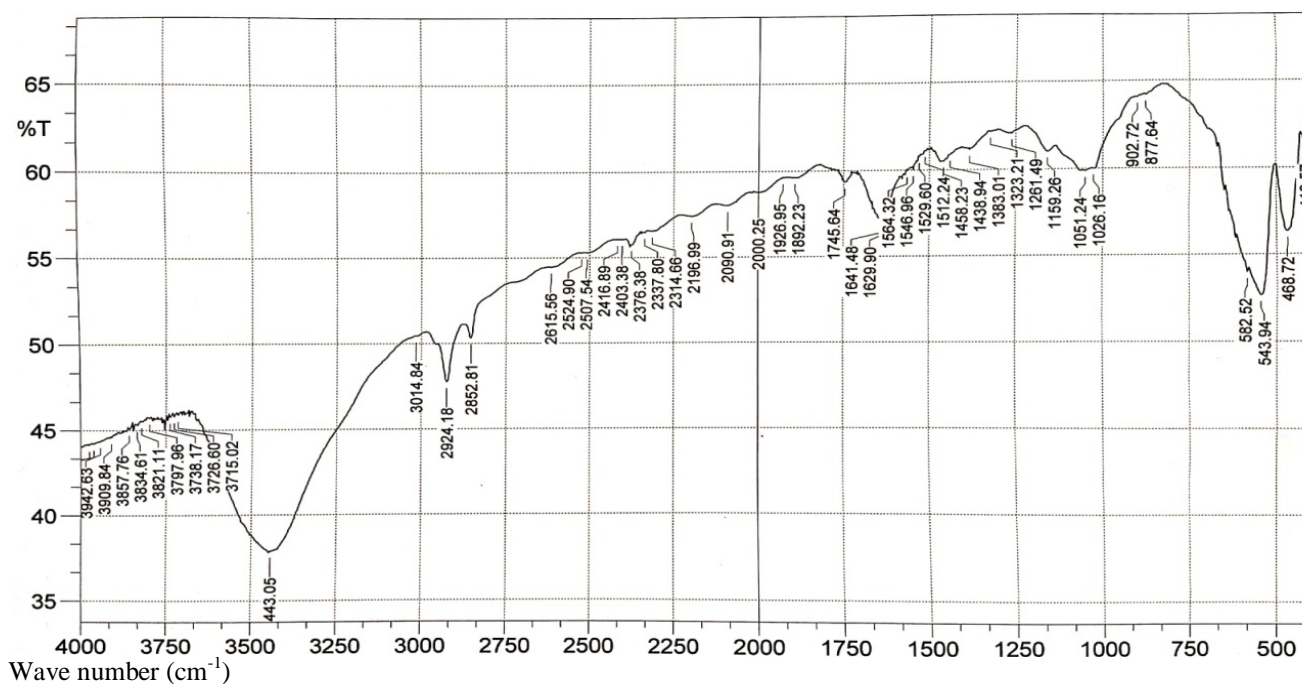


Fig.1. FTIR Spectrum of synthesised iron oxide nanoparticles by combustion method.

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Vol. 8, Issue 3, March 2019

The origination of two sharp absorption bands at 468 cm^{-1} and 544 cm^{-1} are due to the formation of Fe-O bonds which clearly confirms the formation of iron oxide crystals [11, 12]. The appearance of a broad band at 3450 cm^{-1} is due to –OH stretching and a short band at 1628 cm^{-1} can be attributed to H-O-H bending of absorbed water, surface hydroxyl and O-H stretching mode. As the FTIR spectrum exhibits only Fe-O and hydroxyl absorption bands, hence one can confirm that the synthesized product is pure $\alpha\text{-Fe}_2\text{O}_3$.

The splitting of double band into bands at 468 cm^{-1} and 544 cm^{-1} reveals the fact that the synthesized $\alpha\text{-Fe}_2\text{O}_3$ nanoparticles are crystalline in nature. The percent of crystallinity, calculated using the formula $C_{544,469} / (A_{543} + B_{468})$, is 40.6 % and by using the formula $A_1 / A_2 \cdot 100$, is 38.4% both the values are in good agreement with each other.

Finally, it can be concluded that iron oxide synthesized by combustion method is $\alpha\text{-Fe}_2\text{O}_3$ nanoparticles, the crystallinity (%) of which is in between 38.4% to 40.6%.

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