

SYNTHESIS AND XRD CHARACTERIZATION OF Fe₂O₃ NANOPARTICLES

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Abstract : In the present work the iron oxide (Fe₂O₃) nanoparticles were synthesized by the auto combustion method followed by calcination. The synthesized iron oxide nanoparticles were characterized by using X-ray diffraction (XRD) technique. The Degree of crystallinity, Lattice Strain and the crystallite size was calculated from full width at half maximum (FWHM) of the peaks by using the Debye- Scherrer formula.

Keywords : Auto combustion, calcination, XRD, FWHM and Debye-Scherrer.

I. INTRODUCTION

In recent years iron (III) oxide nanoparticles have drawn the interest of many scientists with respect to their particle size, properties and their various industrial and biological applications namely, refrigeration systems, medical imaging, drug targeting and other biological applications, and catalysis (1). There are different chemical methods used to synthesize the iron (III) oxide nanoparticles like, sonochemical method, solvothermal process, micro emulsion technique, precipitation, coprecipitation and auto combustion method, etc. (2,3,4). In the present study the iron(III) oxide nanoparticles are synthesized by using auto combustion method (ref 5. previous publication).

II. MATERIALS AND METHOD

Anhydrous Ammonium ferrous sulphate [FeSO₄ (NH₄)₂SO₄] and anhydrous Oxalic acid were mixed with equimolar concentration and stirred under a magnetic stirrer at 270 to 280 rpm. A homogenous yellow precipitate of ferrous oxalate was obtained. It 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 (5). The sample was subjected to XRD characterization.

III. RESULTS AND DISCUSSION

The data obtained from X-ray diffraction studies are tabulated in the table number (1) and (2) and the peaks obtained are shown in figure (1).

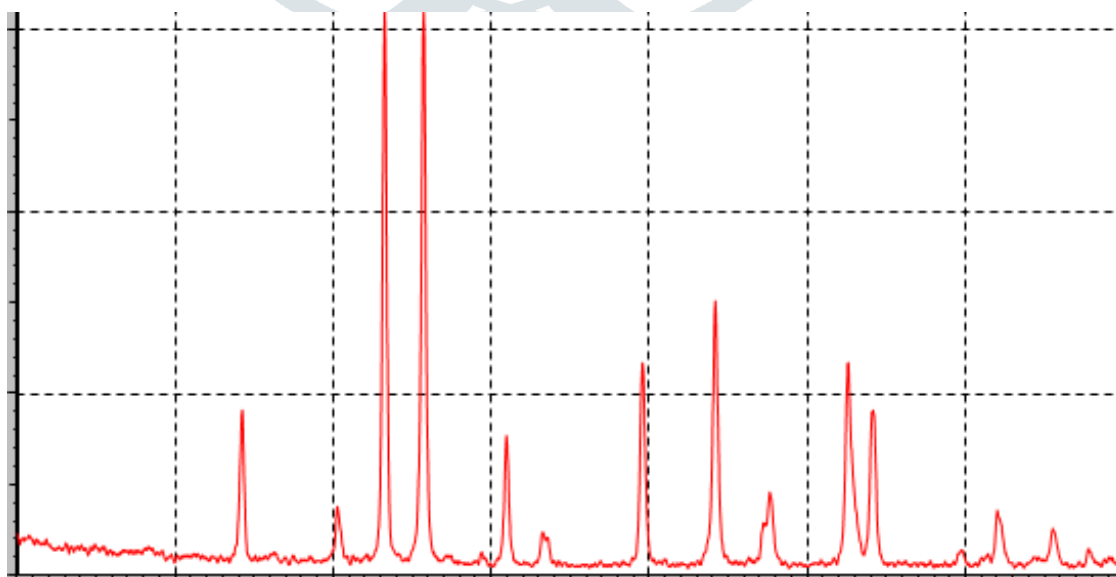


Fig.1: XRD PEAKS (Intensity Vs.2 theta)

Table 1: Peak data list

Peak No.	Theta(deg)	Sin θ	d(A)	I/II	FWHM(rad)	Cos θ	B cos θ
1	12.1	0.2096	3.67368	26	0.03597	0.9777	0.03516
2	15.13	0.26100	2.95028	11	0.2542	0.96533	0.24538
3	16.61	0.2858	2.69421	99	0.2623	0.95827	0.25135
4	17.84	0.30635	2.51356	100	0.1758	0.95191	0.16734
5	20.46	0.34955	2.20287	24	0.14087	0.93691	0.13198
6	24.76	0.4188	1.83864	39	0.27301	0.90807	0.24791
7	27.23	0.4575	1.69291	51	0.25946	0.88917	0.230704
8	28.79	0.4816	1.59899	13	0.15237	0.87639	0.133535
9	31.26	0.5189	1.48419	39	0.22835	0.85482	0.195198
10	32.04	0.5305	1.45167	30	0.28489	0.84767	0.241492
11	36.00	0.5877	1.31030	12	0.21652	0.809016	0.175168

Table 2: Strongest three peaks

Peak No.	Peaks 2 θ (deg)	d(A)	I/II	FWHM(deg)
4	35.6918	2.51356	100	0.28360
3	33.2264	2.69421	99	0.27180
7	54.1318	1.69291	51	0.27860

3.1 Degree of Crystallinity:

Study of the geometric forms and other physical properties of crystalline solids by using X-rays, electron beams and neutron beams etc., constitute the science of crystallography. Solids may be amorphous without any definite arrangement, no definite melting points and are isotropic in nature. They may be crystalline, possessing a regular repeated arrangement of atoms or molecules hence a long range order in their structure. They have a definite melting point and are anisotropic in nature.

The addition of a unit assembly or basis of atoms to every lattice point of the space lattice is called as crystal structure. Bragg's spectrophotometer is used to determine crystal structure of solids. For this a monochromatic x-ray beam with known wave length is made to incident on crystal and the crystal is rotated. Knowing one intensity of diffracted x-ray beam at different glancing angle θ is observed. The glancing angle θ for which a strong diffraction is obtained, the Bragg's condition $2d\sin\theta = n\lambda$ is applied to calculate lattice interval d.

In the present study the observed values are tabulated in the table (1).The strongest peaks obtained at peak numbers 4, 3, and 7 are shown in table (2).The degree of crystallinity is calculated by using diffraction peaks (112)and (300) of hydroxyl apatite using the equation(Landi et al,2000):

$$X_c (\%) = \frac{I(300) - I(112,300).100}{I(300)}$$

$$X_c (\%) = \frac{30.8 - 15.0 \times 100}{30.8}$$

$$= 51.29\%$$

Where X_c is the fraction of crystalline phase or degree of crystallinity, $I_{(300)}$ is the intensity of (300) diffraction peak and $I_{(112,300)}$ is the intensity of the trough between (112) and (300) diffraction peaks of hydroxyl apatite, figure (1). The crystallinity in the sample is calculated as equal to 51.29%.

3.1 Crystallite size and Lattice Strain:

There are some implications for small crystals. The measurements depend upon location of XRD peaks and their widths. Apart from this the peak broadening should also be taken into account. The peak broadening directly depends on (1) Instrument effects, (2) crystalline size and (3) lattice strain. Mathematically,

$$\beta_o = \beta_i + \beta_r$$

Assuming that β_i has been taken care in the measurement of β_r and as such it is neglected, then

$$\beta_o = \beta_r$$

But, $\beta_r = \beta_c + \beta_s$

Where, β_c = Broadening due crystal size

β_s = Broadening due strain

But, $\beta_c = \frac{K\lambda}{L \cos\theta}$

$$\beta_s = 4\epsilon \tan\theta$$

Then,

$$\beta_o = \frac{K\lambda}{L \cos\theta} + 4\epsilon \tan\theta$$

Multiplying the above equation by $\cos\theta$,

$$\beta_o \cos\theta = \frac{K\lambda}{L} + 4\epsilon \sin\theta$$

This equation is in the form of straight line i.e., $y = mx + c$.

If a plot is drawn between $\beta_o \cos\theta$ on y-axis and $\sin\theta$ on x-axis, it is a straight line with a slope = 4ϵ and y intercept = $\frac{K\lambda}{L}$ figure (2).

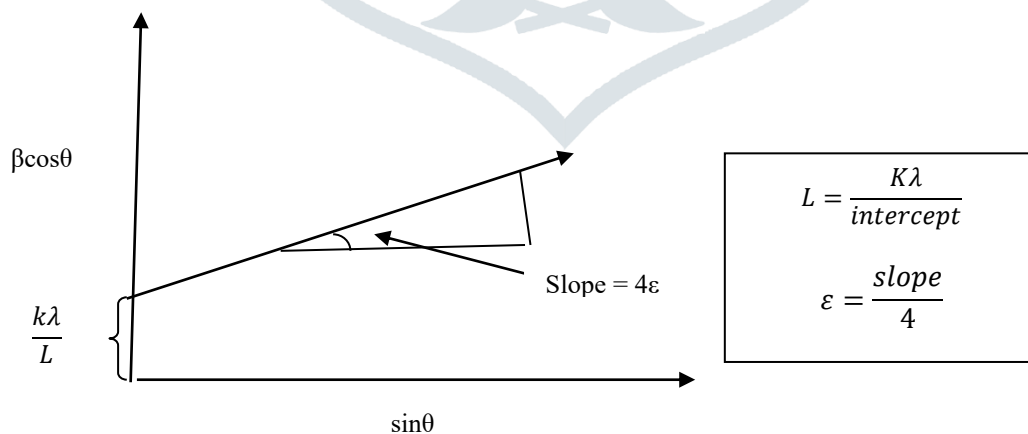


Figure 2 : $\beta_o \cos\theta$ Vs. $\sin\theta$

Therefore,

Crystallite size, $L = \frac{K\lambda}{\text{intercept}}$

Where,

$\lambda = 1.54056 \text{ \AA}$, K is assumed to be 1

$$\text{Lattice Strain, } \varepsilon = \frac{\text{Slope}}{4}$$

In the present study the average Lattice Strain for the three strong peaks is calculated to be as equal to 0.0034.

The crystallite size of the synthesized iron oxide nanoparticles were calculated from the Full Width at Half Maximum (FWHM) of all the peaks in XRD patterns using Debye- Scherrer formula:

$$t = \frac{0.9\lambda}{\beta \cos\theta}$$

The average crystallite size was calculated to be as 30.22nm.

Finally, to conclude the iron oxide nanoparticles synthesized by combustion method are $\alpha - \text{Fe}_2\text{O}_3$ nanoparticles, the crystallinity (%) of which is 51.29. This is in fairly agreement with the crystallinity determined by using FTIR technique (previous published paper, ref.5) which was in between 38.4% to 40.6%. The crystallite size calculated by using Scherrer's equation is 30.22nm and the lattice strain is calculated as equal to 0.0034.

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