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Synthesis and XRD, FTIR Studies of Alumina Nanoparticle using Co-precipitation Method

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Abstract: ***Nano precursor of aluminium hydroxide was synthesized by Co-Precipitation method from aluminium sulphate and sodium carbonate.*** Al_2O_3 ***nanoparticles were prepared by calcinations of the precursor at 500°C for 5 h in an oven. The synthesized samples were characterized by X-ray diffraction (XRD) and Fourier transform infrared spectroscopy (FTIR).***
Keywords: Nanoparticle, Synthesis, XRD, FTIR, Alumina.

I. INTRODUCTION

In a wide variety of basic research and technological applications, metals and semiconductor nano particles received considerable attention because of their improved optical, electrical and magnetic properties compared to their bulk counter-parts [1]. In particular, alumina nano particles are expected to play important role in a variety of relevant applications like high temperature electrical insulator, high voltage insulators, furnace liner tubes, electronic substrates, thermometry sensors, gas laser tubes and anti-bacterial activities. These oxide materials can be synthesized by different methods such as Solution Combustion, Chemical Precipitation, Sol-Gel, Hydrothermal, Solvo thermal, Microwave Assisted Sol-Gel, Green synthesis. Among these methods, Co-precipitation is one of the best methods to synthesis nano particles without agglomeration in the yield. In this paper, Al_2O_3 nano particles are prepared by co-precipitate method using Aluminium Sulphate and Sodium Carbonate with Water as Solvent. The samples are synthesized under standard laboratory condition in clean room and analyzed using X-ray Diffraction (XRD) and Fourier Transform Infrared Spectroscopy (FTIR).

Alumina has two forms namely transition or metastable and stable forms. Of the different forms of aluminas $(\chi, \eta, \delta, \kappa, \theta, \gamma, \rho)$ except α-alumina all other are in the transition forms. The crystal structure of most of the aluminas are hexagonal plate with large surface area. Due to this they are mainly used as catalysis and absorbent[2].

II. EXPERIMENTAL PROCEDURE

A. *Synthesis of Al_2O_3 Nano particles*

To prepare Al_2O_3 nanoparticles, 100 ml of 1 M Sodium Carbonate solution is added drop-wise into a solution containing 100 ml of 0.03 M Aluminium Sulphate solution under constant stirring. Then the resulting solution is kept at room temperature for 12 hours under constant stirring. A white precipitate is formed. It is washed several times with doubly ionized water and filtered by using Whatman filter paper. Then precipitate is dried at 80°C in a hot air oven for more than 24 hours. The obtained samples are calcinated at 500°C for 5 hours to get Al_2O_3 nano particles.

$$Al_2(SO_4)_3 + 3Na_2CO_3 + 3H_2O \rightarrow 2Al(OH)_3 + 3CO_2 + 3Na_2SO_4$$

$$2Al(OH)_3 \xrightarrow{500^oC} Al_2O_3 + 3H_2O$$

III. RESULTS AND DISCUSSION

A. *Fourier Transform infrared analysis*

Fourier Transform Infrared Spectroscopy is used to determine the chemical properties of a compound in a qualitative manner. In Fig. 1, The Vibrational peaks at 517cm^{-1}, 558 cm^{-1}, 625 cm^{-1}, 700 cm^{-1}, 732 cm^{-1} and 881 cm^{-1} are due to Al-O-Al Stretching vibration. According to the author Bustan Afruz et.al., the peaks lie in the range of 400-900 cm^{-1} are assigned to Al_2O_3. [3] The Vibrational peaks at 1024 cm^{-1} and 1160 cm^{-1} in the range 1190-1075 cm^{-1}are corresponding to C-O Stretching vibration of Sodium Carbonate. The Vibrational peaks at cm^{-1}, 1447 cm^{-1} and 1778 cm^{-1} are prescribed to O-H bending vibration of the solvent Water

and these peaks lies in the range 1395-1440 cm^{-1}. The vibrational peaks at 3042 cm^{-1}, 3463 cm^{-1} and 3525 cm^{-1} in the range of 3300-2500 cm^{-1} are 1375 aligned to O-H Stretching vibration.

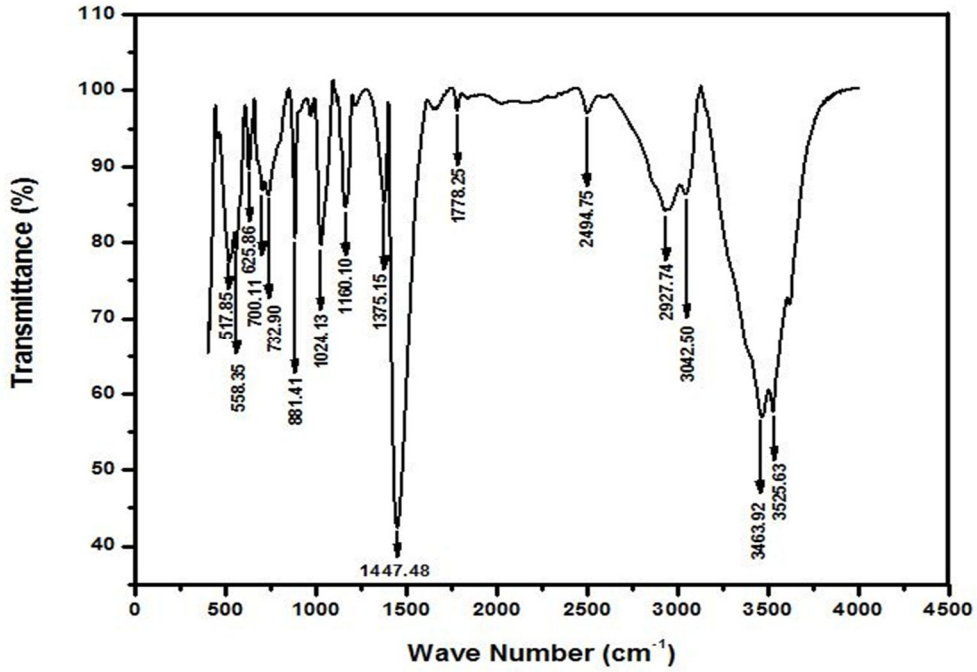

Fig. 1 FTIR spectrum of Al_2O_3 nanoparticle

TABLE 1
Vibrational Peaks, Force Constant and Attributions of Alumina Nanoparticle

Vibrational Peaks (cm^{-1})	Force Constant (N/m)	Attributions	Reference
517	158.20	Al-O-Al (s)	[3]
558	184.28		
625	231.20		
700	290.02		
732	317.14		
881	459.39		
1024	423.73	C-O (s) of Na_2CO_3	[4]
1160	543.76		
1375	105.66	O-H (b) of solvent	[4]
1447	117.02		
1778	176.67		
3042	517.16	O-H (s) of solvent	[4]
3463	670.21		
3525	694.42		

The Vibrational Peaks, Force Constant and Attributions of Al-O, C-O and O-H Vibrations are shown in the Table 1. The force constant is proportional to the strength of the covalent bond linking the masses of the atoms present in the bond. It is calculated using the formula [4].

$$k = 4\pi^2 c^2 \upsilon^2 \mu \text{ ---------- (1)}$$

Where, c is the velocity of light ($3 * 10^{10}\ cm/s$),
υ is the Vibrational frequency,
μ is the reduced mass.

$$\mu = \frac{m_1 m_2}{m_1 + m_2} \text{ ---------- (2)}$$

Where m_1 and m_2 are the mass of atoms in covalent bond. The force constant of Al-O-Al (s), C-O (s) of Na_2CO_3 O-H (b) of solvent is shown in table-2.

B. X-ray Diffraction

X-ray diffraction is a versatile, non-destructive analytical method used to determine the Crystalline Phases of Various Powder and Solid Samples [5]. The X-ray Pattern of Synthesized Alumina Powder is shown in Fig. 2. The diffraction peaks at 2θ are 20.56°, 29.57°, 41.87° and 47.83° corresponding to the lattice planes (104), (024), (303) and (306) of aluminium sulphate [ICSD 073249] and the diffraction peaks at 2θ are 34.94°, 38.04°, 54.61° and 64.19° corresponding to the lattice planes (104), (110), (024) and (214) of alumina [ICSD 025778] according to the author Khamirul Amin Matori et.al., [6].. It reveals that the resultant nano particle contains both Aluminium Sulphate and Alumina. This is due to the boiling property of the Alumina.

The average crystalline size of the nanoparticles is determined by using the Debye-Scherrer equation [7]

$$D = \frac{K\lambda}{\beta cos\theta} \text{ ---------- (3)}$$

Where D is the crystalline size
K is the typical value (0.9),
λ is the wavelength of incident beam
β is the broadening half of its maximum intensity (FWHM)
θ is the Bragg's angle.

The dislocation density (δ) is used to determine the amount of defects presents in the grown samples which are determined using the following equation.

$$\delta = \frac{1}{D^2} \text{ ---------- (4)}$$

The lattice strain (ε) has been determined by using the tangent formula

$$\varepsilon = \beta / (4 \tan\theta) \text{ ---------- (5)}$$

The obtained value of dislocation density and lattice strain is found to be $1.87 * 10^{15}$ $(lines/m^2)$ and 0.0074 respectively. The broadness in the XRD is due to the presents of impurity. XRD and FTIR studies certify the nano particle nature of the prepared sample.

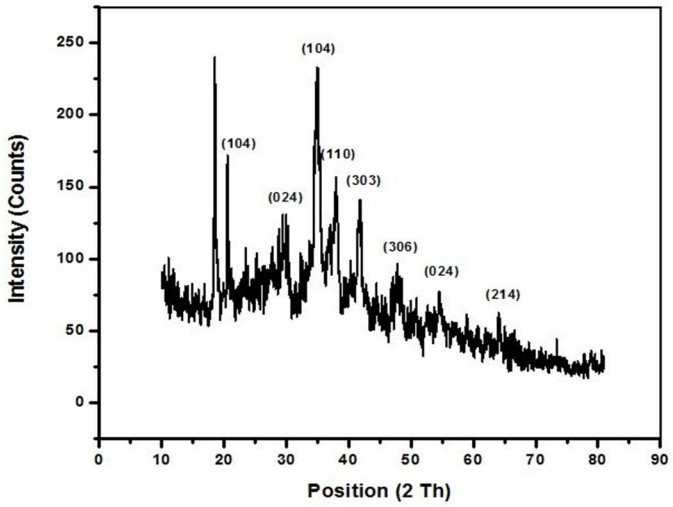

Fig. 2 XRD Spectra of Al_2O_3 nanoparticle

TABLE 2
Crystalline Size, Dislocation Density and Lattice Strain of Alumina Nanoparticle

Nanoparticle	Crystalline Size (D) nm	Dislocation Density (δ) 10^{15} $(lines/m^2)$	Lattice Strain (ε)
Alumina	23.1	1.87	0.0074

The Crystalline Size, Dislocation Density and Lattice Strain of prepared Alumina nanoparticle is shown in the Table 2. The average crystalline size of the prepared sample Alumina has been found to be 23.1 nm. It confirms that the prepared sample is alumina nanoparticle.

IV. CONCLUSIONS

Aluminium oxide (Al_2O_3) Nanoparticles were successfully prepared by Co-precipitation method. The FTIR analysis confirmed the formation of Aluminium oxide bonds and all other bonds by the solvent water and reactant (Na_2Co_3). From the FTIR analysis, the Force Constant was calculated. The XRD analysis confirmed the crystalline nature of Aluminium oxide (Al_2O_3) Nanopaticle. From this study the size of the particle, dislocation density and lattice strain are determined. The grain size of the sample has been found to be 23.1 nm which lies in the nanometer range. It suggests that prepared sample is nanoparticle.

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