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Synthesis of Nanoparticle (Alumina) and its Characterization

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Abstract— Nanotechnology is an important field of modern research and manipulation of Nanoparticles (matter with at least one dimension sized from 1 to 100 nanometers).This paper consists of synthesis of Nano adsorbent (Alumina) and its characterization. Nano Alumina can be used as catalysts , adsorbents and also wear resistant coating. A variety of preparation techniques have been reported for the synthesis of alumina nanoparticle; Deionized water using Laser ablation technique, using agarose as Template , Solvo thermal Method, Using Sol Gel method. Out of all these methods this study focused on Sol Gel process. Nano Alumina (Al_2O_3) were synthesized by 0.1 M $AlCl_3$ Ethanolic Solution. The aim of this present work is to carry out characterization that includes X-ray Diffraction, FTIR (Fourier Transform Infrared Spectroscopy) , SEM (Scanning Electron Microscope) and thus comparing synthesized nanoparticle to the standard one. The aim of this article to reflect on the current state and future prospects especially the potential of the above mentioned technique for Nano adsorption in the chemical industries.

Keywords: *Sol-Gel,Sintering, FT-IR, XRD, SEM, SCHERRER'S EQUATION.*

I. INTRODUCTION

Alumina is one of the inert biomaterials used in implants. It is therefore, a biodegradable material, well tolerated by the biological environment. In literature, there is information on obtaining Al_2O_3 by sol-gel method using the different precursors .The Sol-gel method is based on the phase transformation of a sol obtained from metallic alkoxides or organometallic precursors. Some of the advantages of the sol-gel method are its versatility and the possibility to obtain high purity materials, allowance of the synthesis of special materials and energy savings by using low processing temperature.

II. METHODS OF SYNTHESIS OF NANO PARTICLES

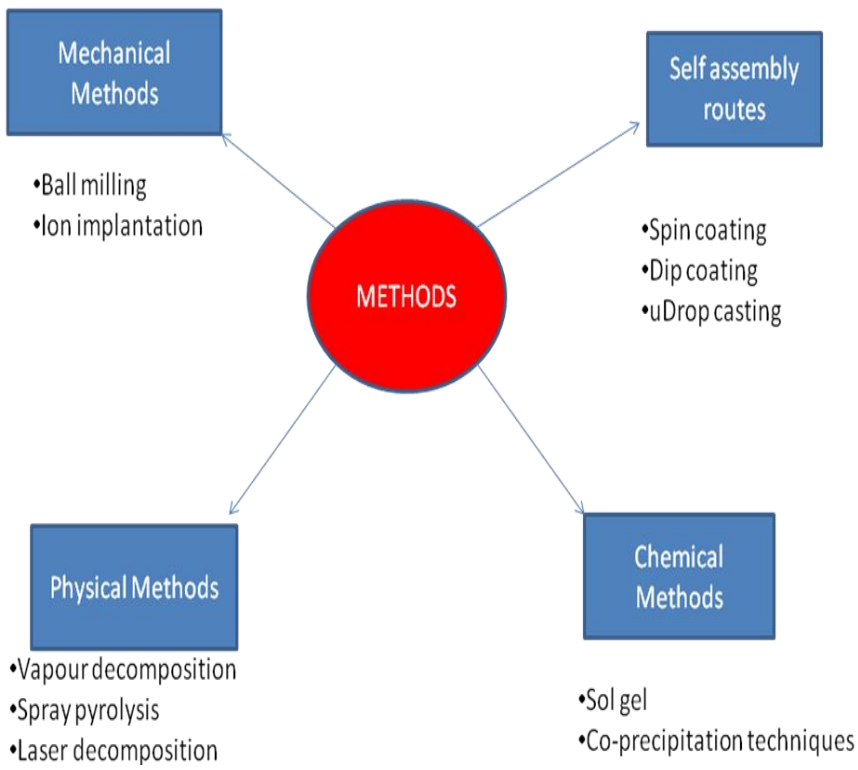

fig:1 Methods of synthesis of Nano particles

A. Mechanical Methods

- *Ball milling*

A tool for grinding hard materials and mixing solids and liquids (prepare suspensions and emulsions) by means of spherical grinding media. The internal device of the ball mill grinds material into powder-like substances, and can rotate continuously for optimal grinding and refinery production.

- *Ion implantation*

Ion implantation is a process of impacting of extrinsic atoms into a solid by bombarding its surface with accelerated ions. This process is used to change the physical, chemical, or electrical properties of the solid.

B. Self Assembly Routes

- *Spin coating*

A process to form thin films of material by placing a solution of the material on the centre of a disc and spinning very rapidly allowing the material to spread out evenly and thereby forming a thin film.

- *Dip coating*

Immersion and controlled removal of a solid from a solution to cover the surface with a uniform layer. A film deposition method where a substrate is dipped into a solution containing a polymer and a solvent. After evaporation of the solvent on a hotplate, a thin layer is formed.

- *uDrop casting*

For small substrates (~ 1 cm^2), an easy and tunable deposition method is drop-casting – spreading a nanoparticle dispersion over a substrate and allowing it to dry under controlled conditions, i.e. pressure and temperature.

C. Physical Methods

- *Vapour decomposition*

Chemical vapor deposition (CVD) is a chemical process used to produce high quality, high-performance, solid materials. In CVD, the wafer (substrate) is exposed to one or more volatile precursors, which react and/or decompose on the substrate surface to produce the desired deposit.

- *Spray pyrolysis*

Spray pyrolysis is a process in which a thin film is deposited by spraying a solution on a heated surface, where the constituents react to form a chemical compound. The chemical reactants are selected such that the products other than the desired compound are volatile at the temperature of deposition.

- *Laser decomposition*

Complex laser-gas interactions generally precede the deposition of solids on a substrate, especially in laser chemical vapor deposition.

D. Chemical Methods

- *Sol gel*

Sol-gel process consists in the chemical transformation of a liquid (the sol) into a gel state and with subsequent post-treatment and transition into solid oxide material.

- *Co-precipitation Techniques*

Co-precipitation method, which produces a mixed precipitate comprising two or more insoluble species that are simultaneously removed from solution. The precursors used in this method are mostly inorganic salts (nitrate, chloride, sulfate, etc.) that are dissolved in water or any other suitable medium to form a homogeneous solution with clusters of ions.

E. Alumina Synthesis Methods

- *Deionized Water Using Laser Ablation Technique*

Laser ablation is the process of removing material from a solid (or occasionally liquid) surface by irradiating it with a laser beam. At low laser flux, the material is heated by the absorbed laser energy and evaporates or sublimates. At high laser flux, the material is typically converted to a plasma. Usually, laser ablation refers to removing material with a pulsed laser, but it is possible to ablate material with a continuous wave laser beam if the laser intensity is high enough.

- *Using agarose as Template*

Agarose gels have been applied as templates for the formation of macroporous metal oxide structures. The preparation of the agarose template is extremely simple, with variation of the agarose content, control over morphology is demonstrated: The average pore size decreases from 180 to 55 nm and the surface area increases from 238 to 271 $m^2 g^{-1}$ with increasing agarose content in the gel.

- *Solvo Thermal Method*

Solvo-thermal synthesis is a method for preparing a variety of materials such as metals, semiconductors, ceramics, and polymers. The process involves the use of a solvent under moderate to high pressure (typically between 1 atm and 10,000 atm) and temperature (typically between 100 °C and 1000 °C) that facilitates the interaction of precursors during synthesis. If water is used as the solvent, the method is called "hydrothermal synthesis.

- *Using Sol Gel Method*

In materials science, the sol-gel process is a method for producing solid materials from small molecules. The process involves conversion of monomers into a colloidal solution (sol) that acts as the precursor for an integrated network (or gel) of either discrete particles or network polymers.

- *Advantages of Sol Gel Method*

It produces high purity products and uniform nanostructure achievable at low temperatures. It has low sintering capability. It can produce thick coating to provide corrosion protection performance. It is also possible to generate ceramic material at a temperature close to room temperature.

- *Disadvantages of sol gel method*

A very high temperature furnace or heating device is required. There is often a large volume shrinkage and cracking during drying.

- *Synthesis Of Alumina By Sol Gel Method*

1. 1M $AlCl_3$Ethanolic solution was taken 28% NH_3 aqueous solution was added drop wise and a turbid solution Al $(OH)_3$ is obtained.
2. After this it is subjected to 100^0C-120^0C for about 24hrs.Further this is calcinated for 2hrs at 1000^0C-1200^0C at heat rate of 20^0C/min. The powder obtained is Nano alumina (Al_2O_3)

- *Chemical Reaction*

$$AlCl_3.6H_2O + C_2H_5OH \xrightarrow{\text{liq. } NH_3} Al(OH)_3 \text{ (white geletinous ppt)}$$

$$Al(OH)_3 \xrightarrow{\triangle} Al_2O_3 \xrightarrow{\text{calcination}} \text{Nano Alumina}$$

III. FLOW CHART OF SYNTHESIS OF NANO ALUMINA

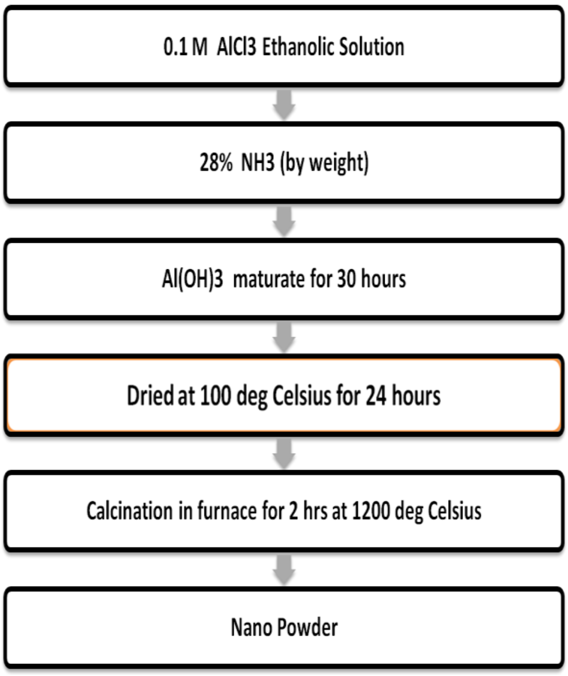

fig: 2 synthesis of nano alumina

- *Calculation*

Molar mass of $AlCl_3.6H_2O$= 241.5 g
For Preparation of 0.1 M AlCl3 Ethanolic Solution
Mass of $AlCl_3.6H_2O$ required= 24.15 g
Volume of C_2H_5OH required= 1 lit.

- *Characterization*

1. FT-IR
2. X-ray Diffraction (XRD)
3. Scanning electron microscopy (SEM)

IV. FT-IR

Fourier Transform Infra-Red (FTIR) spectroscopy is a measurement technique for recording IR-spectra. Infra-red light pass through an interferometer and an sample (a gas cell). A movable mirror inside the interferometer changes the distribution of infra red light that passes through the interferometer. The raw signal, the so-called "interferogram" represents the intensity of light as a function of the position of the mirror. A mathematical technique called Fourier Transform then transforms the raw data into the spectrum of the sample, the optical signal as a function of the infra-red wave-length (or the equivalent, Wavenumber). The optical signal of the spectrum depends on the gas components present. Finally, then the concentrations of the different gas components are determined based on the optical signal of the spectrum.

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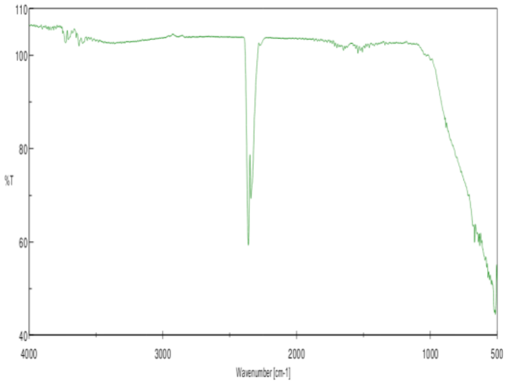
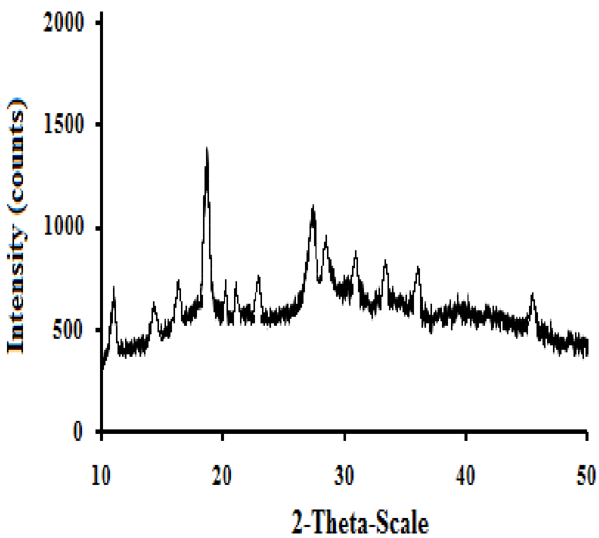


graph: 1 FT-IR of nano alumina

A. X-ray Diffraction

X-ray diffraction (XRD) is a powerful technique used to identify the crystalline phases present in materials and to measure the phase composition, preferred orientation, grain size, strain state and defect structure of these phases.

A beam of X-rays of wavelength λ is directed to the crystal at an angle θto the atomic planes. The interaction between X-rays and the electrons of the atoms is visualized as a process of X-ray reflections by the atomic planes. This is an equivalent description of the diffraction effects produced by a three dimensional grating. The atomic planes are considered to be semitransparent, that is, they allow a part of the X-ray to pass through and reflect the other part, the incident angle ş being equal to the reflected angle (called Bragg angle).



graph:2 XRD of nano alumina

Table: X-ray diffraction data of Al2O3 nanoparticle

d (Å)	**I/I_0**	**$\sin^2\theta$ Obs.**	**$\sin^2\theta$ Calc.**	**h k l**	**2θ Obs.**
8.0223	31.38	92.2	92.2	010	11.02
6.0956	12.33	159.7	159.7	110	14.52
5.4955		196.5	196.5	011	16.11
4.7551	100	262.4	262.4	101	18.64
4.4066	28.74	305.5	303.3	-102	20.13
4.0695	14.51	358.3	354.6	111	21.82
3.8754	17.45	395.0	395.4	-112	22.93
3.2586	49.11	558.7	561.7	-221	27.35
3.1359	29.64	603.3	607.4	300	28.44
				-	

B. Scanning electron microscopy (SEM)

The scanning electron microscope (SEM) is capable of producing high resolution images of a sample surface. Due to the manner in which the SEM images are created, they have characteristic three-dimensional appearance and are useful for judging the surface morphology of the sample. In a typical SEM, electrons are emitted from a tungsten and fly towards an anode; alternatively, electrons can be emitted via field emission (FE). The whole setup is maintained under a very high vacuum. The electron beam which has an energy ranging from a few hundred eV to 50 keV is focused by one or two condenser lenses into a beam with very fine focal spot sized 1nm to5 nm.

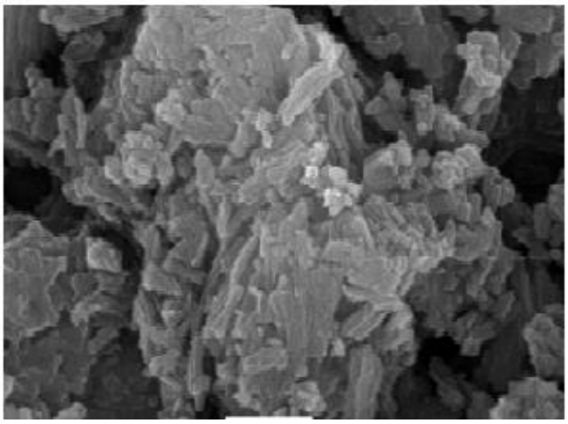


fig: 3 SEM of nano alumina

V. PRACTICAL RESULT

Mass of $AlCl_3.6H_2O$ taken= 25 g
Mass of Nano Alumina obtained= 19.5 g
Practical Yield is 78%

VI. CONCLUSION

Mostly the yield obtained from sol gel method is 90-92% but 78% yield is obtained due to experimental errors and reduction in mass during refluxing(calcination) of nano alumina.

A. FT-IR

FT-IR graph analysis obtained is similar to the standard Nano Alumina.
In FTIR the depression observed at $2400cm^{-1}$ is due to presence $CO_2$and water of hydration as impurities.

B. X-Ray Diffraction

- A comparison of values of 2θ and $\sin^2\theta$ for the Al_2O_3 reveals that, there is good agreement between the calculated and observed values of 2θ and $\sin^2\theta$ on the basis of assumption of monoclinic structure.
- The structure of Al_2O_3 yields values for lattice constant a=11.1427Å, b=8.0223Å, and c= 8.9645Å; α=γ =90° and β= 122.7°, unit cell volume V=674.30Å^3. In conjugation with these lattice parameters the conditions such as a ≠b ≠c and α=γ ≠β required for the samples to be monoclinic were tested and found to be satisfactory.
- The particle size of the samples under study was determined by using equation Scherrer's equation
  $P = 0.9\lambda/B\cos\theta$

As a result average particle size is found to be 87 nm by XRD

C. SEM

The SEM of Al_2O_3 shows aggregation of Nano rods with average size of ~89 nm.

REFERENCES

[1]. Majid Farahmandjou and Nazafarin golabiyan March 2015 Journal of "Ceramic Processing Research"
[2]. Veeradate Piriyawong,Voranuch Thongpool,Piyapong Asanithi, and Pichet Limsuwan 2012 Journal of Nanomaterials "Preparation and Characterization of Alumina Nanoparticles in Deionized Water Using Laser Ablation Technique"
[3]. S.A Hassanzadeh-tabrizi,, E. Taheri –Nassaj ,Sarpoolaky, May 2008,"Synthesis of an alumina–YAG nanopowder via sol–gel method"
[4]. M.J. Ribeiro, J.C. Abrantes, J.M. Ferreira, J.A. Labrincha, Ceram. Int. 28 (2002) 319-326
[5]. I. Nettleship, Key Engg. Mat.122-124 (1996) 305-324.
[6]. L.M. Sheppard, Ceram. Trans. 31 (1992) 3-23
[7]. J.F Poco, J.H Satcher Jr., L.W Hrubesh, J. Non Cryst Solide 285 (2001) 57-63.
[8]. R.W. Rice, "Porosity of Ceramics," Marcel Dekker Inc New York (1998) 539