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Synthesis and Characterization of Alumina Aerogel by Epoxide Assisted Method and Subcritical Drying.

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ABSTRACT : *The increasing industrial application of aerogels in the fields of catalysts, thermal and acoustic insulators, dielectric substrates and in space applications have generated considerable interest in developing aerogels through alternative methods in addition to supercritical drying. To make aerogels interesting for larger scale commercial application, one must look for alternative process for the most expensive and risky part of the preparation: supercritical drying. The present work relates to the synthesis of Alumina aerogel by Epoxide assisted sol-gel process and subcritical drying. Characterization by Scanning Electron Microscope and X-Ray Diffraction Methods and testing of properties such as Porosity, Density, Refractive index and Load Bearing capacity have further enhanced the use of subcritical drying as an effective alternative for the synthesis of Alumina Aerogel.*
Keywords *-Aerogel, Alumina, Catalyst, Nanomaterial, Subcritical.*

I. INTRODUCTION

Man has mastered almost every naturally occurring material and has begun to explore materials that can satisfy his requirements by artificial production. One such artificial material group is Aerogels. Aerogels are open-celled, mesoporous, solid foam that is composed of a network of interconnected nanostructures and exhibit porosity of not less than 50%. There are a variety of aerogels in which Alumina Aerogel stands apart for its application in the fields of catalysts, thermal and acoustic insulators, dielectric substrates and in space applications.

Aerogels are synthesized in a two-step process. (i) Sol-gel process (ii) Gel-Aerogel drying process. For Alumina Aerogel, the sol-gel process can be done by two variants (i) Alkoxide assisted method (ii) Epoxide assisted method. The gel-aerogel drying can be done in three different ways (i) Supercritical drying (ii) Freeze drying (iii) Subcritical drying. Supercritical drying [1]is currently employed to synthesize Alumina Aerogel. This method is risky and expensive. Freeze drying is expensive and cannot be used for large scale production. To make aerogels in large scale, one must look for a process with less expensive and less risk involved preparation i.e., Subcritical drying.The present work relates to the synthesis of Alumina Aerogel by Epoxide assisted sol-gel process and subcritical drying.

II. DRAWBACKS OF DIFFERENT FORMS OF DRYING

2.1 Normal drying

When a liquid changes into a gas in a heterogeneous environment, surface tension in the liquid body and capillary forces pulls against any solid structures the liquid is in contact with. Due to this, the delicate nanostructured networks tend to be broken apart. When a gel is subjected to such heterogenic drying i.e. drying in normal environment, it collapses and forms xerogel.

2.2 Supercritical drying

The method that has been used since the invention of aerogels is supercritical drying (SCD). SCD involves high pressure and high temperature. In such an environment, the transformation from liquid to gas does not cross any phase boundary but instead passes through the supercritical region. In this region, the distinction between gas and liquid ceases to apply because the densities of liquid and gas become equal at the critical point of drying. However, it is expensive and the risk factor is high.

2.3 Freeze drying

Freeze drying involves low temperature and low pressure. This works by freezing the gel and then reducing the surrounding pressure to allow the frozen fluid in the material to sublimate directly from the solid to gas phase. It is expensive and cannot be employed for large scale production.

III. SUBCRITICAL DRYING

It was discovered by Dr Doug Smith, Dr Ravindra Deshpande and Prof Jeffrey Brinker. This method was discovered 50 years after the discovery of supercritical drying. This method does not require high pressure, high temperature and can be done continuously.

3.1 General procedure of subcritical drying

In this process, the gel is purified with acetone or alcohol. Then it is soaked in pentane / hexane / toluene, to replace the pore fluid with the aprotic solvent. The gel is chemically modified to replace its polar surface groups with non-polar groups by diffusing a solution of aprotic solvent and waterproofing agent into its pores. The gel is purified by exchanging into a pure aprotic solvent. The liquid in the gel is gently evaporated, causing the gel to partially collapse. The gel springs back once the liquid has finished evaporating from its pores and forms an aerogel.

IV. EXPERIMENTALDETAILS

4.1 Rawmaterials

The rawmaterialsused for the ssynthesis of Alumina Aerogel by epoxide assisted method and subcritical drying areAluminium Chloride hexahydrate ($AlCl_3.6H_2O$), Ethanol (C_2H_5OH), Epicholorohydrin (C_2H_5ClO), Distilled Water, TrimethylChlorosilane (C_3H_9SiCl), Hexane (C_6H_{14}), Acetone (C_3H_60).

4.2 Synthesis of Alumina Aerogel

Aerogels are synthesized in a two-step process. (i) Sol-gel process (ii) Gel-Aerogel drying process. For Alumina Aerogel, the sol-gel process can be done by two variants (i) Alkoxide assisted method (ii) Epoxide assisted method. The gel-aerogel drying can be done in three different ways (i) Supercritical drying (ii) Freeze drying (iii) Subcritical drying.

4.2.1 Sol-gel process

Epoxideassistedmethodisemployed to make the gel.A hydrated metal salt is dissolved in a solvent to which an epoxide is added in drops. The metal salts exists as an aqua-complex in solution. The epoxide then acts as a proton scavenger, takes a proton away from the metal complex and protonates itself, resulting in an irreversible ring-opening reaction. The pH of the system increases gently and aquo-hydroxy metal complexes form. These species, which contain M-OH sites, can then condense together to form M-O-M bridges, the necessary connectivity to form a metal oxide gel.[2]

Aluminium Chloride hexahydrate is dissolved in a 1:1::Ethanol:DistilledWater solvent. The mixture is stirred continuously while adding Epichlorohydrin drop by drop. Taking the precursor in stoichiometry ratio with respect to their molecular weight is important. The gel time is sensitive to the amount of epichlorohydrin. A whitish cloudy coloured gel can be obtained. The gel time is about 45 minutes.

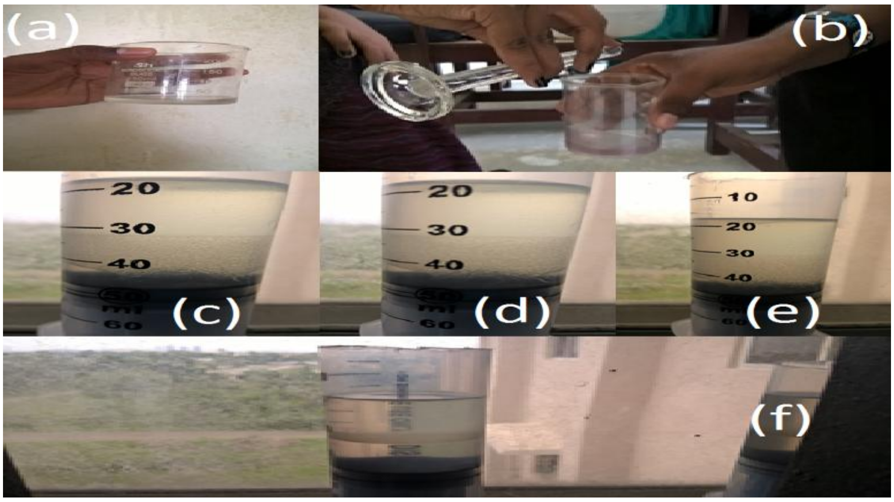

Fig 1 Epoxide assisted sol-gel Method (a)Aluminium Chloride hexahydrate-Ethanol and distilled water Solution (b) Adding Epichlorohydrin to the solution (c)(d)(e) formation of gel (f)Gel-Precursor of Alumina aerogel.

4.2.2 Gel-Aerogel transition

To obtain Alumina aerogel from gel i.e. to replace the liquid phase of the gel by a gas, subcritical method is employed.[3] The following stock solutions are prepared: Ethanol+ water (1:1ratio), Hexane +ethanol(1:3 ratio), Hexane +ethanol (1:1 ratio), Hexane +ethanol(3:1 ratio), Hexane, TMCS + hexane (1:3 ratio). The gel formed by Epoxide Assisted sol-gel method is soaked in each of these solution for 24 hours.Then the gel is finally placed inside the jar with a little hexane and secure the lid only halfway. Let the gel dry out for 1-2 days to obtain Alumina Aerogel.

Fig 2 Alumina Aerogel synthesised by Subcritical drying

V. CHARACTERIZATION

The structure and morphology studies are performed by techniques such as X-ray diffraction analysis (XRD) and Scanning electron microscope (SEM).

5.1X-Ray Powder Diffraction Analysis

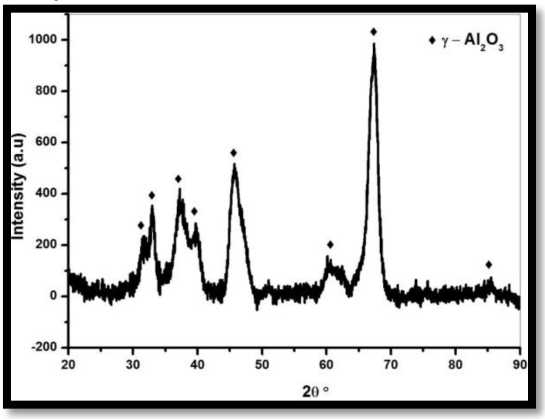

Fig 3 X-Ray Diffraction Analysis of Alumina Aerogel

The formation of γ-Al_2O_3 was confirmed from the characteristic powder XRD shown in Fig.6.1. Analysis of the diffraction pattern reveals the formation single phase cubic final structure as indexed and compared with standard diffraction pattern JCPDS reference no. 00-010-0425. The data shows considerable line broadening, indicating that the particles are Nano-sized.

5.2 Scanning Electron Microscope

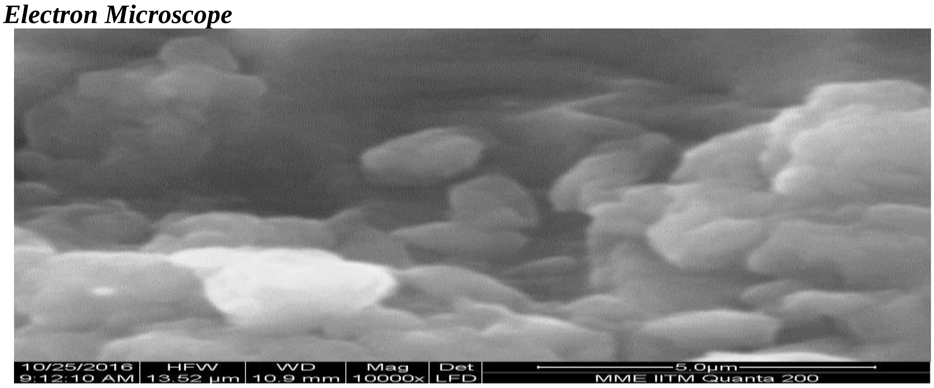

Fig 4 Scanning electron microscope images of Alumina Magnification 10000x

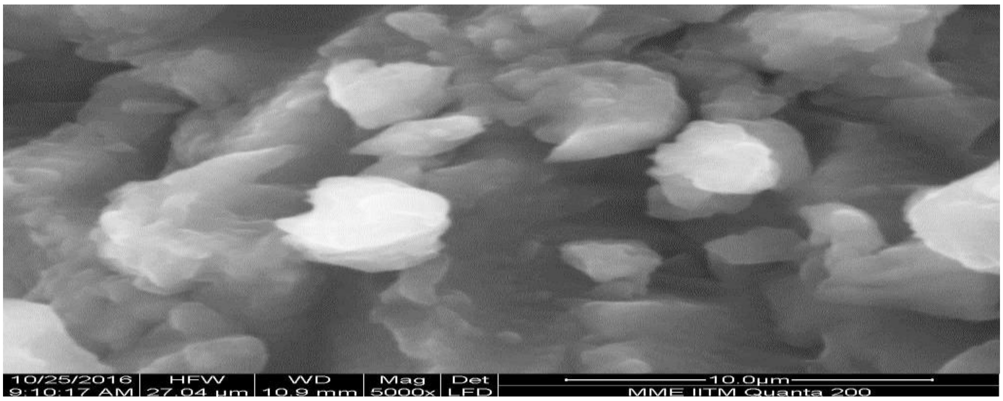

Fig 5 Scanning electron microscope images of Alumina Magnification 5000x

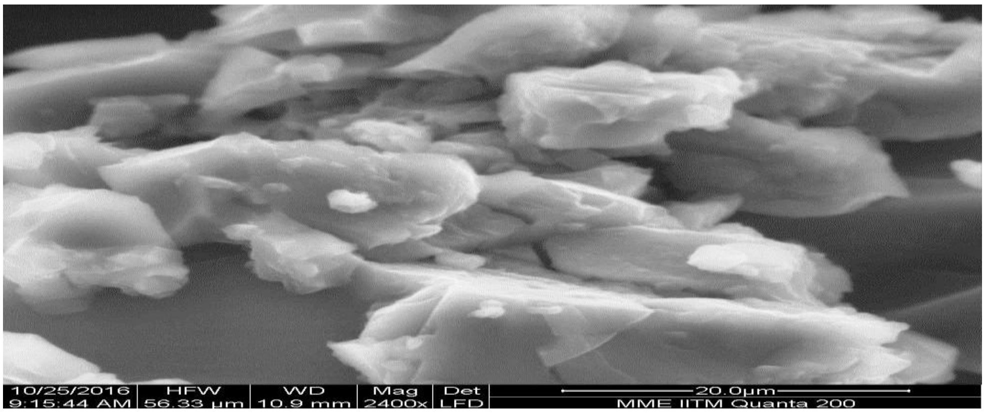

Fig 6 Scanning electron microscope images of Alumina Magnification 2400x

From the SEM images, a porous network with γ-Alumina particles is seen . The particle size of γ Alumina is measured as 85 nm . The pore size is measured as 26 nm . Aerogels are open porous and the pore size in a standard aerogel should be in the range of 1-100 nm and usually >20 nm. Mesoporous materials must have pore size ranging between 2-50 nm.

VI. PROPERTIES

Porosity, density, refractive index and load bearing capacity were measured.

Table 1 Comparison of Properties between Alumina Aerogel synthesized by supercritical method and Subcritical method

Properties	Alumina Aerogel (supercritical) (values as seen in reference papers)	Alumina Aerogel (subcritical) (values as measured)
Porosity (%)	90-97%	81.4%
Density (g/cm^3)	0.06-0.1	0.13
Refractive Index (no unit)	1-1.4	1.4
Load bearing capacity	2g will withstand 2kg (i.e., 1000 times its own weight)	0.07g was able to withstand 51g (i.e., 729 times its own weight)

VII. CONCLUSION

The subcritical method favors the formation of Alumina Aerogel. Structural Analysis of the synthesised Aerogel by Scanning Electron Microscope and X-Ray Diffraction Analysis indicate the formation of nano-sized porous networks similar to SEM image of Supercritically dried Aerogel. The properties of Subcritical drying assisted Alumina Aerogel have properties such as porosity, density, refractive index and load bearing capacity more or less like those of Supercritical drying assisted Alumina aerogel. Subcritical drying is less expensive and the risk factor is very low compared to other drying methods. This enhances the fact and encourages the use of subcritical drying as an effective alternative for the present supercritical drying method. The Alumina Aerogel can be used as catalysts in NO reduction by hydrocarbon, thermal and acoustic insulators, dielectrics, dust collectors in space applications (such as Stardust by NASA) and many more. The work can be extended to make the synthesis process less time consuming and to improve the properties further.

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