SYNTHESIS NANO ALUMINA

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ABSTRACT

The Nano alumina have been synthesized employing a novel eco-friendly route from natural bauxite ore. The synthesis of alumina powder from natural bauxite is of great interest owing to the fact that it enables mass production without the use of expensive chemical resources and processing techniques. Employing the Bayer process, synthesis of Nano alumina has been made from natural bauxite followed by sol-gel route. The ultrafine alumina powder with high surface area is obtained through the digestion of gel followed by sintering. The size of the particle and its morphology was controlled by calcination temperature and processing time. The crystallite size of the Nano alumina powder has been measured using XRD pattern and the FTIR spectrum of Nano powder after calcination at different temperatures (873 K) were measured. The results from the energy dispersive X-ray analysis (EDAX) and morphological studies reveal interesting information. The observed results indicate that one can control the particle size by controlling the aging and calcination temperature. The obtained results indicate that the above process technique is an unique method for the preparation of Nano alumina (Al₂O₃) from natural source such as bauxite.

KEYWORDS: Nano Alumina, Precipitation, sol-gel method, XRD, FTIR, EDAX
1. INTRODUCTION

1.1 NANO ALUMINA

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$_2$O$_3$ by sol-gel method using the different precursors: aluminum triisopropylate in a hydrolysis system consisting of octanol and acetonitrile, aluminum nitrate – in aqueous medium, aluminum secondary butoxide - in an alcoholic medium. The sol-gel method is based on the phase transformation of a sol obtained from metallic alkoxides or organometallic precursors. This sol which is a solution containing particles in suspension is polymerized at low temperature, in order to form a wet gel. The solvent is removed by drying the gel and the next step is a proper heat treatment. Some of the advantages of the sol-gel method are its versatility and the possibility to obtain high purity materials, the provision of an easy way for the introduction of trace elements, allowance of the synthesis of special materials and energy savings by using low processing temperature. The aim of the present paper is to prepare alumina by sol–gel method, starting from different chemical nature precursors. It is expected that the obtained alumina powders have nano metric dimensions and can be utilized as biomaterials.

Alumina ceramics have found various technological applications as high-strength materials and in electronics and catalysts because of their distinctive combination of physicochemical properties including hardness, resistance to aggressive media, refractoriness and electrical and thermal insulation. In recent years, increasing attention has been focused on the development of alumina powders with particle sizes at the nano scale for advanced engineering applications such as transparent armors for ballistic performance. Reducing the dimensions of a particulate material from microscale to nanoscale leads to considerable changes, both in the physical properties, such as electronic conductivity and optical absorption, and the mechanical properties since a much greater fraction of the atoms are located on the surfaces of the particles. Nano-sized powders have an extremely high surface area, resulting in changes in both surface energy and surface morphology. These parameters then alter the basic properties and the chemical reactivity of the nanomaterials. For instance, some nanocrystalline ceramics have demonstrated superplastic behavior at elevated temperatures, enabling these ceramics to undergo up to 300% elongation before failure. They have also been used as solid state bonding agents for joining large-grained commercial ceramics together at moderate temperatures because of having many short-circuit diffusion paths as a result of the high fraction of grain boundaries. There are indications that nano ceramics could have extremely low thermal conductivity due to phonon scattering caused by the grain boundaries.
1.2 NANO ALUMINA

Non-agglomerated ultra fine ceramic powders are important for many applications due to their extreme uniform structures. Alumina (Al₂O₃) is one of the most commonly used oxide ceramics for the packing of distillation tower, the catalyst of reactions and the additive of paints and pigments. Owing to their promising thermal, chemical, electrical, optical and catalytic properties, it has been used in many fields of engineering and technology such as coatings, heat-resistant materials (refractories), abrasive grains, coated/super abrasives, cutting materials and advanced ceramics. This is mainly due to important physico-chemical properties of alumina like hardness, highly resistance towards bases and acids, high thermal resistance, dimensional stability up to 1773 K and an excellent mechanical strength and wears resistance. Comparing micron-sized and nano-sized alumina particles, the nano-alumina has many advantages due to their larger surface area at their nano scale range. Ultra fine abrasive grains find new applications in nano-machining and nano-probes. The uses of nano-sized alumina particles have significantly improved the quality and reproducibility of fine coatings. Nanometre-sized particles of transition (t)-alumina are important for the fabrication of high-quality alumina ceramics. Nano alumina ceramic/polymer composites have been used for drug delivery applications due to their smaller particle size, spherical morphology and a homogeneous size distribution of α- alumina in polymer composite. In view of the unique properties like high surface area, porosity and chemical activity, the nano alumina particles are applied for high temperature applications, adsorbents, coatings and soft abrasives. Porous alumina template has been attracted much attention owing to nanofabrication by self-organization methods using nano hole arrays in porous alumina matrix which has versatile applications, including electronic devices, magnetic storage disks, sensors, and biological membranes. Synthesis of ultra fine alumina through numerous solution based techniques such as sol-gel, hydrothermal microwave and micro emulsions have been revealed extensively.

Adsorbent to capture hydrocarbon impurities from the air for extracting fluorine from a variety of media the ability of aluminium oxide to chemosensitivity fluorine ions used for the purification of water with increased fluorine content for vapor recovery of hydrogen fluoride from gases of super phosphate and electrolysis. Adsorption purification of oil (first transformer).

Al₂O₃ powder has wide range of applications such as electronic ceramics, high strength materials and catalysts. Among the seven polymorphs of transition alumina identified so far, namely, phases γ, η, θ, form is one of the most extensively used in industrial catalysis owing to its comparatively large surface area, unique surface characteristics, and exceptional structural stability. γ -alumina contains the same ratio of Al to O atoms as in α- alumina the only difference is that it has a tetragonal structure where there are 8 cation vacancies for every 160 atoms. Conventional γ -alumina formed through the thermal dehydration of a crystalline aluminum oxyhydroxide (boehmite) at a temperature above 723K. The γ -phase is a metastable phase and on heating it will form the thermodynamically stable α-phase. Recently many researchers are
showing interest on the preparation and application of nano-sized alumina considering their diverse properties. The property of such alumina particles are depends on particle size, morphology, surface and phase homogeneity and these properties can be controlled by selecting a proper synthetic route. γ -alumina with high surface properties such as high surface area and mesoporous properties is commonly used as a high temperature catalyst or catalytic support and as a membrane. A mixture of alumina phases are generally obtained by sol-gel technique and on heating at high temperature these transform to a stable γ -alumina phase. In our study, we have chosen aqueous based method which is simple and economic for the synthesis of pure alumina and modified alumina nanomaterials.

2. EXPERIMENTAL PROCEDURE

2.1. Precursor synthesis employing Bayer process

The commercial raw bauxite obtained from DISIR, Rajgangpur has used as the starting material. Adry mixture containing 66.67 wt. % of raw bauxite and 33.33 wt. % of sodium hydroxide (Merck, 99.9%) were digested at 873 K for 3 h using muffle furnace. The resultant product is leached with double distilled water. The obtained solution mixture is adjusted to a pH of 14 using sodium hydroxide followed by vigorous magnetic stirring at 353 K for 1 h. Thus, the stirred solution is filtered by filter paper (What Mann No.1) and then, the filtrate is cooled at 283 K for 6 h. The filtrate (Bayer liquor) is used as a precursor for sol-gel process.

2.2. Synthesis of nano alumina powder employing sol-gel route

The filtrate (Bayer liquor) is cooled to room temperature and consecutively added with N-cetyl-N,N,N, trimethyl ammonium bromide (Loba, 99%) which is maintained at 1mM (millimolar) concentration. Hydrochloric acid is added to the filtrate to initiate the flocculation and the effective flocculation has been achieved in the pH range between 7.5 to 8.5. The obtained fine aluminium hydroxide gel is further digested and aged respectively at 353 K for 8 h and 393 K for 4 h. Finally, the aged gel was washed with water to remove sodium chloride from the gel matrix. Subsequently, the water was replaced by ethanol from the gel and then, it is dried at 673 K for 1 h. This is calcinated at an elevated temperature 873 K and 1273 K to obtain respectively α- and K- phase nanoalumina products.

3. RESULT AND CONCLUSION

A nano a-Al₂O₃ powder is produced from a low-cost synthetic Bayer liquor (sodium aluminate) precursor. The process includes calcination of the dried gel stabilized with N-cetyl-N, N, N, trimethyl ammonium bromide at two different temperatures respectively 873 K and 1273 K. Calcination of dried gel leads two different crystallographic phases such as a-alumina (873 K for 6h) and K-alumina at two different temperatures. The average crystallite size of the nano
powder was estimated to be 12 nm for a-alumina phase and 76 nm for K-alumina phase. The grain size of the both a and K-alumina concludes that the five and six grains of a-phase have grown into one K-phase alumina particle.

The presence of nano crystalline alumina powder in synthesized sample was recognized by FTIR studies. The EDAX result confirms that the 67.59 wt. % of nano alumina is present in the synthesized sample.

Fig:1 EDAX RESULT OF SAMPLE

Fig:2 FTIR RESULT OF SAMPLE.

Fig:2 show the FTIR spectra of nano alumina powder calcinated at 873 K for 3h. The obtained FTIR spectrum reveals that the broad band ranging from 750 to 1000 cm\(^{-1}\) in Figs.2 is assigned to the existence of characteristic vibration of Al\(_2\)O\(_3\) Further, the obtained sharp peak in the range of 750 to 1000 cm\(^{-1}\) (Fig:2).

The vibration bands observed at 1200-1250 cm\(^{-1}\) (Fig.2) leads to the stretching and bending vibration of Al-O bond. The peaks observed around 1450 cm\(^{-1}\) has been correlated to the bending vibration of weakly bonded water molecules. The absorption bands observed around 3460 cm\(^{-1}\) (Fig:2) is due to the stretching and bending modes of hydroxyl groups on alumina surface.