



SYNTHESIS OF ALUMINUM OXIDE NANOPARTICLES AND EVALUATION OF THEIR PROPERTIES AS ABRASIVES

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ABSTRACT

Alumina, owing to its distinctive physical and chemical properties — including wear resistance, corrosion stability, high refractoriness, and excellent dielectric behavior — is extensively used across various industries. One of its most significant applications is as an abrasive. However, because of its inherent brittleness, its performance under standard conditions requires further improvement. Reducing the particle size can enhance its properties, but this also increases the tendency for particle agglomeration due to van der Waals forces. This agglomeration reduces the packing density of the nanoparticles and can lead to a non-uniform structure. In this study, alumina nanoparticles were synthesized using a drying method. The resulting powder was subjected to SEM, XRD, and FTIR analyses. The results indicated that this drying method is not suitable for producing alumina nanoparticles, as the sample aggregated into coarse clumps after drying.

Keywords: Alumina, nanoparticle, synthesis, drying, abrasive.

INTRODUCTION

The fundamental structure of the alumina unit cell consists of both hexagonal and octahedral faces that coordinate and hold the atoms together. Corner atoms occupy the hexagonal centers, while octahedral centers lie between two vertically stacked layers. Due to its unique properties—such as a high melting point, uniform pore channels, large surface area, and uniform pore size distribution—alumina is extensively used across various industries and research fields. Moreover, its porous nature, high thermal stability, good dispersibility, and affordability make alumina an ideal catalyst and catalyst support in the chemical industry. Alumina exists in several thermodynamically stable phases, including γ , θ , and α , with the α -phase being the most stable at high temperatures. Of these, gamma-alumina is the most commonly utilized in catalysis and plays a crucial role in the petroleum, automotive, aerospace composites, thermal insulation, and thermal-resistant coatings industries. Gamma-alumina is especially valued for its large surface area and Lewis acid sites, making it an effective catalyst support (1).

Activated alumina, produced by the controlled dehydration of various aluminum hydroxides under heat, is also highly sought after for its high surface area, porous structure, and enhanced surface reactivity.

Several techniques can be used to prepare nanoparticles, including the sol-gel process, evaporation–condensation, gas-phase reactions (e.g. plasma- or laser-induced reactions), and other wet-chemical methods. In general, these techniques can be divided into two major approaches: bottom-up and top-down (2).

In a top-down approach, a bulk material is mechanically or physically manipulated to produce nanosized particles. Essentially, a larger material is broken down into smaller pieces until nanoscale dimensions are achieved. This method often, though not always, involves material removal as waste—for example, machining metallic components or reducing metal particles to nanoscale sizes. Within the literature, the boundary between top-down and bottom-up processes is often referred to as nanotechnology. Various examples of top-down methods are discussed below. Mechanical milling is one of the most common top-down methods for producing nanostructured materials. Unlike bottom-up methods, which involve assembling clusters of atoms, this process reduces particle size solely through intense plastic deformation and grinding. Its relatively simple setup, low equipment cost, and scalability make it popular at both laboratory and industrial scales. However, contamination from the milling environment and particle agglomeration remain significant challenges during this process (3, 4).

Severe plastic deformation (SPD) is an advanced top-down technique for producing bulk nanocrystalline materials. Despite its limitations—such as contamination and increased particle surface oxidation—SPD allows for the production of nanocomposites by combining different metallic powders. This method is applicable only to metals, and by applying high pressure and severe shear stress, the metallic grains can be refined to as small as 20 nm. Reducing grain size greatly enhances the strength and, in many cases, ductility of the material. Given its scalability, SPD is especially promising for commercial and industrial applications.

High-shear mixing is another top-down technique employed to produce nanoemulsions. Under high-speed stirring, two immiscible liquids (e.g. oil and water) can be forced to mix into a stable nanoemulsion. The size of the resulting nanostructures depends on the shear rate and ambient temperature (5).

Powder compaction is another well-known top-down method in which pre-synthesized nanoparticles are densified using static or dynamic presses. However, this technique faces similar challenges with contamination and particle surface oxidation, which can limit its effectiveness (5).

In most cases, high-energy planetary ball mills are used for this process. Intense shear and compressive forces are applied to the particles as they collide with milling media, reducing their size. Milling parameters—including milling speed, ball size, ball-to-powder weight ratio, milling time, and milling environment—have a significant impact on particle size and product structure. Milling under cryogenic conditions, for example, enhances particle brittleness and thus improves grinding efficiency. Additionally, milling under inert gas can minimize oxidation for reactive materials, making it suitable for producing oxide-free powders. When energy input is sufficient, uniform nanocomposites can be synthesized. Depending on the milling energy and reaction thermodynamics, the resulting powders can be single-phase or multi-phase and can be either crystalline or amorphous.

Finally, melt-spinning is a different process that converts a polymer melt into nanoscale fibers. The polymer is heated to a high viscosity and loaded into a metal chamber with a spinneret. Under pressure, the melt is forced through fine holes, which may have different shapes depending on the application. The extruded fibers rapidly cool and are collected on a rotating drum. Fibers produced this way typically have diameters of a few hundred nanometers.

Bottom-up methods involve assembling a final product from simpler components much like putting together an engine from its individual parts. Essentially, this approach arranges atoms and molecules, smaller than the nanoscale, into the desired nanostructured material. Imagine being able to see individual atoms and molecules and placing them precisely where you want to form a target structure. Generally, bottom-up methods produce little or no waste, although this is not universally true. For example, some self-assembly processes require an auxiliary component that guides the assembly but is removed and

discarded once the product is complete. The following sections introduce several bottom-up fabrication techniques (6).

Pulsed laser deposition is one such thin-film deposition technique, where a focused laser beam vaporizes the source material, which then re-deposits as a thin film. This process is usually carried out under high vacuum. The high-energy laser pulses instantly ablate the material, converting it directly into a plasma. The resulting plasma plume expands and cools rapidly as it moves away from the target surface, eventually condensing into a gas phase. Under vacuum, this vapor travels without reacting until it reaches a cold substrate, where it deposits as a uniform thin film.

Self-assembly is a bottom-up fabrication process that holds tremendous promise due to its potential cost-effectiveness and scalability. However, the process is more complex than it might initially seem. Self-assembly is not a new concept — biological systems have relied on it for millions of years to form highly intricate structures, and even traditional chemical reactions can sometimes involve self-assembly principles. Recently, much attention has been given to the possibility of precisely controlling self-assembly to produce advanced nanostructures such as functional films (e.g. for surface coatings or nanoparticles), nanocapsules, nanowires, and even molecular electronic devices. Dendrimers, for example, are a classic case of relatively complex self-assembled structures, although some may argue they are simply an extension of polymer chemistry. In nature, self-assembly enables the formation of extraordinarily complex systems. Designing dendrimers and other multilayered self-assembled structures requires careful hierarchical planning. In the future, it may even become feasible to construct complex architectures — like computer processors — purely through self-assembly. Achieving this will require designing a sequence of carefully orchestrated steps. The potential benefits of self-assembly are considerable, including the ability to produce large quantities of sophisticated structures in a manner that mimics industrial-scale processes like those used in pharmaceutical manufacturing (7).

Due to aluminum and its alloys' relatively low wear resistance and mechanical strength, considerable research has focused on enhancing these properties. Hydrothermal deposition, performed at relatively high temperatures and pressures, enables direct synthesis of alpha-alumina powder. By adjusting the process parameters, nanoscale corundum particles can also be produced. Alumina may also be synthesized via thermal decomposition of aluminum salts — for instance, aluminum nitrate nonahydrate converts into different alumina morphologies upon heating to around 181°C.

Dong et al. (2018) investigated the synthesis of uniform hollow alumina-silica microspheres and their thermal insulation properties in adiabatic materials. Initially, Al_2O_3 was synthesized on ceramic foam supports to provide a robust thermal barrier with good mechanical strength and thermal properties. By doping with tetraethyl orthosilicate (TEOS), uniform hollow microspheres were produced via a novel and efficient sol-gel process using aluminum chloride hexahydrate and propylene oxide as a gel initiator. The effects of TEOS on the morphology and high-temperature stability of these hollow microspheres were clearly observed. With the optimal TEOS content, the Al_2O_3 foam acted as a support material for the hollow alumina-silica microspheres. The resulting Al_2O_3 ceramic foam composites displayed outstanding mechanical and thermal properties and exhibited minimal shrinkage after heating at 1200°C. Density, flexural strength, and thermal conductivity of these composites were measured and showed notable performance improvements (8).

Torres et al. (2018) synthesized biofunctionalized nanostructured alumina using the antimicrobial peptide BP100, studying its properties and biological applications. Alumina nanoparticles functionalized with antimicrobial peptides offer a promising strategy for fabricating medical devices and packaging materials for food, pharmaceuticals, or cosmetics because they can help reduce contamination and infection. In this research, fibrous alumina nanostructures were synthesized and modified with the BP100 antimicrobial peptide. The antibacterial activity of these functionalized nanoparticles was tested against different bacterial strains. Covalent attachment of the peptide to the nanoparticles was achieved by reacting the peptide's glutamate side-chain carboxyl groups with amine groups on the alumina surface, which had been modified with 3-aminopropyltriethoxysilane. Functionalized nanoparticles were characterized by zeta potential measurements, FTIR spectroscopy, and other physicochemical analyses. Despite the relatively lower stability of alumina modified with the

EAAA-BP100 peptide, these structures displayed much higher antibacterial activity against *Escherichia coli* and *Salmonella typhimurium* compared to the free peptide. These findings could inform future designs of hybrid nanobiomaterials utilizing antimicrobial peptide-functionalized nanoparticles (9).

Zianrong et al. (2017) synthesized nano-diamond/alumina composites using an explosive synthesis technique. In their study, nano-diamond/alumina composites were prepared via detonation using nano-diamond powder, aluminum isopropoxide, and RDX as raw materials. The phase, morphology, and structure of the resulting explosive products were investigated by X-ray diffraction (XRD), field-emission scanning electron microscopy (FE-SEM), and transmission electron microscopy (TEM). The findings revealed that the intense pressure and temperature generated by the explosion enabled alumina to coat the nano-diamonds, forming nano-diamond/alumina composites. The nano-diamond particles remained 2–10 nm in size, while the composite particles ranged between 50 and 200 nm. The alumina coating enhanced the antioxidant properties of the nanoparticles. Furthermore, the boron present in the oxide coating played a significant role in preventing the diamond particles from oxidizing during synthesis, reducing damage to the graphitic structure, increasing the explosion temperature, and improving the high-temperature stability of the alumina composites (10).

Aluminum oxide, also referred to as alumina or corundum, is an inorganic compound with the chemical formula Al_2O_3 . It is characterized by its very high melting point ($\sim 2054^\circ\text{C}$) and remarkable chemical stability. These properties make alumina ideal for use in high-temperature applications. It is also commonly used as a catalyst support and as a material in biological implants. Among oxides, alumina exhibits some of the greatest hardness, strength, and wear resistance, which is why it is often employed as an abrasive, bearing material, and cutting tool. Doping alumina with chromium or titanium yields gemstones such as sapphire and ruby, and these doped forms also serve in laser components. Alumina can exist in multiple crystalline phases as well (11).

In 2015, Yusheng et al. investigated the synthesis of coarse alumina particles through precipitation of aluminum ammonium sulfate with ammonia. Uniform, coarse-grained alumina was successfully synthesized by precipitating aluminum ammonium sulfate with ammonia under controlled conditions. The effects of temperature and ammonia concentration on the resulting precipitates were analyzed by XRD, SEM, laser particle size analysis, and FTIR spectroscopy. The results showed that the particle size distribution was strongly influenced by both temperature and ammonia concentration. The as-precipitated product was identified as boehmite, and its particle size remained stable after calcination. Optimal precipitation was achieved using 1 mol/L ammonia at 90°C , resulting in D10, D50, and D90 particle sizes of 12.54 μm , 28.14 μm , and 78.19 μm , respectively (12).

Numerous methods can be used to fabricate nanostructured materials, including mechanical milling, vapor-phase techniques (13), precipitation (14), sol-gel processes (15), and combustion synthesis. Mechanical milling is time-consuming and may introduce contamination into the final product. Vapor-phase methods require expensive equipment and often yield low production rates. In contrast, wet-chemical methods stand out due to their capacity to produce highly pure, uniform materials with controllable process parameters. One of the most crucial factors influencing the final powder's properties in wet-chemical routes is the pH of the synthesis medium. Given the significant impact of pH on powder characteristics, investigating this parameter is particularly important. Accordingly, the present study aims to synthesize alumina nanoparticles and evaluate their properties for abrasive applications.

Materials and Methods

First, 100 mL of 0.1 M aluminum chloride solution was mixed with 300 mL of 0.1 M sodium hydroxide in a 500 mL Erlenmeyer flask. The resulting mixture was then filtered, and the solid residue on the filter paper was washed twice with double-distilled water. After several rinsing steps, the washed precipitate was carefully transferred into a clean porcelain crucible using a glass stirring rod. The crucible was then placed in a furnace and dried at 80 °C for 5 hours. Finally, the dried sample was sent to the laboratory for further analysis.

Sample Analysis

Scanning Electron Microscopy (SEM) is one of the most effective analytical tools commonly employed across various disciplines, including nanotechnology. This microscope allows detailed examination of the sample's morphology, composition, surface features, and internal microstructure at both micro- and nanoscales.

Fourier-Transform Infrared (FTIR) spectroscopy is based on the principle of infrared light absorption and the study of vibrational transitions of molecules and polyatomic ions. This highly advanced and versatile technique is extensively used for structural characterization and quantitative analysis of chemical species. It is especially valuable for identifying organic compounds, as these materials produce complex spectra with distinctive absorption peaks that can be utilized for comparative studies.

X-Ray Diffraction (XRD) is a well-established and extensively used technique for analyzing the crystalline structure of materials. In this technique, the diffraction pattern produced by X-rays passing through the sample is measured, providing information about the crystal lattice parameters, phase composition, crystalline size, preferred orientation, stress, strain, lattice defects, and other important crystallographic properties.

Findings

Scanning Electron Microscopy (SEM)

As shown in Figure (1), the nanoparticle sizes ranged from approximately 66 to 129 micrometers, with more than 80% of the particles between 70 and 100 micrometers. Furthermore, the particles appeared as aggregates. This agglomeration indicates that the nanoparticles clumped together during synthesis, making this method less effective. Typically, alumina nanoparticles should exhibit a well-defined cubic structure (17).

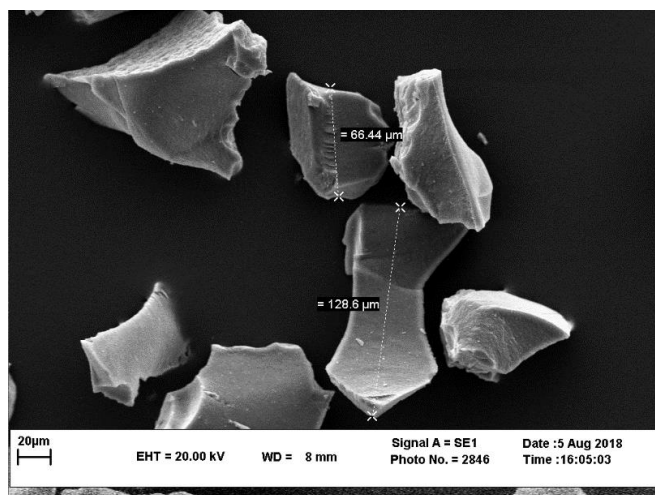


Figure 1: SEM image of alumina nanoparticles at 20× magnification.

Fourier-Transform Infrared Spectroscopy (FTIR)

FTIR spectroscopy not only provides insights into the chemical structure of a sample but is also widely used for identifying organic functional groups. Additionally, it can reflect the physical or mechanical presence of particles. In the FTIR study, specific absorption bands corresponding to key functional groups were identified for both the pure nanoparticles and nanoparticles encapsulated within carriers. The IR spectra shown in Figure (2) exhibit absorption peaks at 1633 cm^{-1} , attributed to carboxylic acid C=C stretching, and at 3449 cm^{-1} , corresponding to Al–O stretching (17). These findings indicate that alumina nanoparticles were not successfully formed.

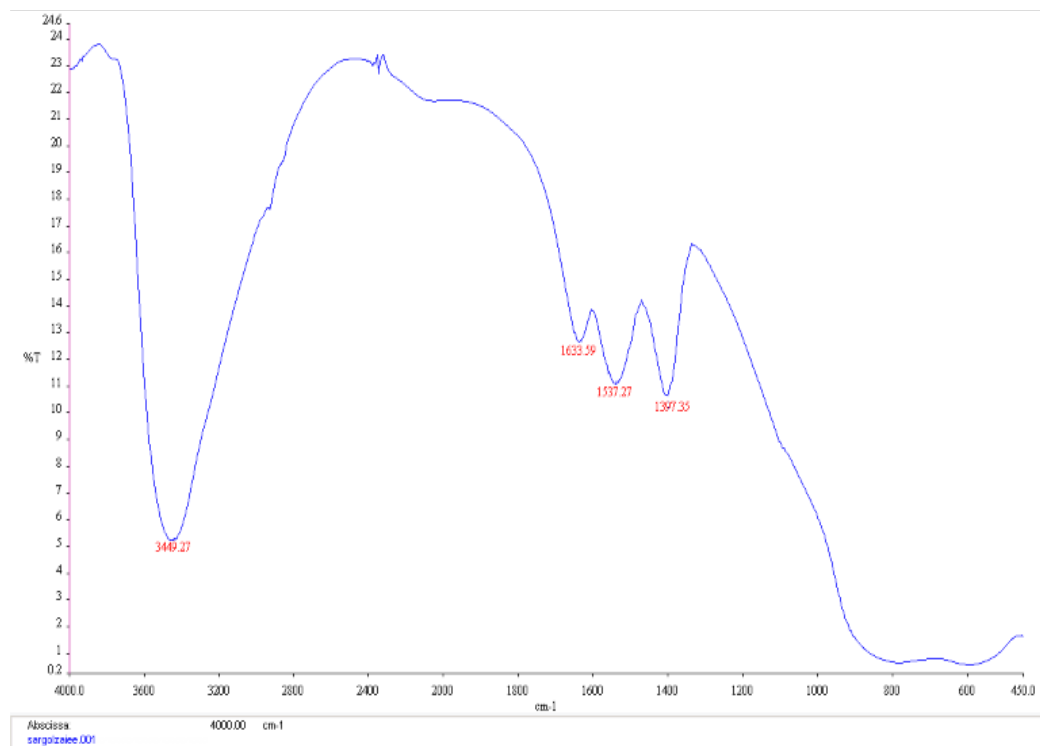


Figure 2: Fourier-Transform Infrared (FTIR) spectrum of alumina.

X-Ray Diffraction (XRD)

The results of the structural analysis of magnetic nanoparticles using X-ray diffraction are shown in Figure (3). The main diffraction peaks of alumina appear at 2θ values corresponding to the cubic crystal pattern based on JCPDS No. 2303-72. According to the XRD pattern, the particle sizes correspond well with those observed in the SEM analysis, indicating that this method is not suitable for synthesizing alumina nanoparticles.

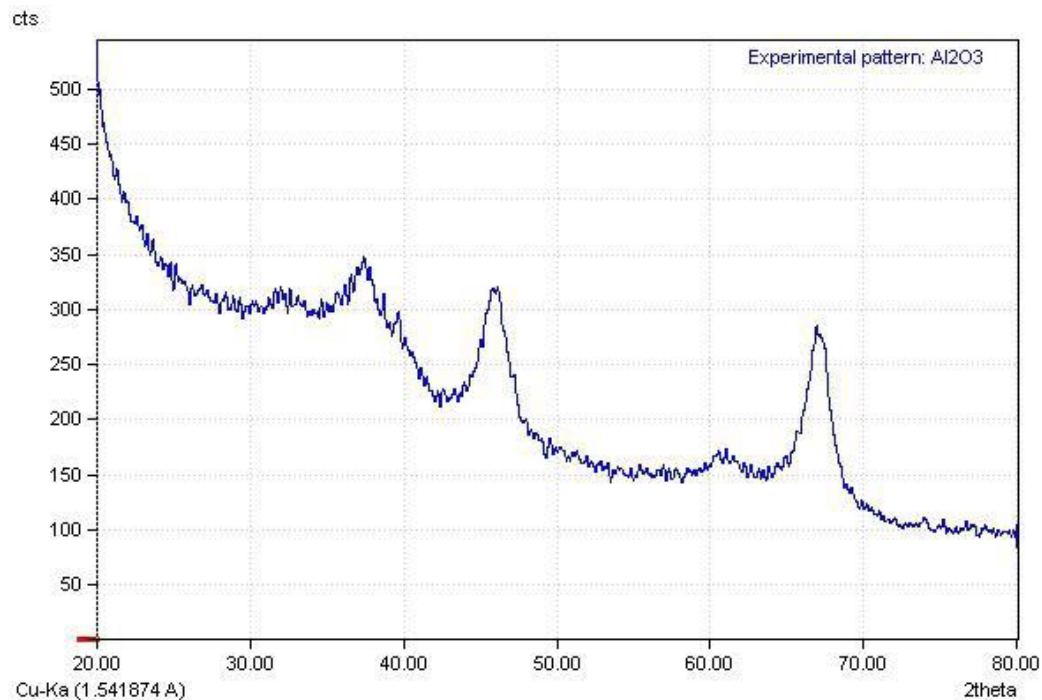


Figure 3: XRD analysis of alumina nanoparticles.

CONCLUSION

Due to the agglomeration of the nanoparticles, this method is not suitable for synthesizing alumina nanoparticles. According to the FTIR spectrum, the necessary bonds for alumina formation were not observed. The SEM images show that the particle size is very large, on the order of micrometers, and the particles are agglomerated. Considering the results from synthesizing other nanoparticles using this method and comparing them with alumina nanoparticles, it is recommended to synthesize alumina nanoparticles via the hydrothermal method and to study the effect of temperature on the nanoparticle size.

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