

Review

Properties and Preparation of Alumina Nanomaterials and Their Application in Catalysis

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Abstract

Nanomaterials are materials in which at least one dimension in three-dimensional space is at the nanoscale. In recent years, nano-alumina has attracted much attention due to its large specific surface area and pore volume, as well as novel optical, magnetic, electronic, and catalytic properties. This review summarizes the preparation methods of nano-alumina based on the basic phases and properties of alumina materials, focusing on one-dimensional, two-dimensional, and three-dimensional nano-alumina preparation methods, which can provide some theoretical guidance for the subsequent development of efficient nano-alumina materials. Finally, the application of nano-alumina materials in catalysis is reviewed, and some suggestions are provided for improving the use of nano-alumina in the catalysis field.

Keywords: nanomaterial; alumina; preparation; catalytic application



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1. Introduction

Nanomaterials have nanoscale dimensions in a certain dimension, and when the size of the particles is reduced to the nanoscale, the physical and chemical properties of a material change significantly. Due to their unique size, nanomaterials have made great progress in recent years in the fields of optical sensors, solar energy utilization and fuel cells, biological science, catalysis [1–3], smart materials, etc. Alumina is one of the most widely used materials, with excellent performance in catalysis, optics, biomedicine, ceramic materials, and adsorbents. Particularly in the catalytic field, alumina serves as an excellent support material, offering outstanding thermal and hydrothermal stability. Its variable pore sizes and diverse structural facets provide an advantageous microenvironment for active metal species modification and material functionalization. Nano-alumina can not only inherit these advantages but also exhibit a high specific surface area and abundant defect sites [4–8], enhancing its applications in the catalysis field.

Nano-alumina can be classified into one-dimensional (1D), two-dimensional (2D), and three-dimensional (3D) nanomaterials based on its morphological structure. Nano-alumina with different dimensions has its own characteristics in terms of structure, properties and preparation methods, and shows different advantages in applications. One-dimensional nano-alumina typically appears as nanowires, nanorods, or nanotubes with high aspect ratios [9–12]. These structures exhibit excellent strength and toughness and pronounced size effects due to their unique geometry. Two-dimensional nano-alumina includes nanosheets and thin films with large surface areas, ultrathin structures, and tunable surface properties. Unlike 1D structures, 2D nano-alumina provides a higher density of active sites and

showcases strong mechanical and electronic properties. Three-dimensional nano-alumina often exhibits network-like or hierarchical porous structures characterized by higher specific surface areas and more complex pore architectures. These structures facilitate efficient mass transport and offer stable support functionality, expanding their application scope for advanced catalysts, energy storage, and environmental materials (Figure 1).

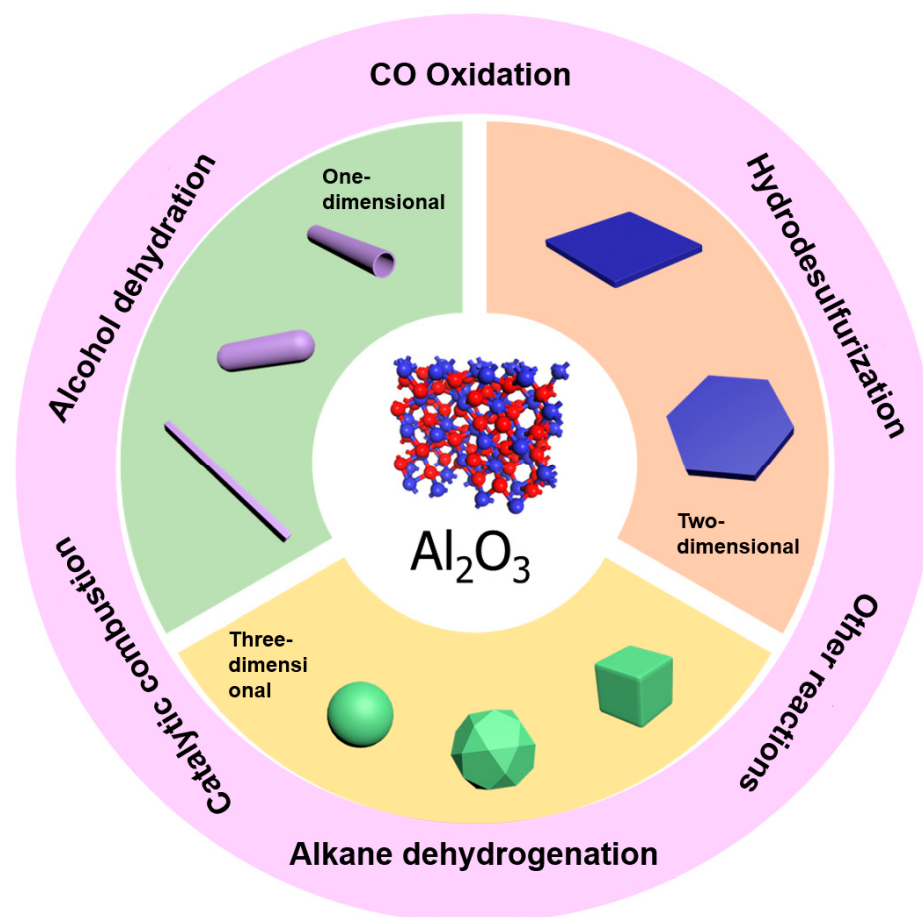


Figure 1. Nano-alumina and its applications in the catalytic field.

The synthetic approaches for nano-alumina and other nanostructured metal oxides broadly fall into two strategies: “top-down” and “bottom-up” [13–17]. The top-down strategy involves deriving nanomaterials from bulk materials through methods such as lithography, grinding, quenching, etching, or continuous cutting [18]. The bottom-up strategy builds nanostructures from atomic or molecular precursors via self-assembly [19], leveraging non-covalent interactions such as hydrophobic forces, π - π stacking, electrostatic interactions, and hydrogen bonding to form stable nanostructures [20]. The bottom-up strategy is often more cost-effective and reproducible than the top-down and is suited for large-scale production with precise control over material dimensions and shapes [21].

Nano-alumina’s unique properties, including high specific surface area, excellent thermal stability, and tunable acidity and basicity, make it a versatile material in fields like catalytic supports, refractory materials, and electronic components. This review provides a concise introduction to the fundamental properties of nano-alumina. Emphasis is placed on elaborating the commonly employed preparation methods for 1D, 2D, and 3D nano-alumina at present (Table 1), along with the impacts of different methods on the reaction. The characteristics of materials with different dimensional structures, as well as the challenges and technical highlights of their respective preparation techniques, are comprehensively outlined. It highlights the challenges and technical differences associated with

each preparation method and evaluates their potential for future applications. Furthermore, the review provides an in-depth discussion of nano-alumina's role in catalysis, particularly in enhancing catalytic performance and material stability. Suggestions for overcoming existing challenges, such as improving activity and selectivity through modification and compositing methods, are proposed. The findings aim to guide the development of efficient nano-alumina materials and lay a foundation for their broader application in catalysis.

Table 1. Summary of preparation methods.

Synthesis Method	Definition	Advantage	Disadvantage
Template method (hard/soft template)	Using template agents to guide the growth of aluminum precursors in a specific direction, resulting in the formation of nanostructures	Advanced technology, strong controllability of morphology, suitable for large-scale production	Difficult to remove the template, not reusable, and may cause the structure to collapse
Electrospinning method	Under a high-voltage electric field, the aluminum-containing solution is stretched into fibers and then calcined to obtain one-dimensional aluminum oxide	Uniform structure, continuous fiber length, capable of producing hollow structure	High equipment cost, high energy consumption, limited output, and low mechanical strength
Hydrothermal/solvothermal method	Under high temperature and high pressure in a sealed reaction vessel, the precursor is made to self-assemble into a special structure	No template required, adjustable crystal phase, good product dispersion	Strict conditions, low yield, difficulty in scale-up, and limited ability to control morphology
Precipitation-deposition method (including electro-deposition)	A precipitant is added to the metal salt solution and the conditions are adjusted to obtain the desired morphology	The process is simple, the cost is low, and it is suitable for industrial production	Poor morphology control, poor product stability, and low yield of the electro-deposition method
High-energy ball mill method	In the ball mill, the bulk materials are ground into nanoparticles through intense mechanical action	Simple process, high output, easy to scale up	High energy consumption, uneven particle size, prone to contamination, and difficult to control the structure
Sol-gel method	By forming a sol and then transforming it into a gel, and through drying and calcination, nano-alumina is obtained	Uniform composition, compatible with the template method	The synthesis process is lengthy, particles tend to agglomerate easily, and the calcination treatment is complex
Pyrolysis method	The aluminum salt precursor undergoes high-temperature decomposition to produce aluminum oxide	Low-cost and suitable for mass production	It is prone to generating toxic gases (such as SO ₂) and the equipment requirements are high
Chemical vapor synthesis method (CVS)	Nanometer-sized aluminum oxide particles are directly generated through chemical reactions in the gas phase	Narrow particle size distribution, not prone to agglomeration, and highly controllable	The equipment is complex, the raw material costs are high, and the yield is limited

Table 1. *Cont.*

Synthesis Method	Definition	Advantage	Disadvantage
Combustion method	Melting the metal in the burning flame and oxidizing it into aluminum oxide	Low energy consumption, short time, high product purity	It is difficult to precisely control the particle size, prone to aggregation, and has a relatively high safety risk
Wet chemical method	Liquid-phase precipitation reactions (such as aluminum salts + precipitating agent) can control the morphology through additives (nanoscale aluminum powder) or ultrasonic treatment	Low-temperature synthesis, with low energy consumption, high specific surface area and activity, great industrialization potential	Easy to stack/aggregate, poor process stability, difficult to fabricate ultra-large single wafers
Sodium nitrate solution method	Using low-melting-point salts (such as Na_2SO_4) as the medium, the reactants dissolve and precipitate at high temperatures, forming sheet-like Al_2O_3 (with a thickness of 400–720 nm)	High crystallinity, controllable morphology (hexagonal lamellae), good high-temperature stability	High energy consumption, difficult to control uniformity, dependence on additives
Carbon fractionation method	CO_2 /air was introduced into the aluminum salt solution, resulting in the formation of aluminum hydroxide precursors. After calcination, hexagonal plate-like Al_2O_3 (with a thickness of 150–200 nm) was obtained	Edge regularization, smooth surface, the morphology can be controlled by flow rate/pH	The process is complex, the gas flow rate is sensitive, the pH control is demanding
Microemulsion method	Using two immiscible solvents (such as an oil phase and a water phase) under the action of surfactants, a thermodynamically stable nanoscale microemulsion is formed. The water-phase core serves as a “nanoreactor”, and the aluminum precursor undergoes hydrolysis/reaction in it to generate nanoparticles	Uniform particle size, good dispersibility, low-temperature synthesis, and adjustable morphology	High cost, contamination by organic solvents, low yield, and great difficulty in achieving industrialized continuous production

2. Preparation and Applications of One-Dimensional Nano-Alumina

One-dimensional (1D) nanomaterials refer to nanostructures confined within two dimensions or quasi-one-dimensional systems, such as nanowires, nanorods, nanotubes, nanofibers, nanowhiskers, nanodendrites, etc. [22,23]. These materials are nanoscale in one dimension and exhibit anisotropic properties (Figure 2a–c) [24]. Their typical width ranges from 5 nm to 20 nm, while their lengths can vary from tens of nanometers to several micrometers. Among these, tubular structures (nanotubes) have garnered significant attention due to their ability to provide access to distinct regions, such as inner surfaces, outer surfaces, and the ends of the tubes [25]. One-dimensional nano-alumina features excellent properties, including high strength, a large specific surface area, wear resistance, high-temperature tolerance, and a high elastic modulus. These characteristics make it widely applicable in fields such as mechanics, electronics, metallurgy, ceramics, chemical engineering, and aerospace [24]. In catalysis, 1D nano-alumina, with its high specific surface area and remarkable stability, is commonly used as a catalyst support, playing a critical role in various catalytic processes.

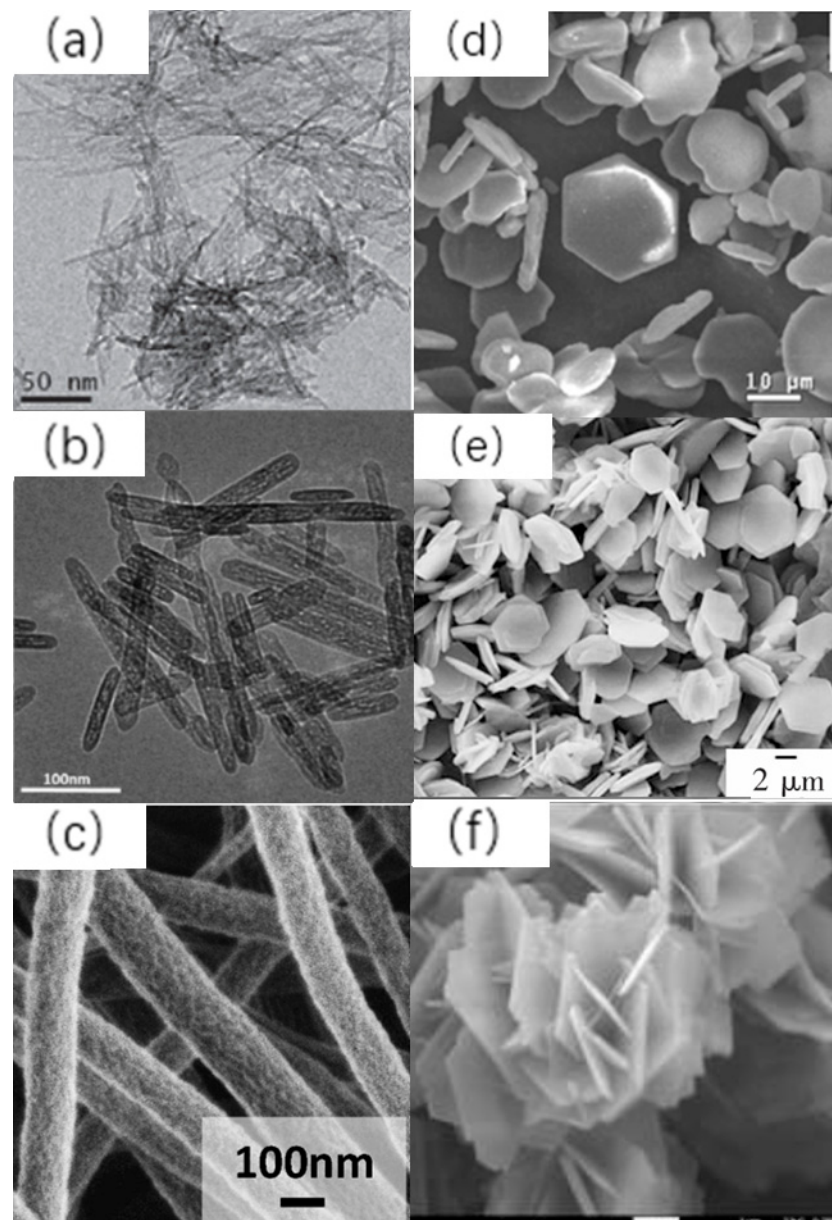


Figure 2. (a–c) One-dimensional nano-alumina; (d–f) two-dimensional nano-alumina [26–31].

2.1. Preparation Methods of One-Dimensional Nano-Alumina

Due to the limitations of existing “top-down” methods, such as high equipment costs, reliance on external control, long reaction times, high-temperature requirements, complex reactions, and the generation of by-products, along with the unclear reaction mechanisms involved in material synthesis [32], the preparation of one-dimensional (1D) nanomaterials primarily relies on the more cost-effective “bottom-up” methods. Common bottom-up methods for synthesizing 1D nano-alumina include the template method [33], the hydrothermal method [34–37], the liquid-phase method [6], the gas-phase method [26], and the electrospinning method [11,38,39] (Table 2).

Table 2. Common methods for the preparation of one-dimensional nano-alumina materials.

Synthesis Method	Precursor	Template	Synthesis Temperature (°C)	Length-to-Diameter Ratio	Specific Surface Area (m ² ·g ^{−1})	Morphology	Ref.
Template method	Al(NO ₃) ₃	F127	165	5–10	123	Nanorods	[27]
Template method	Al ₂ (SO ₄) ₃	P123	165	5–20	218	Nanorods	[27]
Electrospinning method	Al ₂ (SO ₄) ₃	PVP	Room temperature	0.6–2.1	217	Nanofiber	[28]
Hydrothermal method	Al(NO ₃) ₃	-	200	5	87	Nanorods	[34]
Hydrothermal method	Al(NO ₃) ₃	-	100	4–25	347	Nanorods	[35]
Template method	Boehmite sol	P123	100	-	88	Nanorods	[40]
Template method	Al(NO ₃) ₃	Glucose	Room temperature	-	429	Nanoparticles	[41]
Solvothermal method	C ₉ H ₂₁ AlO ₃	-	300	-	226	Nanoparticles	[42]
Solvothermal method	AlCl ₃	-	170	-	-	Nanoparticles	[43]
Electrodeposition method	C ₆ H ₉ AlO ₆	-	1200	10	-	Nanocrystals	[44]
Sol–gel method	C ₉ H ₂₁ AlO ₃	-	Room temperature	10–50	-	Nanorods	[45]
Chemical vapor-phase synthesis method	C ₁₂ H ₂₇ O ₃ Al	-	900	-	-	Nanoparticles	[46]

2.1.1. Template Method

The template method is a common approach for preparing one-dimensional (1D) alumina, primarily utilizing a template agent as a crystal plane modifier to guide the aluminum precursor to grow in a specific direction, forming a one-dimensional structure. Ghosh et al. [40] employed P123 as the template agent and boehmite sol as the aluminum precursor for the hydrothermal synthesis of 1D alumina nanorods and/or nanofibers at different temperatures (100–165 °C). After calcining at 400–1000 °C, the as-prepared 1D alumina materials were effective in adsorbing Congo red. Additionally, the authors explored the growth mechanism and found that nanofibers were gradually transformed into nanorods with increasing reaction temperature. Under solvothermal conditions, boehmite nanoparticles self-assembled in one direction, with the growth mechanism following the Ostwald ripening process. Nanofibers were formed at lower temperatures (100 °C), while intermediate structures between fibers and rods formed around 140 °C. With further increases in temperature (up to 165 °C), the self-assembly resulted in rod-like structures. Therefore, the authors concluded that the formation of 1D alumina nanostructures is highly temperature-dependent. Cai et al. [27] added ammonia solution to aluminum sulfate solution at room temperature, stirring vigorously to form a white precipitate, which was filtered, washed, and dried to obtain a gel. The gel was then mixed with P123 template agent and distilled water, followed by hydrothermal treatment, drying, and calcining, resulting in alumina nanorods. The authors examined the influence of different aluminum precursors (aluminum chloride and aluminum nitrate) and template agents (P123 and F127) on the samples. The results showed that when no template agent was added, the alumina sample (MA-S) formed a dense and irregular microstructure. When F127 and P123 were added, the samples (MA-S-F and MA-S-P) transformed into well-dispersed nanorods with a length of about 100–200 nm, a diameter of 10–20 nm, and an aspect ratio of approximately ten. The MA-S-F nanorods, with the addition of F127, had a slightly larger diameter and smaller aspect ratio compared to MA-S-P nanorods made with P123, showing that the larger molecular weight of F127 had a more significant effect on the morphology of the nanorods. This synthesis strategy offers mild reaction conditions, mature technological implementation, and significant potential for industrial scalability.

The template method utilizes intermolecular or intramolecular forces, such as hydrogen bonding, molecular interactions, and electrostatic forces, to form aggregates with specific structures during the reaction process. The reactants then use these aggregates as templates to generate particles with certain morphological and structural features. Feng

et al. [41] employed glucose as a soft template and aluminum nitrate as a precursor to synthesize mesoporous γ -Al₂O₃ particles with a surface area as high as 429 m²/g. The results indicated that the introduction of glucose significantly increased the surface area of the as-prepared γ -Al₂O₃. When the as-synthesized samples were used in an ethanol dehydration reaction, the experimental results showed a linear relationship between ethanol conversion rate and the surface area of the material. The higher the surface area, the higher the ethanol conversion rate. γ -Al₂O₃ is currently the only industrial catalyst used for ethanol dehydration to ethylene. The synthesis method using glucose as a soft template is cost-effective, environmentally friendly, and produces materials with a higher surface area, making it a promising approach for industrial catalyst preparation.

The template method demonstrates precise morphological controllability, wherein the diameter and aspect ratio of the resulting alumina nanofibers are determined by the pore structure parameters (e.g., aperture and length) of the templating material. Notably, the method enables structural diversification of alumina products, exhibiting tunable crystalline phases and hierarchical architectures through parameter modulation. However, there are also some problems. The template removal process exhibits significant technological complexity, and high-quality templates cannot be reused. The fiber length is determined by the template, making it difficult to synthesize ultra-long nanofibers. At the same time, removing the template may also cause the morphology of the alumina to lose support and collapse. While soft-template methods eliminate template removal requirements and demonstrate superior morphological controllability for ultrafine fiber fabrication compared to hard-template approaches, they suffer from inherent structural instability in alumina matrices. The synthesized architectures exhibit significant shrinkage and sintering-induced collapse during calcination processes, fundamentally limiting their practical application.

2.1.2. Electrospinning Method

Electrospinning is a specialized fiber manufacturing technique in which polymer solutions or melts are electrostatically spun under a strong electric field. Under the influence of the electric field, the droplets at the needle tip transform from a spherical to a conical shape (known as the “Taylor cone”), and fibers are drawn from the tip of the cone [47]. This method can produce polymer fibers with diameters at the nanoscale and can be used to prepare one-dimensional nanostructured alumina materials with uniform structures. Additionally, when template agents (such as PVP) are introduced, hollow one-dimensional alumina structures can be prepared. Under the action of an injection pump, a solution containing a certain proportion of aluminum precursor and PVP is spun at a fixed rate through the nozzle under a high-voltage electric field, forming fibers with diameters ranging from microns to nanometers, resulting in one-dimensional alumina materials. Rodriguez-Olguin et al. [28] used the electrospinning method through the electrostatic force generated by a high electric field to directly stretch polymer solutions or melts into nanometer-sized ultrafine fibers. The authors prepared a series of one-dimensional alumina materials with varying acidity and porosity with different aluminum precursors. Among them, the one-dimensional alumina prepared from aluminum acetylacetonate butoxide (ASB) exhibited the strongest acidity (0.70 mmol/m²), large pores (4 μ m), and a specific surface area of 180 m²/g, which is higher than that of some commercial γ -Al₂O₃.

The nano-alumina prepared by the electrospinning method has a high specific surface area. Moreover, continuous long fibers can be produced with controllable fiber diameters. However, due to the need for high-voltage electricity in the electrospinning method, the current material preparation cost is relatively high, and the mechanical strength of the prepared alumina structure is low.

2.1.3. Hydrothermal Method

The hydrothermal method is one of the most important techniques for preparing one-dimensional nanomaterials. In the absence of template agents, adjusting hydrothermal conditions can promote the self-assembly of aluminum precursors to form one-dimensional materials with high specific surface areas. For example, Dewangan et al. [34] used aluminum nitrate and urea as precursors to synthesize alumina nanorods with a specific surface area of $87 \text{ m}^2/\text{g}$ through a hydrothermal process at 200°C . The nanorods were further loaded with non-precious metal cobalt (Co) using an impregnation method. Even after cobalt impregnation and calcination at 700°C , their morphology was well-preserved, indicating that the alumina nanorod support had high thermal stability. The one-dimensional nanomaterials in this study had more five-coordinate Al^{3+} ions, which enhanced the metal-support interaction between Co and Al_2O_3 , significantly improving the catalytic performance of the Co-based catalyst. Similarly, Zhang et al. [35] used aluminum nitrate as a precursor, dissolved it in deionized water, and added urea as a precipitant. After hydrothermal treatment and subsequent calcination, they prepared uniform nanorods with lengths ranging from 2 to 5 μm and diameters of 0.2–0.5 μm . The product calcined at 300°C had a specific surface area of $773 \text{ m}^2/\text{g}$. As the calcination temperature increased, the surface area decreased, but the structure became more stable. Experimental results showed that the alumina nanorods, rich in hydroxyl groups, exhibited excellent Cr^{6+} removal capabilities, making them an excellent adsorbent for heavy metal ions in water.

The solvothermal method is a development of the hydrothermal method, where organic or non-aqueous solvents are used instead of water in a closed system, such as a high-pressure reactor, under specific temperature and self-generated pressure conditions [48]. Unlike hydrothermal reactions that use water as a solvent, solvothermal reactions utilize organic solvents. Meephoka et al. [42] used isopropyl-aluminum as a precursor and a mixture of n-butanol and toluene as a solvent to obtain alumina nanoparticles with sizes of 5–6 nm through the solvothermal method. The as-prepared samples had a high specific surface area of $226 \text{ m}^2/\text{g}$. The authors found that using different solvents, such as n-butanol and toluene, produced different phases, including γ -phase and χ -phase alumina. Cao et al. [43] prepared dispersible and thermally stable $\gamma\text{-Al}_2\text{O}_3$ with a nanoporous structure using the solvothermal method. They dissolved aluminum chloride and urea in ethanol, and the mixture was hydrothermally treated at 170°C for 5 h, followed by washing, drying, and calcining in a nitrogen atmosphere at various temperatures (500°C , 700°C , 800°C , and 900°C). The resultant nanoparticles were uniform in size (approximately 4–7 nm) with a narrow size distribution. This result indicated that under high-pressure, high-temperature solvothermal conditions, a complex structure will be formed from aluminum chloride, urea, and ethanol. During calcination, this complex decomposed, ultimately resulting in the formation of $\gamma\text{-Al}_2\text{O}_3$. This special structure of the complex may have two functions: it may prevent the growth of $\gamma\text{-Al}_2\text{O}_3$ grains during calcination, and it may allow the $\gamma\text{-Al}_2\text{O}_3$ grains to pack into a nanoporous structure after high-temperature calcination.

The hydrothermal method enables the preparation of nano-alumina with a complex morphology, the regulation of crystal phases, and the realization of crystallization at low temperatures. However, during the preparation process, the reaction conditions are demanding, the yield is low, and it is difficult to achieve large-scale application. Moreover, it may result in uneven fiber lengths. The use of this method to prepare nano-alumina still presents many challenges. The solvothermal method has the advantages of low energy consumption, minimal agglomeration, and controllable particle shapes. Moreover, the growth of crystals can be controlled by using different solvents. However, this method has problems such as invisible reactions, high equipment costs, and environmental risks.

2.1.4. Precipitation Deposition Method

The precipitation deposition method involves adding a precipitating agent to a metal salt solution and then heat-treating the precipitate to obtain the target product. By adjusting reaction parameters such as temperature and reactant concentration, materials with different shapes can be obtained. Wang et al. [49] used aluminum nitrate as the aluminum source, ammonia solution as the precipitating agent, and a small amount of PEG4000 as a surfactant. After calcining the mixture at 900 °C, they obtained γ -Al₂O₃, and at 1100 °C, α -Al₂O₃ particles with a size of less than 50 nm were obtained. The author found that PEG4000 formed a uniform and thick protective film on the surface of the colloid, effectively inhibiting the aggregation of colloidal particles [50].

In addition, electrodeposition is another deposition–precipitation method for preparing one-dimensional nanostructured aluminum oxide, based on electrochemical theory [51]. It is a film-forming technique that allows the creation of functionalized films on the surface of materials through electrochemical reactions. In the electrodeposition process, positive and negative ions from an electrolyte solution migrate to the electrode surface, leading to the deposition of the material. Qu et al. [44] used co-electrodeposition to prepare Al₂O₃ nanowhisker composite materials with an average diameter of 80 nm and an aspect ratio of about ten. They applied ultrasonic vibration to reduce the aggregation of alumina particles. As the ultrasonic frequency decreased, the number of Al₂O₃ whiskers in the composite coating increased, and at low frequencies, the whiskers were more uniformly distributed. At a frequency of 50 Hz, no whiskers were observed in the coating, and the Al₂O₃ particles transformed into a hemispherical structure. When the frequency was lowered to 10 Hz, a large number of Al₂O₃ whiskers were observed, and at this frequency, the whiskers were well-dispersed with only slight aggregation, while the Al₂O₃ particles remained hemispherical. Further decreasing the frequency to 5.6 Hz led to an increased amount of Al₂O₃ whiskers embedded in the coating, but the whiskers became more aggregated.

The precipitation deposition method is simple and cost-effective, and has a short process flow, making it suitable for the industrial production of nanopowders. However, the morphology of the synthesized alumina is easily affected by the pH value and other synthesis conditions, and the control of the morphology is poor. Electrodeposition is a simple, flexible, and controllable method that allows the deposition of low-concentration solutes onto an electrode, resulting in high-concentration deposits. At the same time, it can precisely control the morphology of alumina. However, its yield is relatively low. Moreover, the products are present in the solute and are difficult to collect. The synthesized alumina has an unstable structure and is prone to collapse during the calcination process.

2.1.5. High-Energy Ball Milling Method

High-energy ball milling involves mixing powders of different materials in specific proportions and subjecting them to intense impacts, compression, and stirring from milling balls to grind the powder into nanometer-sized particles. Al₂O₃ is commonly used in medical devices such as surgical implants and cochlear implants. However, under conventional conditions, alumina requires a high sintering temperature (around 1700 °C) to form dense ceramics, which can present challenges for the metal components of implants due to their high melting points. Ball milling can lower the sintering temperature while improving the sintering density, although the high hardness of alumina can lead to contamination from the milling media during preparation. Reid et al. [52] used a high-energy ball milling method to grind commercial alumina powder for 32 h in air with a ball-to-powder ratio of 10:1. The result was alumina nanoparticles with a grain size of less than 100 nm. The as-prepared nanocrystalline alumina exhibited a low sintering temperature and high sintering density. Li et al. [53] directly milled irregularly shaped, larger commercial alumina particles and

then etched them with hydrochloric acid to obtain high-purity, highly dispersed alumina nanoparticles with an average particle size of 6.2 nm. By using HCl as a flocculating agent for graded precipitation, the products were further separated into narrower particle size distributions. The γ - Al_2O_3 obtained from different concentrations of HCl had average particle sizes of 4.8, 6.6, and 7.7 nm. This method is simple, cost-effective, and capable of producing ultrafine particles, making it suitable for large-scale production. This method operates at relatively low temperatures and offers high yields, and no chemical reagents are needed during the synthesis process. However, it is prone to causing dust pollution. Moreover, the synthesized alumina nanofiber particles have uneven sizes and high energy consumption.

2.1.6. Sol–Gel Method

The sol–gel method is a common approach for preparing metal oxide nanoparticles. A sol (or solution) is a colloidal suspension of solid particles or clusters in a liquid, which can induce bonding between different molecules. A gel, resembling a solid state, indicates the extent to which colloidal particles aggregate to form a filterable, interconnected three-dimensional network [54]. Using isopropyl aluminum and a cationic surfactant (cetyltrimethylammonium bromide) as precursors, Keshavarz et al. [45] applied the sol–gel method to prepare γ - Al_2O_3 . They found that by adjusting the preparation parameters, they could obtain porous alumina particles in the form of nanorods with varying lengths (40–50 nm) and diameters (1–4 nm). Comparing these with the alumina material prepared without the surfactant, they observed that the addition of the surfactant reduced the size of the nanoparticles (from 8.1 nm to 4.3 nm) while simultaneously increasing the surface area and the number of acidic sites on the material. The products synthesized by this method have the characteristic of uniform composition and can be combined with the template method. However, the process has a long synthesis cycle, and the calcination process is prone to causing agglomeration.

2.1.7. Other Methods

In addition to common methods like the template method and electrospinning, there are other techniques such as pyrolysis and vapor-phase methods used for the preparation of one-dimensional Al_2O_3 nanomaterials. The following is a brief overview of these methods.

1. Pyrolysis Method

The pyrolysis method generally uses aluminum salts such as ammonium alum and ammonium carbonate as raw materials. These salts are reacted and purified to obtain aluminum oxide precursors, which are then heated and decomposed to produce nano-alumina [55]. During the pyrolysis of ammonium alum, toxic gases such as SO_2 are released, leading to environmental pollution. As a result, the ammonium carbonate pyrolysis method has gradually become the preferred method. In 2006, Marchal et al. [56] proposed a liquid-feed flame spray pyrolysis (LF-FSP) method for preparing nano-alumina. The authors mixed 1–10 wt% nano-talcum powder with aluminum oxide medium in ethanol and ground it for 24 h. After ultrasonic treatment on a titanium rod at 500 W, the powder was atomized using O_2 and combusted at temperatures close to 1600 °C. This process can convert nano-alumina into α - Al_2O_3 powder with particle sizes ranging from 30 to 80 nm. LF-FSP enables the conversion of various commercially available t- Al_2O_3 nanopowders into α - Al_2O_3 powders without causing aggregation.

2. Vapor-Phase Method

Chemical vapor synthesis (CVS) is a technique in which nanometer-sized powders are generated through chemical reactions in the gas phase. It is essentially an improvement on chemical vapor deposition (CVD), with the process adjusted to produce particles instead of

thin films. Nanopowders produced by this method typically do not aggregate and exhibit a narrow particle size distribution, and their phase composition can be varied by adjusting the process parameters. Lukić et al. [46] used aluminum tri-sec-butoxide and oxygen as raw materials, with helium gas as a support, under low-pressure conditions. An alumina tube was used as a reactor and heated in an electric furnace to 900 °C, where nano-alumina powders were directly synthesized in the gas phase. Characterization results showed that the nanometer-sized alumina powders produced under different helium flow rates and pressure conditions had similar microstructures, consisting of low-density nanoparticles with an average particle size of 6.8 nm.

3. Combustion Method

The combustion method involves the melting and oxidation of metal particles during the combustion process, leading to the formation of stable metal oxides, which then nucleate and aggregate. This method relies on the heat released by redox chemical reactions rather than using high-temperature furnaces, enabling the synthesis of high-purity single-phase or multi-phase composite oxide powders at lower processing temperatures and shorter reaction times. Kathirvel et al. [57] introduced commercial aluminum powder into a flame containing oxygen and acetylene gases. The aluminum powder melted and reacted with oxygen, producing alumina, which was collected by a powder collector. This process synthesized α -Al₂O₃ nanoparticles with grain sizes ranging from 70 nm to 150 nm. By adjusting the oxygen-to-acetylene ratio, materials with varying sizes and aggregation levels were synthesized. The resulting products exhibited high surface areas and abundant oxygen vacancies, making them suitable as catalyst supports in industries such as the automotive sector.

2.2. Applications of One-Dimensional Nano-Alumina in the Field of Catalysis

The high specific surface area, good thermal stability, and unique rich defect sites of one-dimensional nano-alumina make it an excellent catalyst or catalyst support (Table 3). For example, in automotive catalysts (such as the three-way catalytic converter, where the temperature can reach up to 1050 °C), the use of the phosphate-assisted dynamic hydrothermal method results in nanoscale alumina with better thermal stability. Additionally, by optimizing the synthesis (controlling the morphology and crystal planes) and modifying the alumina supports (using chemical promoters such as P, Si, and La) to enhance the catalyst's thermal stability [31], it enables the catalyst to continuously and stably catalyze the conversion of vehicle exhaust at high temperatures. Zhou et al. [58] used aluminum nitrate as the raw material, ethanol as the reaction medium, and ammonia as the precipitant to prepare ultrafine, sponge-like aluminum hydroxide. The aluminum hydroxide was then mixed with iron powder, compacted, and sintered to prepare alumina-enhanced iron-based composite materials. During the reaction, the aluminum hydroxide dehydrates and transforms into high-hardness α -Al₂O₃ particles at high temperatures, which are evenly distributed on the iron matrix. The resulting material exhibits high interfacial activity, with the particle size of nano-alumina significantly influencing the strength of the composite material. Smaller particle sizes result in larger distances between particles, leading to a more uniform distribution and, consequently, increased material strength. Li et al. [59] used zirconium oxychloride octahydrate and pseudo-boehmite as raw materials to prepare a series of ZrO₂-Al₂O₃ composite oxides with different ZrO₂ contents by chemical precipitation. These composite oxides were used as catalyst supports for the hydrodesulfurization (HDS) of dibenzothiophene (DBT). The resulting composite oxides consisted of ultrafine nanoparticles with a size of 10 nm. The introduction of Al₂O₃ made the metastable tetragonal ZrO₂ more stable at room temperature.

Table 3. Applications of one-dimensional nano-alumina in catalysis.

Serial Number	Catalyst	Role of Alumina	Morphology	Preparation Method	Catalytic Application Reaction	Ref.
1	Co/Al ₂ O ₃ -NF	Support	One-dimensional nanorods	Hydrothermal method	Dehydrogenation of non-oxidized propane	[36]
2	Al(NO ₃) ₃	Support	One-dimensional nanorods	Hydrothermal method	Anthraquinone hydrogenation reaction	[36]
3	Catalyst A	Support	One-dimensional nanorods	Hydrothermal method	Hydrogenation demetallization of residual oil	[37]
4	γ-Al ₂ O ₃	Active component	One-dimensional nanofibers	Soft template method	Ethylene is produced by dehydration of ethanol	[41]
5	Pt/Al ₂ O ₃	Support	One-dimensional nanoparticles	Solvothermal method	Oxidation of carbon monoxide	[42]
6	CoMo/ZrO ₂ -Al ₂ O ₃	Support	One-dimensional nanorods	Chemical precipitation method	Hydrodesulfurization of dibenzothiophene (DBT)	[59]

Dewangan et al. [34] synthesized γ-Al₂O₃ of different morphologies by the wet impregnation method, namely, nanosheet-type alumina (Al₂O₃-NS), nanofiber-type alumina (Al₂O₃-NF), and nanoplate-type alumina (Al₂O₃-NP). These were used as supports, and after loading cobalt-based materials, Co-based alumina catalysts were formed. These catalysts were then applied in the non-oxidative dehydrogenation of propane. The research found that the catalyst synthesized by urea and the hydrothermal method at different temperatures presented different micromorphologies, including nanosheet, nanofiber and nanoplate structures. Through TPR analysis, it can be seen that the three catalysts exhibit different reduction temperatures, which indicates that there are differences in the strength of the metal-support interaction. The experimental results showed that the alumina nanofibers had abundant unsaturated Al³⁺ sites, which could effectively anchor the active Co species, forming strong metal-support interactions. Furthermore, the Lewis acidic centers of the catalyst facilitated the efficient progression of the propane dehydrogenation reaction. Similar conclusions have also been reported in the PtSn system [60,61].

Xu et al. [38] used a hydrothermal method to synthesize porous alumina nanoparticles with nitric acid and urea as raw materials. Polyethylene glycol was added as a template to disperse the alumina, resulting in small particles with regular structures. During high-temperature calcination, polyethylene glycol undergoes pyrolysis, and the pore-forming effect leads to the formation of numerous porous structures in the alumina nanoparticles, giving them a high specific surface area. Finally, the porous alumina was used as a support to prepare Pd-based alumina catalysts. The high surface area of the support allowed for better dispersion of Pd species, increasing the number of active catalytic sites. This resulted in higher hydrogenation efficiency in the anthraquinone hydrogenation reaction.

Feng et al. [41] synthesized small-particle g-Al₂O₃ with a well-ordered textural structure using aluminum nitrate and sodium hydroxide as raw materials and glucose as a soft template agent through a precipitation–hydrothermal method. During the preparation, glucose served as both a soft template and structural directing agent, leading to the formation of mesoporous g-Al₂O₃ with a high specific surface area. The g-Al₂O₃ exhibited a high ethanol conversion rate and ethylene selectivity in the ethanol dehydration to ethylene reaction. The authors suggested that the specific surface area of the g-Al₂O₃

catalyst influenced its activity for ethanol dehydration, showing a positive correlation between the specific surface area and the ethanol conversion rate. Meephoka et al. [42] used toluene and n-butanol as raw materials and employed a solvothermal method to prepare nanostructured Al_2O_3 materials with different α -/ γ -phase compositions. These materials were used as supports, and platinum (Pt) was loaded by impregnation. The as-prepared catalysts were evaluated in a carbon monoxide oxidation reaction. The results indicated that the phase composition of the alumina support affected the dispersion of platinum on the alumina, and the addition of the α -phase to γ - Al_2O_3 enhanced the dispersion of platinum and improved the catalytic activity in reactions such as CO oxidation, which are less sensitive to structural changes.

In conclusion, the performance of the catalyst is closely related to its preparation method. Therefore, an appropriate synthesis strategy needs to be selected based on the specific requirements of the target reaction (such as thermal stability, density of active sites, controllability of morphology, etc.). At present, the hydrothermal method remains one of the main methods for preparing alumina-based catalysts. However, it has limitations, such as low yields and difficulty in controlling the morphology. In the future, with the in-depth study of one-dimensional nano-alumina, the limitations of a single method can be overcome through the collaborative optimization of multiple preparation methods. For instance, in the process of hydrothermal synthesis, by combining the soft template method, one can not only leverage the high-crystallinity and thermal-stability advantages of the hydrothermal method, but also precisely control the pore structure and morphology of alumina through the template agent, thereby preparing a catalyst support that possesses a high specific surface area, abundant defect sites, and excellent thermal stability. This multi-method collaborative strategy is expected to further enhance the activity and stability of the catalyst, meeting the requirements of demanding reaction conditions (such as high-temperature catalysis).

3. Preparation and Applications of Two-Dimensional Nano-Alumina

Two-dimensional nanomaterials, such as nanosheets, nanodisks, and nanolayers, are two-dimensional nanomaterials with nanometer thicknesses (Figure 2d–f) [62,63]. Alumina nanosheets have a flat, plate-like structure [64], exhibiting properties such as good adsorption, shielding effects, and strong light-reflection ability. Due to their unique structural characteristics, including smooth surfaces, uniform thicknesses, and large aspect ratios, alumina nanosheets can be widely used in functional ceramics, cosmetics, pearlescent pigments, rubber, plastics, and other products [65,66]. Alumina nanosheets can not only enhance the stiffness and hardness of products but also help adjust the thermal expansion coefficient and shrinkage [67]. Additionally, they can be applied to the surface of aircraft to achieve stealth capabilities.

3.1. Preparation Methods for Two-Dimensional Nanostructured Alumina

The main methods for preparing two-dimensional alumina currently include the sol–gel method [68,69], the molten salt method [70,71], the hydrothermal (solvothermal) method [72–74], the coating method [75,76], and mechanical methods [76,77], as shown in Table 4. These methods are discussed in detail below.

Table 4. Common preparation methods for two-dimensional nano-alumina materials.

Synthesis Method	Precursor	Synthesis Temperature (°C)	Specific Surface Area (m ² ·g ^{−1})	Morphology	Ref.
Spray thermal decomposition method	Al(C ₅ H ₇ O ₂) ₃	350	-	Nanoporous thin film	[26]
Citric acid gel–co-solvent method	Al ₂ (SO ₄) ₃	1150	-	Hexagonal nanosheets	[29]
Organic gel–molten salt method	KAl(SO ₄) ₂ ·12H ₂ O	1150	-	Nanosheet	[30]
Hydrothermal method	C ₁₂ H ₂₇ O ₃	170	80	Nanosheet	[31]
Sol–gel method	Al ₂ O ₃ ·H ₂ O	1100	20	Hexagonal nanosheets	[68]
Sol–gel method	Al ₂ O ₃ ·H ₂ O	1200	0.32	Hexagonal nanosheets	[78]
Hydrothermal method	Al(NO ₃) ₃	200	113	Hexagonal nanosheets	[79]
Wet chemical method	Al(NO ₃) ₃	1100	-	Nanosheet	[80]
Molten salt method	Al ₂ (SO ₄) ₃ ·18H ₂ O	1150	-	Hexagonal nanosheets	[81]
Carbon fraction method	Al(NO ₃) ₃	1200	-	Nanosheet	[82]
Combustion method	γ-AlOOH	1000	-	Hexagonal nanosheets	[83]
Combustion method	Al(NO ₃) ₃	300	-	Nanosheet	[84]
High-energy ball milling method	Al(NO ₃) ₃	Room temperature	-	Nanosheet	[85]
High-energy ball milling method	Al(OH) ₃	Room temperature	-	Hexagonal nanosheets	[86]
Chemical vapor-phase synthesis method	Al(C ₅ H ₇ O ₂) ₃	1150	-	Nanosheet	[87]
Sol–gel method	Al(NO ₃) ₃	500	-	Nanolayer	[88]
Atomic layer deposition method	C ₃ H ₉ Al	120	-	Nanosheet	[89]

3.1.1. Sol–Gel Method

The sol–gel method is a wet chemical synthesis technique that involves the hydrolysis and polymerization of inorganic or organic alkoxides in a solvent to form a sol. During the aging process, the particles aggregate to form a gel with a three-dimensional network structure. After drying and high-temperature calcination, the gel is converted into nanopowder. This method offers several advantages, such as simple equipment requirements, the high purity and uniformity of the powder, the ease of process control, and its low cost [90]. Chen et al. [91] used the sol–gel method to prepare sheet-like alumina with a thickness of 200–500 nm and a length-to-thickness ratio of around ten. The as-obtained alumina nanosheets had regular microstructures, smooth surfaces, and uniform particle sizes and thickness distributions. Zhou et al. [92] used monohydrate gibbsite as the precursor and controlled the pH of the sol with dilute nitric acid, adjusting the AlF₃ content in the gel.

After heat treatment at 1200 °C, they obtained hexagonal-shaped plate-like α -Al₂O₃ with a size of approximately 890 nm. The surface tension of solid surfaces is anisotropic. Generally, more tightly packed crystal planes have lower surface tension values. The (0001) crystal plane of α -Al₂O₃ has a smoother, more uniform surface with higher surface tension, which hinders crystal growth, while other crystal planes grow more rapidly, leading to the formation of plate-like α -Al₂O₃. Li et al. [93] used isopropyl aluminum as the main precursor and applied the sol–gel method to prepare sheet-like nano-alumina. They studied the effects of different water addition methods during the hydrolysis of aluminum alkoxide on the formation process and microstructure of nano-alumina powder. The results indicated that the method of adding water affected the crystallinity and morphology of the hydrolysis products, which in turn influenced the final morphology of the nano-alumina particles. When water was added by spraying, the hydrolysis product was a flocculent amorphous material, while spraying water resulted in needle-like boehmite AlOOH. The authors concluded that the water addition method influenced the supersaturation of the solution, which affected the formation of crystal nuclei and the precursor structure, ultimately affecting the structure of the alumina. The flocculent amorphous product resulted in spherical alumina powder after calcination, while the needle-like hydrolysis product formed sheet-like γ -Al₂O₃ with a length of approximately 30 nm after calcination. However, this method still has problems in the preparation of alumina. When using this method to prepare nano-alumina, the process is complex, and the resulting nano-alumina also has the disadvantage of being prone to agglomeration.

3.1.2. Hydrothermal Method

Hydrothermal synthesis can be carried out at temperatures and pressures below the solvent's critical point. Above this critical point, the difference between liquid and vapor disappears, which is called the supercritical state. Under supercritical conditions, nucleation during crystallization is rapid, hydrolysis occurs quickly, metal oxide dissolution is low, and the product morphology is uniform [93]. Therefore, hydrothermal preparation of oxide nanomaterials allows easy control over the purity, size, and shape of the crystals. Moreover, the hydrothermal method has the ability to generate metal oxide nanostructures with uniform nucleation, growth, and aging, influencing the size, morphology, and aggregation of the oxide nanostructures. Suchanek et al. [8] synthesized α -Al₂O₃ nanosheets with aspect ratios between 7 and 200, thicknesses ranging from 10 to 75 nm, and widths from 0.5 to 3 mm by hydrothermally treating boehmite powder (γ -AlOOH) in the presence of α -Al₂O₃ seed crystals and a morphological modifier (nanometer-sized colloidal silica dispersion). The mixture containing H₂SO₄, boehmite powder, α -Al₂O₃ seed crystals, and the morphological modifier was dissolved and hydrothermally treated at 430–450 °C. In the process, an α -Al₂O₃ phase could be formed at pressures below 15 MPa, while controlling pressure was necessary to avoid the formation of boehmite (α -AlOOH). The two-dimensional nano-alumina synthesized by the author possesses excellent mechanical strength, ultra-high porosity and outstanding thermal stability. Its excellent physical and chemical properties allow for the doping of metals, which can significantly enhance the dispersion of metals and improve the activity of the target catalytic reaction. At the same time, it can meet the requirements of continuous and stable catalytic reactions under extremely harsh reaction conditions. Through a phosphate-assisted hydrothermal method, Wu et al. [31] dissolved PEG 600, sodium nitrate, and sodium phosphate, and then added tetrapropylammonium hydroxide (TPAOH) solution and isopropylaluminum (AIP) into the mixture, followed by further hydrothermal treatment and calcination to synthesize Al₂O₃ nanosheets (see Figure 2f). They also studied the effects of various salts (such as LiCl, NaCl, KCl, CsCl, and Na₂SO₄) and phosphate sources (NaH₂PO₂,

Na_2HPO_4 , and $\text{C}_3\text{H}_9\text{O}_4\text{P}$) on the structure of the as-synthesized Al_2O_3 nanosheets. Al_2O_3 nanosheets with adjustable average thicknesses (approximately 240 nm, 130 nm, 115 nm, and 15 nm) were obtained. The authors hypothesized that the selective adsorption of anions during the synthesis determined the preferential growth and morphology of the inorganic crystals. The thickness of the Al_2O_3 nanosheets decreased with increasing phosphate content, although excessive phosphate led to the formation of irregular nanosheets. Based on this, the authors proposed a new intercalation–swelling–exfoliation pathway to explain the formation mechanism of ultrathin Al_2O_3 sheet structures, which involves the selective adsorption of phosphate ions to further regulate the morphology and thickness of the synthesized ultrathin Al_2O_3 nanosheets. Moreover, the presence of phosphate ions further enhanced the thermal stability of the Al_2O_3 nanosheets, likely due to the occupation or depletion of the highly reactive surface hydroxyl groups and active sites on Al_2O_3 . Zhang et al. [79] used aluminum nitrate and sodium hydroxide as raw materials to synthesize hexagonal–flat nanostructured alumina via the hydrothermal method, investigating the impact of sodium nitrate additives on the morphology and properties of the alumina. Experimental results showed that sodium nitrate did not alter the crystalline phase of the product but influenced the textural properties, such as the specific surface area, pore volume, and pore size of the nanostructured alumina, thus playing a role in regulating the growth of certain crystal faces. Inorganic additives like sodium nitrate are cost-effective, less toxic than organic substances, easy to wash during the cleaning process, and result in minimal environmental pollution, making such raw materials beneficial for improving the preparation process of nanostructured alumina. The two-dimensional nano-sized aluminum oxide prepared by the hydrothermal method has a larger specific surface area compared to the one-dimensional form. Moreover, by controlling the synthesis conditions, the morphology of the aluminum oxide can be finely adjusted to achieve the desired results. However, similarly, the issue of yield and the high temperature and pressure during the reaction also pose challenges for the large-scale application of this technology.

3.1.3. Wet Chemistry Method

Wet chemistry methods refer to material preparation techniques that involve chemical reactions in a liquid phase, such as chemical liquid-phase deposition (CBD), electrochemical deposition (electroplating), etc. Lu et al. [80] successfully prepared plate-like Al_2O_3 particles using a wet chemical method with nano-aluminum powder as an additive. The authors first added nano-sized aluminum powder to an aluminum nitrate solution and used ammonia as a precipitant to synthesize Al_2O_3 . The experimental results showed that Al_2O_3 nanosheets with an average diameter of about 80 nm and a length of about 300–500 nm were prepared. The authors speculated that during the preparation process, the nano-aluminum powder additives could form Al_2O_3 seed crystals at the grain boundaries and liquid phases, promoting anisotropic grain growth and forming plate-like Al_2O_3 grains. The additives also decreased the temperature for the formation of $\alpha\text{-Al}_2\text{O}_3$. Chen et al. [94] used an aluminum ammonium vanadate solution as the precipitation mother liquid and ammonium bicarbonate solution as the precipitant, employing ultrasonic radiation to wet-synthesize Al_2O_3 nanosheets with a thickness of approximately 200 nm. The authors found that, regardless of whether ultrasonic radiation was applied during the synthesis phase, the precursor particles would change from a spherical shape at the end of the reaction to a rod- or bar-like shape during aging. This transition corresponded to the change from precipitation to gelation. During the aging process, precursor particles could adhere to each other via molecular attractive forces (Van der Waals forces), forming connection necks. Subsequently, due to the difference in surface curvature between the connected particles, the necks were gradually filled by a dissolution–precipitation mechanism, and the two particles

merged into one, forming rod-like structures. As the aging time increased, the particles further grew and gradually coalesced into a plate-like structure, eventually forming a three-dimensional network structure. However, when ultrasonic radiation was applied during the initial reaction stage, the intense cavitation effect caused by the ultrasound disrupted the hydrogen bonds formed on the particle surface, causing the particles to re-disperse and preventing aggregation. This made it particularly difficult to form a gel network structure during the aging process.

In conclusion, the wet chemical method has unique advantages in the controllable preparation of two-dimensional alumina. It has high controllability of morphology, can be synthesized at low temperatures with low energy consumption, and the synthesized nano-alumina has a high specific surface area and activity. Compared with the hydrothermal method, it also has the potential for large-scale application. However, there are still some problems. The synthesized alumina is prone to agglomeration or stacking, it is difficult to prepare single-crystal two-dimensional alumina with an extremely large lateral area, and its process stability is poor. These are all areas that need to be improved in the future.

3.1.4. Molten Salt Method

The molten salt method typically uses one or more low-melting-point salts as the reaction medium, allowing the reactants to have a certain solubility in the molten salt, thus facilitating atomic-level reactions. The synthesized nano-alumina has the advantages of good high-temperature stability, high crystallinity, controllable morphology, and the ability to produce ultrathin nanosheets. However, the process needs to be carried out at high temperatures, which results in high energy consumption and higher requirements for equipment. Moreover, the uniformity of the two-dimensional morphology is difficult to control, which also restricts its development. Wang et al. [81] applied the molten salt method using aluminum sulfate octadecahydrate as the precursor and sodium sulfate as the molten salt. By dry-mixing the molten salt with γ - Al_2O_3 obtained from aluminum sulfate, they successfully synthesized dispersed two-dimensional plate-like alumina. After the decomposition of aluminum sulfate, the resulting γ - Al_2O_3 was well-dispersed and could dissolve thoroughly in the molten salt. After cooling, the plate-like alumina with a 563 nm thickness was obtained. The type and amount of molten salt, the reaction pH, the heating process, and the presence of different additives significantly affected the morphology of the two-dimensional plate-like alumina. In the study of Chen et al. [95], it was found that adding chloride salts to the molten salt helped obtain larger-sized two-dimensional plate-like alumina powders. The addition of Zn^{2+} during the preparation process led to well-formed, uniformly sized hexagonal plate products. Under the influence of molten salt, the surface energy required for the growth of the alumina (0001) crystal plane was reduced, causing an increase in the growth rate of the (0001) plane. Consequently, the driving force required to form the same area of the (0001) plane was smaller than that for the (1010) plane, which made the growth morphology of the alumina particles more likely to form regular hexagonal plate layers. By dissolving the aluminum sulfate octadecahydrate and mixed salt and then mixing with sodium carbonate solution, the reaction was allowed to proceed fully, followed by stirring to form a gel. The gel was dried, ground evenly, and calcined in a furnace. After calcination, the product was treated with hot water to dissolve residual salts, and the undissolved solids were separated by filtration. The product was then washed with water and dried to obtain two-dimensional plate-like alumina with a thickness of approximately 400 nm.

Chen et al. [30] employed an organic gel–molten salt method to prepare two-dimensional plate-like alumina. In their process, PEG600 served as the organic gel, while potassium sulfate and sodium chloride were used as fluxing agents. Due to the weak

spatial hindrance of PEG600, it allowed for the uniform mixing of the precursors without impairing the effectiveness of the fluxing agents. The authors observed that the as-resulted two-dimensional plate-like alumina had a well-defined morphology with a thickness of approximately 720 nm and a diameter-to-thickness ratio of about 21.4. They hypothesized that during the early stage of the reaction, when the concentration of alumina precursors was high, the crystal growth was governed by interface-controlled reactions. In the later stages, when the concentration decreased, crystal growth became dominated by screw dislocation mechanisms. Over time, the alumina plates evolved into a board-like structure with a consistent thickness of around 720 nm.

The nano-alumina synthesized by the molten salt method has the advantages of good high-temperature stability, high crystallinity, controllable morphology, and the ability to produce ultrathin nanosheets. However, the process needs to be carried out at high temperatures, which results in high energy consumption and higher requirements for equipment. Moreover, the uniformity of the two-dimensional morphology is difficult to control, which also restricts its development.

3.1.5. Pyrolysis

The pyrolysis method is simple and often used in industrial production. In addition to liquid-feed flame spray pyrolysis, spray pyrolysis technology is also a method for preparing nano-alumina, particularly nanofilms. Alumina films prepared using spray pyrolysis technology exhibit high quality, strong adhesion, and a pinhole-free structure. Chandrashekar et al. [96] used aluminum acetylacetonate as a precursor and dimethylformamide as a solvent to prepare Al_2O_3 - TiO_2 films on silicon substrates at 350 °C via spray pyrolysis. The results showed that the grain size of the Al_2O_3 - TiO_2 films was 11.9 nm. The nanostructured Al_2O_3 - TiO_2 films primarily consisted of the Al_2TiO_5 phase and a mixed Al_2TiO_5 phase, with a uniformly distributed nanoporous surface morphology.

3.1.6. Subsubsection

In addition to the common methods such as sol-gel and hydrothermal methods, other approaches like the carbon template method, the combustion method, and high-energy ball milling are also employed for the preparation of two-dimensional nano-alumina materials. A brief overview of these methods is provided below:

1. Carbonation Method

The carbonation method is an approach used to synthesize ultrafine, flaky Al_2O_3 powders. Typically, this method involves introducing a mixture of carbon dioxide and air into a solution containing sodium aluminate, sodium hydroxide, aluminum fluoride, and titanium tetrachloride. This process precipitates aluminum hydroxide precursors, which would yield smooth-surfaced, well-defined ultrafine hexagonal nano-flaky Al_2O_3 after high-temperature calcination. For instance, Wu et al. [82] used ammonium bicarbonate and aluminum nitrate as precursors and synthesized two-dimensional flaky α - Al_2O_3 with a diameter of approximately 700 nm with the addition of urea. This study found that the formation of the α - Al_2O_3 flakes was significantly influenced by the urea additive. Urea could help control the agglomeration of Al_2O_3 powders and play a critical role in directing the growth of the α - Al_2O_3 crystals, thereby promoting the formation of distinct crystallographic orientations. Similarly, Sun et al. [97] explored the effects of mixed gas flow rate, calcination temperature, and pH value on product morphology using the carbon deposition method. They employed sodium aluminate, ferric chloride, and aluminum fluoride as precursors, with carbon dioxide and air precipitating the precursor. This resulted in ultrafine, two-dimensional hexagonal Al_2O_3 flakes with a thickness of 150–200 nm, smooth surfaces, and well-defined edges. The study revealed that the flow rate of the mixed

gas directly impacted the precipitation rate, crystal phase, and microscopic morphology of aluminum hydroxide [98]. When the gas flow rate is too low, insufficient nucleation occurs, leading to excessive crystal growth. This results in a lower yield of flaky Al_2O_3 grains, with particles exhibiting rough edges and poor morphology. Conversely, at excessively high flow rates, the rapid precipitation inhibits proper crystallization, often yielding amorphous or very small microcrystalline structures. These products typically exhibit irregular shapes and a loose, porous structure. When the gas flow rate increases to 200 L/h, most of the grains exhibit a flaky morphology, with well-dispersed particles and smooth, regular edges. However, when the mixed gas flow rate exceeds 200 L/h, the number of flaky Al_2O_3 grains decreases, and amorphous agglomerated structures may start to appear. The morphology of the resulting Al_2O_3 particles varies depending on the final reaction pH. This is because different pH values lead to different dominant reactions in the solution system. When the pH exceeds 12, the primary reaction is the self-decomposition of NaAlO_2 , resulting in products predominantly composed of gibbsite structures. These structures, when well-crystallized, typically appear as hexagonal platelets or prismatic shapes. At a pH value around 11, the crystallization of the product in the solution is incomplete, with most of the product being a mixture of gibbsite and gibbsite with pseudoboehmite (a type of hydrated aluminum oxide, which appears as fibrous particles). When the pH is lower than 9, the resulting aluminum hydroxide is amorphous. These different types of aluminum hydroxide, formed under varying pH conditions, lead to significant differences in the morphology of the aluminum oxide after calcination.

2. Burning Method

The combustion method uses organic substances during preparation, which would release heat from their combustion to lower the final calcination temperature. At the same time, the gases produced during combustion can reduce the aggregation of the product, resulting in smaller particle sizes. This method yields products with small particles and a uniform composition, and the lower synthesis temperature reduces energy consumption. However, the method has some drawbacks, including small-batch processing and a high cost, due to the addition of organic materials. Hsiang et al. [99] used a mixture of boehmite and potassium sulfate and calcined it at 1000 °C to produce hexagonal $\alpha\text{-Al}_2\text{O}_3$ with a diameter of about 200 nm and a thickness of about 25 nm. They found that adding potassium sulfate as a diluent to the precursor material and calcining the mixture below its melting temperature helped separate $\alpha\text{-Al}_2\text{O}_3$ nanoparticles, preventing microcrystal growth and aggregation [83,100]. This led to the synthesis of nanoscale $\alpha\text{-Al}_2\text{O}_3$ microcrystals, which self-assembled into single-crystal hexagonal $\alpha\text{-Al}_2\text{O}_3$ nanosheets through a process of polycrystalline to single-crystal transformation. Potassium sulfate acted as a template to direct the attachment of nanoscale $\alpha\text{-Al}_2\text{O}_3$ microcrystals, forming hexagonal single-crystal $\alpha\text{-Al}_2\text{O}_3$ sheets, while also promoting the phase transition from $\gamma\text{-Al}_2\text{O}_3$ to $\alpha\text{-Al}_2\text{O}_3$. Bharthasaradhi et al. [84] mixed aluminum nitrate solution with glycine solution and heated it in a muffle furnace at 300 °C for 10–15 min. They then used the combustion method to prepare carbon-doped aluminum oxide thermoluminescent dosimetric materials. X-ray diffraction (XRD) showed that the synthesized material was nanocrystalline (with cell parameters $a = 4.75$ nm and $c = 12.99$ nm), and scanning electron microscopy (SEM) confirmed the sample's sheet-like morphology. Liu et al. [101] dissolved polyvinyl alcohol (PVA) in deionized water until it became a uniform gel. They then added aluminum nitrate and glycine to the PVA gel, transferred the mixture to a furnace, and heated it to obtain nanostructured aluminum oxide. The as-synthesized oxide was a two-dimensional nanosheet with a thickness ranging from 200 to 400 nm. XRD results showed that as the calcination temperature increased, the diffraction peaks became sharper, indicating improved crystallinity. Additionally, products calcined at 1273 K and 1473 K contained $\beta\text{-Al}_2\text{O}_3$.

Al_2O_3 and $\alpha\text{-Al}_2\text{O}_3$ phases, respectively, which offered a variety of Al^{3+} coordination structures, including four-fold, five-fold, and six-fold coordination. These different coordination forms of Al^{3+} species laid the foundation for their participation in catalytic reactions and anchoring active species.

3. High-Energy Ball Milling Method

High-energy ball milling involves mixing powdered materials in specific proportions and grinding them into nanoparticles through intense impact, compression, and stirring with milling balls. This method is efficient for reducing particle size and achieving nanoscale materials. Wu et al. [85] utilized aluminum nitrate and ammonia water as raw materials and employed a high-energy ball milling process with in situ seeding to prepare $\alpha\text{-Al}_2\text{O}_3$ agglomerates with a sheet-like morphology and an average particle size of less than 50 nm. The in situ introduction of crystal seeds during milling promoted controlled crystal growth and refinement, leading to the formation of nanoscale $\alpha\text{-Al}_2\text{O}_3$. The effectiveness of this method highlights its potential for synthesizing high-performance nanomaterials with precise control over size and shape.

Su et al. [86] employed a co-precipitated $\text{Al}(\text{OH})_3$ product as the precursor and potassium sulfate as a fluxing agent to synthesize nanoplatelet alumina. The materials were mixed via ball milling, enabling the formation of nanoscale platelet structures. During synthesis, the Al_2O_3 grains were sensitive to surface energy conditions, especially in the presence of a liquid phase, facilitating their anisotropic growth into platelets. The slow growth rate of the (0001) plane of $\alpha\text{-Al}_2\text{O}_3$ promoted the formation of top and bottom surfaces, yielding high-crystallinity, hexagonal platelet alumina with a thickness of 50–100 nm. This method provides a cost-effective route for synthesizing platelet alumina nanocrystals. To further refine the grain size of the alumina platelets, Su et al. [102] introduced $\alpha\text{-Al}_2\text{O}_3$ seeds during the ball milling process. This approach reduced the activation energy for nucleation, lowered the transformation temperature, and enhanced the nucleation rate of $\alpha\text{-Al}_2\text{O}_3$. As a result, the average diameter of the synthesized alumina nanoparticles decreased to 220 nm. However, this work noted that while the addition of seeds increased the nucleation sites and reduced grain size, the resulting products exhibited some twinning and stacking issues. These findings highlight the balance between enhanced nucleation and potential aggregation when optimizing the process for uniform, high-quality alumina nanoplatelets.

4. Microemulsion Method

The microemulsion method refers to a technique for preparing nano-alumina within a system composed of a water phase, an oil phase, and additives. The reaction liquid is introduced into the oil phase in the form of tiny droplets, which are uniformly dispersed with the assistance of surfactants. The water phase is surrounded by a microscale monolayer interface formed by the additives, eventually forming microemulsion particles, with sizes controllable at the nanoscale. These particles are then subjected to precipitation, washing, drying, and calcination to obtain nano-alumina powders. The microemulsion method has advantages such as a simple experimental setup, controllable particle size and shape, as well as the ability to reduce the average particle size of catalysts, thereby improving their dispersion and reduction efficiency [103]. However, the residual additives may mix with the nanoproducts, affecting the product purity. In 2008, Liu et al. [103] employed a microemulsion–hydrothermal method to prepare core–shell nano- $\text{TiO}_2/\text{Al}_2\text{O}_3$ particles. They created a microemulsion system by adding emulsifier OP-10, n-butanol, cyclohexane, and water. TiCl_4 solution was then introduced into this system to form a TiCl_4 microemulsion. Concentrated ammonia was slowly added to the microemulsion to obtain $\text{Ti}(\text{OH})_4$. Subsequently, AlCl_3 solution and ammonia were added dropwise to precipitate

Al^{3+} onto the $\text{Ti}(\text{OH})_4$ surface, forming a coating. After calcination and other treatments, core-shell nano- $\text{TiO}_2/\text{Al}_2\text{O}_3$ particles with an average grain size of 13.7 nm were obtained.

5. Chemical Vapor Synthesis

The chemical vapor synthesis (CVS) method involves the generation of nanopowders through chemical reactions in the gas phase. Dhonge et al. [87] utilized this technique to fabricate alumina coatings with a nanosheet structure. The authors dissolved aluminum acetylacetonate in ethanol at various concentrations as a precursor. After ultrasonic atomization, the precursor was fed coaxially into a flame, where it evaporated and decomposed at a temperature of approximately 1150 ± 15 °C, forming nanosheet-structured alumina deposits. Characterization results showed that the alumina prepared using chemical vapor deposition featured mesocrystalline nanosheet layers. The as-synthesized alumina had a polycrystalline nanosheet structure, with a sheet width of 60–200 nm and an aspect ratio ranging from 0.03 to 0.1. Moreover, the study revealed that the stacking morphology of the alumina nanosheets depended on the precursor concentration. High precursor concentrations led to aggregated hexagonal alumina sheets, while low concentrations resulted in isolated hexagonal alumina sheets.

3.2. Applications of Two-Dimensional Nano-Alumina in the Catalysis Field

Two-dimensional sheet-like alumina, with its unique planar structure and regular hexagonal morphology, exhibits notable characteristics such as an extremely low thickness and a high diameter-to-thickness ratio. Its thickness can reach the nanoscale, while its diameter extends to the microscale, offering dual benefits of both nanoscale and microscale properties [104]. Besides its inherent excellent properties, such as high melting point, good wear resistance, chemical corrosion resistance, and oxidation resistance [105–107], it also finds applications in catalysis (Table 5). Dewangan et al. [34] synthesized two-dimensional Al_2O_3 nanosheets as catalyst supports using aluminum nitrate and urea via the hydrothermal method. During the reaction, metallic cobalt tends to deposit carbon at active sites, leading to catalyst deactivation. The tetrahedral-coordinated Al^{3+} in the nanostructured Al_2O_3 acts as a strong Lewis acid center, enhancing the interaction between the metal component (Co) and the Al_2O_3 support. This strong metal-support interaction effectively suppresses the formation of metallic Co during the pre-reduction and reaction stages, thus improving catalyst stability. In addition, the nano-alumina synthesized by the hydrothermal method has a large specific surface area, which significantly improves the dispersion of the metal component (Co). Similarly, Dai et al. [108] used urea and aluminum nitrate to synthesize two-dimensional $\gamma\text{-Al}_2\text{O}_3$ nanosheets with a thickness of <6 nm via the hydrothermal method. These $\gamma\text{-Al}_2\text{O}_3$ nanosheets, employed as Co-based catalyst supports for propane dehydrogenation to propylene, contain a high proportion of octahedral Al^{3+} sites. Due to their high lattice energy, these octahedral Al^{3+} sites enhance the stability of Co-based catalysts, limit the isolated tetrahedral Co^{2+} sites, and promote propylene desorption. This suppresses the formation of coke and other by-products while releasing active Co sites, resulting in high specific catalytic activity.

The hydrothermal method is one of the main synthesis methods for nano-alumina. The nano-alumina synthesized by this method has the characteristics of a large specific surface area, a controllable crystal phase, and high thermal stability. After loading active metals, its large specific surface area, high thermal stability, and strong interaction between the metal supports significantly enhance the activity of the catalytic target reaction.

Table 5. Applications of 2D nano-alumina in catalysis.

Serial Number	Catalyst	Role of Alumina	Morphology	Preparation Method	Catalytic Application Reaction	Ref.
1	1%Pd/ANSx-T	Support	Two-dimensional nanosheet	Hydrothermal method	Catalytic combustion of propane	[31]
2	Co/Al ₂ O ₃ -NS	Support	Two-dimensional nanosheet	Hydrothermal method	Dehydrogenation of non-oxidized propane	[34]
3	catalyst E.1	Active component	Two-dimensional nanosheet	Electrospinning method	Hydrogenation of carbon dioxide	[39]
4	Co-Al ₂ O ₃ -HT	Support	Two-dimensional nanosheet	Hydrothermal method	Propane dehydrogenation to produce propylene	[108]
5	NiMg/Al	Support	Two-dimensional nanosheet	Hydrothermal method	Methanation of carbon dioxide	[109]
6	ALC-NH ₃	Active component	Two-dimensional nanosheet	Wet chemical method	Ethylene is produced by dehydration of ethanol	[110]
7	V ₂ O ₅ /Al-C	Support	Two-dimensional nanosheet	Sol–gel method	Ethylbenzene dehydrogenates to form styrene	[111]

Wu et al. [31] synthesized layered Al₂O₃ nanosheets with a regular leaf-like structure and high thermal stability using a phosphate-assisted kinetic hydrothermal method. The authors proposed a novel intercalation–swelling–exfoliation pathway to explain the formation of these sheet-like structures and further adjusted their morphology and thickness via phosphate-induced dissolution–growth. The presence of phosphates enhanced the thermal stability of the Al₂O₃ nanosheets. The as-synthesized two-dimensional γ -Al₂O₃ nanosheets, rich in pentacoordinated Al³⁺ centers, exhibit strong interactions with supported metals, effectively stabilizing noble metals. As a result, thermally stable γ -Al₂O₃ nanosheets with medium anchoring sites were used as palladium (Pd) supports for propane catalytic combustion. Experimental results showed that Pd/Al₂O₃ nanosheets demonstrated excellent catalytic activity for propane combustion, achieving complete conversion below 300 °C. Notably, the redispersion of Pd particles and the strong metal-support interaction (SMSI) at high temperatures endowed the Pd/Al₂O₃ nanosheets with exceptional high-temperature resistance, maintaining performance even at 1000 °C.

Yang et al. [109] developed a Ni-MgO/Al₂O₃ catalyst with a high specific surface area, large pore size, and high pore volume by in situ growth of NiMgAl-LDH precursors on macroporous alumina. This catalyst was applied in the CO₂ methanation reaction. The macroporous alumina provided excellent mass and heat transfer properties, reducing pressure drops during the reaction and mitigating the formation of hotspots. Hao et al. [110] used a chelate-assisted co-assembly method based on the chemical etching effect of ammonia to fabricate a hierarchical leaf-like alumina–carbon nanosheet catalyst. This catalyst enhanced the catalytic performance in ethanol dehydration reactions. The uniformly dispersed leaf-like nanosheet structure of the alumina-carbon catalyst offered a large specific surface area, exposing more active sites conducive to the ethanol dehydration process. The addition of ammonia played a critical role in forming the leaf-like nanosheet

structure and generating Brønsted acid active centers, significantly promoting the ethanol dehydration reaction.

4. Preparation and Applications of 3D Nano-Alumina

Three-dimensional nano-alumina is a nano-alumina material with a complex three-dimensional structure, usually presenting a network, hierarchically porous spherical [112–114] or flower-like [115,116] structure. It is characterized by a highly developed pore system and a large specific surface area, which can provide abundant active sites and efficient material transfer paths for reactions. This three-dimensional structure enables three-dimensional nano-alumina to exhibit excellent performance in a variety of applications. Due to its rich surface hydroxyl functional groups, multi-level pore structure distribution, and high specific surface area, three-dimensional nano-alumina has a wide range of applications in petrochemicals [117,118], pollutant adsorption [119], and machinery.

4.1. Methods for Preparing 3D Nano-Alumina

Similar to one-dimensional and two-dimensional alumina, the main methods for preparing three-dimensional alumina include the sol–gel method [120,121], the hydrothermal method [115,121–123], and the template method [124] (Table 6). The following sections will discuss these preparation methods in detail.

Table 6. Common methods for preparing three-dimensional nanostructured alumina materials.

Synthesis Method	Precursor	Synthesis Temperature (°C)	Specific Surface Area (m ² ·g ^{−1})	Morphology	Ref.
Hydrothermal method	Al(NO ₃) ₃	150	>130	Nano hollow sphere	[112]
Hydrothermal method	Al ₂ (SO ₄) ₃	150	-	Nano hollow sphere	[113]
Microemulsion method	C ₁₂ H ₂₇ O ₃ Al	Room temperature	-	Nano hollow sphere	[114]
Hydrothermal method	AlCl ₃	160	-	Nano-flower-like	[115]
Solvothermal method	AlCl ₃	200	166.8	Nano-flower-like	[116]
Sol–gel method	Na ₃ AlO ₃	100	268	Nano hollow sphere	[120]
Hydrothermal method	Al(NO ₃) ₃	100	283	Leaf cluster structure	[121]
Sol–gel method	C ₉ H ₂₁ AlO ₃	Room temperature	-	Nanosphere	[121]
Hydrothermal method	AlCl ₃	200	137.2	Nano-flower-like	[122]
Hydrothermal method	Al(NO ₃) ₃	100	528	Velvet–spherical cluster structure	[123]
Hydrothermal method	Al(NO ₃) ₃	100	447	π-bond-type cluster structure	[123]
Hydrothermal–co-precipitation method	Al(NO ₃) ₃	150	250	“Cross” shape	[125]
Hydrothermal method	Al ₂ (SO ₄) ₃	165	269	Nano hollow sphere	[126]
Hydrothermal method	KAl(SO ₄) ₂	180	-	Nano hollow sphere	[127]

4.1.1. Sol–Gel Method

The synthesis via the sol–gel method typically requires three steps: (i) hydrolysis and condensation of the precursor to form a sol, (ii) gelation (also known as sol–gel transformation), and (iii) drying or calcination [128,129]. During this process, gelation and drying steps would promote the formation of various phases, structures, and shapes of metal oxide nanostructures, such as dense powders, porous structures, nanoparticles, fibers, wires, rods, thin films, and coatings [130–138]. Zhang et al. [139] used isopropyl aluminum as the precursor and employed a two-step alcoholysis sol–gel method in an organic phase to synthesize spherical (≤ 10 nm) and fibrous ($100\text{ nm} \times 4\text{ nm}$) alumina nanoparticles. The role of functional organic molecules in controlling the morphology of inorganic oxide particles is achieved by coordinating with inorganic ions or adsorbing onto the surface of crystal nuclei, thus altering the crystal face energy and growth rates, resulting in changes in particle morphology. Through characterizations, the authors found that organic compounds with carboxyl/ester groups could effectively modify the microstructure of nano-alumina. In an organic phase, acetic acid (AcOH) adsorbs more easily on the crystal nuclei or around the isopropyl aluminum molecules than toluene and isopropanol, changing the growth pattern or hydrolysis mechanism of the nuclei. Therefore, a small amount of AcOH can effectively modify the particle morphology. In an aqueous solution, however, the solvation of water and the adsorption of AcOH compete, and water's hydration effect on inorganic ions and AcOH reduces the interaction force between AcOH and the crystal nuclei, thereby weakening its modification effect. However, if polyamide carboxylates (PAMAMs) are used, their rich negative surface charges effectively adsorb onto the positively charged hydrated alumina crystal nuclei. As a result, even a small amount of PAMAMs can achieve morphology control. Additionally, large organic molecules like PAMAM not only alter the crystallization process due to coordination or adsorption but also suppress particle aggregation due to steric hindrance.

4.1.2. Hydrothermal Method

The hydrothermal method is a material preparation method with water serving as the reaction medium, and high temperature and pressure are needed in a sealed reactor to promote the dissolution and recrystallization of poorly soluble or insoluble raw materials. The hydrothermal method includes hydrothermal synthesis, hydrothermal treatment, and hydrothermal reactions. Nanomaterials prepared using the hydrothermal method have many superior characteristics, such as diverse shapes, high crystallinity, a uniform distribution, and low aggregation [8,140–149]. The formation of crystal faces and the morphology of crystals are closely related to reaction conditions such as hydrothermal temperature and pressure. Lan et al. [125] used a hydrothermal co-precipitation method with aluminum nitrate as the precursor, urea as the precipitant, and hydrothermal treatment to prepare a highly crystalline boehmite precursor. After calcination, they obtained a three-dimensional cross-shaped $\gamma\text{-Al}_2\text{O}_3$ ultrafine powder with a high surface area ($250\text{ m}^2/\text{g}$), a large pore diameter (229 nm), and a high porosity (87.7%). This novel morphology of nanostructured alumina can be used for the design and manufacture of new nano-functional materials. Liu et al. [115] used AlCl_3 as the precursor, cetyltrimethylammonium bromide (CTAB) as the surfactant, and an ethanol/water mixture as the solvent to synthesize three-dimensional nano-flower-shaped $\gamma\text{-AlOOH}$ through hydrothermal treatment at $160\text{ }^\circ\text{C}$ for 12 h. After calcination at $550\text{ }^\circ\text{C}$ for 2 h, they obtained Al_2O_3 while retaining the three-dimensional microstructure. The authors speculated that the formation of the nanoleaf-shaped alumina was the result of in situ assembly during crystallization. During the preparation process, $\gamma\text{-AlOOH}$ flakes with a thickness between 60 and 90 nm were first formed. Since the system contained CTAB, hydrophobic CTA^+ chains adsorbed on the

surface of the flakes, preventing their stacking. Eventually, the flakes interspersed and grew together, forming a three-dimensional nano-flower structure. Although this synthesis method is advantageous for forming three-dimensional microstructures, the use of organic solvents increases production costs and also raises the safety risks associated with the production process.

4.1.3. Template Method

The template method is a synthesis method that uses a template to control the morphology, structure, and particle size of the product. Depending on the type of template structure, the template method can be divided into hard template and soft template methods [93].

In the hard template method, the precursor is uniformly dispersed in the pores of the hard template or adsorbed on its surface to form a complex structure. The template is then removed by appropriate methods, such as dissolution, sintering, or etching, to obtain the target product. The unique structure of the hard template can restrict the crystallization or polymerization of the precursor, and the final product's shape will complement the template. Zhang et al. [150] prepared aluminum–carbon composite spherical shells using a one-step method (glucose-catalyzed polymerization followed by in situ ion adsorption) and a two-step method (glucose polymerization into spheres followed by ion adsorption) to make hollow alumina spheres by calcining to remove carbon and water. The one-step method is simpler but results in larger particle sizes and looser structures, whereas the two-step method produces smaller, more compact, and uniformly sized hollow spheres (Figure 3). This hollow structure is lightweight, high-strength, has a large specific surface area, and is resistant to heat and corrosion, making it widely used in drug release, radar stealth materials, and micro-reactors, among other applications.

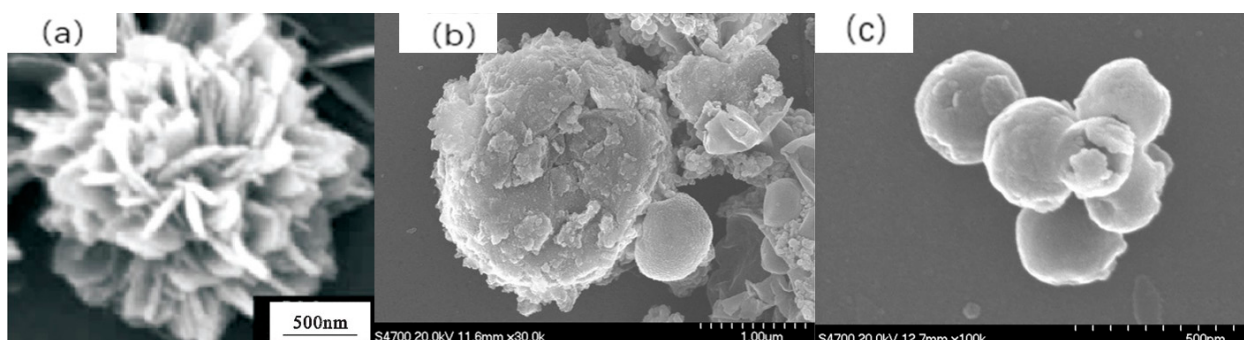


Figure 3. (a) Three-dimensional “cross-shaped” γ -AlOOH precursor, (b) morphology of hollow spheres prepared by the one-step method, and (c) morphology of hollow spheres prepared by the two-step method [125,150].

4.2. Three-Dimensional Nanostructured Alumina in Catalysis

Three-dimensional nanostructured alumina has a nanometer-scale structure in all three dimensions, which provides a higher specific surface area and superior physicochemical properties. It is characterized by a high specific surface area, excellent conductivity, and chemical reactivity, and is widely applied in fields such as catalysis, electrode materials, and sensors (Table 7). Zhao et al. [151] investigated the catalytic effect of nanostructured Al_2O_3 on heavy oil pyrolysis. Using techniques such as SEM, EDS, and TG-MS-DSC, they analyzed the impact of adding spherical nanostructured Al_2O_3 particles (30 nm in diameter) on the combustion behavior, activation energy changes of combustion products, and product performance in both pre- and post-combustion chambers and combustion tubes. The results showed that nanostructured Al_2O_3 particles reduced the activation energy of cracking and high-temperature oxidation (HTO) processes, while they increased the

peak temperature in low-temperature oxidation and high-temperature oxidation processes. Furthermore, the authors found that nanostructured Al_2O_3 reduced fuel consumption, thus decreasing carbon monoxide and carbon dioxide formation and oxygen consumption during the regeneration and combustion tube experiments.

Martínez et al. [125] were the first to use a surface templating method to synthesize large-pore mesoporous nanofiber $\gamma\text{-Al}_2\text{O}_3$ ($\text{Al}_2\text{O}_3\text{-nf}$), which formed a porous network structure. This porous alumina with a hierarchical structure was used as a support to prepare a series of Ru- and Co-loaded catalysts, which were then evaluated for Fischer–Tropsch synthesis (FTS) in a fixed-bed reactor. The synthesized nanostructured alumina porous network exhibited the highest specific surface area ($321\text{ m}^2/\text{g}$) and the largest macropores, which improved the dispersion of Co and thus enhanced FTS activity. The large macropores of this $\gamma\text{-Al}_2\text{O}_3$ also improved the selectivity. Kim et al. [152] prepared aluminum oxide nanospheres smaller than 50 nm and carbon-based composite catalysts ($\text{Al}_2\text{O}_3\text{-S}$) by reacting aluminum nitrate with a polyether surfactant. As the surfactant concentration increased, the carbon content, the specific surface area, and the concentration of acidic/basic sites in the resulting $\text{Al}_2\text{O}_3\text{-S}$ catalysts also increased. The results showed that the sample with the highest carbon content exhibited the highest acidity/basicity and the highest specific surface area ($97.6\text{ m}^2/\text{g}$), as well as the best catalytic activity for the hydrogenation of furfural to furfuryl alcohol. The presence of acidic centers prevented the aldol condensation of furfural and isopropanol, thereby enhancing the selectivity.

Furthermore, as the requirements for the catalytic performance of catalysts continue to increase, in the field of propane dehydrogenation, the preparation of catalysts with high activity and high stability and which are capable of meeting industrial conditions has become a key challenge in current research. Zhou et al. [153] used ML-100(V) as the precursor, and through the gum arabic-assisted sol–gel method and two-step calcination with N_2 and air, a spherical vanadium-based catalyst with high stability was successfully synthesized. The study found that the thermal decomposition and carbonization of VMOF and the subsequent decarbonization generated micron-sized macropores, enabling the formation of a layered porous structure on the spherical nano-alumina layer. The generated spherical nano-alumina significantly dispersed the V species, and its layered porous structure significantly enhanced the dehydrogenation activity. Dong et al. [154] synthesized alumina microspheres with different pore diameters using the ammonium alginate-assisted sol–gel method and the emulsion templating method. Subsequent studies have shown that the PtSn-La/ Al_2O_3 catalyst with large pore diameters can accelerate the diffusion of gas molecules within the channels, significantly enhance the catalytic performance of the catalyst, and also feature smaller Pt particles and lower surface acidity.

Umegaki et al. [118] used surfactant micelles to control the particle size distribution within the shells of hollow spheres and prepared copper-loaded porous silica–alumina hollow-sphere catalysts, which were then applied to the decomposition of dinitrogen monoxide (N_2O). The results showed that the catalytic activity was influenced not only by the dispersion of the active copper species but also by the size of the interparticle space within the hollow-sphere shell. The hollow spheres prepared with a high water-to-alcohol ratio exhibited highly dispersed copper species on their surfaces and showed high decomposition activity. Additionally, catalysts with small-sized particles within the hollow sphere shell also demonstrated relatively high catalytic activity. Ahmed et al. [155] used reverse microemulsion to prepare aluminum oxide hollow spheres (HSs) and used them as supports for loading nickel (Ni), which was applied to methane decomposition for hydrogen production. The prepared aluminum oxide hollow spheres had an average diameter of 15 nm, with a uniform shell thickness and no nanoparticles inside or outside the spheres. The results showed that NiO was uniformly adsorbed on the Al_2O_3 (HS) support.

The small size of the Al_2O_3 nanoparticles allowed for the prevention of strong interactions between the active component (NiO) and the Al_2O_3 support during calcination, making the active component more easily reducible and enhancing the catalytic decomposition activity.

Table 7. Applications of three-dimensional nanostructured alumina in catalysis.

Serial Number	Catalyst	Role of Alumina	Morphology	Preparation Method	Catalytic Application Reaction	Ref.
1	$\text{SiO}_2\text{-Al}_2\text{O}_3/\text{Cu}$	Active component	Three-dimensional nanospheres	Wet chemical method	Oxidation decomposition of N_2O	[118]
2	$\text{RuCo}/\gamma\text{-Al}_2\text{O}_3$	Support	Three-dimensional nanocluster	Template method	Fischer–Tropsch	[125]
3	$\text{Al}_2\text{O}_3\text{-S}$	Active component	Three-dimensional nanospheres	Wet chemical method	Glycolaldehyde is converted into sugar alcohol	[152]
4	$\text{Ni}/\text{Al}_2\text{O}_3$	Support	Three-dimensional nanospheres	Microemulsion method	Hydrogen production through the decomposition of methane	[155]
5	$\text{Al}(\text{NO}_3)_3$	Support	Three-dimensional nanocluster	Hydrothermal method	Catalytic reduction of NO	[156]
6	$\text{KW}/\text{Al}_2\text{O}_3$	Support	Three-dimensional nanospheres	Hydrothermal method	Catalysis of the production of methyl mercaptan from thiothiol	[157]

5. Conclusions and Prospectives

In summary, as an inorganic nanomaterial with multiple applications, nano-alumina's core value stems from its unique intrinsic properties: high melting point, hardness, corrosion resistance, thermal stability, oxidation resistance, high resistivity, and excellent thermal conductivity. The nanoscale effect endows it with a high specific surface area, a large pore volume, a tunable electronic structure, and enhanced catalytic activity, laying a broad application foundation. It should be emphasized that the physical and chemical properties of the material (including surface reactivity, mechanical strength, and catalytic efficiency) are influenced by the microscopic morphology (1D/2D/3D), and the controllability of the morphology depends on the precise regulation of the preparation parameters—by optimizing key variables such as reaction temperature/time, precursor chemical composition, structure-directing agents, and crystal-plane modifiers, the directional design of the material structure and properties can be achieved.

This review summarizes the preparation methods for nano-alumina with different morphologies and introduces their unique properties. It also provides an overview of the catalytic applications of these types of nano-alumina materials. One-dimensional nano-alumina, with its high specific surface area and unique structures such as nanowires and nanotubes, exhibits excellent physical and chemical properties, such as high thermal stability, good mechanical strength, and corrosion resistance. Therefore, in catalysis, one-dimensional nano-alumina can offer more active sites or anchoring points for active components, improving catalytic efficiency. Additionally, the special morphology of one-dimensional nano-alumina facilitates the adsorption of reactants and desorption of

products, further promoting catalytic reactions. Two-dimensional alumina, with its smooth, flat, and uniform thickness, as well as large aspect ratio, possesses characteristics such as high adsorption, high specific surface area, and exposure of specific crystal planes. It can be used as an efficient catalyst or catalyst support in the field of catalysis. Due to the tunable surface structure, particularly the exposed crystal planes, two-dimensional alumina can alter the catalytic activity and product selectivity in certain catalytic reactions. Three-dimensional nano-alumina, composed of nano-sized particles or nanosheets, possesses unique characteristics such as small-size effects, surface/interface effects, and quantum size effects. In catalysis, three-dimensional nano-alumina, with its high specific surface area and rich pore structure, provides a large number of active catalytic sites. This unique structure favors the adsorption and conversion of reactants. Additionally, its excellent thermal and chemical stability allow it to maintain catalytic activity under harsh catalytic conditions. Based on these structural features, nano-alumina materials are widely applied as catalysts and catalyst supports in various chemical reactions. However, compared to the preparation of conventional alumina powders, one-dimensional, two-dimensional, and other multidimensional nanomaterials have higher requirements for raw materials and preparation processes. For example, the template method used for one-dimensional nano-alumina requires the use of template agents, but the residual template agents are often difficult to remove completely, which can reduce catalytic performance. Similarly, electrospinning methods require a high voltage and incur higher costs, while vapor-phase methods often involve high temperatures and harsh preparation conditions.

Nanometer-sized aluminum oxide, due to its inherent characteristics, such as high thermal stability, excellent mechanical strength, and abundant adjustable active sites, has become a highly promising high-performance material in the field of catalysis. It can significantly enhance reaction efficiency and the lifespan of catalysts. The technical bottlenecks in the current preparation process (such as residual contamination from template agents, cost limitations of high-pressure electrospinning, and energy consumption issues in gas-phase synthesis) and the challenges related to the sustainability of large-scale production still need to be overcome. With the development of green synthesis processes, the advancement of in situ characterization techniques, and the application of precise control methods for reactions, these constraints will gradually be reduced. In the future, by establishing a quantitative structure–activity relationship between preparation parameters, microstructure, and catalytic performance, the synthesis process of nano-alumina will be precisely designed, promoting its large-scale application in energy catalysis, environmental remediation, and the synthesis of high-end chemicals, ultimately enabling the technological iteration and upgrading of related industries.

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