

Effect of Aluminum Precursors and Precipitating Agents on the Thermal, Morphological, and Textural Properties of γ - Al_2O_3 Synthesized by Homogeneous Precipitation

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Abstract— The effects of aluminum precursor salts and homogeneous precipitating agents on the thermal, morphological, and textural properties of γ - Al_2O_3 synthesized by homogeneous precipitation were investigated. The precursor salts used were aluminum sulfate, nitrate, and chloride, and the precipitating agents were ammonium bisulfite and urea. Thermal analysis revealed that materials derived from ammonium bisulfite were more thermally stable, with lower weight losses (28–37%) and higher DTG peak temperatures (280–350 °C), than those derived from urea, which exhibited weight losses of 40–46% and DTG maxima between 220 and 260 °C. XRD confirmed that all precursors were completely transformed into γ - Al_2O_3 after calcination at 450 °C. SEM observations showed that materials synthesized with sulfate and ammonium bisulfite developed highly open fibrillar aggregates, whereas materials synthesized with nitrate exhibited compact, platelet-like morphologies. Nitrogen adsorption-desorption analyses revealed Type IV isotherms, which are characteristic of mesoporous aluminas. The highest BET surface area and pore volume were obtained for sulfate-derived γ - Al_2O_3 synthesized using ammonium bisulfite: 332 m² g⁻¹ and 0.64 cm³ g⁻¹, respectively. These results demonstrate that thermal stability, morphological retention and pore development of the final product are governed by the choice of precursor chemistry and precipitation route. The aluminum sulfate–ammonium bisulfite system is the most effective for producing high-surface-area, mesoporous γ - Al_2O_3 .

Keywords—Ammonium bisulfite; urea; homogeneous precipitation; aluminum sulfate; aluminum nitrate; aluminum chloride; pseudoboehmite; γ - Al_2O_3 .

I. INTRODUCTION

Pseudoboehmite (γ - AlOOH) is one of the most important precursors for the production of transition aluminas, particularly γ - Al_2O_3 , owing to its high specific surface area, controlled pore structure, thermal stability, and excellent peptization behavior.

These characteristics have made pseudoboehmite an indispensable material in heterogeneous catalysis, catalyst supports, adsorbents, advanced ceramics, environmental remediation, and petrochemical processes. The structural properties of pseudoboehmite strongly influence the textural characteristics of the resulting transition aluminas after thermal treatment; therefore, controlling its synthesis remains an important research topic in materials science and chemical engineering. Recent developments have focused on tailoring crystallinity, morphology, pore structure, and thermal stability through precise control of synthesis parameters and precursor composition [1–3].

Numerous synthesis routes have been developed for the preparation of pseudoboehmite, including sol-gel processing, hydrothermal synthesis, precipitation from aluminum salt solutions, alkoxide hydrolysis, rehydration of transition alumina, and neutralization methods. Among these approaches, precipitation techniques remain particularly attractive because they allow control over particle size, morphology, pore development, and crystallinity while maintaining relatively simple experimental procedures and low production costs. Furthermore, precipitation routes are readily scalable for industrial production, making them suitable for manufacturing catalyst-grade materials. Recent studies have demonstrated that subtle modifications in precipitation conditions significantly affect the structural evolution of pseudoboehmite and its subsequent transformation into γ - Al_2O_3 [4–6].

The selection of the aluminum precursor constitutes a critical parameter during pseudoboehmite synthesis. Aluminum sulfate, aluminum nitrate, and aluminum chloride are among the most commonly employed precursor salts because they provide different solution chemistries and hydrolysis behaviors.

The precursor anion influences aluminum speciation, nucleation kinetics, particle growth mechanisms, and aggregation phenomena during precipitation. Consequently, variations in precursor chemistry can lead to substantial differences in crystallinity, morphology, water content, thermal stability, pore structure, and surface area. Recent investigations have reported that chloride-, nitrate-, and sulfate-derived aluminum species exhibit distinct condensation pathways, producing pseudoboehmite with markedly different textural properties [2,7–10]. Because the physicochemical characteristics of pseudoboehmite are largely established during nucleation and growth, variations in precursor anions may also affect the subsequent thermal evolution, morphological organization, and pore development of the γ - Al_2O_3 obtained after calcination. Therefore, understanding the influence of precursor selection is essential for rationally tailoring the properties of transition aluminas for catalytic and adsorption applications [2,4,13].

In addition to precursor selection, the nature of the precipitating agent plays a fundamental role in controlling particle formation. Traditional precipitants include sodium hydroxide, ammonium hydroxide, ammonium carbonate, carbonate salts, urea, and several homogeneous precipitation reagents. Among these, homogeneous precipitation has attracted increasing attention because the gradual generation of hydroxyl ions promotes controlled nucleation and minimizes local supersaturation. The use of urea as a homogeneous precipitating agent is well established owing to its thermal hydrolysis, which slowly releases ammonia and progressively increases solution pH. However, alternative precipitation systems have gained interest because they can offer lower precipitation temperatures, faster reaction kinetics, and improved control over phase formation. The influence of precipitating agents on the formation of pseudoboehmite, boehmite, or gibbsite remains an active field of investigation due to its direct impact on the final properties of transition aluminas [1,11,12].

Although urea is one of the most widely employed homogeneous precipitating agents for aluminum oxyhydroxide synthesis, alternative systems capable of generating ammonia in situ have received considerably less attention. Among them, ammonium bisulfite represents an attractive precipitation route because ammonia is generated at relatively low temperatures, promoting rapid and homogeneous nucleation while minimizing local supersaturation effects.

Previous studies have demonstrated the feasibility of using ammonium bisulfite for the synthesis of pseudoboehmite; however, comprehensive evaluations of its influence on thermal stability, morphology, and textural development relative to conventional homogeneous precipitants such as urea remain limited [11,15].

The properties of pseudoboehmite obtained by different synthesis routes are highly dependent on the precipitation mechanism and aging conditions. Sol-gel synthesis generally produces highly pure materials with large surface areas and good reproducibility, whereas hydrothermal treatments frequently enhance crystallinity and thermal stability. Neutralization and precipitation routes allow wider control over pore structure and particle morphology. Reported surface areas for synthetic pseudoboehmite commonly exceed $250 \text{ m}^2 \text{ g}^{-1}$ and may reach values above $330 \text{ m}^2 \text{ g}^{-1}$ depending on synthesis conditions. Likewise, pore volumes and pore size distributions vary significantly according to precursor chemistry, pH, aging temperature, and drying procedure. These differences are important because they determine the textural characteristics of the resulting γ - Al_2O_3 after calcination [4,13–16].

The industrial importance of pseudoboehmite originates primarily from its role as a precursor to high-surface-area alumina supports. γ - Al_2O_3 derived from pseudoboehmite is extensively used in fluid catalytic cracking (FCC), hydrosulfurization, hydrocracking, catalytic reforming, automotive catalytic converters, adsorption systems, and environmental catalysis. In addition, pseudoboehmite has attracted increasing attention in adsorption processes, advanced ceramics, coatings, flame-retardant formulations, and nanostructured materials where control of surface area and pore architecture is essential for performance [17–20].

Understanding the thermal evolution of pseudoboehmite is essential because the thermal decomposition pathway directly affects pore development and phase transformation. Thermogravimetric analysis (TGA) and derivative thermogravimetry (DTG) are among the most widely used techniques for characterizing dehydration, dehydroxylation, thermal stability, and phase-transition behavior in aluminum oxyhydroxides. These methods provide valuable information regarding the removal of physisorbed water, the loss of structural hydroxyl groups, and the transformation of pseudoboehmite into transition aluminas. Recent investigations have demonstrated that TGA and DTG analyses allow discrimination among pseudoboehmite, boehmite, gibbsite, and amorphous hydroxide phases while simultaneously providing insight into the stability and degree of structural condensation of synthesized materials [21–25].

The textural properties of γ -Al₂O₃ are commonly evaluated through nitrogen adsorption-desorption measurements using the Brunauer-Emmett-Teller (BET) method. BET analysis provides quantitative information regarding specific surface area, total pore volume, pore size distribution, and mesoporosity. These parameters are particularly relevant because catalytic activity, adsorption capacity, and mass-transfer characteristics strongly depend on the accessible surface area and pore architecture of the material. Pseudoboehmite-derived γ -Al₂O₃ often exhibits Type IV adsorption-desorption isotherms characteristic of mesoporous solids and may develop surface areas exceeding 300 m² g⁻¹ under optimized synthesis conditions. Consequently, BET characterization is widely used to establish relationships between synthesis parameters, precursor morphology, pore development, and final functional performance [26–29].

Scanning electron microscopy (SEM) has become an essential technique for investigating the morphological evolution of pseudoboehmite and γ -Al₂O₃. SEM provides direct visualization of particle shape, aggregation state, surface roughness, porosity, and microstructural organization over a broad range of length scales, making it particularly useful for evaluating morphological changes associated with precursor chemistry and thermal transformation processes [24,31]. Morphological observations frequently reveal substantial differences in particle architecture arising from changes in precursor chemistry, pH conditions, precipitating agents, and thermal treatments. When combined with TGA, DTG, and BET measurements, SEM enables the establishment of correlations among morphology, thermal stability, pore development, and surface area. Such integrated characterization approaches are increasingly employed to elucidate the mechanisms governing the formation and transformation of alumina precursor materials [1,5,30–32].

Despite the extensive literature devoted to pseudoboehmite synthesis, the combined influence of aluminum precursor anions and homogeneous precipitating agents on thermal evolution, morphological development, and textural properties remains insufficiently understood. Most previous investigations have focused either on the effect of precursor chemistry or on the influence of precipitation methods separately, whereas systematic comparisons involving aluminum sulfate, aluminum nitrate, and aluminum chloride combined with different homogeneous precipitating agents under identical synthesis conditions are still scarce.

Furthermore, limited information is available regarding how these variables collectively affect the transformation of pseudoboehmite into γ -Al₂O₃ and the development of its final mesoporous structure. Therefore, the present investigation aims to evaluate the effect of different aluminum precursors and precipitating agents on the formation of pseudoboehmite and the resulting γ -Al₂O₃. Special attention is devoted to correlating thermal behavior, morphology, pore structure, and surface area in order to establish relationships between precursor selection, precipitation route, and the final characteristics of high-surface-area mesoporous aluminas [2,4,11,13].

II. EXPERIMENTAL PROCEDURE

2.1 Materials

Aluminum sulfate hydrate (Al₂(SO₄)₃·18H₂O), aluminum nitrate nonahydrate (Al(NO₃)₃·9H₂O), aluminum chloride hexahydrate (AlCl₃·6H₂O), ammonium bisulfite (NH₄HSO₃), urea ((NH₂)₂CO), and ammonium hydroxide (NH₄OH) were obtained from Merck (Darmstadt, Germany). All reagents were analytical grade and were used without further purification. Distilled water was used throughout the experimental procedure.

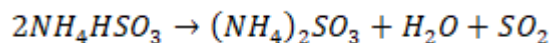
2.2 Synthesis of Pseudoboehmite Precursors

Aluminum sulfate, aluminum nitrate, and aluminum chloride were dissolved separately in distilled water to prepare aluminum precursor solutions with a concentration of 0.65 M.

Synthesis using ammonium bisulfite

Ammonium bisulfite solution was prepared by absorption of sulfur dioxide in aqueous ammonia. The aluminum salt solution and ammonium bisulfite solution were mixed at a volumetric ratio of 3:1 and heated at 90 °C for 30 min under continuous stirring.

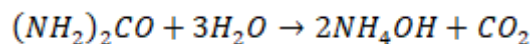
The gradual thermal decomposition of ammonium bisulfite generated ammonia in situ, promoting homogeneous precipitation of basic aluminum salts. The overall reaction may be represented as:



Synthesis using urea

For comparison, homogeneous precipitation was also carried out using urea as the precipitating agent. Aluminum salt solutions were mixed with an aqueous urea solution at a volumetric ratio of 2.5:1 and heated at 95 °C for 2.5 h under continuous stirring.

The gradual hydrolysis of urea generated ammonia and promoted a controlled increase in pH, resulting in the homogeneous precipitation of basic aluminum salts. The overall reaction may be represented as:



2.3 Preparation of Pseudoboehmite

The basic aluminum salts obtained by homogeneous precipitation were converted into pseudoboehmite through a solid-liquid neutralization process. The precipitated solids were filtered, washed with hot distilled water, and subsequently dispersed in distilled water using a solid-to-liquid ratio of 1:8.

Neutralization was carried out using aqueous ammonium hydroxide at 75 °C under continuous stirring. The resulting solids were filtered, repeatedly washed with hot distilled water to remove residual soluble species, and dried at 110 °C for 12 h.

The pseudoboehmites derived from aluminum sulfate, nitrate, and chloride synthesized using ammonium bisulfite were designated SPB-B, NPB-B, and CPB-B, respectively. Similarly, the pseudoboehmites synthesized using urea were designated SPB-U, NPB-U, and CPB-U.

2.4 Preparation of γ -Al₂O₃

The dried pseudoboehmite powders were calcined in air at 450 °C for 3 h using a heating rate of 10 °C min⁻¹.

The corresponding γ -Al₂O₃ samples obtained from SPB-B, NPB-B, CPB-B, SPB-U, NPB-U, and CPB-U were designated SA-B, NA-B, CA-B, SA-U, NA-U, and CA-U, respectively.

2.5 Characterization

Thermogravimetric Analysis

Thermogravimetric analysis (TGA) and derivative thermogravimetry (DTG) were performed using a TGA/DTA (TA Instruments: DMA, SDT 2960) in a simultaneous thermal analyzer under a flowing air atmosphere. Approximately 10 mg of sample was heated from room temperature to 1200 °C at a heating rate of 10 °C min⁻¹.

X-ray Diffraction

The crystalline phases of the calcined γ -Al₂O₃ samples were identified by X-ray diffraction (XRD) using a Siemens D500 diffractometer with Cu K α radiation (λ = 1.5406 Å) over the 2 θ range from 10° to 80°. Diffraction patterns were used to evaluate phase formation and structural ordering of the alumina samples.

Scanning Electron Microscopy

The morphology of the pseudoboehmite precursors and the corresponding γ -Al₂O₃ samples was examined by Field Emission Scanning Electron Microscopy (FE-SEM; JEOL JSM 7401F). Before observation, the samples were sputter-coated with a thin gold layer to improve electrical conductivity [31].

Nitrogen Adsorption-Desorption Analysis

Textural properties were determined by nitrogen adsorption-desorption measurements at 77 K. Prior to analysis, the samples were degassed under vacuum at 200 °C for 4 h. The specific surface area was calculated by single-point BET measurements (ASAP 2010 Micromeritics Instrument Corp., USA), whereas pore volume and pore size distributions were determined from the adsorption branch of the isotherms using the Barrett-Joyner-Halenda (BJH) method.

All samples were synthesized, dried, calcined, and characterized under identical experimental conditions. Therefore, the differences observed in thermal behavior, morphology, phase development, and textural properties can be attributed exclusively to the aluminum precursor and the precipitating agent employed during homogeneous precipitation.

III. RESULTS AND DISCUSSION

3.1 Effect of Aluminum Precursors and Precipitating Agents on the Thermal Behavior of Pseudoboehmite

TGA and DTG analyses were used to investigate the thermal decomposition behavior of pseudoboehmite powders synthesized from aluminum sulfate, nitrate, and chloride using ammonium bisulfite or urea as precipitating agents. The corresponding thermograms are presented in Figure 1.

As shown in Figure 1A, all samples exhibit a multistep decomposition process from room temperature to 1200 °C. The thermal events can be divided into three main regions: (i) dehydration below 150 °C, (ii) dehydroxylation between 150 and 500 °C, and (iii) high-temperature transformations above 800 °C.

The thermal decomposition parameters obtained from the TGA and DTG curves are summarized in Table 1.

The first weight-loss step occurred below 150 °C and is attributed to the removal of physically adsorbed water and moisture retained within the porous structure. The DTG curves reveal broad peaks centered between approximately 50 and 100 °C, indicating the release of weakly bound water molecules.

This process is more pronounced in the materials synthesized using urea, suggesting a higher degree of hydration. This thermal event is characteristic of pseudoboehmite and boehmite materials, in which weakly adsorbed water species are progressively removed at relatively low temperatures [21,23,24].

The second thermal region, extending from approximately 150 to 500 °C, corresponds to the dehydroxylation of aluminum hydroxide and oxyhydroxide species. Similar behavior has been reported for pseudoboehmite and nanocrystalline boehmite, where the removal of structural hydroxyl groups promotes the formation of transition alumina phases during calcination [22,23,24,25]. Significant differences were observed depending on both the aluminum precursor and the precipitating agent. Samples synthesized with ammonium bisulfite exhibited broad DTG peaks distributed over a relatively wide temperature interval. The main DTG maxima were located at approximately 350, 310, and 280 °C for SPB-B, CPB-B, and NPB-B, respectively. The progressive shift of the maximum decomposition temperature toward lower values indicates a reduction in thermal stability in the order:

$$\text{SPB-B} > \text{CPB-B} > \text{NPB-B}$$

This trend suggests that sulfate-derived pseudoboehmite possesses a more condensed hydroxide network and a higher degree of structural organization than the chloride- and nitrate-derived materials. Sulfate ions may influence hydrolysis and condensation reactions during precipitation, promoting the formation of thermally more stable aluminum oxyhydroxide structures. Similar effects of precursor anions on the thermal stability and structural organization of pseudoboehmite have been reported previously [2,9].

In contrast, materials synthesized using urea showed markedly sharper DTG peaks, particularly CPB-U, which presented an intense decomposition maximum near 220 °C. The lower decomposition temperatures and larger weight losses observed for the urea-derived materials indicate the presence of more highly hydroxylated phases. These results may indicate the presence of more highly hydroxylated aluminum hydroxide species, which generally dehydroxylate at lower temperatures than pseudoboehmite [21,23].

The total weight loss values presented in Table 1 further support this interpretation. The bisulfite-derived samples exhibited total weight losses between 28 and 37%, whereas the urea-derived materials showed considerably larger losses ranging from 40 to 46%.

The lowest total weight loss was observed for SPB-B (28%), while CPB-U exhibited the highest value (46%).

The higher residual masses observed for the bisulfite-derived solids indicate that ammonium bisulfite promotes the formation of less hydrated and thermally more stable pseudoboehmite structures. Conversely, urea favors the formation of phases containing larger amounts of structural hydroxyl groups, resulting in greater mass losses during thermal treatment.

Table 1. Thermal decomposition parameters of pseudoboehmite synthesized from different aluminum precursors.

Sample	Residual mass at 1200 °C (%)	Total weight loss (%)	Main DTG peak (°C)	High-temperature event (°C)
SPB-B	72	28	350	900
CPB-B	69	31	310	900
NPB-B	63	37	280	900
NPB-U	60	40	260	900
SPB-U	57	43	240	900
CPB-U	54	46	220	900

The effect of the aluminum precursor is particularly evident when comparing materials synthesized using the same precipitating agent. For the bisulfite route, the residual mass decreased from 72% for SPB-B to 69% for CPB-B and 63% for NPB-B. This behavior suggests that the precursor anion influences the hydrolysis and condensation reactions occurring during precipitation, thereby modifying the structural organization of the resulting pseudoboehmite.

Among the studied systems, CPB-B displayed an intermediate thermal behavior between sulfate- and nitrate-derived pseudoboehmites. This observation is relevant because it demonstrates that chloride ions do not simply reproduce the behavior of nitrate or sulfate systems but rather promote a distinct precipitation pathway that leads to a material with different thermal characteristics.

A third thermal event was detected around 900 °C for all samples. Thermal events within this temperature range have been associated with structural rearrangements of transition aluminas and the progressive evolution toward thermodynamically more stable alumina phases during high-temperature treatment [24,25]. The low intensity of the corresponding DTG peak indicates that only a small fraction of the material undergoes structural evolution at this temperature. The phenomenon is more evident in sulfate-derived samples, which may suggest the persistence of residual sulfate-containing species or differences in structural rearrangement during high-temperature treatment.

TGA and DTG results demonstrate that both the aluminum precursor and the precipitating agent strongly affect the thermal stability and degree of hydroxylation of the synthesized materials. The thermal stability sequence determined from the DTG maxima follows:

SPB-B > CPB-B > NPB-B > NPB-U > SPB-U > CPB-U

whereas the total weight loss follows the reverse order:

SPB-B < CPB-B < NPB-B < NPB-U < SPB-U < CPB-U

These results suggest that the precursor anion plays a critical role during nucleation and growth of aluminum oxyhydroxide phases, producing structures with different degrees of condensation and hydration. Such differences are expected to influence not only the morphological evolution but also the preservation of porosity during calcination. Consequently, variations in thermal stability may play a decisive role in determining the final surface area and pore volume of the resulting γ -Al₂O₃.

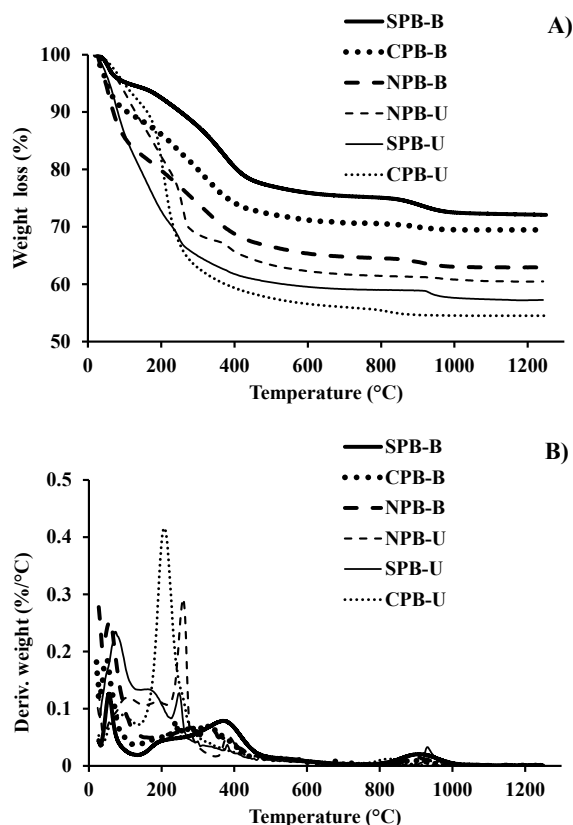


Figure 1. A) TGA and B) DTG analyses of pseudoboehmite powders synthesized from aluminum sulfate, nitrate, and chloride using ammonium bisulfite or urea as precipitating agents

3.2 Formation and Structural Characteristics of γ -Al₂O₃

The X-ray diffraction patterns of the calcined samples are presented in Figure 2. All materials exhibit diffraction peaks located at approximately $2\theta = 36.5^\circ$, 45.9° , and 66.7° , corresponding to the (311), (400), and (440) crystallographic planes, respectively, which are characteristic of the γ -Al₂O₃ phase. The observed reflections are in good agreement with the standard diffraction pattern reported for γ -Al₂O₃ in the ICDD reference card No. 10-0425, confirming the successful formation of transition alumina after calcination.

The absence of detectable diffraction peaks associated with residual hydroxide or oxyhydroxide phases suggests that the calcination treatment effectively promoted the transformation of the precursor materials into γ -Al₂O₃, regardless of the aluminum precursor or precipitating agent employed during synthesis.

A common feature observed in all diffractograms is the broad character of the diffraction peaks. Such peak broadening is typical of transition aluminas and is generally associated with limited crystallite size, structural disorder, and a relatively low degree of long-range ordering. Similar diffraction characteristics have been reported for boehmite-derived γ -Al₂O₃ synthesized under controlled calcination conditions, where the preservation of nanocrystalline domains contributes to the broadening of the characteristic reflections and to the defective spinel structure commonly observed in transition aluminas [25,35].

Although the diffraction patterns are qualitatively similar, differences in peak definition can be observed among the samples. The aluminas synthesized using ammonium bisulfite (SA-B, NA-B, and CA-B) exhibit broader reflections and lower peak intensities than the corresponding materials obtained using urea. In contrast, the urea-derived samples display slightly sharper and better-defined reflections, suggesting the development of a somewhat higher degree of structural ordering during calcination.

These differences may be associated with variations in the decomposition behavior of the precursor materials. As previously observed in the TGA and DTG analyses, the materials synthesized using ammonium bisulfite exhibited lower overall weight losses and broader dehydroxylation processes. Such gradual thermal transformations could limit crystallite growth during calcination, resulting in less ordered γ -Al₂O₃ structures. Conversely, the more pronounced thermal decomposition observed in the urea-derived samples may favor localized structural rearrangements, leading to slightly greater crystallinity. Similar relationships between calcination-induced structural evolution and diffraction peak definition have

been reported for boehmite-derived γ - Al_2O_3 synthesized through different preparation routes [25,35].

Among the bisulfite-derived materials, SA-B exhibits the broadest reflections, indicating the lowest degree of structural ordering. CA-B and NA-B display similar diffraction profiles, although slight differences in peak intensity suggest that the precursor anion influences the structural evolution occurring during thermal treatment. For the urea-derived samples, SA-U, CA-U, and NA-U present comparable diffraction patterns, all corresponding to the γ - Al_2O_3 phase. Nevertheless, the reflections appear somewhat sharper than those observed for the bisulfite-derived aluminas, suggesting that the choice of precipitating agent affects the structural organization developed during phase transformation.

The broad diffraction peaks observed for all samples indicate the formation of poorly crystalline γ - Al_2O_3 , which is characteristic of transition aluminas prepared from pseudoboehmite precursors. This behavior is consistent with the preservation of nanocrystalline domains inherited from the precursor material during the pseudomorphic transformation process and agrees with previous studies describing the structural evolution of boehmite-derived γ - Al_2O_3 [24,25,35].

XRD results demonstrate that all synthesized materials were successfully converted into γ - Al_2O_3 under the applied calcination conditions. Furthermore, the observed differences in peak broadening indicate that both the aluminum precursor and the precipitating agent influence the degree of structural ordering of the resulting alumina. These findings provide structural evidence that the synthesis conditions affect not only phase formation but also the crystallographic characteristics developed during the transformation of pseudoboehmite into γ - Al_2O_3 .

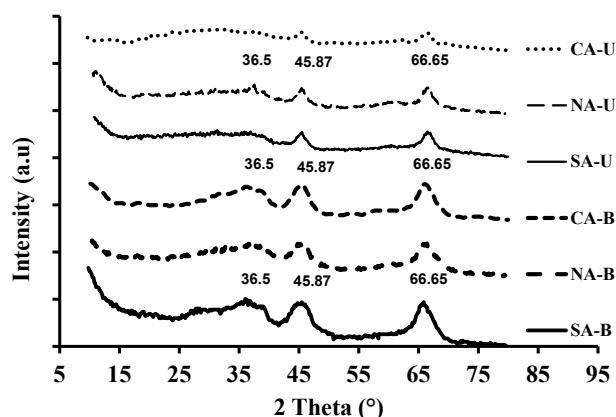


Figure 2. X-ray diffraction patterns of γ - Al_2O_3 synthesized from sulfate, nitrate, and chloride precursors using ammonium bisulfite and urea as precipitating agents.

3.3 Morphological characterization of Pseudoboehmite (AlOOH) and γ - Al_2O_3 synthesized from sulfate, chloride, and nitrate precursors using ammonium bisulfite and urea as precipitating agents

3.3.1 Morphological characterization of pseudoboehmite

Figure 3 presents the SEM micrographs of pseudoboehmite synthesized by homogeneous precipitation using different aluminum salts and two precipitation agents, namely ammonium bisulfite (SPB-B, NPB-B, and CPB-B) and urea (SPB-U, NPB-U, and CPB-U). Previous XRD characterization of the precursor materials confirmed the formation of pseudoboehmite, although the diffraction patterns are not shown in the present work. Significant differences in particle morphology and aggregation state can be observed as a function of both the aluminum precursor and the precipitation agent. These observations suggest that differences in hydrolysis kinetics, nucleation behavior, and crystal growth mechanisms strongly influence the resulting microstructure. Similar relationships between synthesis conditions and morphological evolution have been reported for pseudoboehmite and boehmite-derived alumina systems prepared through different precipitation and calcination routes [24,35].

The pseudoboehmite synthesized using aluminum sulfate and ammonium bisulfite (SPB-B) (Figure 3A) exhibits aggregates composed of intertwined fibrillar particles forming a highly open three-dimensional network. The individual crystallites are difficult to distinguish because of their extensive intergrowth, indicating the formation of finely divided pseudoboehmite domains. This morphology is characteristic of poorly crystalline pseudoboehmite and is consistent with the structural disorder typically associated with highly dispersed oxyhydroxide phases. The open arrangement of the fibrillar aggregates suggests that nucleation occurred extensively throughout the precipitation process, limiting the growth of large individual particles. Similar fibrillar structures have frequently been reported for pseudoboehmite synthesized under conditions favoring rapid nucleation and restricted crystal growth [33].

SPB-U (Figure 3B), synthesized from aluminum sulfate using urea as the precipitating agent, exhibits a markedly different morphology. The material consists of well-defined flower-like agglomerates composed of radially oriented nanosheets or nanoplates. Compared with SPB-B, the aggregates appear more organized and structurally differentiated. This behavior may be attributed to the slow decomposition of urea, which gradually generates hydroxyl ions and favors controlled crystal growth during aging.

The preservation of discrete nanosheet structures suggests the development of a hierarchical architecture before drying. Similar flower-like and hierarchical morphologies have been reported for boehmite and pseudoboehmite synthesized through homogeneous precipitation and hydrothermal routes, where controlled hydroxyl release promotes anisotropic crystal growth [15,33,35].

The sample NPB-B, obtained from aluminum nitrate and ammonium bisulfite (Figure 3C), exhibits a markedly different morphology characterized by compact platelet-like particles. The surface appears relatively uniform and dense compared with the sulfate-derived samples. Such morphology suggests that crystal growth became more significant than nucleation during precipitation, leading to the formation of larger coherent domains. This observation agrees with the comparatively greater structural organization inferred from the XRD patterns. Furthermore, the denser appearance of the particles indicates a lower degree of structural heterogeneity than observed in the fibrillar sulfate-derived pseudoboehmites. Similar platelet-like morphologies have been described in pseudoboehmite systems where particle growth is favored during synthesis [24,34].

The morphology becomes even more compact in NPB-U (Figure 3D), synthesized using aluminum nitrate and urea. Large lamellar agglomerates with smooth surfaces dominate the microstructure. The absence of fibrillar features and the presence of extensive flat regions indicate substantial particle growth and aggregation during synthesis. The slow generation of hydroxyl ions by urea appears to have promoted the formation of larger structural entities rather than numerous small nuclei. Consequently, the resulting pseudoboehmite exhibits the highest degree of morphological consolidation among the investigated materials. Similar compact lamellar morphologies have been associated with advanced crystal growth and particle densification processes in aluminum oxyhydroxides synthesized under conditions of slow hydroxyl generation and prolonged aging [15,24,35].

The pseudoboehmite synthesized using aluminum chloride and ammonium bisulfite (CPB-B) (Figure 3E) exhibits porous agglomerates composed of fine fibrillar units. The morphology resembles that observed for SPB-B, although the aggregates appear somewhat more compact and irregular. Nevertheless, the presence of abundant fibrillar structures indicates that nucleation remained an important process during precipitation.

The resulting morphology suggests the formation of nanocrystalline pseudoboehmite assemblies with significant structural development. Similar aggregated fibrillar structures have been described for chloride-derived pseudoboehmite materials prepared through homogeneous precipitation.

In contrast, CPB-U (Figure 3F) presents a considerably denser morphology. The micrograph reveals a compact surface where individual crystallites are difficult to identify due to extensive aggregation. Compared with CPB-B, the use of urea appears to promote a greater degree of particle consolidation during synthesis and aging. Although remnants of the original fine particle structure can still be distinguished, the material exhibits a substantially lower level of morphological definition than the corresponding bisulfite-derived sample. This behavior further demonstrates the strong influence of precipitation chemistry on the organization of pseudoboehmite particles. Similar microstructural evolution has been reported for boehmite-derived materials synthesized through slow hydrolysis routes.

The morphological differences observed by SEM are also consistent with the thermal behavior discussed previously. Samples exhibiting highly developed fibrillar structures, such as SPB-B and CPB-B, displayed thermal events associated with the removal of adsorbed water and structural hydroxyl groups characteristic of poorly crystalline pseudoboehmite. In contrast, the more compact morphologies of NPB-U and CPB-U suggest greater structural organization before calcination, which may influence their dehydration and dehydroxylation pathways. Similar correlations between morphology, crystallinity, and thermal evolution have been reported for pseudoboehmite and boehmite systems [24].

SEM observations reveal two clear morphological trends. Samples synthesized using ammonium bisulfite generally exhibit fibrillar and highly developed aggregated structures, whereas samples synthesized using urea tend to exhibit more organized, lamellar, or compact morphologies. Additionally, sulfate-derived materials favor the formation of fibrillar and hierarchical architectures, whereas nitrate-derived materials promote particle growth and densification. Chloride-derived samples exhibit intermediate behavior. These results demonstrate that both the precursor anion and the precipitation mechanism play a decisive role in controlling the microstructural evolution of pseudoboehmite, in agreement with the structural and thermal characteristics identified by XRD and TGA/DTG analyses [8,24,34].

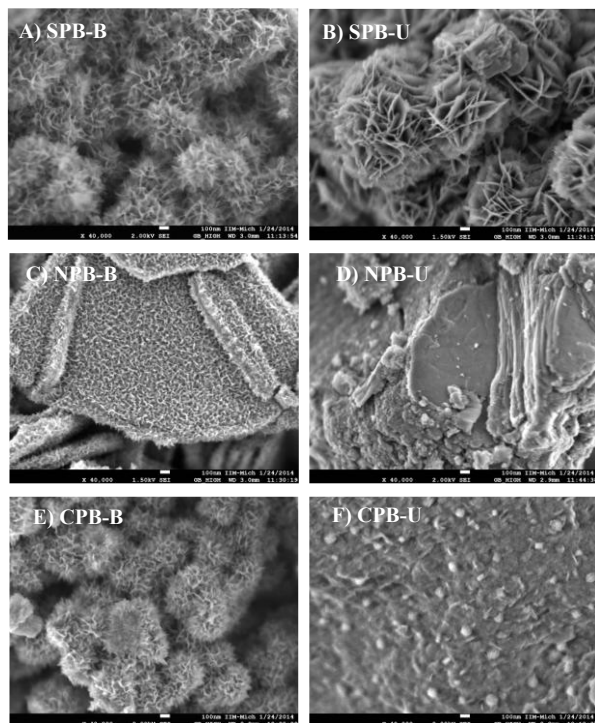


Figure 3. SEM micrographs of pseudoboehmite synthesized by homogeneous precipitation using different aluminum precursors and precipitating agents

3.3.2 Morphological Characterization of γ - Al_2O_3

Figure 4 shows SEM micrographs of the γ - Al_2O_3 samples obtained after calcining the corresponding pseudoboehmite precursors at 450 °C, which were synthesized using different aluminum salts and precipitation agents. As discussed above, the XRD results indicate that all calcined materials correspond to the γ - Al_2O_3 phase. SEM observations show that thermal treatment partially preserved key morphological features developed during pseudoboehmite synthesis. This behavior is characteristic of the pseudomorphic transformation of pseudoboehmite into γ - Al_2O_3 , in which structural reorganization occurs through dehydroxylation while retaining significant features of the precursor architecture. Similar morphology-preserving transformations have been widely reported for boehmite-derived transition aluminas and are considered a key factor controlling the final porosity and surface development of γ - Al_2O_3 [24,25,35].

The γ - Al_2O_3 sample designated SA-B, obtained from pseudoboehmite synthesized using aluminum sulfate and ammonium bisulfite (Figure 4A), exhibits agglomerates composed of intertwined fibrillar particles forming a highly open three-dimensional network.

Comparison with the corresponding pseudoboehmite precursor reveals that the fibrous morphology remains largely preserved after calcination. The formation of γ - Al_2O_3 therefore appears to occur through dehydroxylation and structural rearrangement without significant collapse of the aggregated architecture. Such morphology retention has been associated with limited structural collapse during dehydroxylation and is frequently correlated with the development of high-surface-area aluminas [24,35]. The rough surface texture observed can be attributed to the release of structural water during the thermal decomposition process identified in the TGA and DTG analyses. Similar retention of fibrillar morphology has been reported during the transformation of poorly crystalline pseudoboehmite into transition aluminas.

A particularly distinctive morphology is observed for SA-U (Figure 4B), obtained from pseudoboehmite synthesized using aluminum sulfate and urea. The sample consists of spherical aggregates formed by radially distributed platelet-like structures that preserve the flower-like morphology previously observed in the precursor. The persistence of these hierarchical arrangements indicates that the architecture generated during homogeneous precipitation remained stable during calcination. The well-defined nanosheets suggest that the gradual generation of hydroxyl ions during urea hydrolysis promoted controlled crystal growth during pseudoboehmite formation, producing structures capable of maintaining their integrity throughout the pseudoboehmite-to- γ - Al_2O_3 transformation. Similar hierarchical morphologies have been reported for boehmite-derived aluminas synthesized through controlled hydrolysis and hydrothermal routes [15,35].

The morphology of NA-B, obtained from aluminum nitrate and ammonium bisulfite (Figure 4C), differs markedly from that observed in sulfate-derived samples. The material exhibits relatively compact platelet-like particles with a rough surface texture generated during calcination. The overall morphology closely resembles that observed in the corresponding pseudoboehmite precursor, indicating that structural reorganization occurred without significant modification of aggregate geometry. The preservation of these dense particles suggests that crystal growth processes during precipitation had already established a stable morphology before calcination. Similar observations have been described for boehmite-derived aluminas obtained from precursors exhibiting higher degrees of particle aggregation and structural ordering [24,35].

The sample NA-U (Figure 4D) exhibits the most compact morphology among all analyzed materials. Large agglomerates with relatively smooth external surfaces and limited evidence of fibrillar features can be observed. Compared with the other γ - Al_2O_3 samples, this morphology suggests extensive particle coalescence and a lower degree of microstructural heterogeneity. The resemblance between the calcined sample and its precursor further supports the pseudomorphic nature of the transformation process. The compact appearance of the particles indicates that the combination of nitrate ions and the slow decomposition kinetics of urea favored crystal growth during pseudoboehmite synthesis, generating aggregates that remained stable throughout calcination. Similar morphologies have been associated with advanced particle densification and reduced pore development in boehmite-derived aluminas [24,35].

The γ - Al_2O_3 sample identified as CA-B (Figure 4E), obtained from pseudoboehmite synthesized using aluminum chloride and ammonium bisulfite, exhibits irregular agglomerates composed of interconnected fibrillar units. The rough and highly developed surface texture indicates that the microstructural organization established during pseudoboehmite formation was largely retained after calcination. Although the aggregates appear more compact than those observed in SA-B, the persistence of fibrillar features suggests that dehydroxylation occurred without complete morphological collapse. Such morphology retention is consistent with the preservation of interparticle voids that may contribute to the development of mesoporosity after calcination [24,35].

In contrast, CA-U (Figure 4F) presents irregular compact aggregates with poorly defined particle boundaries. The surface appears denser and smoother than that observed for CA-B, suggesting a greater degree of particle coalescence during thermal treatment. Unlike the well-developed fibrillar structures observed in SA-B and CA-B, the original morphology of the precursor is more difficult to identify after calcination. This observation suggests that the precursor generated using urea was more susceptible to local structural rearrangement during the pseudoboehmite-to- γ - Al_2O_3 transformation. Similar microstructural evolution has been reported for boehmite-derived aluminas undergoing thermal dehydration and progressive crystallographic reorganization [24,25].

The SEM observations reveal that the morphology of γ - Al_2O_3 is strongly influenced by both the aluminum precursor salt and the precipitation agent employed during pseudoboehmite synthesis.

Sulfate-derived samples (SA-B and SA-U) retained well-developed fibrillar and hierarchical structures after calcination, whereas nitrate-derived materials (NA-B and particularly NA-U) exhibited more compact morphologies. Chloride-derived samples displayed intermediate behavior. The morphological similarities observed between each pseudoboehmite precursor and its corresponding γ - Al_2O_3 product provide further evidence of the pseudomorphic nature of the transformation confirmed by XRD and thermal analyses. These results demonstrate that the synthesis conditions employed during pseudoboehmite formation play a decisive role in defining the final microstructure of γ - Al_2O_3 . Furthermore, the strong morphological correspondence observed between precursor and calcined materials indicates that precursor morphology constitutes one of the primary factors governing pore preservation, surface development, and the final textural properties of the resulting alumina. This relationship is examined in greater detail in the nitrogen adsorption-desorption analyses presented in the following section.

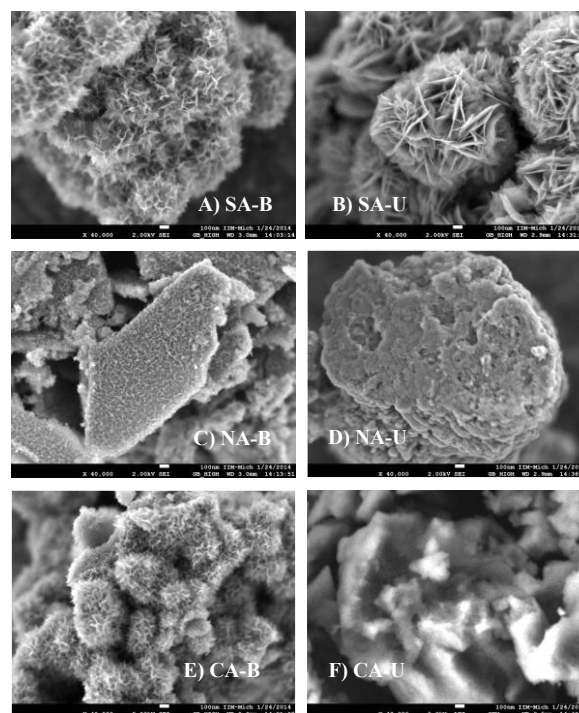


Figure 4. SEM micrographs of γ - Al_2O_3 obtained from pseudoboehmite precursors synthesized using aluminum sulfate, aluminum nitrate, and aluminum chloride with ammonium bisulfite or urea as precipitation agents, followed by calcination at 450 °C.

3.4 Textural Properties of γ - Al_2O_3 Synthesized from Sulfate, Chloride, and Nitrate Precursors Using Ammonium Bisulfite and Urea as Precipitating Agents

3.4.1 Nitrogen Adsorption-Desorption Isotherms of γ - Al_2O_3 Figure 5A shows the N_2 adsorption-desorption isotherms of γ - Al_2O_3 synthesized from sulfate, chloride, and nitrate precursors using ammonium bisulfite and urea as precipitating agents. All materials exhibit Type IV isotherms according to the IUPAC classification, indicating the formation of mesoporous aluminas. The presence of hysteresis loops in the intermediate and high relative pressure region ($P/P_0 \approx 0.4$ -1.0) confirms capillary condensation within interconnected mesoporous networks generated during the thermal transformation of the aluminum oxyhydroxide precursors. Such adsorption behavior is characteristic of pseudoboehmite-derived γ - Al_2O_3 materials possessing well-developed mesoporosity and high surface area [26,27,29].

The adsorption capacity strongly depends on both the aluminum precursor and the precipitating agent. The highest nitrogen uptake was observed for SA-B, followed by NA-B and CA-B, whereas the urea-derived materials exhibited significantly lower adsorption capacities. These observations are quantitatively confirmed by the BET surface areas listed in Table 2. SA-B exhibited the highest specific surface area ($332 \text{ m}^2 \text{ g}^{-1}$), followed by NA-B ($320 \text{ m}^2 \text{ g}^{-1}$) and CA-B ($296 \text{ m}^2 \text{ g}^{-1}$). In contrast, the materials synthesized using urea showed considerably lower values ranging from 109 to $165 \text{ m}^2 \text{ g}^{-1}$.

The superior adsorption behavior of SA-B can be directly associated with the highly open fibrillar morphology observed for both SPB-B and the corresponding calcined alumina. SEM analysis revealed loosely packed aggregates composed of interconnected nanofibrils that remained largely preserved after calcination. Such an architecture promotes the formation of a highly accessible pore network and contributes to the largest pore volume ($0.64 \text{ cm}^3 \text{ g}^{-1}$) among all investigated samples. Similar relationships between precursor morphology, morphology retention during calcination, and the development of high-surface-area γ - Al_2O_3 have been reported for boehmite-derived aluminas synthesized under controlled thermal conditions [24,35].

Although NA-B exhibited a more compact platelet-like morphology than SA-B, its nitrogen adsorption capacity remained remarkably high. This result suggests that a substantial fraction of the available surface area originates from nanoscale porosity not readily visible by SEM.

Consequently, external particle morphology alone does not completely determine the final BET surface area; rather, the internal pore structure generated during calcination also plays an important role in governing texture development.

The materials prepared using urea displayed considerably lower adsorption capacities. The reduced nitrogen uptake observed for SA-U, CA-U, and NA-U indicates a less developed porous network. This tendency is consistent with the TGA and DTG results, which demonstrated that urea-derived precursors underwent larger weight losses during thermal treatment. The greater extent of dehydroxylation and structural rearrangement likely promotes partial pore collapse and reduced surface-area development during calcination.

The adsorption isotherms therefore indicate that ammonium bisulfite promotes the formation of precursor structures capable of preserving their pore architecture during thermal transformation, whereas urea favors the formation of more compact precursor morphologies that undergo greater shrinkage upon calcination. These findings highlight the critical role of precursor morphology in controlling the accessibility of the resulting mesoporous network.

3.4.2 Pore Size Distribution

Figure 5B shows the BJH pore size distributions of γ - Al_2O_3 synthesized from different aluminum precursors and precipitating agents. The pore size distributions obtained by the BJH method are shown in Figure 5B. All samples exhibit pore diameters within the mesoporous range, confirming the conclusions derived from the adsorption-desorption isotherms. The presence of mesopores is consistent with the pseudomorphic transformation of pseudoboehmite into γ - Al_2O_3 and with the preservation of interparticle voids developed during precursor formation [24,25].

SA-B displayed a broad pore size distribution extending approximately from 4 to 12 nm, with a maximum centered around 6-8 nm. This broad distribution, together with the highest pore volume ($0.64 \text{ cm}^3 \text{ g}^{-1}$), indicates the formation of a highly interconnected mesoporous network. The result agrees well with the fibrillar morphology observed in the corresponding pseudoboehmite precursor and with the gradual dehydroxylation process identified by DTG analysis.

CA-B exhibited a broader distribution shifted toward larger pore sizes. This sample showed the largest average pore diameter (4.6 nm) of the entire series and a relatively high pore volume ($0.44 \text{ cm}^3 \text{ g}^{-1}$).

The intermediate thermal stability observed by TGA/DTG suggests that partial restructuring during calcination generated larger interparticle mesopores while maintaining significant pore connectivity. NA-B presented a narrower pore size distribution and a lower pore volume ($0.31 \text{ cm}^3 \text{ g}^{-1}$) despite exhibiting a very high BET surface area ($320 \text{ m}^2 \text{ g}^{-1}$). This result indicates that the surface area originates primarily from a large number of small mesopores and nanometer-scale voids rather than from extensive pore volume. The platelet-like morphology observed by SEM is consistent with this interpretation.

Among the urea-derived materials, SA-U and CA-U exhibited moderate mesoporosity with pore volumes of 0.19 and $0.13 \text{ cm}^3 \text{ g}^{-1}$, respectively. Their pore distributions are narrower than those observed for the bisulfite-derived samples, suggesting that pore generation during calcination was more limited. NA-U displayed the poorest textural development. The extremely low pore volume ($0.04 \text{ cm}^3 \text{ g}^{-1}$) and reduced surface area ($109 \text{ m}^2 \text{ g}^{-1}$) indicate extensive structural contraction during thermal transformation. This observation is consistent with the compact morphology observed by SEM, as well as with the larger weight losses detected by thermal analysis.

Interestingly, the average pore diameters vary only slightly between 3.4 and 4.6 nm . Therefore, the major differences among the samples arise primarily from pore volume and pore density rather than from major changes in average pore size.

3.4.3 Correlation Between Thermal Stability, Morphology, and Textural Properties

The combined analysis of adsorption-desorption isotherms, pore size distributions, SEM observations, XRD results, and thermal analyses reveals a strong correlation between precursor chemistry and the final textural properties of $\gamma\text{-Al}_2\text{O}_3$. The most significant trend observed in Table 2 is that all bisulfite-derived aluminas exhibit substantially higher BET surface areas and larger pore volumes than their urea-derived counterparts.

SA-B combines the highest pore volume and the highest surface area, demonstrating that the highly open fibrillar morphology generated during precipitation was effectively preserved during calcination. The superior thermal stability identified by TGA and DTG likely minimized structural collapse during dehydroxylation, allowing extensive pore development and surface-area retention.

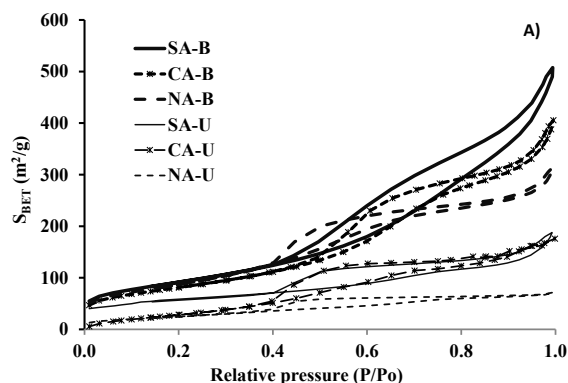
NA-B represents a particularly interesting case because its surface area is nearly as high as that of SA-B despite possessing only about half its pore volume.

This behavior suggests that a large fraction of the available surface area originates from numerous small mesopores and nanoporosity within the platelet-like particles rather than from large interparticle voids.

CA-B occupies an intermediate position. Although its surface area is lower than that of SA-B and NA-B, it exhibits the largest average pore diameter and a relatively high pore volume, indicating a mesoporous structure dominated by wider pore channels.

In contrast, the urea-derived materials exhibit significantly lower surface areas and pore volumes because of the stronger structural rearrangements occurring during dehydroxylation. The lower thermal stability observed in the TGA/DTG analyses and the compact morphologies detected by SEM suggest that greater shrinkage occurred during calcination, reducing pore accessibility and limiting the development of porous networks. These observations are consistent with previous studies describing the influence of precursor morphology and calcination behavior on the textural evolution of $\gamma\text{-Al}_2\text{O}_3$ [24,35].

Taken together, the TGA/DTG, XRD, SEM, and nitrogen adsorption results indicate that precursor-derived morphology and thermal stability are the primary factors governing pore preservation during calcination. Consequently, the highest surface areas and pore volumes were obtained for materials exhibiting both superior thermal stability and significant morphology retention, particularly the sulfate-derived aluminas synthesized using ammonium bisulfite. These findings demonstrate that the combined selection of aluminum precursor and precipitating agent plays a decisive role in tailoring the pore structure and surface characteristics of high-surface-area mesoporous $\gamma\text{-Al}_2\text{O}_3$.



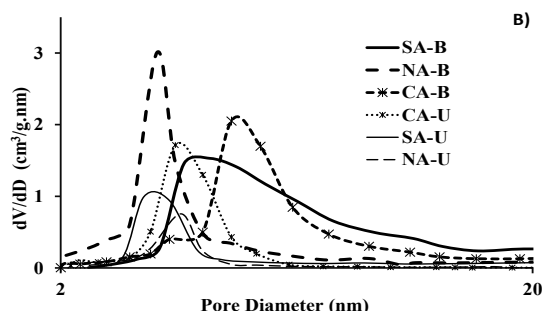


Figure 5: A) N_2 adsorption-desorption isotherms of γ - Al_2O_3 prepared from sulfate, nitrate, and chloride precursors; and B) Mesopore size distribution of γ - Al_2O_3 obtained from sulfate-, nitrate-, and chloride-derived pseudoboehmite precursors.

Table 2. TEXTURAL PROPERTIES OF γ - Al_2O_3 .

Sample	S_{BET} (m^2/g)	Pore Vol. (cm^3/g)	Average diameter (nm)
SA-B	332	0.64	4.14
CA-B	296	0.44	4.6
NA-B	320	0.31	3.5
SA-U	152	0.19	3.4
CA-U	165	0.13	3.5
NA-U	109	0.04	3.6

IV. CONCLUSION

The effects of aluminum precursor salts and homogeneous precipitating agents on the thermal behavior, morphological evolution, structural characteristics, and textural properties of γ - Al_2O_3 synthesized through homogeneous precipitation were systematically investigated. Based on the experimental results, the following conclusions can be drawn:

The thermal behavior of the precursor materials was strongly influenced by both the aluminum precursor and the precipitating agent. Samples synthesized using ammonium bisulfite exhibited lower total weight losses (28-37%) and higher dehydroxylation temperatures than those prepared using urea (40-46%), indicating the formation of less hydrated and thermally more stable pseudoboehmite structures. These results demonstrate that the precipitation route plays a critical role in determining the degree of structural condensation of the precursor material.

X-ray diffraction analysis confirmed the successful transformation of all precursor materials into γ - Al_2O_3 after calcination at 450 °C. The broad diffraction peaks characteristic of transition alumina indicate the formation of poorly crystalline γ - Al_2O_3 with a defective spinel structure.

Differences in peak broadening revealed that both precursor chemistry and precipitating agent influence the degree of structural ordering developed during calcination.

Significant differences in morphology were observed as a function of precursor composition and precipitation route. Sulfate-derived materials synthesized using ammonium bisulfite developed highly open fibrillar aggregates, whereas nitrate-derived samples exhibited compact platelet-like structures and chloride-derived samples displayed intermediate morphologies. Moreover, materials synthesized using urea generally exhibited more compact and consolidated particle arrangements than their bisulfite-derived counterparts.

SEM observations demonstrated that the transformation of pseudoboehmite into γ - Al_2O_3 occurred through a largely pseudomorphic process, preserving many of the morphological characteristics established during precursor formation. The degree of morphology retention strongly influenced the development of mesoporosity and surface area in the resulting aluminas.

All γ - Al_2O_3 samples exhibited Type IV adsorption-desorption isotherms characteristic of mesoporous materials. However, pronounced differences in textural properties were observed. The highest BET surface area and pore volume were obtained for SA-B, reaching 332 $m^2 g^{-1}$ and 0.64 $cm^3 g^{-1}$, respectively, whereas the lowest values were observed for NA-U. These differences demonstrate that the textural properties of γ - Al_2O_3 are highly dependent on both precursor chemistry and precipitation conditions.

The combined TGA/DTG, XRD, SEM, and BET analyses revealed a direct relationship between thermal stability, morphology preservation, and textural development. Materials exhibiting greater thermal stability experienced less structural collapse during calcination, retained a larger fraction of their original fibrillar architecture, and consequently developed higher surface areas and pore volumes. This relationship explains the superior performance of the aluminas synthesized using ammonium bisulfite.

Among the investigated synthesis routes, the combination of aluminum sulfate and ammonium bisulfite proved to be the most effective for producing high-surface-area mesoporous γ - Al_2O_3 . The results demonstrate that careful control of both precursor selection and homogeneous precipitation conditions provides an effective strategy for tailoring the structural and textural properties of γ - Al_2O_3 intended for catalytic and adsorption applications.

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