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Electrolytic synthesis of aluminum hydroxide nanocrystals

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Over recent years, interest in synthetic aluminum hydroxides in nanostructured form has grown significantly. Metastable alumina phases represent highly important technological materials characterized by high specific surface areas and promising applications in modern catalysis, battery technology, absorbents, medicine, targeted drug delivery, composite materials, and advanced ceramics.

This study explores the synthesis of aluminum hydroxide nanoparticles via electrochemical methods and examines their structural and physical properties. It is demonstrated that aluminum hydroxide nanocrystals can be successfully synthesized using an electrolytic method employing aluminum electrodes and an aqueous solution of sodium chloride (NaCl) as the electrolyte. The investigation revealed that synthesis parameters, particularly electrolyte temperature, significantly influence the crystallite size of the resulting aluminum hydroxide nanoparticles. The dimensions of the synthesized $\text{AlO}(\text{OH})$ and $\text{Al}(\text{OH})_3$ nanocrystals were calculated using the Scherrer and Williamson-Hall methods; additionally, internal microstrains (relative elongations) for $\text{AlO}(\text{OH})$ nanocrystals were evaluated using Williamson-Hall analysis.

Keywords: boehmite, $\text{AlO}(\text{OH})$, gibbsite, $\text{Al}(\text{OH})_3$, nanocrystals, electrochemistry, electrolytic method, XRD, Crystallite size determination.

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Introduction

The natural minerals boehmite ($\gamma\text{-AlO}(\text{OH})$) and gibbsite ($\alpha\text{-Al}(\text{OH})_3$) are primary components of bauxite ores, which are conventionally refined into alumina for metallic aluminum production via molten salt electrolysis. Beyond their metallurgical significance, recent studies [1,2] have highlighted the feasibility of utilizing natural gibbsite and boehmite as promising active materials for Li^+ intercalation, owing to their distinct layered crystal structures. At a discharge voltage of 2.7 V, their theoretical specific capacities reach 425 mAh/g for boehmite and 300 mAh/g for gibbsite – values that are more than double those of conventional commercial cathode materials.

Interest in synthetic aluminum hydroxides has grown substantially with the advancement of nanostructuring techniques. Nanostructured boehmite $\text{AlO}(\text{OH})$ and its

transitional alumina phases are highly dispersed materials with high specific surface areas and pronounced surface acid–base properties [3]. These metastable alumina derivatives typically form during the calcination of aluminum hydroxides below the temperature threshold required for transformation into thermodynamically stable corundum ($\alpha\text{-Al}_2\text{O}_3$) [4]. Due to these tailored surface characteristics, nanostructured boehmite and metastable alumina phases offer vast prospects for applications in heterogeneous catalysis, high-capacity adsorbents, structural composites, and functional ceramics [4,5].

In biomedical engineering and pharmaceuticals, the scope of application for aluminum hydroxides is notably extensive. Applications include topical formulations (powders, ointments, and pastes) for dermatological treatments; antacid formulations for peptic ulcers, hyperacid gastritis, and food poisoning; and vaccine adjuvants that enhance and prolong the immune response

[3]. Targeted drug delivery represents another promising frontier for aluminum hydroxide nanoparticles [6,7]. For instance, specialized studies have examined the delivery capabilities of hollow $\text{AlO}(\text{OH})$ nanospheres produced via microemulsion templating. Following template removal, hollow spheres with average diameters ~ 30 nm and wall thicknesses of 5-6 nm are obtained [6,7], enabling controlled drug release triggered by organic acids and osmotic pressure changes. Furthermore, nano-aluminum compounds have been utilized to engineer matrix-type tablets with prolonged release profiles, ensuring precise and long-term therapeutic dosing.

In polymer science and nanocomposite engineering, incorporating $\text{Al}(\text{OH})_3$ and related nanostructures into polymer matrices enhances their mechanical, thermal, and chemical resistance, yielding multifunctional hybrid materials. Layered double hydroxides (LDHs) and $\text{Al}(\text{OH})_3$ nanostructures have attracted significant attention as selective sorbents, drug carriers, ceramic precursors, and flame-retardant or functional additives in plastics, rubber, and paper manufacturing [8,9].

Despite their broad applicability, a major scientific and technological challenge lies in achieving precise control over the phase evolution (e.g., tuning the balance between $\text{AlO}(\text{OH})$ and $\text{Al}(\text{OH})_3$ and lattice strain during nanostructure growth under mild conditions. Conventional synthetic routes – such as hydrothermal, solvothermal, and sol-gel synthesis [9–11] – typically require elevated temperatures, high pressures, prolonged reaction times, or toxic chemical precursors and complex organic surfactants. To overcome these limitations, electrochemical synthesis offers a controllable, simple, eco-friendly, and cost-effective alternative. In contrast to multi-step chemical methods, this work demonstrates a green electrolytic synthesis route operating under ambient pressure using standard aluminum electrodes and a low-concentration aqueous sodium chloride (NaCl) solution.

The scientific novelty of this study lies in establishing a direct correlation between key synthesis parameters and both the phase composition and lattice strain state of the resulting nanostructures. Specifically, this single-step electrolytic approach allows for precise phase control: yielding a biphasic mixture of boehmite ($\text{AlO}(\text{OH})$) and gibbsite ($\text{Al}(\text{OH})_3$) at room temperature, or phase-pure boehmite at elevated temperatures, accompanied by a predictable transition from tensile to compressive lattice microstrains. Building upon our prior success in the electrochemical synthesis of various functional nanostructured materials – including including ZnO [12], CdS [13], ZnS [14], $\text{Cd}(\text{OH})_2$ [15], PbS [16], hydrozincite $\text{Zn}_5(\text{CO}_3)_2(\text{OH})_6$ [17], and the ZnS-ZnO heterostructure system [18], we investigate a green, cost-effective electrolytic route for aluminum hydroxide nanoparticle fabrication and systematically evaluate their physical and structural properties.

I. Experiment

Samples of aluminum hydroxide nanocrystals were synthesized via an electrochemical method in a 1-liter glass electrolytic cell. High-purity aluminum rods were employed as both the cathode and anode electrodes.

Aqueous sodium chloride (NaCl) solutions in distilled water at concentrations of 0.75 g/L and 2.1 g/L served as the electrolyte. The synthesis temperature was systematically varied from room temperature ($\sim 18^\circ\text{C}$) up to 100°C in different experimental runs.

The electrolytic cell was powered by a stabilized, regulated DC power supply providing a constant current density of 5×10^{-3} A/cm². The electrolysis was carried out for 3 hours, with the current polarity reversed every 30 minutes to ensure uniform electrode erosion and minimize passivation. Following synthesis, the liquid electrolyte containing the precipitate was filtered through quantitative paper filters. The retrieved powder was thoroughly washed five times with distilled water to remove residual $\text{Na}^+ \text{Cl}^-$ ions and subsequently air-dried at room temperature. For each experimental run, the net yield mass of the precipitate and the mass loss of the aluminum electrodes were precisely recorded.

X-ray diffraction (XRD) structural characterization was conducted on a DRON-4 diffractometer at room temperature. The scanning step was set to 0.05° (2θ) with a counting exposure time of 5 seconds per step. An X-ray tube with a copper anode served as the X-ray source, operating at an anode voltage of 41 kV and a current of 21 mA. To significantly reduce the intensity of K_β radiation, a nickel foil filter was used.

II. Results and discussion

Figure 1 illustrates the experimental XRD patterns of three samples recorded using CuK_α radiation. All three diffraction patterns exhibit pronounced peak broadening at full width at half maximum (FWHM), confirming the nanoscale dimensions of the crystallites. Notably, in the diffraction pattern shown in Figure 1a, alongside the substantially broadened reflections, three distinct peaks display a visibly smaller FWHM.

To perform a detailed analysis of the X-ray diffraction patterns, each peak was fitted using a Gaussian function, allowing the precise determination of its key parameters: the angular position (2θ), the peak broadening β (FWHM), and the integral intensity (I). Because X-ray diffraction analysis revealed that the synthesized samples consist of a mixture of aluminum hydroxides, namely boehmite ($\text{AlO}(\text{OH})$) and gibbsite ($\text{Al}(\text{OH})_3$), the theoretical angular positions 2θ of the reflections were calculated via Bragg's law [19] using the known interplanar d -spacings for both compounds [20]:

$$2d \sin \theta = k\lambda,$$

where d is the interplanar distance, θ is the Bragg angle, k is the order of the diffraction maximum, and λ is the wavelength of the X-ray radiation.

The results from our calculations and the processing of the experimental patterns are presented in Tables 1 and 2.

Analysis of the data in Tables 1 and 2 revealed that the diffraction patterns of all studied samples contain exclusively reflections characteristic of boehmite $\text{AlO}(\text{OH})$ and gibbsite $\text{Al}(\text{OH})_3$. Certain experimental reflections represent a superposition of two overlapping

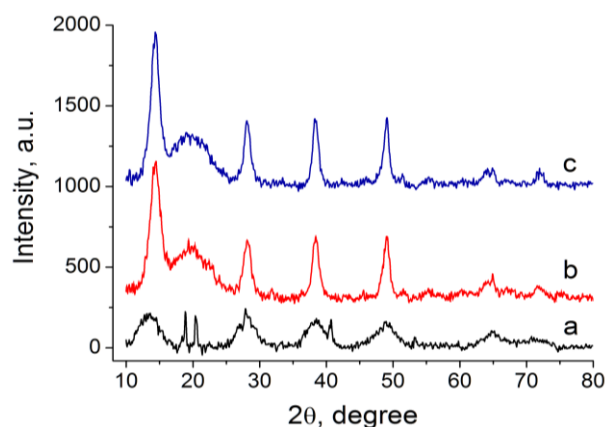


Fig. 1. X-ray diffraction patterns of samples synthesized under different conditions:

- a) $t = 18\text{ }^{\circ}\text{C}$, electrolyte concentration 0.75 g/L;
 b) $t = 93\text{ }^{\circ}\text{C}$, electrolyte concentration 0.75 g/L;
 c) $t = 93\text{ }^{\circ}\text{C}$, electrolyte concentration 2.1 g/L.

diffraction peaks. For $\text{AlO}(\text{OH})$, the reflection at an angular position of $2\theta_{\text{exp}} = 38.40^{\circ}$ is a superposition of reflections with Miller indices (140) and (031). Similarly, the diffraction maximum at $2\theta_{\text{exp}} = 49.00^{\circ}$ corresponds to the overlapping (051) and (200) planes. Furthermore, the former reflection reflection ($2\theta_{\text{exp}} = 38.40^{\circ}$) cannot be resolved even in well-crystallized polycrystalline powders (see Table 1).

In the case of $\text{Al}(\text{OH})_3$, the sample synthesized at a temperature of $18\text{ }^{\circ}\text{C}$ exhibits reflections at angular positions of $2\theta_{\text{exp}}$: 18.90° , 20.50° , and 40.70° . The latter two peaks represent superpositions of the (110)/(200) and (022)/(213) reflections, respectively. For samples obtained at 93°C , only a single reflection is observed at

$2\theta_{\text{exp}} = 19.00^{\circ}$. This peak corresponds to a superposition of three overlapping planes – (002), (110), and (200) – and is characterized by a relatively large peak width.

Similar results were reported in previous studies [11,21]. Specifically, in [11], aluminum hydroxides were synthesized via chemical precipitation from aqueous solutions of sodium aluminate (NaAlO_2) and oxalic acid ($\text{C}_2\text{H}_2\text{O}_4$). When the pH was maintained within the range of 4.0–8.5, only diffraction peaks characteristic of $\text{AlO}(\text{OH})$ were present in the XRD patterns. However, increasing the pH to 9.5–12.0 led to the co-existence of both $\text{AlO}(\text{OH})$ and $\text{Al}(\text{OH})_3$ phases. Furthermore, varying the synthesis temperature from 30°C to 93°C yielded phase-pure $\text{AlO}(\text{OH})$, whereas a biphasic mixture of $\text{AlO}(\text{OH})$ and $\text{Al}(\text{OH})_3$ was formed at temperatures between 96°C and 98°C .

In the study referenced as [21], solutions of aluminum chloride and sodium hydroxide were used for synthesis. XRD analysis revealed that at a pH of 9.6 and synthesis temperatures ranging from 60°C to 100°C , phase-pure $\text{AlO}(\text{OH})$ was obtained.

The results obtained from analyzing the experimental X-ray diffraction patterns shown in Figure 1 were used to calculate the nanocrystal sizes via the Scherrer equation [22], which relates the crystallite size to the diffraction peak broadening:

$$D = \frac{0.89\lambda}{\beta \cos \theta}$$

In this equation, λ represents the wavelength of the X-ray radiation, β corresponds to the structural peak broadening (FWHM), and θ is the Bragg diffraction angle. The corrected FWHM value was calculated using the

Table 1.

Angular positions of reflections of $\text{AlO}(\text{OH})$					
Boehmite $\text{AlO}(\text{OH})$					
d , nm	I	hkl	$2\theta_{\text{calc}}$	$2\theta_{\text{exp}}$	I_{exp}
0.61	100%	020	14.52°	14.3°	100%
0.3164	65%	120	28.2°	28.2°	41%
0.2346	55%	140; 031	38.37°	38.4°	36%
0.186	30%	051	48.97°	49.0°	39%
0.185	25%	200	49.25°		
0.166	14%	151	55.34°	-	-
0.1454	16%	231	64.09°	64.0°	9%
0.1432	10%	002	65.04°	65.0°	4%
0.1312	16%	521	71.97°	72.0°	7%

Table 2.

Angular positions of reflections of $\text{Al}(\text{OH})_3$					
Gibbsite $\text{Al}(\text{OH})_3$					
d , nm	I	hkl	$2\theta_{\text{calc}}$	$2\theta_{\text{exp}}$	I_{exp}
0.485	320%	002	18.29°	18.9°	-
0.437	50%	110	20.32°	20.5°	-
0.432	25%	200	20.56°		
0.3306	16%	112	26.96°	-	-
0.3112	8%	103	28.69°	-	-
0.2454	25%	021	36.62°	-	-
0.242	20%	004	37.15°	-	-
0.2388	25%	311	37.67°	-	-
0.2244	10%	022; 213	40.19°	40.7°	-

following formula:

$$\beta = \sqrt{(\beta_1^2 - \beta_2^2)},$$

where β_1 is the experimental FWHM value of the X-ray reflection, and β_2 is the instrumental peak broadening. The instrumental broadening was determined by analyzing the XRD patterns of *standard materials* (powders of Si and Al_2O_3) measured under identical experimental conditions.

Considering the above information, only the (020), (120), (231), (002), and (521) reflections were used to determine the sizes of $\text{AlO}(\text{OH})$ nanoparticles, while only the (002) reflection was used for $\text{Al}(\text{OH})_3$. For boehmite, the sizes were determined using five reflections, and the arithmetic mean of these results was calculated. The dimensioning of the nanocrystals are presented in Table 3.

Based on the above analysis, only the (020), (120), (231), (002), and (521) reflections were selected to determine the crystallite sizes of the $\text{AlO}(\text{OH})$ nanoparticles, whereas only the (002) reflection was used for $\text{Al}(\text{OH})_3$. For boehmite, the crystallite sizes were determined using these five reflections, and their arithmetic mean was calculated. The calculated dimensions of the nanocrystals are summarized in Table 3.

Table 3.

The dimensioning of the nanocrystals

Sample synthesis conditions	Nanocrystal sizes, nm	
	$\text{AlO}(\text{OH})$	$\text{Al}(\text{OH})_3$
$t = 18^\circ\text{C}$, electrolyte concentration 0.75 g/L	2.7	48.4
$t = 93^\circ\text{C}$, electrolyte concentration 0.75 g/L	9.8	1.3
$t = 93^\circ\text{C}$, electrolyte concentration 2.1 g/L	10.7	1.3

From Table 3, it is evident that the size of $\text{AlO}(\text{OH})$ nanoparticles increases with rising temperature, while the size of $\text{Al}(\text{OH})_3$ nanocrystals decreases sharply. As noted in reference [23], the peak broadening at half the maximum intensity of X-ray reflections can increase due to two factors: a reduction in crystal size and the presence of mechanical stresses. The Scherrer equation accounts only for the decrease in crystal size. Therefore, to accurately determine both the size and the mechanical stresses affecting $\text{AlO}(\text{OH})$ nanocrystals, we employed the Williamson–Hall method [24].

In the Williamson–Hall method, the FWHM of a reflection β is described by the following relation:

$$\beta = \frac{0.89 \lambda}{D \cos \theta} + 4\varepsilon \tan \theta,$$

where ε represents the lattice microstrain (relative elongation).

In this equation, the first term accounts for the peak broadening due to the crystallite size effect, whereas the second term represents the broadening contribution arising from lattice microstrains (mechanical stresses). The resulting relationship is expressed as:

$$\beta \cos \theta = \frac{0.89 \lambda}{D} + 4\varepsilon \sin \theta.$$

When plotted in coordinates of $4 \sin \theta$, $\beta \cos \theta$, the data points yield a linear relationship. Applying a linear least-squares fit allows for the determination of both the crystallite size (D) from the y -intercept and the lattice microstrain (ε) from the slope. Furthermore, the sign of the microstrain reveals the nature of the internal mechanical stresses, where positive values denote tensile stress and negative values indicate compressive stress. The linear regression shown in Figure 2 was calculated using the least-squares method.

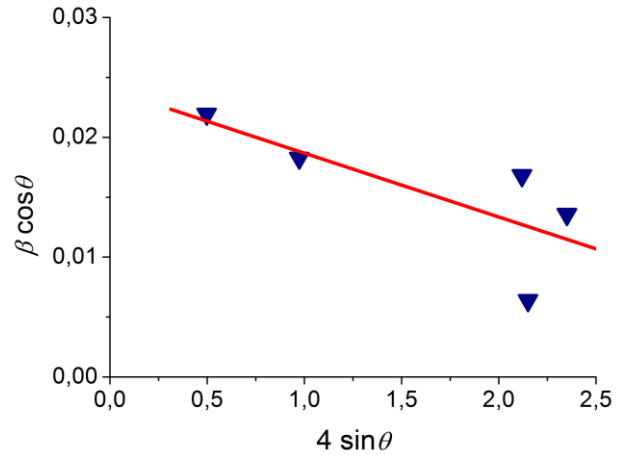


Fig. 2. Williamson–Hall plot for the $\text{AlO}(\text{OH})$ nanocrystal sample synthesized at an electrolyte temperature of 93°C and a concentration of 2.1 g/L.

By applying the Williamson–Hall method to the $\text{AlO}(\text{OH})$ nanocrystals, their average crystallite sizes and lattice microstrains (relative elongations) were determined (Table 4).

Table 4.

Crystallite sizes (D) and lattice microstrains (ε) of the $\text{AlO}(\text{OH})$ nanocrystals determined via the Williamson–Hall method

Sample synthesis conditions	Nanocrystal sizes D , nm	Relative elongation, ε
$t = 18^\circ\text{C}$, electrolyte concentration 0.75 g/L	3.5	0.003
$t = 93^\circ\text{C}$, electrolyte concentration 0.75 g/L	5.5	-0.003
$t = 93^\circ\text{C}$, electrolyte concentration 2.1 g/L	5.7	-0.005

The negative sign of the lattice microstrain (ε) indicates the presence of internal compressive stresses in the samples. Furthermore, increasing the synthesis temperature induces a transition from tensile to compressive stress state.

Conclusions

1. Aluminum hydroxide nanocrystals were successfully synthesized via an electrochemical method using aluminum electrodes and an aqueous sodium

chloride (NaCl) electrolyte.

2. The synthesized products consist of a biphasic mixture of boehmite $\text{AlO}(\text{OH})$ and gibbsite $\text{Al}(\text{OH})_3$ nanocrystals.

3. The synthesis temperature plays a critical role in controlling the nanocrystal size. Increasing the temperature from room temperature (18 °C) to 93 °C leads to an increase in the crystallite size of $\text{AlO}(\text{OH})$, whereas the size of the $\text{Al}(\text{OH})_3$ nanocrystals decreases sharply.

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Електролітичний метод синтезу нанокристалів гідроксиду алюмінію

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В останні роки зріс інтерес до синтетичних гідроксидів алюмінію в наноструктурній формі. Метастабільні фази оксиду алюмінію є надзвичайно важливими технологічними матеріалами з високорозвинutoю поверхнею, що створює перспективи застосуванням у сучасних каталізаторах, акумуляторах, абсорбентах, медицині, цільовій доставці ліків, композитних матеріалах, кераміці тощо.

Метою статті було синтезувати наночастинки гідроксиду алюмінію за допомогою електрохімічних методів і вивчити їхні фізичні властивості. Авторами було отримано нанокристали гідроксиду алюмінію $\text{AlO}(\text{OH})$ та $\text{Al}(\text{OH})_3$ електролітичним методом із застосуванням алюмінієвих електродів і розчину кухонної солі як електроліту. Показано, що параметри синтезу, зокрема температура, значно впливають на розмір нанокристалів гідроксиду алюмінію. Розміри отриманих нанокристалів $\text{AlO}(\text{OH})$ та $\text{Al}(\text{OH})_3$ визначали за методами Шеррера та Вільямсона-Холла.

Ключові слова: беміт, $\text{AlO}(\text{OH})$, гібсит, $\text{Al}(\text{OH})_3$, нанокристали, електрохімія, електролітичний метод, рентгенівська дифракція, визначення розмірів.