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IMPACT OF ANNEALING CONDITIONS ON THE CRYSTAL QUALITY OF HYDROTHERMAL TREATED γ-AlOOH

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Abstract

Among different material families, oxide hydroxides have emerged as promising cadidates for different advanced applications such as catalysis, adsorption, and ceramics. In this paper, the synthesis of Boehmite (γ-AlOOH) powder via the hydrothermal technique and the influence of annealing temperature and duration on its crystal structure is reported. Different annealing temperatures (100, 200 and 300^{o}C) and durations (1, 2, and 3 hours) were applied on synthesized AlOOH powder. The crystal structure and crystallite size of the samples were evaluatedvia X-ray diffraction patterns. The proportion of atoms in the powder sample was ascertained utilizing Energy Dispersive X-ray Spectroscopy. The sample's molecular compositionswere meticulously investigated using Fourier-transform infrared spectroscopy. Experimental results indicate that AlOOH generated via the hydrothermal technique exhibits crystallite sizes between 8.27 nm and 15.31 nm. Besides, The hydrothermal time was significantly reduced, and the crystal quality and properties of AlOOH powder were improved by using precursors such as $Al(NO_3)_3$, CTAB, and $NH_3{\cdot}H_2O$ in combination with annealing conditions. The 300 °C samples have the highest particle size and minimal microstrain, signifying a solid crystal structure with few imperfections.

Keywords: *aluminium, annealing conditions, crystal structures, hydrothermal, oxide hydroxide, powder.*

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ẢNH HƯỞNG CỦA ĐIỀU KIỆN Ủ ĐẾN CHẤT LƯỢNG TINH THỂ CỦA γ-ALOOH XỬ LÝ THỦY NHIỆT

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Tóm tắt

Trong số các họ vật liệu khác nhau, oxit hydroxit đã nổi lên như những ứng dụng đầy hứa hẹn cho các ứng dụng tiên tiến khác nhau như xúc tác, hấp phụ và gốm sứ. Bài báo này trình bày quá trình tổng hợp bột Boehmite (γ-AlOOH) bằng kỹ thuật thủy nhiệt và ảnh hưởng của nhiệt độ và thời gian ủ lên cấu trúc tinh thể của nó. Các nhiệt độ ủ khác nhau (100, 200 và 300oC) và thời gian ủ (1, 2 và 3 giờ) đã được áp dụng trên bột AlOOH tổng hợp.. Cấu trúc tinh thể và kích thước tinh thể của các mẫu được đánh giá thông qua các mẫu nhiễu xạ tia X. Tỷ lệ các nguyên tử trong mẫu bột được xác định bằng phương pháp quang phổ tia X tán xạ năng lượng. Thành phần phân tử của mẫu được nghiên cứu tỉ mỉ bằng phương pháp quang phổ hồng ngoại biến đổi Fourier. Kết quả thực nghiệm cho thấy AlOOH được tạo ra bằng kỹ thuật thủy nhiệt thể hiện kích thước tinh thể từ 8,27 nm đến 15,31 nm. Bên cạnh đó, Thời gian thủy nhiệt được giảm đáng kể, chất lượng tinh thể và tính chất của AlOOH powder bằng việc sử dụng các tiền chất phản ứng như $Al(NO_3)_3$, CTAB, $NH_3·H_2O$ và kết hợp với điều kiện nung. Các mẫu ở nhiệt độ 300 °C có kích thước hạt lớn nhất và độ biến dạng vi mô tối thiểu, cho thấy cấu trúc tinh thể rắn với ít khuyết tật.

Từ khóa: *bột, cấu trúc tinh thể, điều kiện ủ, nhôm oxit hydroxit, thủy nhiệt.*

1. Introduction

Boehmite (γ-AlOOH) is a natural component of bauxite ore, including kaolinite, gibbsite, and diaspore. This material possesses a multitude of exceptional properties, making it extensively applicable in a variety of fields, including ceramics, metallurgy, fuel cells, flame retardants , LEDs, coating materials, and refined catalysts (Bai et al., 2014; Islama et al., 1818; Laachachi et al., 2009; Q. Liu et al., 2008; Oliveira et al., 2014; Raybaud et al., 2001; C. Zhang et al., 2021; Zhong et al., 2019). The hydrothermal treatment of aluminum salts or aluminum trihydroxide has recently enabled the synthesis of Boehmite with a high crystallinity (Ohta et al., 2017). The structure, morphology, and size of AlOOH continue to pose a substantial challenge, and their synthesis and regulation are essential for optimizing the practical application potential of this material.

Significant attention is being paid to the production of γ-AlOOH nanomaterials by the research community. Numerous solution-based synthesis methods have been devised, including hydrothermal, precipitation, and sol-gel techniques (Lee et al., 2005; Park et al., 2005). The morphology of Boehmite is significantly influenced by the pH of the solution. In an acidic environment, this material frequently generates 1D structures, such as nanorods or nanofibers, whereas in an alkaline environment, it frequently generates 2D structures, such as nanosheets, or 3D structures, such as nanotubes (Chen et al., 2007; Chen & Lee, 2007; He et al., 2009; Pardo, Montoya, et al., 2015). The pH value can be used to effectively regulate this diversity during the hydrothermal procedure (Chen et al., 2008). Furthermore, the final size and morphology of Boehmite are also substantially influenced by the temperature and aging time involved in the synthesis process (Pardo, Montoya, et al., 2015; Pardo, Serrano, et al., 2015).

In the research conducted by Fang Wang et al. (Wang et al., 2021), γ-AlOOH was produced through a hydrothermal process involving aluminum sulfate, CTAB (with a molar ratio of 5:1), and urea in an aqueous solution maintained at 165°C for 24 hours. The resulting product consisted of γ-AlOOH hollow microspheres characterized by a highly active surface and composed of hexagonal nanoflake structures that predominantly grew along the (020) crystallographic plane, with a crystal size approximately 13.8 nm. The authors highlighted that CTAB was essential in directing the morphology of γ-AlOOH, indicating its potential uses in catalysis, as catalyst supports, and as adsorbents. Similarly, Li et al.(2008) synthesized γ-AlOOH nanofibers via a hydrothermal method lasting 6 hours at 200 °C, using $AlCl_3 \cdot 6H_2O$, CTAB, distilled water, and $NH_3 \cdot H_2O$, adjusting the pH to 8 and 10. Their findings revealed that γ-AlOOH primarily grew along the (020) plane, with improved crystal quality at pH 8 and a crystal size around 11 nm. The nanofibers produced were uniform and pure, with an average diameter of 10 nm and lengths ranging from 150 to 250 nm. The study also demonstrated the potential application of AlOOH nanofibers in the ceramics industry.

In this investigation, our primary objective is to investigate the impact of temperature and annealing time on the formation of powder Boehmite. At the same time, the hydrothermal time is significantly reduced, making it more suitable for industrial-scale production, while also improving the crystal quality and properties of its. The changes in crystal structure, particle size, and properties of Boehmite have been extensively investigated. The findings not only elucidate the material's formation mechanism but also establish a foundation for the creation of a variety of Boehmite types with optimal properties, thereby enabling the development of practical applications that are pervasive.

2. Experiments

Boehmite powder (AlOOH) was synthesized from the precursor $AlCl_3.6H_2O$ and Cetyltrimethylammonium Bromide (CTAB) at a molar ratio 80 : 1 ($AlCl_3\cdot 6H_2O$: CTAB) using the hydrothermal method. Initially, a homogeneous solution was produced by dissolving $AlCl_3$. $6H_2O$ and CTAB in distilled water and stirring it uniformly. Subsequently, the NH_4OH solution was gradually incorporated into the aforementioned mixture until the pH reaches the desirable level (8.0) to produce a gelatinous precipitate of $Al(OH)_3$. The stirring process was then maintained for an additional 10 minutes. The obtained $Al(OH)_3$ (with controlled pH and CTAB) would be deposited in a hydrothermal tube and subjected to a hydrothermal treatment at 200°C for 6 hours after complete dissolution. Under these conditions, $Al(OH)_3$ undergoes a solid-state phase transformation into γ-AlOOH (boehmite). Through hydrothermal, AlOOH is fabricated, as illustrated by equations 1 and 2 (Li et al., 2006; X. M. Sun et al., 2003):

$$AlCl_3 + 3NH_4OH \rightarrow Al(OH)_3\downarrow + 3NH_4Cl \qquad (1)$$

$$Al(OH)_3 \rightarrow AlOOH + H_2O \qquad (2)$$

The sample would be washed and the powder obtained by centrifuging and sonicating the product five times at the conclusion of the hydrothermal process. The samples were dried at 80°C. Finally, the dried samples were subjected to annealing under different temperatures (100, 200 and 300 ºC) and durations (1, 2 and 3 hours). The structural properties were analyzed by X-ray diffraction (XRD, D8-ADVANCE). The elemental composition and ratios were analyzed using Energy Dispersive X-ray Spectroscopy (EDX, S-4800, Hitachi). The identification of functional groups was carried out through Fourier-transform infrared spectroscopy (FTIR, Nicolet 6700, Thermo).

3. Results and Discussion

The X-ray diffraction (XRD) patterns of the samples obtained under various processing conditions are depicted in Figure 1. We present the standard diffraction peaks of AlOOH from the databases of PDF#21-1307, PDF#48-0890, and PDF#49-0133. The pronounced (020) peaks were evident in all AlOOH samples. The crystal structure of Boehmite preferentially develops along the (020) plane, as evidenced by the diffraction angle of $2\theta \sim 14.5^o$ for the (020) plane of the AlOOH sample (Zhang et al., 2018). In addition, the peaks (110), (120), (021), (031), and (200), corresponding to the diffraction angles 2θ at 23.2°, 28.6°, 33.0°, 38.7° and 49.5° are also significant characteristics of Boehmite. The 1-hour sample demonstrates distinct diffraction peaks at 100 °C, which suggests a high degree of crystallinity. The (021) peak is considerably enhanced when the annealing time is increased to 2 hours and 3 hours, which suggests a change in the crystal structure. The crystallization process continues, and the crystal particles reorganize into more stable structures. The diffraction peaks are significantly sharper at processing temperatures of 200°C and 300°C, particularly at the (020) and (120) positions. This suggests that the crystal structure of Boehmite is preferentially developed in these crystallographic orientations. The Boehmite phase maintains the fundamental crystal structure of the material, as evidenced by the minimal variation in the peaks at the 2θ positions across various thermal treatment conditions. This is in accordance with the development direction of the network surface, which has been investigated and documented in prior publications (Karouia et al., 2016; Rani & Sahare, 2015).

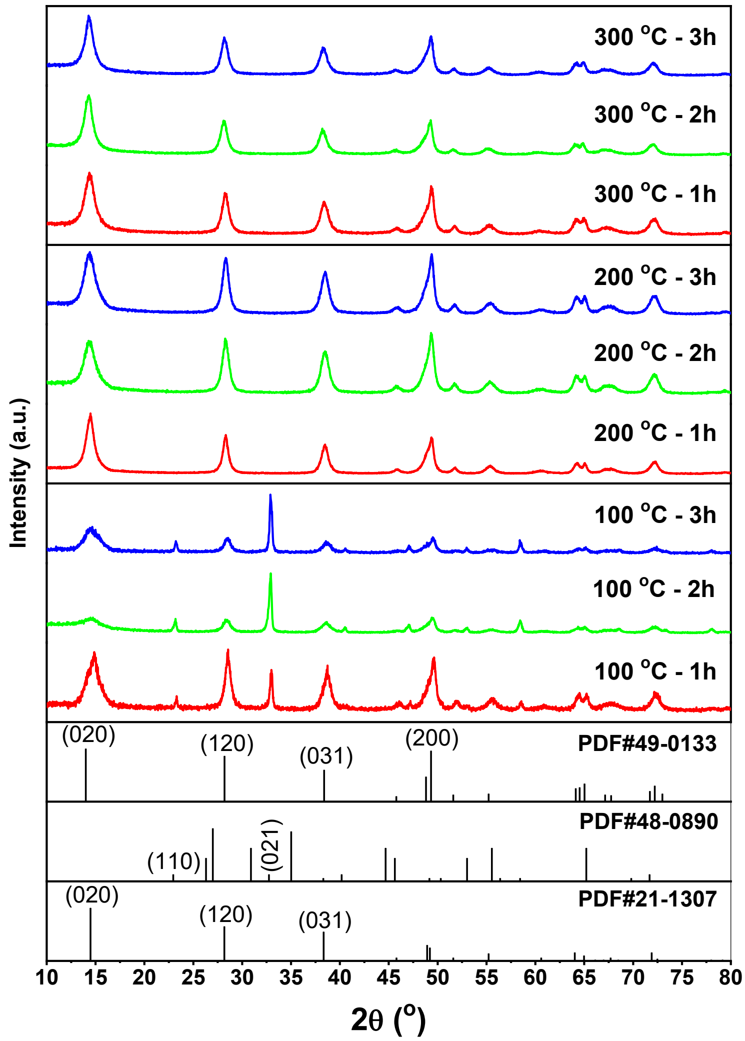

Figure 1. X-ray diffraction patterns of Boehmite at temperatures of 100°C (a), 200°C (b), 300°C (c) at 1 hour, 2 hours, 3 hours.

The crystallite size changes under various temperature conditions are illustrated in Figure 2. Sample 101 (annealed at 100 ºC in 1 hour) exhibits the lowest full width at half maximum (FWHM) (0.73º) at 100 °C, which corresponds to the greatest crystallite size (10.99 nm). In sample 102, the crystallite size decreases to 8.27 nm as the annealing time increases, while it marginally increases to 9.70 nm in sample 103. The 2h sample's substantial reduction in crystallite size can be attributed to the Boehmite particles' propensity to restructure or disintegrate, resulting in a reduction in crystallite size (Y. Liu et al., 2008). The largest particle size (14.00 nm) is corresponding to the lowest FWHM (0.57º) at 200 °C. The particle size decreases gradually over time, with the 2h sample measuring 12.92 nm and the 3h sample measuring 12.67 nm. The FWHM increases from 0.52º (1h sample) to 0.61º (3h sample) when the temperature reaches 300 °C, suggesting that the structure commences the phase transition process with longer sintering times or progressively stabilizes. This is also indicative of the significant increase in grain size at elevated temperatures, which is a result of the phenomenon of diffraction peak broadening or narrowing in accordance with the Scherrer equation. The FWHM is inversely proportional to crystallite size (Jiao et al., 2010).

The values such as crystallite size (D), micro-strain (ε), and dislocation density (δ) are calculated using the formulas (3, 4 and 5) (Hang Pham et al., 2025; Pham et al., 2025):

$$D = \frac{0.9\lambda}{FWHM.\cos(\theta)} \quad (3)$$

$$\delta = \frac{1}{D^2} \quad (4)$$

$$\varepsilon = \frac{FWHM}{4\tan(\theta)} \quad (5)$$

Table 1. Structural property parameters of Boehmite powder samples at the (020) plane.

Sample	Annealing temperature (°C)	Annealing time (hour)	2θ (°)	FWHM (°)	d-spacing (Å)	Crystallite size (nm)	Micro-strain (10^{-2})	Dislocation density (10^{-3} nm^{-3})
B-101	100	1	14.87	0.73	5.96	10.99	2.44	8.27
B-102		2	14.73	0.97	6.01	8.27	3.27	14.64
B-103		3	14.67	0.83	6.04	9.70	2.80	10.62
B-201	200	1	14.48	0.57	6.11	14.00	1.97	5.10
B-202		2	14.42	0.62	6.14	12.92	2.14	5.99
B-203		3	14.41	0.63	6.14	12.67	2.18	6.23
B-301	300	1	14.36	0.52	6.16	15.31	1.81	4.27
B-302		2	14.32	0.59	6.18	13.55	2.05	5.45
B-303		3	14.28	0.61	6.20	13.19	2.11	5.75

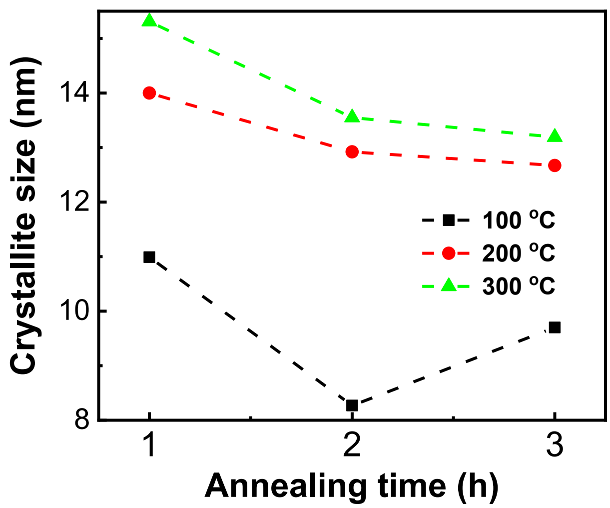

Figure 2. Variations in the crystallite size of Boehmite with different annealing durations at 100, 200, and 300 °C.

Additionally, Figure 2 illustrates that the Boehmite particle size tends to increase as the sintering temperature rises, despite the fact that the annealing time remains constant. This can be accounted for by the fact that the thermal energy delivered at elevated temperatures is adequate to induce particle agglomeration or growth into larger particles. This is a prevalent occurrence during thermal treatment, as small particles tend to recrystallize and merge to form larger particles in order to reduce surface energy (Kang et al., 2015). Furthermore, managing the annealing temperature and time promotes an increase in crystallite size, resulting in better crystal quality. The atoms are arranged in a more orderly arranged, which decreases micro-strain and dislocation density within the Boehmite crystal lattice. The XRD results indicate that the crystal quality of Boehmite is optimal when thermally treated at 300 °C for 1 hour.

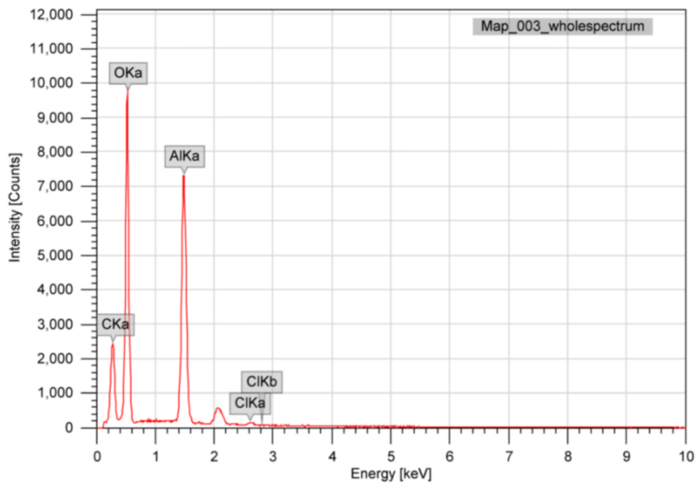

Figure 3. EDX spectrum of the γ-AlOOH sample.

Figure 3 presents the EDX spectra of the γ-AlOOH sample subjected to 300°C for one hour. The distinctive peak of aluminum (Al) is located at an energy level of around 1.5 keV, whereas the peak of oxygen (O) is observed at around 0.5 keV, signifying the presence of oxygen inside the structure of the γ-AlOOH particles. The results of the EDX quantitative analysis of the elemental components are showed In Table 2, with the mass percentage of Oxygen accounting for 44.21% and Aluminum for 30.09%. This result indicates that the two main elements in the γ-AlOOH structure are relatively evenly distributed, consistent with the chemical composition and characteristic structure of the material.

Table 2. Quantitative analysis table of the elements in the γ-AlOOH sample.

Element	Line	Mass %	Atom %
C	K	25.00 ± 0.11	34.81 ± 0.15
O	K	44.21 ± 0.18	46.21 ± 0.19
Al	K	30.09 ± 0.18	18.65 ± 0.11
Cl	K	0.70 ± 0.05	0.33 ± 0.02
Total		100.00	100.00

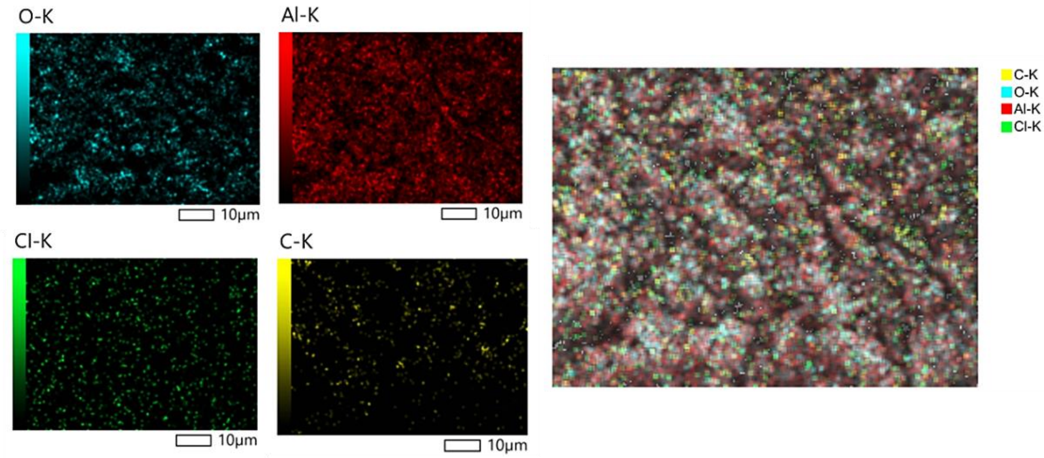

Figure 4. Distribution of aluminum and oxygen elements in the sample.

Figure 4 shows the distribution of elements in the obtained γ-AlOOH sample. Observing the distribution of elements in the sample, including aluminum (red spots) and oxygen (blue spots) as the dominant components. Additionally, the presence of some impurities such as carbon (yellow spots) and chlorine (green spots) interspersed within the sample is noted. These impurity elements may originate from the remnants of the initial precursors in the reaction, form during the sample annealing process, or be unwanted elements from the external environment. The EDX analysis results show that the distribution density of the main elements in the γ-AlOOH sample is quite uniform, but the presence of impurities in the sample remains a factor that needs to be addressed and controlled to enhance the purity and quality of the material during the synthesis process.

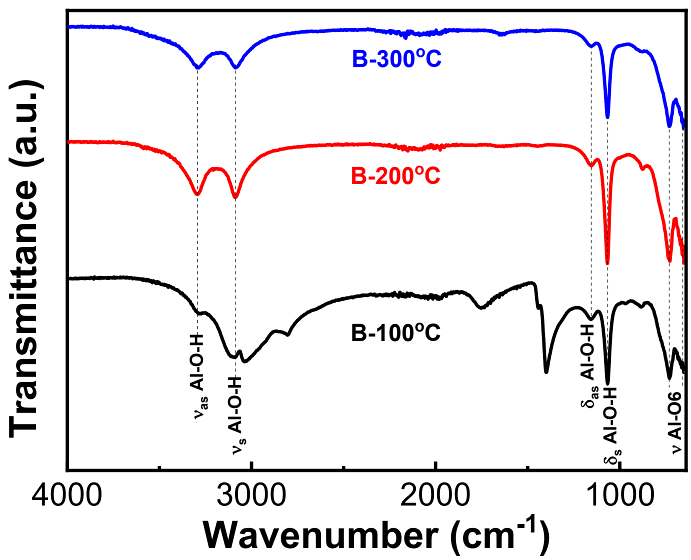

Figure 5. FT-IR spectrum of γ-AlOOH at different annealing temperatures.

The FT-IR spectra of samples heated at temperatures of 100°C, 200°C, and 300°C in the wavelength range of 600 - 4000 cm^{-1} are depicted in Figure 5. Observing The absorption bands at 3293, 3086, 1153, 873, 727, and 645 cm^{-1} are in agreement with the values that have been previously investigated and published (Sun et al., 2015). The stretching vibrations of ν_{as} (Al)–OH and ν_s(Al)O–H are responsible for the strong absorption peaks at 3277 and 3083 cm⁻

[1]. The bending vibrations δ_s Al–O–H and δ_{as} Al–O–H of Boehmite are identified by the absorption peak at 1067 cm^{-1} and the shoulder peak around 1153 cm^{-1}, respectively. This suggests that the hydroxyl group interacts with aluminum atoms in the structure. The characteristic vibrations of AlO6 are represented by the bands between 600 and 750 cm^{-1} (Chicot et al., 2011; Choi et al., 2018). Additionally, the sample treated at 100 °C exhibits a strong absorption peak at 1398 cm^{-1}, which may be attributed to water molecules bound to tetrahedral coordination aluminum ions or to residual nitrate, as a result of the AlOOH formation reaction (Yan et al., 2017; Y. Zhang & Binner, 2002). Thus, the AlOOH sample at 100 ºC still contains other compounds or the AlOOH structure has not yet reached a stable state, suggesting that the crystallization process is not yet complete. This results in discrepancies in the FT-IR spectrum, consistent with the XRD analysis. The absorption peaks in the FT-IR spectrum become more stable and clear as the temperature increases to 200 °C and 300 °C, as the crystallization process of the sample continues to develop. As the sintering temperature increases, the structure of Boehmite endures gradual changes, as evidenced by the shift and intensity of the FT-IR peaks, as demonstrated by FT-IR spectral analysis. These alterations are indicative of the dehydration and phase transition of Boehmite as the temperature rises. The research indicates that temperature and time are critical elements in regulating the creation of full AlOOH structures while concurrently eliminating undesirable impurities during synthesis.

4. Conclusion

In this report, the Boehmite material used $AlCl_3$. $6H_2O$ and CTAB as precursors of the reaction were synthesized using the hydrothermal method. When the temperature increases, the crystallization process grows significantly, the crystallite size increases, but under the same temperature conditions with different time intervals, the crystallite size decreases. Through FT-IR spectra, strong absorption peaks at 3277 and 3083 cm^{-1} are assigned to the stretching vibrations of ν_{as}(Al)–OH and ν_s(Al)O–H. The obtained results show that the vacuum thermal treatment process has significantly affected the properties of AlOOH. Optimizing the temperature and annealing time conditions is a crucial factor in controlling and optimizing the properties of these materials in practical applications.

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