

# Preparation and Properties of Pseudo-boehmite Obtained from High-Alumina Fly Ash by a Sintering–CO<sub>2</sub> Decomposition Process

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A sintering–CO<sub>2</sub> decomposition process for preparation of pseudo-boehmite is proposed for clean valorization of high-alumina fly ash. The effects of different synthesis conditions on the crystal structure and textual properties of the pseudo-boehmite were studied systematically. The results showed that the main product was NaAlCO<sub>3</sub>(OH)<sub>2</sub> when the decomposition terminal pH was below 9.5, and the optimal terminal pH for the pseudo-boehmite (PB) product was 10.5. The optimal aging time was 4 h, and the peptization ratio of PB reached 97.5% when the aging temperature was 90°C. Under decomposition conditions of 30 g/L initial alumina concentration in liquid phase and temperature of 40°C, followed by 4 h of aging treatment, the specific surface area of PB was 425 m<sup>2</sup>/g. The pore volume of PB product reached 0.6920 cm<sup>3</sup>/g at decomposition conditions of 40 g/L and 30°C.

## INTRODUCTION

High-alumina fly ash (HAFA) is mainly generated in Northwest China, being regarded as one of the most important energy bases.<sup>1</sup> Annually, some 50 million metric tons of fly ash are generated, only 20% of which is comprehensively utilized. This results in significant soil, air, and water pollution.<sup>2</sup> Fly ash has an alumina content of over 45 wt.%, and the total accumulated amount is approximately 15 billion metric tons.<sup>3</sup>

Annual production of alumina in China exceeded 60 million tons by the end of 2017. Although bauxite resources in China are abundant, reserves of high-grade bauxite are limited, with the majority (> 95%) being diasporic bauxite with high contents of aluminum and silica.<sup>4–6</sup> The proportion of bauxite resources with mass ratio of Al<sub>2</sub>O<sub>3</sub> to silica (A/S) higher than 7 is 29.8%, with A/S between 4 and 7 is 45.9%, and with A/S less than 4 is 24.3%. This shortage of available bauxite resources is one of the key problems faced by the aluminum industry, and

HAFA is regarded as a valuable and cleaner mineral resource as a substitute for bauxite for alumina production.

Considerable work has been carried out on alumina extraction from HAFA.<sup>7–9</sup> However, the process for alumina recovery has always been complex. This is because HAFA is formed at very high temperatures and is mainly composed of mullite, corundum, and amorphous silica.<sup>10</sup> In previous studies, removal of amorphous silica was the main method used to improve the A/S ratio, whereas mullite as another major source of silica in HAFA has been considered nonreactive owing to the stability of Al–O–Si bonds.<sup>11</sup>

Alkali sintering and acid leaching methods have been demonstrated to be effective for separation of Al from Si during alumina production using coal fly ash.<sup>12–15</sup> A number of water or alkaline leaching processes for recovery of alumina have been reported and can be broadly classified into sintering, hydrochemical, acid, and some other special processes.<sup>9</sup> The sintering processes usually couple a

reaction of coal ash with a sintering agent powder to form soluble alumina compounds under high temperature.<sup>12</sup> The alkali sintering process involves the following steps: desilication of fly ash, calcination, digestion, and carbon dioxide decomposition.<sup>13</sup> One of the greatest advantages of this technology is its solid industrial foundation.<sup>14</sup> Fundamental knowledge of and experience with the sintering process can be drawn on to improve the process. It also has the advantage of being a simple process that uses mature equipment systems.<sup>15</sup> However, its disadvantages include high energy consumption and cost. Therefore, a possible economical way to solve this problem is to produce high-value alumina-containing products from HAFA.

Pseudo-boehmite ( $\alpha'$ -AlOOH, PB) is poorly crystallized boehmite, consisting of  $\text{Al}(\text{O},\text{OH})_6$  octahedral layers with crystal water content of 1.25–2. This material has a large pore volume and high specific surface area, and also displays strong adhesion, good peptization, and thixotropic gelling behavior. PB is the main raw material for preparation of a activated alumina ( $\gamma$ - $\text{Al}_2\text{O}_3$ ) for use as a catalyst support. Many methods for preparation of pseudo-boehmite have been reported,<sup>16</sup> among these, mainly the neutralization method and organic alcohol hydrolysis process can reach actual industrialization. In the neutralization method, different aluminum-containing raw materials and corresponding precipitation agents are subject to a neutralizing reaction, generating amorphous aluminum hydroxide under certain conditions, followed by a multistep procedure to obtain pseudo-boehmite.<sup>17–20</sup> The organic aluminum alkoxide hydrolysis method is based on the characteristics of aluminum and can generate organic compounds, and under certain conditions form organic aluminum alkoxides, such as  $\text{Al}(\text{OR})_3$  (or alkoxy) first, followed by pseudo-boehmite through a hydrolysis reaction.<sup>21,22</sup>

The PB materials prepared by the aforementioned methods have the characteristics of high chemical purity, fewer impurity phases, high crystallinity, and perfect peptization. However, it is difficult to obtain such high-quality products when using a simple or low-cost process.

Previous studies by Wefers and Misra on the gel method for special alumina-containing products provided a comprehensive and macroscopic overview of the relationship between aging conditions and the structure of the product,<sup>23</sup> but the effects of the gelation conditions on the structure, surface, and pore characteristics of the product require further investigation.

The gelation behavior of colloidal and polymeric gels, in general, and the influence of variables such as the solvent type and concentration, pH, catalyst concentration, temperature, etc. on several oxide systems including silica have also been considered by others.<sup>24</sup> Previous research illustrates the feasibility of the  $\text{CO}_2$  decomposition method for

preparing PB products.<sup>25</sup> In the work presented herein, PB was prepared by a sintering–carbonation precipitation method using HAFA as raw material. The effects of different synthesis conditions on the crystal structure and textual properties of the pseudo-boehmite were studied systematically.

## EXPERIMENTAL

### Raw Material

The HAFA used in this research was from Inner Mongolia, China. After desilication and sintering treatment, the mass contents of alumina, silica, sodium oxide, and calcium oxide in the sintering clinker were 29.66%, 16.85%, 19.12%, and 27.21%, respectively, the calcium ratio ( $\text{CaO}/\text{SiO}_2$  molar ratio) was 1.73, and the alkali ratio ( $\text{Na}_2\text{O}/\text{Al}_2\text{O}_3$  molar ratio) was 1.06. Moreover, the main impurities in the sintering clinker were magnesium oxide (1.5%), ferric oxide (1.43%), and others (4.23%).

In this experiment, NaOH and  $\text{Al}(\text{OH})_3$  analytical reagents were used to prepare the decomposition medium, and industrial-grade (purity > 99%)  $\text{CO}_2$  was used in the carbonation precipitation experiments.

### Experimental Procedures

There are three main steps in this process (Fig. 1): (1) the dissolution and desilication process of coal fly ash sintering clinker, (2) carbonation decomposition of sodium aluminate solution, and (3) treatment of the decomposition product followed by an aging process to produce PB with better crystallinity, larger pore size, and higher specific surface area. Moreover, the main content of the dissolution residue of this process is  $\text{CaSiO}_3$ , which can be used as a raw material in the cement industry. Clean and valuable utilization of HAFA is achieved using this process.

The sintering clinker was digested with the decomposition medium at temperature of 80°C for dissolution time of 60 min at dissolution liquid–solid ratio of 4. The alumina and alkali dissolution ratio exceeds 80% under these conditions.

To obtain pseudo-boehmite products that meet the purity requirements (Na, Fe, and Si content below 0.3%), digestion of coal fly ash sintering clinker was needed to remove impurities. Silicon oxide is the most abundant impurity in sodium aluminate solution. Therefore, the silicon index ( $\text{Al}_2\text{O}_3/\text{SiO}_2$  mass ratio in the liquid phase) is an important parameter to measure the quality of the sodium aluminate solution. The stripping time in the autoclave desilication was 120 min, the desilication temperature was 170°C, the amount of silicon slag added was 100 g/L, the deep desilication time was 120 min, the desilication temperature was 90°C, the CaO content was 15 g/L, and the silicon index of the green liquor reached 838, all meeting the requirements for production of pseudo-boehmite.

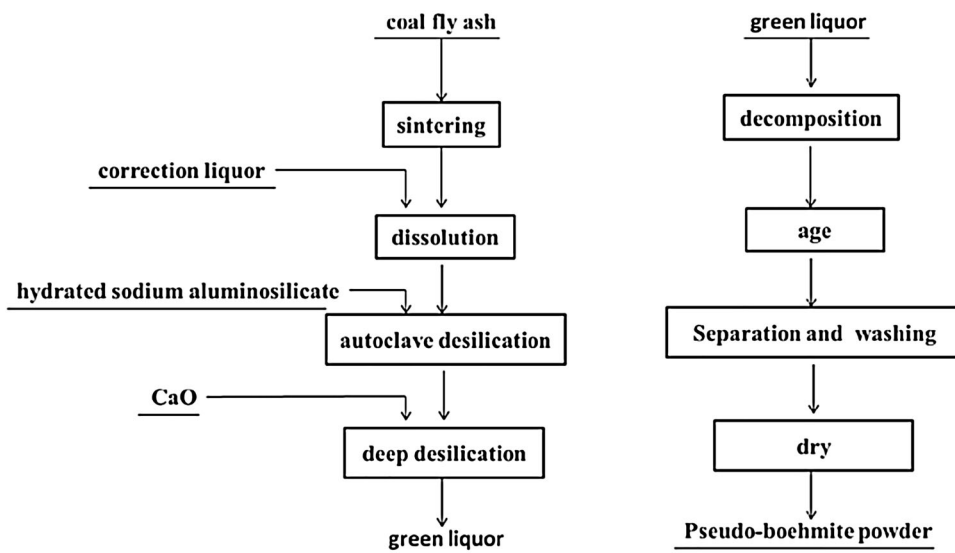


Fig. 1. Process steps in this method.

Carbonate decomposition of sodium aluminate solution is an exothermic process. With continuing decomposition, the temperature of the decomposition system rises sharply. This increase in the decomposition temperature may result in a dramatic increase in the amount of gibbsite. There are many mixed phases in pseudo-boehmite, which will influence the final quality of the pseudo-boehmite. A constant-temperature water bath device was added to the decomposition system to prevent the temperature from increasing rapidly.

Green liquor was decomposed into a gel through a gelling process, and after a certain duration of aging, separation, and washing, the filter cake was dried at 70°C, and pseudo-boehmite powder was obtained.

X-ray diffraction (XRD) analysis was carried out using a D8 Advance (Bruker, German) with a Cu target with step size of 0.0095°, scanning the spectrum between 10° and 90°. Thermogravimetry–differential thermal analysis (TG–DTA) was carried out using an SDT-Q600 STA (TA, USA). The surface area and pore structure were evaluated by the Brunauer–Emmett–Teller (BET) method using an ASAP 2020 Physisorption Analyzer (Micromeritics, USA). Chemical analysis was carried out by inductively coupled plasma (ICP) spectroscopy (prodigy XP, Leeman, USA).

The peptization ratio was tested as follows: (1) PB product with alumina content of 20.0 g was mixed in deionized water to a total weight of 200.0 g; (2) 1.65 mL formic acid was added into the PB–water mixture liquid at 27°C while stirring at 500 rpm; (3) when the viscosity of the mixed slurry reached 0.5 Pa s, the stirring was stopped. The peptization ratio (DI) was calculated as

$$DI = \frac{w}{20} \times 100\%, \quad (1)$$

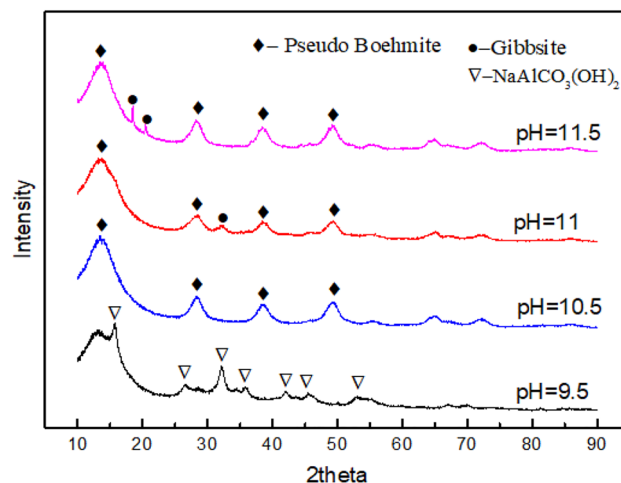


Fig. 2. XRD patterns of carbonation products for different terminal pH levels.

where  $w$  is the total mass of alumina in the solution (g).

## RESULTS AND DISCUSSION

### Effect of the Terminal pH of the Carbonation Process

To investigate the effect of the terminal pH, we carried out a series of experiments with concentration of alumina in the liquid phase of 40 g/L at decomposition temperature of 40°C and CO<sub>2</sub> volume fraction of 33%. The decomposed slurry was treated by aging at 90°C with holding time of 4 h after the decomposition process.

The results in Fig. 2 indicate that the main solid product was NaAlCO<sub>3</sub>(OH)<sub>2</sub> when the terminal

decomposition pH was lower than 9.5 with aging treatment. When the terminal pH was above 11, gibbsite phase appeared in the product. The optimal terminal pH for pseudo-boehmite production was found to be 10.5.

### Influence of the Aging Process

The aging process had a significant influence on the quality (such as the crystallinity and grain size) of the PB product. The aging process causes loose flocs generated during the decomposition process to further crystallize. This is followed by grain growth and stabilization of the sediments (such as amorphous hydrated alumina and fine-grain pseudo-boehmite) after dehydration and shrinkage. The quality of the pseudo-boehmite is directly affected by the parameters controlling the aging process.<sup>26</sup> In the new method, the aging process follows the decomposition process, but it is difficult to characterize the decomposition products without a suitable aging treatment, so in this research, we first investigated the optimal aging parameters.

### Effects of the Aging Temperature

The preparation of different types of hydrated alumina or the crystal transition of hydrated alumina is closely related to the pH during the aging process. The effect of the aging medium is essentially the effect of the aging pH on the crystal form of hydrated alumina. In this research, two kinds of medium were investigated: circulatory liquor and deionized water. The circulatory liquid used in this experiment was sodium salt solution formed by reaction between  $\text{NaAlO}_2$  solution and  $\text{CO}_2$ . The experimental method was direct aging of the precipitated slurry without solid-liquid separation, the main product of which is pseudo-boehmite at 80°C after 4 h. However, if the aging medium is deionized water, after a certain aging time, part of the PB was converted to gibbsite. Therefore, the suitable aging medium was found to be the circulatory liquor.

A series of experiments to investigate the effect of the aging temperature were carried out with the following conditions: decomposition solution concentration ( $\text{Al}_2\text{O}_3$ ) of 40 g/L, decomposition temperature of 40°C,  $\text{CO}_2$  volume fraction of 33%, decomposition terminal pH of 10.5, with circulatory liquor as aging medium and aging time of 4 h. The XRD analysis results shown in Fig. 3 illustrate that the product was in amorphous state without aging treatment. With increase in the aging temperature, the intensity of the diffraction peaks gradually increased. When the aging temperature reached 90°C, the intensity of the diffraction peaks reached their maximum value, and the crystallinity (calculated using JADE 6.5) of the PB product increased from 26.50% to 61.75% from 30°C to 90°C (26.50%, 36.05%, 47.58%, 51.34%, and 61.75% at 30°C, 50°C, 70°C, 80°C, and 90°C, respectively).

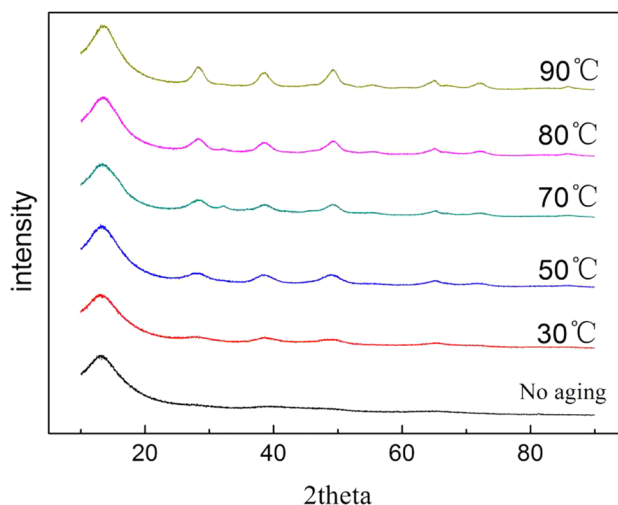


Fig. 3. XRD patterns of products for different aging temperature.

When the aging temperature was less than 70°C, the peptization ratio of PB was quite low and difficult to measure. The peptization ratio of the PB product increased in the temperature range between 70°C and 90°C, reaching 97.5% at 90°C. Increase in the aging temperature accelerates the transformation from a disordered to ordered state. Furthermore, with increase in the aging temperature, the Fe and Si contents did not change significantly and remained lower than the quality requirements for pseudo-boehmite (< 0.3%), while the Na content gradually decreased (from 3.88% at 30°C to 0.26% at 90°C). Therefore, considering the crystallinity, peptization ratio, and impurity contents of the PB product, the optimal aging temperature was found to be 90°C.

### Effects of the Aging Time

To research the effect of the aging time, a series of experiments were carried out under the following conditions of the carbonation decomposition process: alumina concentration of 40 g/L, decomposition temperature of 40°C,  $\text{CO}_2$  volume fraction of 33%, terminal pH of 10.5, and aging temperature of 90°C. The XRD patterns of the products obtained with different aging times are shown in Fig. 4, indicating that the intensity of the diffraction peaks gradually increased with increasing aging time. The crystallinity of PB was 31.90% when the aging time was 2 h, reaching 57.39% after aging for 4 h. Moreover, the increasing trend in the crystallinity was not significant (from 61.75% after 8 h to 62.93% after 20 h).

With increasing aging time, the peptizing ratio increased (94.7%, 97.2%, 98.3%, 98.1%, and 98.5% for 2 h, 4 h, 8 h, 12 h, and 20 h). However, it did not improve much when the aging time was extended beyond 4 h. The Fe and Si contents in all the products were below 0.3% after aging at 90°C for

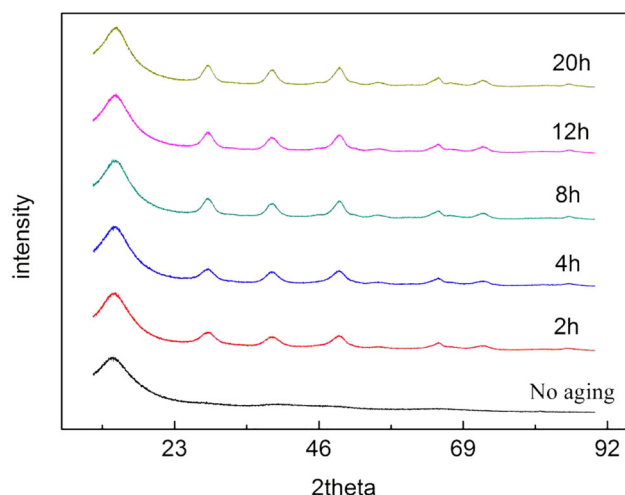


Fig. 4. XRD patterns of products at different aging times.

different holding times, but when the aging time was more than 8 h, the Na content exceeded 2.30%.

The TG-DTA results for the samples aged for different times (Fig. 5) indicate: (1) for the unaged sample, a broad endothermic peak appeared below 300°C and there was no endothermic peak at 300–500°C, which indicates that the sample contained absorbed water and PB crystal was not formed; (2) each of the aged samples showed an obvious endothermic peak between 300°C and 500°C, indicating that they contained PB crystal and lost water at 300–500°C. Moreover, with increasing aging time, the endothermic effects grew stronger and the endothermic peak increased, from 404.6°C to 434.4°C. During the aging process, dehydration contributes to formation of pores in the inner layer, hence the specific surface area increases. However, when the aging time was longer than 12 h, the dehydration ratio increased as well, which affected the PB peptizing ratio.

The results in Fig. 5e and f show that the mass loss of PB products with treatment for 12 h and 20 h were 17.17% and 18.83%, respectively, being higher than the values of 14.03% (after 4 h of aging) and 14.69% (after 8 h of aging). The measured mass loss of all the PB products meets the quality requirement for catalyst carriers in China (e.g., mass loss for P-DF-05-LSi of  $\leq 24\%$ ).

In conclusion, considering the crystallinity, impurities content, and mass loss of the PB products, a suitable aging condition was between 4 h and 8 h at temperature of 90°C.

### Effect of the CO<sub>2</sub> Volume Fraction

The effect of the CO<sub>2</sub> volume fraction on the PB products was tested using an alumina concentration in the initial decomposition liquid of 40 g/L, decomposition temperature of 40°C, and decomposition terminal pH of 10.5, followed by aging treatment at 90°C for 4 h.

The crystal form, crystallinity, and peptization ratio results for the PB products decomposed using different CO<sub>2</sub> volume fractions are as follows: (1) the main products were  $\alpha$ -Al(OH)<sub>3</sub> and PB when the CO<sub>2</sub> volume fraction was 25%; (2) the main product was PB when the CO<sub>2</sub> volume fraction was higher than 35%; however, the PB crystallinity decreased from 65.4% to 35.6% when the CO<sub>2</sub> volume fraction was increased from 35% to 100%; and (3) the peptization ratio of the PB product was 56.4% at CO<sub>2</sub> volume fraction of 25%, and all peptization ratios of the PB products were higher than 97.5% and changed little when the CO<sub>2</sub> volume fraction was higher than 35%. In the sintering process for alumina production, CO<sub>2</sub> is recovered in a rotary kiln for the limestone roasting process for preparation of calcium oxide, which is one of the raw materials in sintering. The volume fraction of recovered CO<sub>2</sub> is about 40% after purification and concentration; therefore, the recovered CO<sub>2</sub> can be used for PB preparation without further treatment.

### Effect of the Decomposition Alumina Concentration and Temperature

These experiments were carried out using the following conditions: terminal pH of 10.5, CO<sub>2</sub> volume fraction of 35%, followed by aging treatment at 90°C for 4 h and 8 h. The XRD analysis results are shown in Fig. 6.

For the PB samples produced after 4 h of aging treatment, when the decomposition temperature was below 60°C, x-ray diffraction peaks appeared at 2 theta values of 13.933°, 28.332°, 38.477°, and 49.214°, corresponding to crystal surfaces of (020), (120), (140), and (200), which are the typical diffraction peaks of PB. Diffraction peaks emerged at 2 theta values of 18.946° and 20.461°, indicating presence of the by-product  $\alpha$ -Al(OH)<sub>3</sub> for decomposition above 60°C. When the aging time was increased to 8 h, the intensity of the typical diffraction peaks of the decomposition product strengthened compared with 4 h of aging time under the same decomposition conditions; however, no diffraction peaks for  $\alpha$ -Al(OH)<sub>3</sub> appeared after 8 h of aging treatment. It can be estimated that the gibbsite phase in the decomposition product transformed into PB with longer aging treatment, motivating further research on this phenomenon.

When the decomposition temperature was between 30°C and 50°C and the alumina concentration was below 60 g/L, the main phase in the PB products after aging times of 4 h and 8 h was PB, and there was no significant change in the crystallinity (55.45%, 52.48%, and 58.64% for 30°C, 40°C, and 50°C), peptization ratio (97.2%, 98.3%, and 96.5% for 30°C, 40°C, and 50°C), or impurity contents (all of the Na, Fe and Si contents in the products were lower than 0.3%) among the decomposition products, which all meet the requirements for catalyst carriers (peptization ratio > 95%).

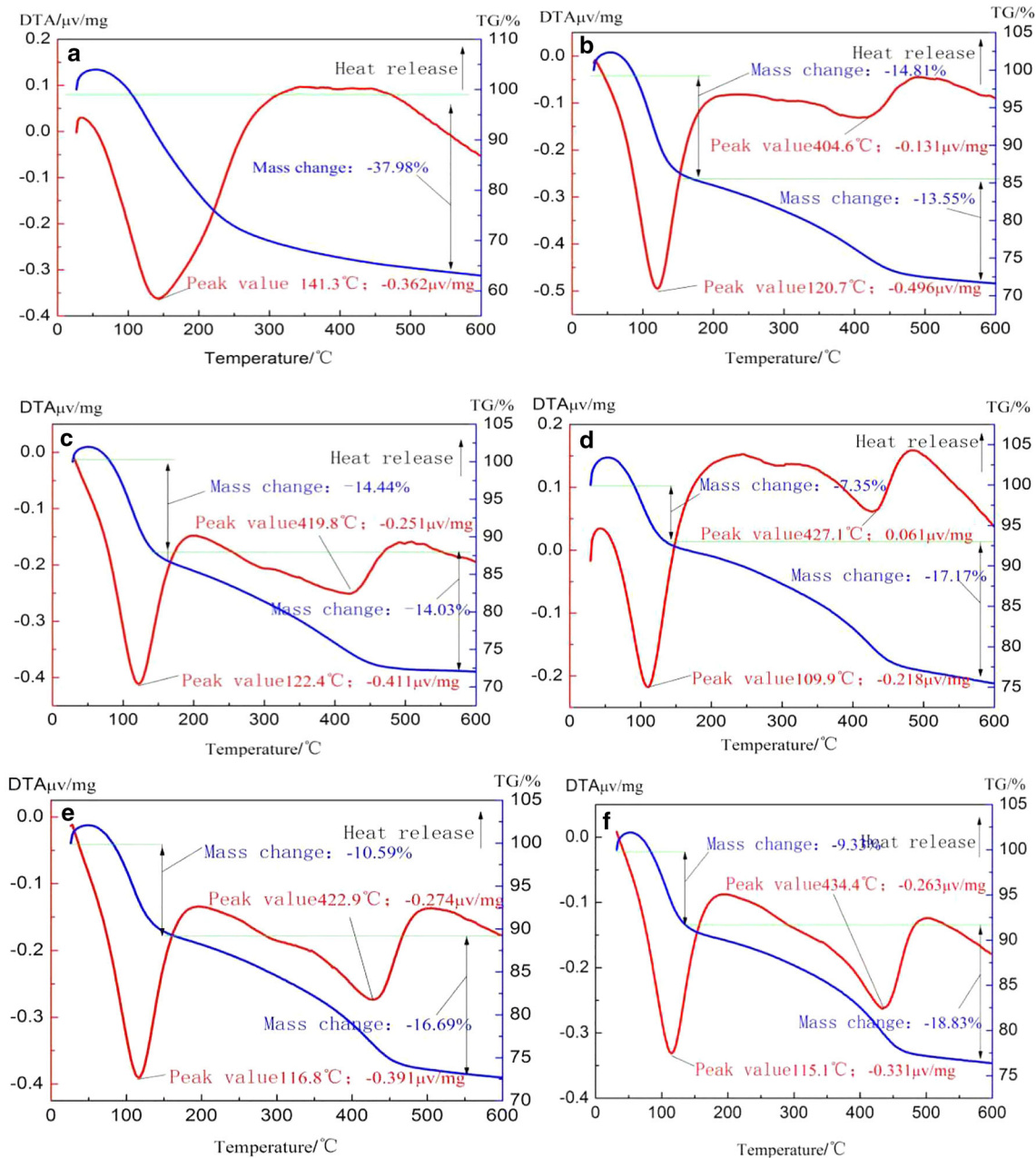


Fig. 5. Effect of aging time on the TG–DTA behavior of PB: (a) unaged, and aged for (b) 2 h, (c) 4 h, (d) 8 h, (e) 12 h, and (f) 20 h.

When the decomposition temperature was increased to 60°C with aging time of 4 h, the peptization ratio of the decomposition products decreased to 75.2%, and gibbsite was also present. The reason for this phenomenon can be estimated as due to the progress of sodium aluminate hydrolysis during decomposition, leading to generation of gibbsite. Moreover, when the initial alumina concentration reached 60 g/L, the peptization ratios of the PB products were 91.3% and 90.3% for 4 h and 8 h of aging treatment, respectively.

In summary, there was no significant improvement in the crystallinity, peptization ratio, or chemical composition of the PB products on

extending the aging time to 8 h. Based on economic considerations, the optimal aging time is 4 h, and the optimal initial alumina concentration and decomposition temperature should be lower than 60 g/L and 60°C, respectively.

#### Characteristics of the Decomposition Products at 4 h of Aging Time

The changes in the specific surface area and pore volume of the PB products formed at different decomposition temperatures (20°C to 50°C) and initial alumina concentrations (30 g/L to 50 g/L) followed by 4 h of aging treatment were analyzed.

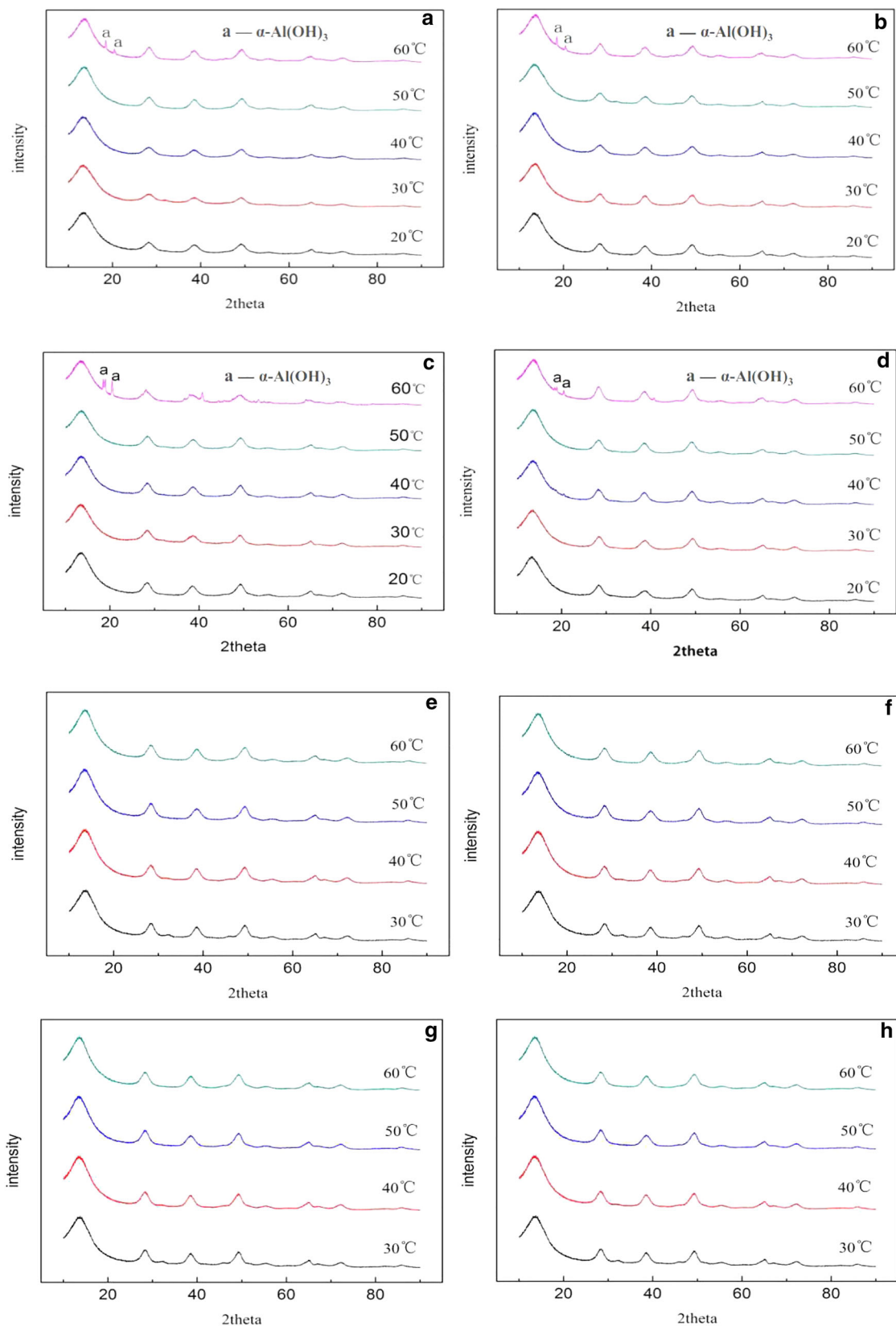


Fig. 6. XRD analysis results for PB products with different initial alumina concentrations and temperatures: (a) 30 g/L, 4 h; (b) 40 g/L, 4 h; (c) 50 g/L, 4 h; (d) 60 g/L, 4 h; (e) 30 g/L, 8 h; (f) 40 g/L, 8 h; (g) 50 g/L, 8 h; and (h) 60 g/L, 8 h.

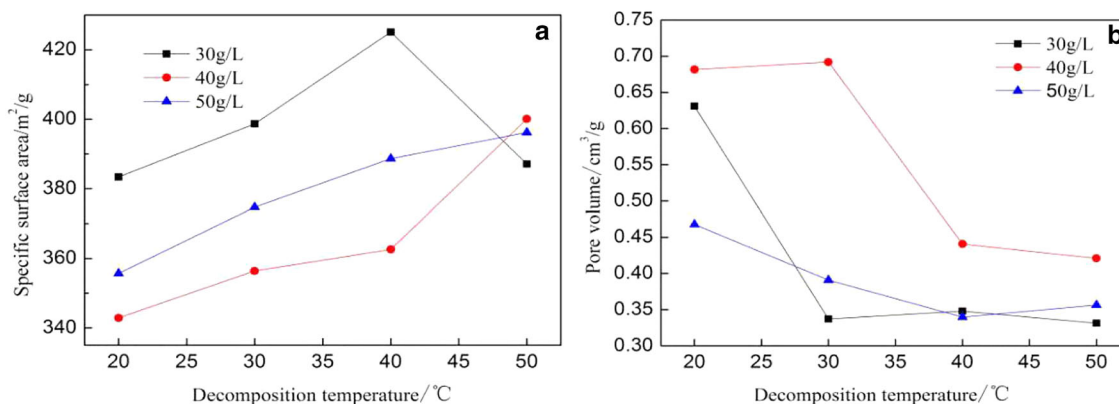


Fig. 7. Change in (a) specific surface area and (b) pore volume of PB products.

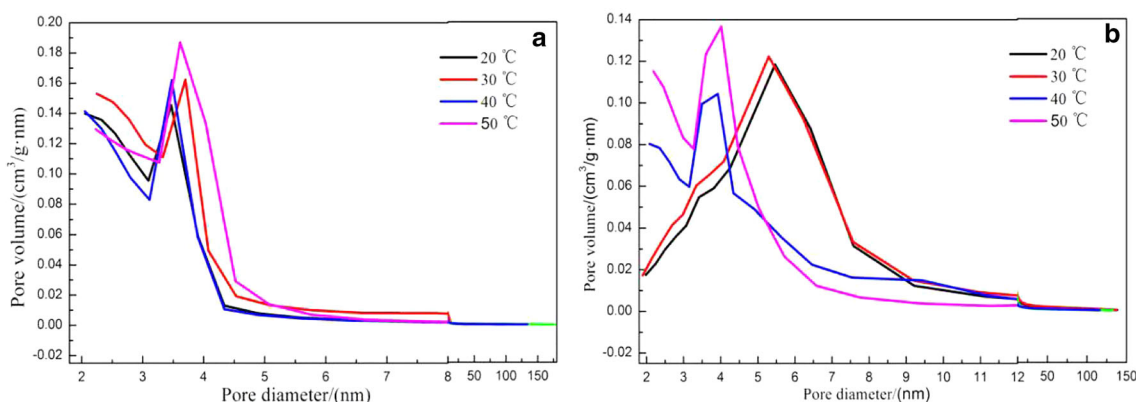


Fig. 8. Pore diameter distribution of PB products with initial alumina concentration of (a) 30 g/L and (b) 40 g/L.

The results in Fig. 7a indicate the following: (1) the specific surface area of the PB products increased with increasing temperature except for the 30 g/L sample, and (2) the maximum surface area was obtained for decomposition concentration of 30 g/L and decomposition temperature of 40°C.

The results in Fig. 7(b) illustrate that the general trend is a decrease in the pore volume with increasing temperature, although the results at 30 g/L show an increasing trend at temperatures between 30°C and 50°C. Moreover, an initial increase in the pore volume between 20°C and 30°C is seen for the samples produced with alumina concentration of 40 g/L. Conclusively, a maximum of 0.6920 cm<sup>3</sup>/g was observed at 40 g/L and 30°C. Moreover, the surface area and pore volume of the PB products meet the industrial requirements for mesoporous PB (e.g., surface area and pore volume for P-DF-05-LSi product of  $\geq 250$  m<sup>2</sup>/g and 0.5–0.7 cm<sup>3</sup>/g, respectively).

The results in Fig. 8 illustrate that the pore size of the PB products exhibited a somewhat narrow distribution between 2 nm and 5 nm for decomposition alumina concentration of 30 g/L followed by 4 h of aging treatment. However, the size lay between 2 nm and 15 nm when using 40 g/L

alumina at 20°C or 30°C, and 2 nm and 5 nm for the samples obtained at 40°C and 50°C. Moreover, the contribution of the pores in the > 5 nm range to the total pore volume appears minor in comparison with the pores in the 2 nm to 5 nm range for the samples obtained using 40 g/L alumina and 20°C or 30°C (as shown in Fig. 8).

In summary, when using 30 g/L alumina at 40°C followed by 4 h of aging, the specific surface area of PB was 425 m<sup>2</sup>/g. The pore volume of PB was 0.6920 cm<sup>3</sup>/g when using 40 g/L alumina at 30°C. Moreover, the Na, Si, and Fe contents of both samples were below 0.3%, meeting the requirement for PB products (e.g., the purity requirement for P-DF-05-LSi product is Na, Si, and Fe contents below 0.3%). The peptization ratio and chemical composition of the PB products prepared under both conditions meet the requirements of PB for use as a catalyst carrier.

## CONCLUSION

Based on the results of the experimental preparations and the properties of PB products obtained from HAFA, the following conclusions can be drawn:



The sintering-CO<sub>2</sub> decomposition process is suitable for preparation of PB using HAFA as raw material. The main product was NaAlCO<sub>3</sub>(OH)<sub>2</sub> when the decomposition terminal pH was lower than 9.5. When the terminal pH was higher than 11, gibbsite phase appeared in the product. The optimal terminal pH for production of pseudo-boehmite was 10.5.

With increase in the aging temperature, the crystallinity of the PB product increased. When the aging temperature was 90°C, the PB crystallinity reached 97.5%.

The main product was PB when the CO<sub>2</sub> volume fraction was higher than 35%; however, the crystallinity of PB decreased with an increase in the CO<sub>2</sub> volume fraction. Therefore, the CO<sub>2</sub> recovered from the sintering process can be used for PB preparation without further treatment.

When the decomposition temperature was below 50°C and the alumina concentration lower than 60 g/L with aging time of 4 h or 8 h, the main phase in the product was PB, and there was no significant change in the crystallinity, peptization ratio, or impurity contents among the decomposition products.

Under the decomposition conditions of alumina concentration of 30 g/L and temperature of 40°C followed by 4 h of aging treatment, the specific surface area of PB was 425 m<sup>2</sup>/g. The pore volume of the PB product was 0.6920 cm<sup>3</sup>/g for decomposition conditions of 40 g/L and 30°C. The peptization ratio and chemical composition of the PB products prepared using both of the above-mentioned conditions meet the requirements for use of PB as a catalyst carrier.

#### ACKNOWLEDGEMENTS

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