A New Route to the Synthesis of Fine-Grain Gibbsite

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A fine-grain gibbsite (<20 μm) is obtained in 2 days compared with the usually reported several weeks. One of the parameters, which considerably affected the nature of the resulting aluminum hydroxide, has been the concentration of ammonia solution used to precipitate the aluminum gel together with the amount of aged sample. The dehydration sequence of this gibbsite was via χ-alumina. This is the path characteristic of gibbsites with fine grain. XRD, BET, TG, and SEM techniques were employed to characterize the solids obtained. A seeding experiment shows that the gibbsite prepared can be used in the Bayer process. Pure α-aluminas of 13 and 18 m^2 g^{-1} were obtained after calcination at 1223 K for 1.30 h of the gibbsites prepared in 2 and 4 days, respectively.

Introduction

Gibbsite, bayerite, and nordstrandite are aluminum hydroxides with Al(OH)$_3$ stoichiometry. Gibbsite is technically the most important hydroxide of the three because it is an intermediate in the Bayer process. In this process,[1] bauxite is refined to smelting grade alumina (Al$_2$O$_3$). The production of this alumina first requires that gibbsite crystals with a precise size distribution and strength be obtained from seeded (with gibbsite or bayerite nuclei[2,3]) sodium aluminate solutions. The agglomeration step is an important size enlargement process during crystallization since the growth rates of gibbsite are very low. The degree of agglomeration of gibbsite crystals increases as the size of seed decreases.[4,5]

Gibbsite is also a good alternative for producing high-area α-Al$_2$O$_3$, which can be used as ceramic or catalyst support,[6–8] and activated aluminum oxide (AAO), which can be used as an adsorbent or catalyst support, mainly in petrochemistry.[9] Only a few methods for preparing AAO from gibbsite are known. One of the more promising ones is flash calcination (FCAL) followed by rehydration. The porous nature of the prepared alumina strongly depends on the initial grain size of gibbsite,

and the pore-structure parameters of the AAO obtained by FCAL from fine-grain gibbsite (<50 μm) are similar to those of the commercial precipitated alumina adsorbents and catalyst supports.[9]

While the dehydration sequence of gibbsite has been extensively studied,[10–18] there is less information about the synthesis of gibbsite at the laboratory scale.[8,19–21]

In a previous work,[8] we studied how experimental conditions, such as precipitation temperature of the gels, ammonia concentration, gel aging, and pH, affect the formation rates of several aluminum hydrates. Small changes of these parameters lead to different alumina hydrate phases. Gibbsite was obtained in 8 days by precipitating an amorphous gel at 348 K and then aging it at room temperature without stirring, first in basic medium (pH $\approx$ 8) and with a later treatment in acid medium (pH $\approx$ 2). This poor crystalline gibbsite calcined at 1223 K for 3 h gave place to a high-area α-Al$_2$O$_3$ (42 m^2 g^{-1}).

In this work, we report the synthesis of gibbsite of small particle size by a new route which allows one to prepare gibbsite in 2 days. The interest of this contribution lies in the short time of obtention of crystalline gibbsite compared with the about weeks time reported in the literature and also in the importance of obtaining

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fine-grain size of crystalline gibbsite which can be used as an intermediate in the production of activated aluminas and as seed in the Bayer process (to favor the agglomeration of the gibbsite crystals). Also, the formation of α-Al_2O_3 from this fine-grain size gibbsite has been studied.

Experimental Section

Aluminum Hydroxide Preparation. The synthesis procedure was as follows: One series of aluminum hydroxides was prepared from the precipitation of 100 mL of a 0.1 M aluminum nitrate hexahydrate aqueous solution with a 0.25% ammonia aqueous solution up to pH 8 at 348 K. The resultant gel was then loaded into a flask and heated under constant stirring at 348 K for different times: 1 day; 2 days; 4 days; 6 days; 1 week; 2 weeks. The corresponding samples are designated as D1, D2, D4, D6, W1, and W2, respectively. A reflux was used to prevent the volume modification. After cooling to room temperature, the white gel was recovered by filtration, washed with deionized water and dried overnight in an oven at 393 K.

To see the effect of the ammonia concentration, two samples were prepared by precipitating 100 mL of a 0.1 M aluminum nitrate hexahydrate aqueous solution with a 0.5% ammonia solution up to pH $\approx$ 8 at 348 K and later refluxing of the precipitant gel under the above conditions for different times: 1 week (sample AW1); 2 weeks (sample AW2).

Also, to determine how the amount of aged sample affects the nature of the resulting aluminum hydroxide, a gel was synthesized by precipitating 50 mL of a 0.1 M aluminum nitrate hexahydrate aqueous solution with a 0.25% ammonia solution up to pH $\approx$ 8 at 348 K and later refluxing of the precipitant gel for 2 days (sample M2). All samples were characterized by XRD, BET, TG, and SEM techniques.

Seeding Experiment of Sample D2. One seeding experiment tested whether sample D2 could be used as seed in the Bayer process. We prepared a supersaturated aqueous solution containing 5 M sodium aluminate and 3 M excess NaOH. Seeding was performed by adding gibbsite. We select the sample D2 which was synthesized after 2 days of refluxing. Crystallization was performed at atmospheric pressure and room temperature for 48 h. The solid was then filtered out and dried. The phases were identified by carrying out a powder diffraction pattern of the product.

Calcination of the Gibbsites. Several aluminas were obtained from the calcination of the gibbsites previously prepared. The calcination procedures were performed in a furnace at 1223 K under air for different times between 0.30 and 16 h.

The nomenclature used to refer to the aluminas will be the name of the initial gibbsite and the calcinations time (hours in parentheses) at 1223 K; i.e., D2(1.30) corresponds to the alumina obtained by calcining the gibbsite D2 at 1223 K for 1.30 h.

X-ray Diffraction (XRD). Powder X-ray diffraction patterns (XRD) were obtained with a Siemens D5000 diffractometer using nickel-filtered Cu Kα radiation. Samples were dusted on double-sided sticky tape and mounted on glass microscope slides. The patterns were recorded over a range of 2θ angles from 10 to 70°, and crystalline phases were identified using the Joint Committee on Powder Diffraction Standards (JCPDS) files. The 2θ angles of the main peaks ($I/I_{max} = 20\%$) of gibbsite (with the relative intensities in parentheses) are the following: 18.28 (100), 20.30 (70), 20.55 (50), 26.89 (30), 28.01 (25), 36.41 (25), 36.62 (40), 37.68 (55), 40.11 (20), 41.69 (27), 44.17 (40), 45.44 (28), 50.55 (30), 52.17 (30), 54.42 (30), and 63.80 (30) for the gibbsite phase;[22] 18.83 (90), 20.40 (70), 27.86 (30), 40.57 (100), and 53.11 (40) for the bayerite phase;[23]

25.58 (75), 35.14 (90), 37.78 (40), 43.36 (100), 52.55 (45), 57.52 (80), 66.55 (30), and 68.20 (50) for the α-Al_2O_3 phase;[24] 14.27 (30), 19.71 (20), 29.36 (40), 32.05 (60), 33.15 (20), 34.88 (80), 37.28 (30), 38.78 (40), 42.82 (80), 43.92 (30), 45.55 (40), 46.53 (20), 48.65 (60), 50.08 (30), 52.55 (20), 56.03 (60), 62.26 (30), 64.18 (30), 65.19 (80), and 67.31 (100) for the κ-Al_2O_3 phase;[25] 37.44 (40), 39.67 (20), 42.82 (30), 45.79 (20), and 67.31 (100) for the χ-Al_2O_3 phase.[25]

This technique was also used to determine the percentage of α-Al_2O_3 in the mixtures of aluminas by the Rietveld method.[26] This method enables a quantitative phase analysis of multicomponent mixtures from the X-ray powder diffraction data.

BET Areas. BET areas were calculated from the nitrogen adsorption isotherms at 77 K using a Micromeritics ASAP 2000 surface analyzer and a value of 0.164 nm² for the cross section of the nitrogen molecule.

Scanning Electron Microscopy (SEM). Scanning electron micrographs were obtained with a JEOL JSM-35C scanning microscope operating at an accelerating voltage of 35 kV, a work distance (wd) between 14 and 16 mm. and magnification values in the range 200−50 000×.

Thermogravimetric Analysis (TG). Thermal decompositions of alumina hydrates were carried out in a Perkin-Elmer TG 7 microbalance, with an accuracy of 1 μg, equipped with a 273−1273 K programmable temperature furnace. Each sample (50 mg) was heated in a N_2 flow (80 cm³/min) from 50 to 900 °C at 10 °C min^{-1} and maintained at 900 °C for 1.30 h. The weight change obtained is a measure of the water loss of the sample after calcination.

Results and Discussion

From the results obtained in a previous work,[8] the rateThermal decompositions of alumina hydrates were carried out in a Perkin-Elmer TG 7 microbalance, with an accuracy of 1 mg, equipped with a 273−1273 K programmable temperature furnace. Each sample (50 mg) was heated in a N2 flow (80 cm3/min) from 50 to 900 °C at 10 °C min-1 and maintained at 900 °C for 1.30 h. The weight change obtained is a measure of the water loss of the sample after calcination. of gibbsite formation was found to be faster if (a) the gel precursor is prepared by hot precipitation (348 K), (b) the ammonia concentration is low (0.5% NH_3), and (c) the gel is first aged in basic medium and then in acid medium at 298 K (room temperature). The increase in the aging time at pH $\approx$ 2 (from 8 h until 1 day) gives place to a bit more crystalline gibbsite although only the two more intense peaks of the gibbsite phase (2θ at 18.28 and 20.30°) were detected by XRD.[8]

Now, a gel precipitated at 348 K by using lower ammonia concentration (0.25%) was aged in its precipitating solution at 348 K at different refluxing times. Figure 1 shows the X-ray diffraction of the samples refluxed at (a) 1 day, (b) 2 days, (c) 4 days, (d) 6 days, (e) 1 week, and (f) 2 weeks. One day was not long enough to transform the initial amorphous gel totally to gibbsite, and there are some peaks (marked with an asterisk on the plot) identified as a bayerite phase. On the other hand, the samples refluxed for 2 or more days are identified as pure gibbsite with particle sizes, observed by SEM, in the 10−20 μm range.

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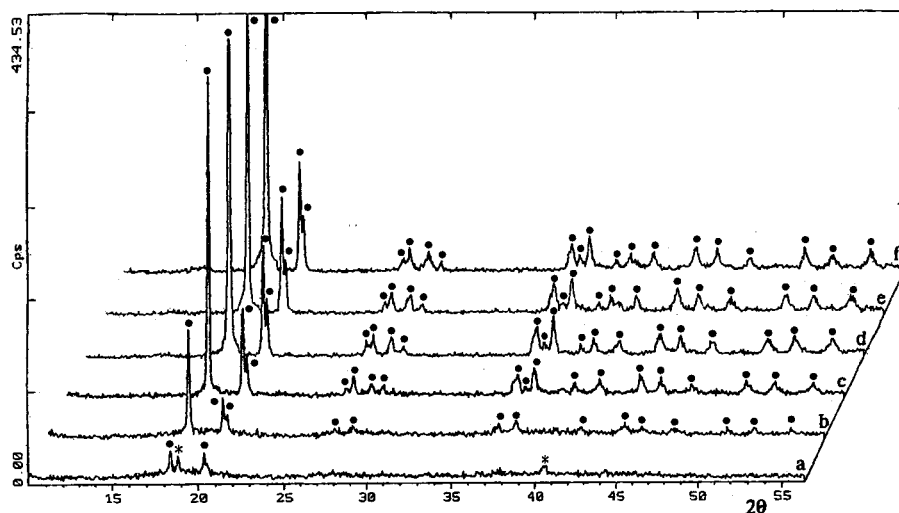(26) Bish, D. L.; Howard, S. A. *J. Appl. Crystallogr.* **1988**, *21*, 86.

Figure 1. Powder X-ray diffraction patterns of the samples prepared with 0.25% ammonia concentration by refluxing the precipitant gel at 353 K for (a) 1 day, (b) 2 days, (c) 4 days, (d) 6 days, (e) 1 week, and (f) 2 weeks: ∗, bayerite phase; ●, gibbsite phase.
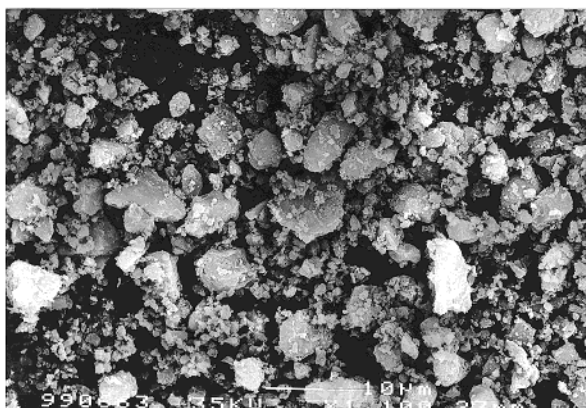

Figure 2. Scanning electron micrograph taken from the surface of the synthesized gibbsite obtained in 2 days designated as D2 (magnification ×1100).

Figure 2 shows a scanning electron micrograph of the gibbsite D2 with around 90% of the particle sizes <20 μm. The specific properties of this fine-grain size gibbsite means that good adsorbents AAO and catalyst supports can be prepared from it.

The surface area of sample D2 was determined as 4.7 m^2 g^{-1} by a full adsorption−desorption nitrogen isotherm using BET analysis.

One seeding experiment, described above in the Experimental Section, tested whether this fine-grain size gibbsite (D2) could be used as seed in the Bayer process. The powder diffraction pattern obtained for the seeded sample (see Figure 3) shows a primary crystalline gibbsite phase and some peaks, which were identified as a bayerite phase. This agrees with the results of other authors.[2]

One of the parameters which considerably affects the nature of the resulting aluminum hydroxide (as we reported in a previous work[8]) is the concentration of ammonia solution used to precipitate the aluminum gel. There is also another factor to take into account that we had not previously tested, which is the amount of aged sample. The effect of these factors has been verified for this new method proposed here.
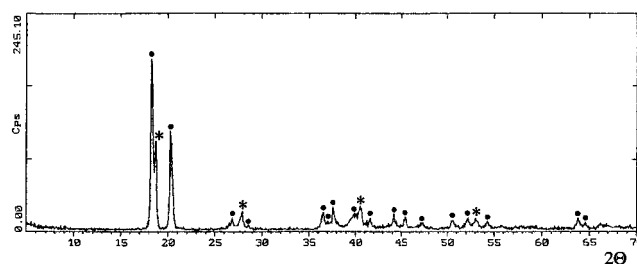

Figure 3. Powder X-ray diffraction pattern of the sample obtained by seeding the gibbsite D2: ∗, bayerite phase; ●, gibbsite phase.
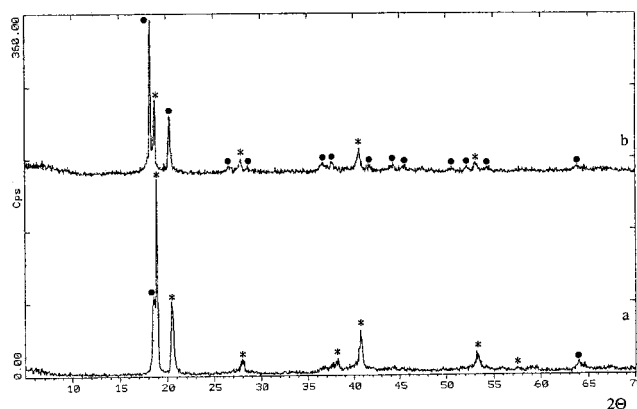

Figure 4. Powder X-ray diffraction patterns of the samples prepared with 0.5% ammonia concentration by refluxing the precipitant gel at 348 K for (a) 1 week (sample AW1) and (b) 2 weeks (sample AW2): ∗, bayerite phase; ●, gibbsite phase.

Precipitating with 0.5% ammonia solution up to a final pH ≈ 8 and later refluxing the precipitant gel under the above conditions did not allow us to identify gibbsite as a single phase even after 2 weeks of refluxing. A mixture of gibbsite and bayerite was obtained in all cases. Figure 4 shows the X-ray diffraction pattern obtained after (a) 1 week (sample AW1) and (b) 2 weeks of refluxing (sample AW2). This is probably related to the slower precipitation rate of the gel precipitated with less ammonia concentration that gives place to smaller particles with low water content, and in agreement with previous studies,[8] this gel evolves quicker toward the
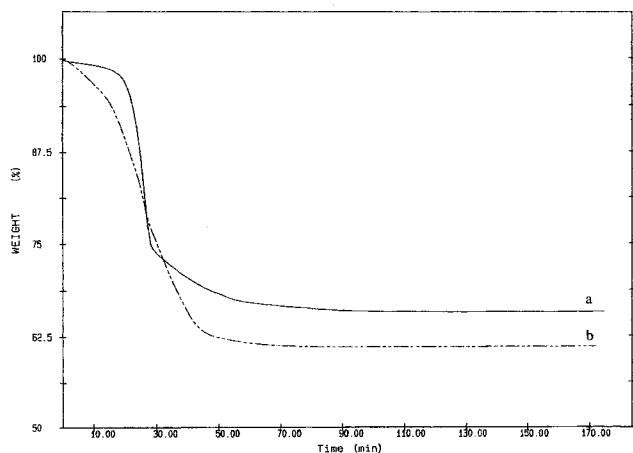
Figure 5. TG plot of dehydration for the gels recent precipitated at 348 K with different ammonia concentrations: (a) 0.25%; (b) 0.5%.

Figure 6. Scanning electron micrograph taken from the surface of the gel obtained by precipitating 50 mL of aluminum nitrate hexahydrate aqueous solution with 0.25% ammonia (magnification ×200).

gibbsite phase. To confirm this idea, some TG studies were performed to investigate the loss of water from the gels. Figure 5 shows the TG plot for the recent precipitated gels at 348 K (without refluxing) obtained with (a) 0.25% ammonia and (b) 0.5% ammonia. As observed, the gel precipitated with less ammonia concentration has a smaller loss of total water and consequently the gibbsite phase is obtained more easily.

Regarding the amount of aged sample, samples D2 and M2, prepared from precipitating 100 and 50 mL, respectively, of 0.1 M aluminum nitrate hexahydrate solution with 0.25% ammonia solution and later refluxing for 2 days, were compared. The recent precipitated gels (without aging) show very similar particle sizes, as observed by SEM (Figures 6 and 7), but after 2 days of refluxing under the same conditions, sample D2 was identified as pure gibbsite whereas sample M2 was a mixture of mainly gibbsite and small amounts of bayerite. It seems clear that the gel evolves quicker to a highest ordering state in a major volume of sample and therefore this affects the nature of the resulting aluminum hydroxide.

A number of studies using a wide variety of techniques have examined the dehydration sequences of aluminum hydroxides during calcination.[10-18] Scheme 1 shows the generally accepted thermal sequences for
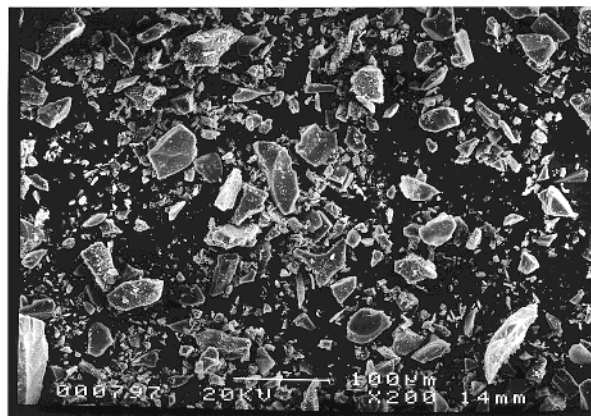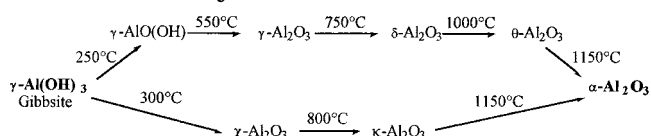

Figure 7. Scanning electron micrograph taken from the surface of the gel obtained by precipitating 100 mL of aluminum nitrate hexahydrate aqueous solution with 0.25% ammonia (magnification ×200).

Scheme 1. Dehydration Paths of Gibbsite in Air

the transformation of gibbsite in air.[12,15-18] The relative proportions following the two paths depend on a number of factors such as moisture, alkalinity, pressure, bed depth, and heating rate and especially on the particle size of the gibbsite. Fine gibbsite dehydrates to α-alumina via χ- and κ-aluminas, while coarse gibbsite dehydrates to α-alumina by two simultaneous routes, via χ-alumina and via boehmite during atmospheric heating and only via boehmite and γ-, δ-, and θ-aluminas under hydrothermal conditions.[15]

Figure 8 shows the powder diffraction patterns of the aluminas obtained from the calcination of several gibbsites at 1223 K for 1.30 h. The aluminas D2(1.30) and D4(1.30) were identified as crystalline α-alumina while the aluminas D6(1.30), W1(1.30), and W2(1.30) were identified as crystalline κ + α-alumina, poorly crystallized κ + α-alumina, and a mixture of gibbsite and poorly crystallized χ + κ-alumina, respectively. The gibbsites D2 and D4 were calcined at 1223 K for times less than 1.30 h, but no pure α-alumina was obtained. A mixture of κ + α-alumina was formed in all cases. From these results, we can conclude that the path of dehydration of these fine-grain size gibbsites is via χ-alumina, in agreement with that reported in the literature.[15] Also, the evolution to α-alumina is faster when the time of refluxing is lower. This can be explained because a longer time of aging produces gibbsites better crystallized, which evolve slower to α-alumina during calcination.

Table 1 shows the BET areas calculated for the aluminas obtained. The two pure α-aluminas D2(1.30) and D4(1.30) have areas of 13 and 18 m^2 g^{-1}, respectively. These values are slightly higher than the surface areas of less than 10 m^2 g^{-1} usually obtained from calcination of bayerite and lower than the surface area of the α-alumina (42 m^2 g^{-1}) obtained by calcining at the same temperature, for at least 3 h, a less crystalline gibbsite prepared in 8 days which dehydrates via boehmite.[8]
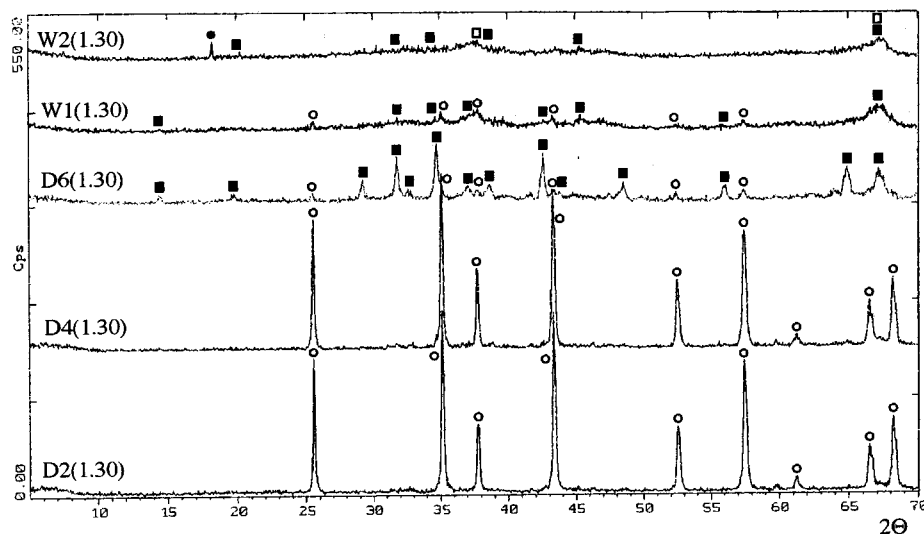
Figure 8. Powder X-ray diffraction of the aluminas obtained from calcination of the gibbsites at 1223 K for 1.30 h: ●, gibbsite phase; ○, α-alumina; ■, κ-alumina; □, χ-alumina.

Table 1. Surface Characterization by Nitrogen Physisorption and XRD of the Aluminas Obtained by Calcination at 1223 K for 1.30 h

alumina	phases detected (XRD)	BET area ($m^2\,g^{-1}$)
D2(1.30)	$\alpha\text{-}Al_2O_3$	13
D4(1.30)	$\alpha\text{-}Al_2O_3$	18
D6(1.30)	$\kappa\text{-}Al_2O_3 + \alpha\text{-}Al_2O_3$	49
W1(1.30)	$\kappa\text{-}Al_2O_3 + \alpha\text{-}Al_2O_3$	82
W2(1.30)	gibbsite $+ \chi\text{-}Al_2O_3 + \kappa\text{-}Al_2O_3$	105

From these results, it seems clear that the path of dehydration definitively affects the rate of formation of the α-alumina phase. This might be due to the different transition aluminas obtained for each dehydration path, which have different degrees of order of Al atoms in a more or less perfect close packing of O. There are two transition aluminas from gibbsite to α-alumina via χ-alumina while there are one oxide−hydroxide and three transition aluminas from gibbsite to α-alumina via boehmite. Therefore, there are less transitional steps via χ-alumina than via boehmite.

The initial step in the thermal decomposition of gibbsite is the diffusion of protons and the reaction with hydroxyl ions to form water. This process removes the binding forces between the strata of the gibbsite structure and changes in the chemical composition and density within the layers. The fact that coarse gibbsite dehydrates first via an oxide−hydroxide and fine gibbsite dehydrates directly via an aluminum oxide leaves us to assume that the elimination of water is slower for coarse gibbsite than for fine gibbsite.

Moreover, γ-, δ-, and θ-aluminas have spinel-related structures with ccp (cubic close packed) oxygens and aluminums in octahedral and tetrahedral sites (with an increasing in the proportion of tetrahedral aluminums through the sequence γ, δ, and θ) whereas χ- and κ-aluminas have hcp (hexagonal close packing) oxygens with also partial occupation of octahedral and tetrahedral sites by Al atoms. The structure of gibbsite contains double layers of hydroxyl ions (each layer in hexagonal close packing) with aluminums in octahedral coordination inside the layers in a pattern of hexagonal rings. The double layers stack to give an AB BA anion sequence. α-Alumina contains oxygen in approximate
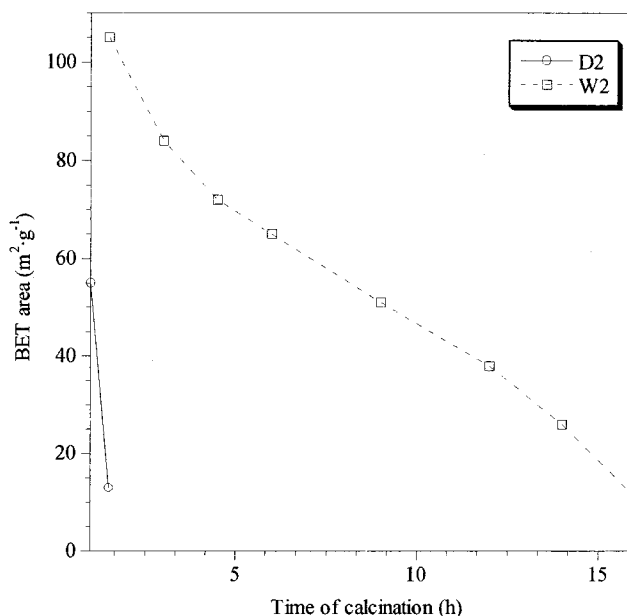

Figure 9. Plot of the BET areas of the calcined gibbsites D2 and W2 as a function of the calcination time.

hexagonal close packing with aluminums in two-thirds of the octahedral sites. Therefore, it seems that the transition from gibbsite to α-alumina can be more efficient though other hcp structures (κ- and χ-alumina) than through spined-related structures with ccp (γ-, δ-, and θ-alumina), where the reorganization of the O and Al atoms should be more difficult. Moreover, χ-alumina shows a fairly diffuse X-ray powder diffraction patterns with wide bands[12] and contains a large number of stacking faults. This might be related to more amorphous structures, which can lose water quicker than the more crystalline γ-, δ-, and θ-aluminas. All these factors could explain the higher rate of formation of α-alumina observed when gibbsite dehydrates through χ- and κ-aluminas than through the sequence boehmite and γ-, δ-, and θ-alumina.

Figure 9 shows the evolution of the BET area in function of the calcination time for gibbsites D2 and W2. The phases of the different aluminas obtained from D2

Table 2. Surface Characterization of Several Aluminas by Nitrogen Physisorption and XRD

alumina	phases detected (XRD)	BET area (m^2g^{-1})
D2(0.30)	κ-Al_2O_3 + α-Al_2O_3 (10%)[a]	83
D2(1.00)	κ-Al_2O_3 + α-Al_2O_3 (45%)[a]	55
D2(1.30)	α-Al_2O_3	13
W2(1.30)	gibbsite + χ-Al_2O_3 + κ-Al_2O_3	105
W2(3.00)	κ-Al_2O_3 + α-Al_2O_3 (5%)[a]	84
W2(4.30)	κ-Al_2O_3 + α-Al_2O_3 (17%)[a]	72
W2(6.00)	κ-Al_2O_3 + α-Al_2O_3 (25%)[a]	65
W2(9.00)	κ-Al_2O_3 + α-Al_2O_3 (49%)[a]	51
W2(12.00)	κ-Al_2O_3 + α-Al_2O_3 (62%)[a]	38
W2(19.00)	κ-Al_2O_3 + α-Al_2O_3 (86%)[a]	26
W2(16.00)	α-Al_2O_3	11

[a] α-Al_2O_3 content, in parentheses, determined by the Rietveld method.[26]

and W2 are indicated in Table 2. Gibbsite W2 has a slower dehydration rate and needs more calcination time (16 h) to become pure α-Al_2O_3 phase than gibbsite D2 (1.30 h). This is due to its higher cristallinity that makes the elimination of water slower. The variation in the surface area values can be explained by the kind of alumina formed. It is well-known[12] that χ- and κ-aluminas are less crystalline than α-alumina and therefore they have higher BET areas. With increase of the time of calcination, the α-alumina content increases, and consequently, the BET area decreases.

Last, the lower BET area observed for the α-aluminas obtained in this work (13–18 m^2/g) than that obtained previously[8] (42 m^2/g) can also be explained by the cristallinity of the starting gibbsite. The gibbsites obtained here are more crystalline because the precipitated gels were aged at higher temperature (348 K) than the less crystalline gibbsite (obtained by aging at room temperature), which is the precursor of the α-alumina of 42 m^2/g. The aging of the gel at higher temperature evolves quicker to a more crystalline gibbsite that gives place to α-alumina of low BET area.

Conclusions

This new method allows one to synthesize crystalline gibbsite with smaller particle sizes (<20 μm) in a shorter time (2 days) than under other conditions reported in the literature. These gibbsites can be used in a great number of industrial fields: in the Bayer process; to obtain aluminas with different properties to be used as ceramics, adsorbents, or catalyst supports.