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Synthesis of Aluminum Oxides from the Products of the Rapid Thermal Decomposition of Hydrargillite in a Centrifugal Flash Reactor: III. Properties of Aluminum Hydroxides and Oxides Obtained via the Mild Rehydration of the Products of the Centrifugal Thermal Activation of Hydrargillite

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Abstract—The interaction between the amorphous product of the centrifugal thermal activation of hydrargillite (CTA HG) and aqueous electrolytes (pH 5–11) under mild conditions (15–35°C, atmospheric pressure) has been investigated by a variety of physicochemical methods. This interaction causes material morphologic and phase changes in CTA HG, and the product composition is governed by the pH of the electrolyte and by the hydration temperature and time. The product that forms in a basic medium or water in <24 h contains up to 50% pseudoboehmite. Raising the pH or temperature or extending the hydration time results in the formation of bayerite as the major phase (~80%). An X-ray amorphous hydroxide forms in acid media. The heat treatment of this hydroxide at 550°C yields aluminum oxides differing from alumina prepared via hydroxide reprecipitation. Products with new, unusual properties can thus be obtained.

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INTRODUCTION

The thermal activation (heat treatment) of hydrargillite/gibbsite (HG) at the dehydration temperature (T_{dehydr}) for a short time is one of the main methods of obtaining active aluminum hydroxide oxide, $Al_2O_3 \cdot nH_2O$ ($n < 1.5$). The interaction between the resulting material and water or an aqueous solution is the basic process in the low-waste pseudoboehmite ($Al_2O_3 \cdot 1.5H_2O$) and γ-Al_2O_3 production technology that does not involve reprecipitation [1–3]:

$$Al(OH)_3\ (T_{dehydr} \approx 300^\circ C) \longrightarrow Al_2O_3 \cdot nH_2O + H_2O\uparrow,$$

$$H_2O(l) + Al_2O_3 \cdot nH_2O\ (T, pH, P) \longrightarrow Al_2O_3 \cdot 1.5H_2O,$$

$$Al_2O_3 \cdot 1.5H_2O\ (T) \longrightarrow \gamma\text{-}Al_2O_3 + H_2O\uparrow.$$

The properties of the thermally activated product, $Al_2O_3 \cdot nH_2O$, are determined to a large extent by activation parameters, the nature and temperature of the heat-transfer agent, the heating rate, the heat treatment time, the composition of the gas medium, cooling (quenching) conditions, storage conditions, etc.

Several thermal activation techniques are known, which include thermal activation in counterflowing flue gas (thermochemical activation) [2], thermal activation in a fluidized bed of a catalyst or a solid heat-transfer agent (thermal dispersion) [3], and thermal activation of a thin fixed bed in an exhaust gas flow.

The quick heat treatment of powders in a TSEFLAR™ centrifugal flash reactor, a new process designed at the Institute of Catalysis, Siberian Branch, Russian Academy of Sciences, yields the so-called centrifugal thermal activation (CTA) product [4, 5].[1] This process opens up new possibilities for obtaining aluminum hydroxides and oxides and is, therefore, expected to afford a wide variety of alumina-based supports and catalysts. The operation of the TSEFLAR™ reactor is based on short-time contact between the initial reactant and a rotating hot surface followed by quenching the heat-treatment product. Both the heat-transfer (rotating) surface temperature and the heat-treatment time can be controlled. The rate at which hydrargillite is heated to its dehydration temperature can exceed 1000 K/s. It is essential that the resulting solid does not contain extraneous impurities, such as products of incomplete fuel combustion.

[1] The product resulting from the heat treatment of hydrargillite in a TSEFLAR™ reactor was given [6] the individual name CTA for the reason that some of its properties differentiate it from the thermal activation products obtained using other techniques.

The physicochemical properties of CTA HG were studied in earlier works [6, 7]. This material can be viewed as a poorly ordered aluminum(III) hydroxide oxide having an imperfect structure and high reactivity. In particular, it was found that the CTA product reacts with water and aqueous alkali under mild conditions, specifically, at temperatures below 50°C and atmospheric pressure [6].

Here, we present a more detailed description of the effects of CTA HG hydration conditions (electrolyte nature, pH, temperature, hydration time, and solid-to-liquid ratio (S/L)) on the properties of the resulting hydroxide and oxide phases.

EXPERIMENTAL

In rehydration, we used hydrargillite (PO Glinozem, Pikalevo, Russia) processed in a TSEFLAR™ reactor with a conical plate heat-transfer surface [4, 5]. The processing conditions were the following: plate temperature, 580°C; powder–plate contact time, ~1 s; reactant feed rate, 7 kg/h. The properties of the starting solid, which was largely amorphous to X-rays, are described below.

X-ray powder diffraction patterns from the starting solid and rehydration products were obtained on an HZG-4C diffractometer (Germany) using monochromated CuK_α radiation. The coherent-scattering domain size (D) was calculated using the Selyakov–Scherrer formula. Quantitative phase analysis was carried out according to a procedure described by Shkryabina et al. [8].

Thermal analysis was carried out on a Q-1500D thermal analysis system in the temperature range of 20–1000°C in air (heating rate, 10 K/min; sample weight, 200 mg; weight loss determination accuracy, ±0.5%). The quantitative phase composition of hydrated products was derived from weight loss data (TG curves) obtained while heating the sample in the decomposition temperature ranges corresponding to the constituent phases. The $Al(OH)_3$, pseudoboehmite ($Al_2O_3 \cdot 1.5H_2O$, Pbo), and boehmite (AlOOH, Bo) contents were estimated from weight change data in the ranges ~250–350°C ($Al(OH)_3$) [9], 350–450°C ($Al_2O_3 \cdot 1.5H_2O$), and 450–550°C (AlOOH) [10]. The difference between the initial sample weight and the total weight of the identified phases was assigned to the amorphous oxide phase $Al_2O_3 \cdot xH_2O$ (Am).

Electron microscopic examinations were carried out with a JEM-100C transmission microscope (resolution, 0.5 nm; accelerating voltage, 100 kV). Specimens to be examined were prepared by placing a drop of an ethanolic dispersion of a solid onto the microscopic grid.

The specific surface areas of solids were determined by the BET method (S_{BET}) from argon desorption data obtained at room temperature for hydroxides and at 300°C for oxides.

The fractional makeup of solids was determined by sieving in a Meinzer 2 sieve shaker with sieve openings of 38 to 250 μm or by the Coulter method [11].

Impurity sodium in solids was quantified by atomic absorption spectroscopy.

The initial HG and thermal activation product were very similar in particle size and particle-size distribution: in both materials, the dominant size fraction was 50–100 μm. Furthermore, thermal activation did not change the impurity levels observed in the initial HG, specifically, 0.22 wt % Na_2O, 0.05 wt % Fe_2O_3, and 0.022 wt % SiO_2. The specific surface area increased from 0.5 m^2/g for the initial HG to 127 m^2/g for the thermally activated product owing to the formation of a porous structure as a result of rapid dehydration [6, 7].

Prior to rehydration, the CTA product was ground in a disintegrator to a particle size of ≤30 μm.

According to X-ray powder diffraction data, the initial CTA product does not contain any significant amount of crystalline phases and is largely amorphous to X-rays. Its residual hydrargillite content is not higher than 6%. Thermal analysis data also indicate that the CTA product contains ~6% $Al(OH)_3$. As the CTA product is heated, it loses weight rather monotonically, giving rise to two endotherms at 140 and 300°C, which are due to the release of the chemisorbed water and the decomposition of $Al(OH)_3$ (~6%), respectively. A weak exotherm is observed at 800°C, which is not accompanied by a weight change and is assigned to the crystallization of amorphous aluminum oxide [12].

Since the thermal events associated with pseudoboehmite and boehmite decomposition are not clearly distinguishable against the background of the monotonic weight loss, it is likely that amorphous aluminum hydroxide of variable composition results from centrifugal thermal processing. As follows from the thermal analysis weight loss data collected while heating this substance to 1000°C, the chemical composition of the CTA product dried at 110°C can be represented as $Al_2O_3 \cdot 0.8H_2O$.

The ground CTA HG product was rehydrated at T_{hydr} = 15–35°C and atmospheric pressure for t_{hydr} = 0.5–200 h in water, aqueous ammonia (pH 10–11), or an acid medium (pH 5.0–5.5) during continuously stirring. The S/L ratio was 1 : 2 to 1 : 3. The hydration product was separated from the solution using a Nutsch filter and was washed with water until pH 6.5–7.5. Next, the product was dried at 110°C for 24 h and was ground in a disintegrator to a particle size of ≤30 μm.

RESULTS AND DISCUSSION

Rehydration in an Alkaline Medium

Table 1 presents X-ray diffraction and thermal analysis phase-composition data for rehydrated CTA HG. Figures 2 and 3 show thermal analysis curves for the products obtained at T_{hydr} = 15–20 and 30–35°C and t_{hydr} = 24 and 200 h.

Table 1. Properties of CTA HG products rehydrated under different conditions

Sample no.	Hydration conditions			Phase composition of the product, %									n in $Al_2O_3 \cdot nH_2O$*	S_{BET}, m^2/g	D, Å
	electrolyte	T_{hydr}, °C	t_{hydr}, h	X-ray diffraction					thermal analysis						
				HG	Ba	Bo	Pbo	Am	$Al(OH)_3$	Bo	Pbo	Am			
1	–	–	0	5	–	–	–	95	6	–	–	94	0.8	127	–
2	NH_4OH	15–20	24	6	45	8	10	31	51	8	9	32	2.0	160	300
3	NH_4OH	15–20	200	6	75	8	2	9	86	8	2	4	2.7	120	310
4	NH_4OH	30–35	24	7	70	8	2	13	84	8	3	5	2.6	140	325
5	NH_4OH	30–35	200	7	75	7	2	9	86	7	2	5	2.7	120	350
6	H_2O	15–20	24	7	0	6	40	47	7	6	40	47	2.1	170	60
7	H_2O	15–20	200	7	70	7	7	9	85	6	5	4	2.6	125	350
8	H_2O	30–35	24	7	70	7	6	10	85	5	5	5	2.6	135	350
9	H_2O	30–35	200	7	75	5	3	10	87	5	3	5	2.7	110	400
10**	CH_3COOH	15–20	24	5	–	–	–	95	7	(3)	(50)	40	1.2	120	<30
11**	CH_3COOH	15–20	200	5	–	–	–	95	7	(3)	(55)	35	1.3	180	<30

*n is the total water content of the product, which was calculated as $n = 5.7\Delta m/(100 - \Delta m)$, where Δm (%) is the heat-induced change in the sample weight. The calculation is based on the chemical equation $Al_2O_3 \cdot nH_2O \longrightarrow Al_2O_3 + nH_2O$ (see Figs. 2–6).

**X-ray amorphous oxide hydroxides. The calculated amounts of hydroxide phases are given in parentheses. The formation of these hydroxide phases is confirmed by electron microscopy.

In aqueous ammonia (pH 10) at 15–20°C, CTA HG reacts with water, as follows from the increased heats of rehydration of $Al(OH)_3$, $Al_2O_3 \cdot 1.5H_2O$, and AlOOH and from the increased weight losses indicated by thermal analysis (compare Figs. 1 and 2). Note that these changes are observed even for short dehydration times of about 2 h; however, according to X-ray diffraction data, the product of this short-time rehydration is X-ray amorphous. Prolonging the rehydration time to 8 h results in the formation of pseudoboehmite (Pbo), as is indicated by the diffraction pattern of the product. Further extending the hydration time to 12 h does not result in any increase in the Pbo content, but it leads to a larger D value, which possibly indicates that the Pbo crystallite size has increased. A possible mechanism of the crystallite growth is the formation of fibrils from Pbo needles intergrown at their lateral faces. According to X-ray diffraction data, the highest Pbo content of the rehydration product is ~12%. Extending the aging time to 24 h does not change the Pbo content and yields the bayerite (Ba) phase (45%, D = 300 Å, S_{BET} = 160 m^2/g; Fig. 2a; Table 1, sample 2). According to X-ray diffraction and thermal analysis data, the sample aged for 200 h consists of ~80% Ba having nearly the same D as the material obtained in 24 h (D = 310 Å, S_{BET} = 120 m^2/g; Fig. 2b; Table 1, sample 3).

Raising the rehydration temperature to 30–35°C speeds up bayerite formation. According to X-ray diffraction and thermal analysis data, a product containing ~78% well-crystallized bayerite with D = 325 Å and S_{BET} = 140 m^2/g results even from short-term aging for 24 h (Fig 3a; Table 1, sample 4). Extending the aging time to 200 h results in an insignificantly higher bayerite percentage and in a larger bayerite particle size (D = 350 Å, S_{BET} = 120 m^2/g; Fig. 3b; Table 1, sample 5).

It is expected that further raising the CTA HG dehydration temperature will speed up bayerite formation. A similar trend was reported earlier for the rehydration of HG thermally activated under different conditions [13].

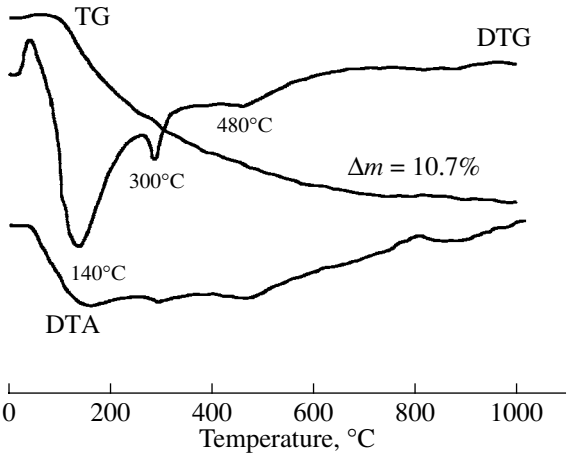

Fig. 1. Thermal analysis data for hydrargillite thermally activated in a TSEFLAR^{TM} reactor and then disintegrated (CTA HG product used as the initial material).

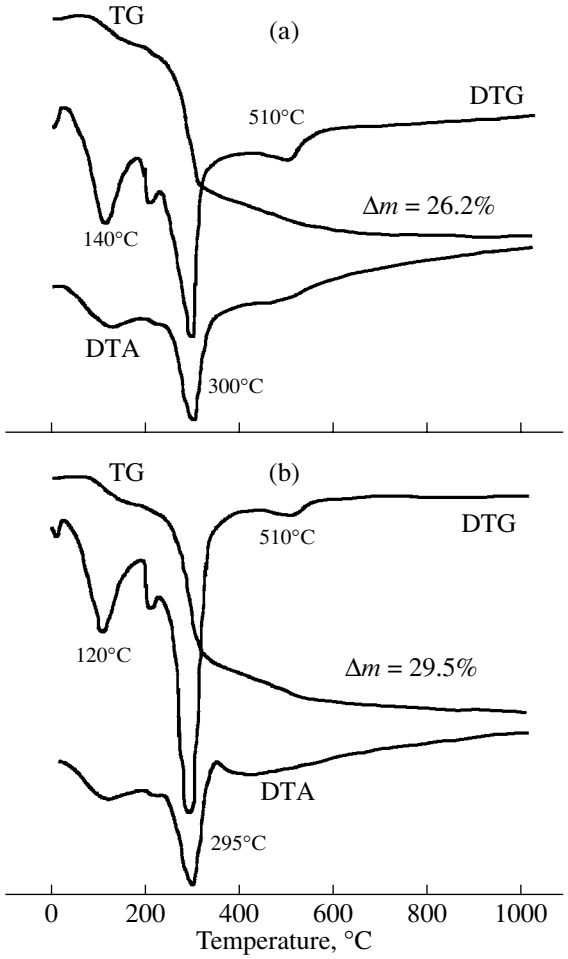

Fig. 2. Thermal analysis data for the CTA HG hydration product obtained in the alkaline medium at T_{hydr} = 15–20°C. t_{hydr} = (a) 24 and (b) 200 h.

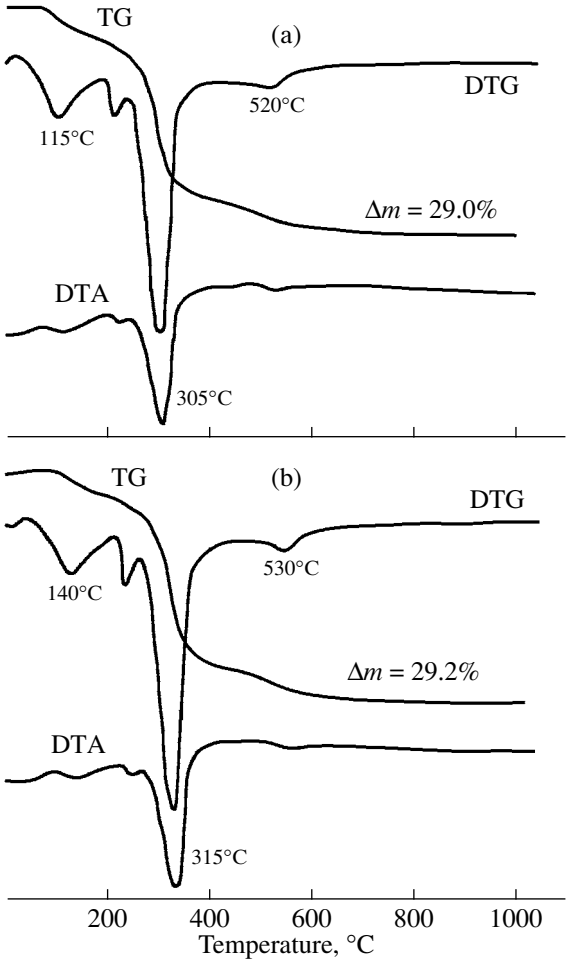

Fig. 3. Thermal analysis data for the CTA HG hydration product obtained in the alkaline medium at T_{hydr} = 30–35°C. t_{hydr} = (a) 24 and (b) 200 h.

Rehydration in Water

As in the case of alkaline rehydration, the rehydration of the CTA product in water at 15–20°C yields an X-ray amorphous hydroxide and then pseudoboehmite, but the formation of the latter phase in water takes longer. Only traces of pseudoboehmite are detected by X-ray diffraction in the sample at the early stages of aging (up to 8 h). The sampled aged for the longer time of 24 h contains 40% pseudoboehmite (Fig. 4a; Table 1, sample 6) with D = 60 Å and is characterized by S_{BET} = 170 m^2/g. Extending the aging time to 200 h reduces the percentage of pseudoboehmite and causes bayerite formation. According to X-ray diffraction data, the sample aged in water for 200 h contains ~78% bayerite with D = 350 Å and is characterized by S_{BET} = 125 m^2/g (Fig. 4b; Table 1, sample 7). Thus, the rehydration of the CTA product in water proceeds in the same way as alkaline rehydration, yielding well-crystallized bayerite as the major phase upon 200-h-long aging.

As in the case of the alkaline medium, raising the rehydration temperature to 30–35°C speeds up bayerite formation. According to X-ray diffraction and thermal analysis data, a product containing ~78% well-crystallized bayerite with D = 350 Å (S_{BET} = 135 m^2/g) is already formed upon 24-h-long aging (Fig. 5a; Table 1, sample 8). Extending the aging time results in a slightly larger amount of bayerite and favors its further crystallization, as is indicated by $D \approx$ 400 Å and S_{BET} = 110 m^2/g (Fig. 5b; Table 1, sample 9).

Rehydration in an Acidic Medium

According to X-ray diffraction data, at 15–20°C, the aqueous electrolyte acidified to pH 5.5 by adding a solution of acetic acid causes no changes in the phase composition of the CTA HG product over a period of time as long as 200 h. The product remains amorphous to X-rays. The solids dried after rehydration have a specific surface area of 120 m^2/g for t_{hydr} = 24 h and 180 m^2/g for t_{hydr} = 200 h.

The acidic-rehydration products show a larger total weight loss (up to 16.5%) in thermal analysis, indicat-

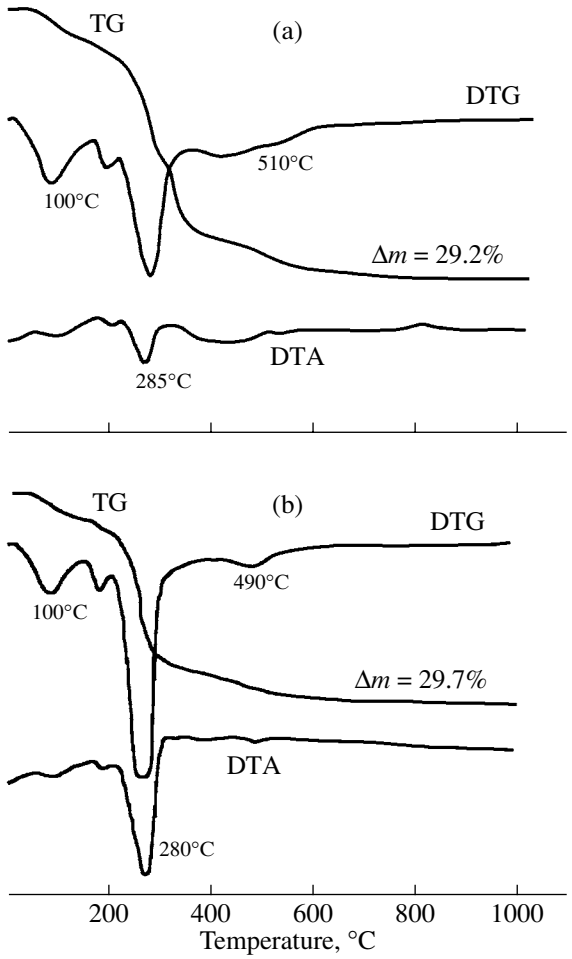

Fig. 4. Thermal analysis data for the CTA HG hydration product obtained in water at T_{hydr} = 15–20°C. t_{hydr} = (a) 24 and (b) 200 h.

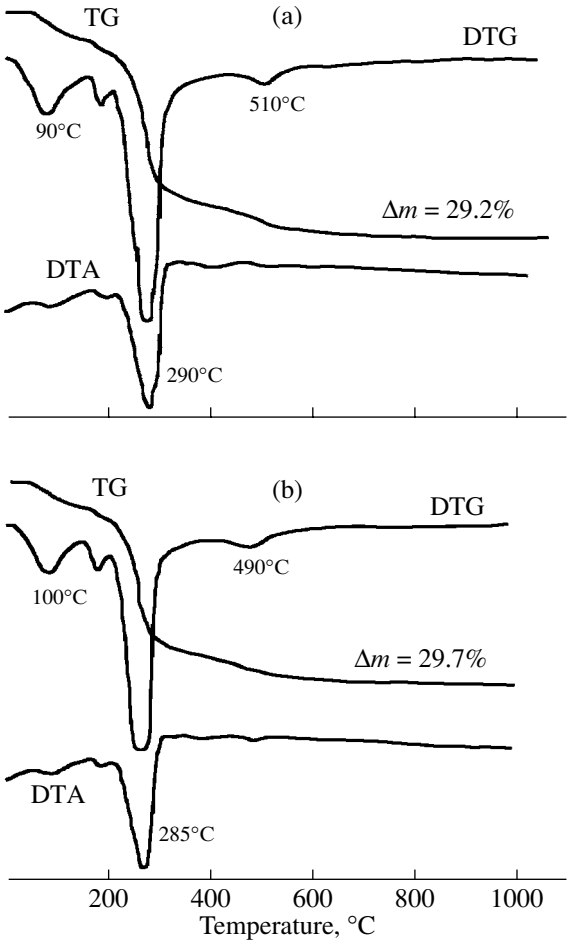

Fig. 5. Thermal analysis data for the CTA HG hydration product obtained in water at T_{hydr} = 30–35°C. t_{hydr} = (a) 24 and (b) 200 h.

ing that the CTA HG product has reacted with water. However, unlike the alkaline- and neutral-rehydration products, the acid-rehydrated products lose weight monotonically, without giving rise to any distinct thermal event. These data suggest that the amorphous oxide hydroxide resulting from CTA changes its water content during acidic rehydration. However, this process is not accompanied by pseudoboehmite, boehmite, or bayerite crystallization. Thus, the acid-rehydrated product is amorphous aluminum hydroxide of variable composition.

Formal calculation of the amounts of pseudoboehmite and boehmite from thermal analysis data indicates that the rehydration product can contain up to 50% pseudoboehmite (Fig. 6a; Table 1, sample 10). It is possible that this phase was not detected by X-ray diffraction because of the small particle size of the rehydration product (size effect).

Note that rehydration in the acid medium is a rather rapid process: according to the formal calculations based on thermal analysis data, a product containing up to 30% pseudoboehmite can form within 2 h. Thermal analysis demonstrated that holding the CTA HG product in the acid solution for a longer time of 200 h results in a larger pseudoboehmite content of ~55% (Fig. 6b; Table 1, sample 11). It is noteworthy that no noticeable amount of bayerite was detected in the product held in the acid medium for 200 h.

Since the amorphous CTA HG product is poorly soluble in water and acid and alkali solutions at the moderate temperatures examined [6], we believe that the rehydration of the CTA product is an S + L reaction involving no redissolution. Therefore, it is possible that the reaction between the amorphous CTA HG product and water first yields primary, amorphous, hydrated particles and that pseudoboehmite or bayerite crystallites result from a secondary process, specifically, the aggregation of primary particles, whose mechanism is possibly similar to oriented growth [14, 15]. If this is the case, the finding that no crystalline phase results from acidic rehydration (X-ray diffraction data) and the fact that the acid-rehydrated product loses less weight

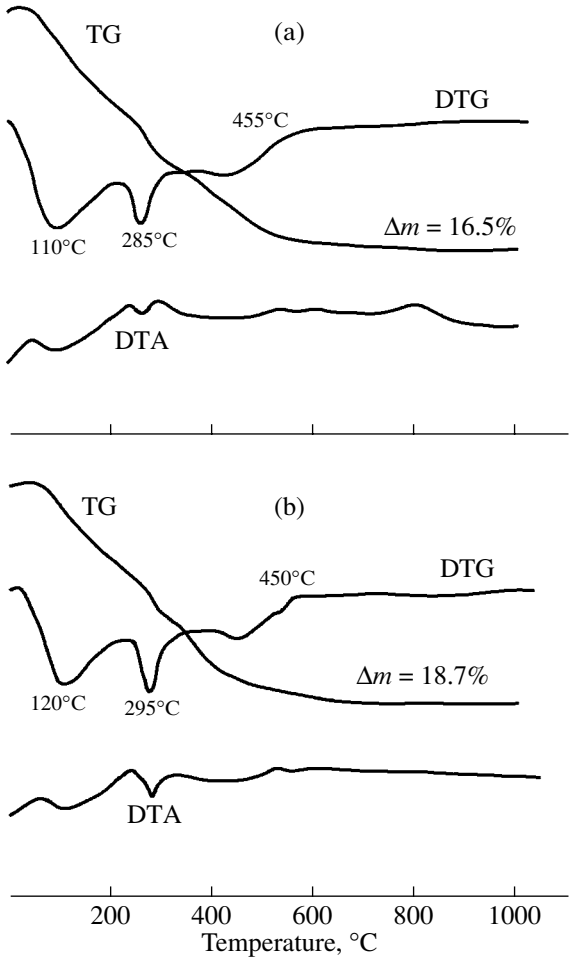

Fig. 6. Thermal analysis data for the CTA HG hydration product obtained in the acidic medium at T_{hydr} = 15–20°C. t_{hydr} = (a) 24 and (b) 200 h.

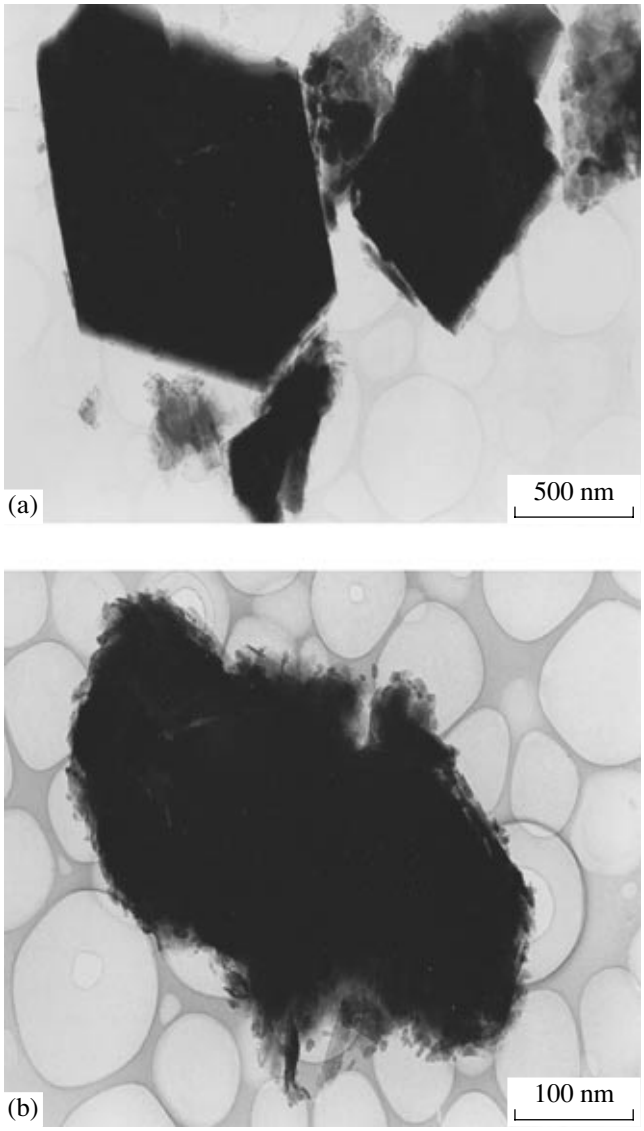

Fig. 7. Electron micrograph of hydrargillite thermally activated in a TSEFLAR™ reactor (CTA HG product used as the initial material).

than the alkaline- or neutral-rehydration product (thermal analysis data) are explained not by the slowdown of the rehydration step but by the slowdown of the aggregation of hydrated particles. This can be due to the formation of anion-modified amorphous oxide hydroxides and changes in the electrical double layer of the hydrated particles in the acidic solutions. Further studies are required to elucidate the factors determining phase formation.

The phase composition data obtained by X-ray diffraction and thermal analysis for the rehydrated CTA HG product are mutually consistent and are in agreement with the known order of phase formation in the aging of fresh, amorphous aluminum hydroxide precipitates, which coarsen by the oriented growth mechanism to yield pseudoboehmite needles and then large particles of bayerite [14].

If the above reaction mechanism is true, it is to be expected that the morphology of the hydration products will be determined by the morphology of the starting CTA product.

Morphology of the Rehydration Products

Electron microscopic and electron microdiffraction data for the rehydrated products are presented in Figs. 7–10.

According to electron microscopic data, the starting material (hydrargillite thermally activated in a TSEFLAR reactor, CTA HG) is a hydrargillite pseudomorphose and appears as faceted particles larger than 1 μm consisting of primary particles smaller than 10 nm. The aggregates have a distinct porous structure (Fig. 7).

The inhomogeneous particles resulting from alkaline rehydration at 20°C for 24 h have a well-crystallized central part and edges as aggregates of smaller, primary particles. The size of some well-crystallized

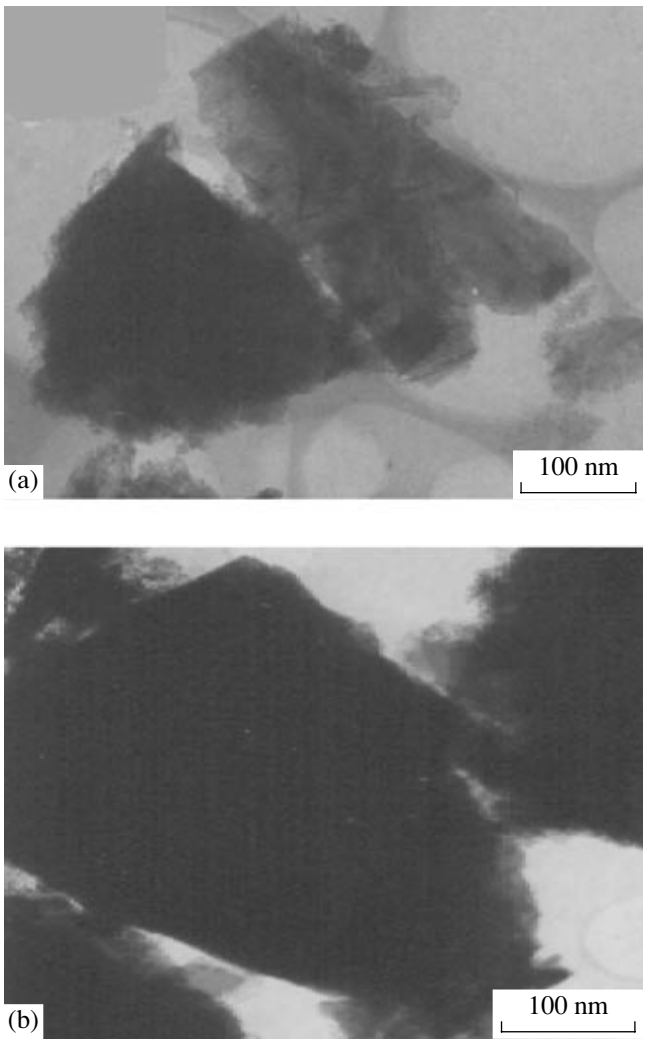

Fig. 8. Electron micrograph of the CTA HG alkaline-hydration product obtained at T_{hydr} = 20°C. t_{hydr} = (a) 25 and (b) 200 h.

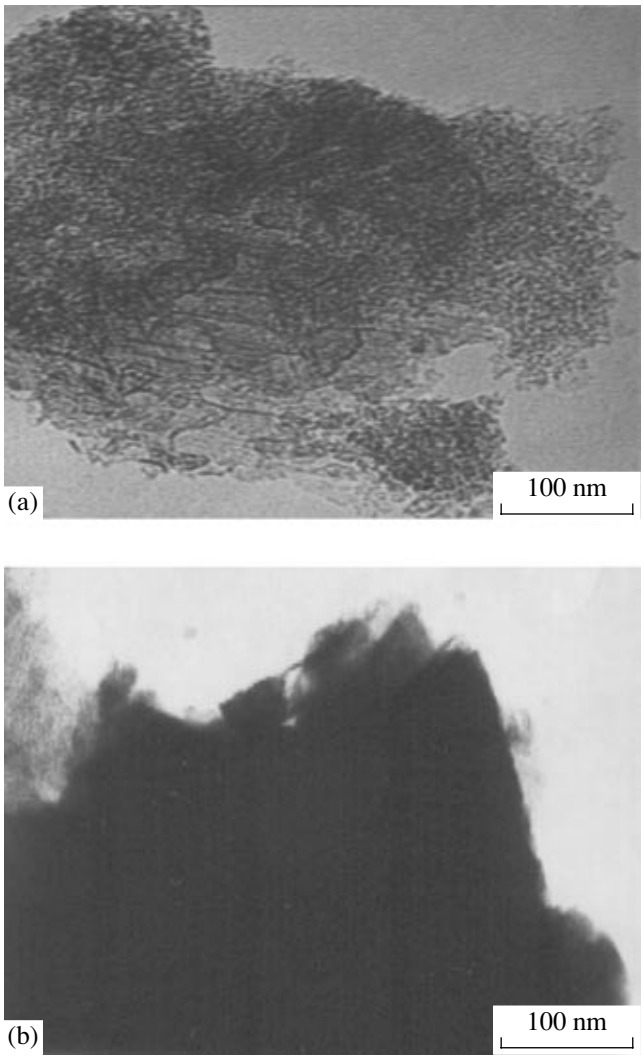

Fig. 9. Electron micrograph of the CTA HG hydration product obtained in water at T_{hydr} = 20°C. t_{hydr} = (a) 25 and (b) 200 h.

bayerite particles is ~300 nm. Few, if any, pseudoboehmite needles are observed (Fig. 8a).

The sample held in the alkaline medium at 20°C for 200 h consists largely of well-crystallized bayerite particles as thin plates with a (001) plane size of ~600 nm. Also present are incompletely crystallized particles consisting of bayerite and aggregates of primary particles. Almost no pseudoboehmite needles are observed (Fig. 8b).

Rehydration in water at 15–20°C for 24 h yields densifiable aggregates of primary particles in which pseudoboehmite whiskers are clearly observed (Fig. 9a). Extending the rehydration time causes the formation of bayerite crystals and the disappearance of pseudoboehmite needles (Fig. 9b).

Rehydration in the acid medium at 15–20°C for 24 h yields densifiable aggregates of primary particles in which pseudoboehmite whiskers can be observed. No crystalline bayerite particles are present (Fig. 10a). Extending the rehydration time to 200 h results in further densification of the layers of ordered primary particles and in a buildup of pseudoboehmite, affording no bayerite particles (Fig. 10b).

The above data confirm the view that the rehydration of the X-ray amorphous particles of the CTA product that are an HG pseudomorphose yields primary, X-ray amorphous, hydrated particles within the initial pseudomorphose. This is followed by the aggregation of the primary hydrated particles yielding a variety of hydroxide phases. The degree of crystallinity of the hydration products depends on the temperature and pH of the medium. Raising T_{hydr} and the pH of the solution speeds up the formation of well-crystallized bayerite particles. Reducing the pH likely slows down the particle aggregation stage, allowing pseudoboehmite needles to be obtained. The rehydration process as a whole proceeds rather rapidly under normal conditions; how-

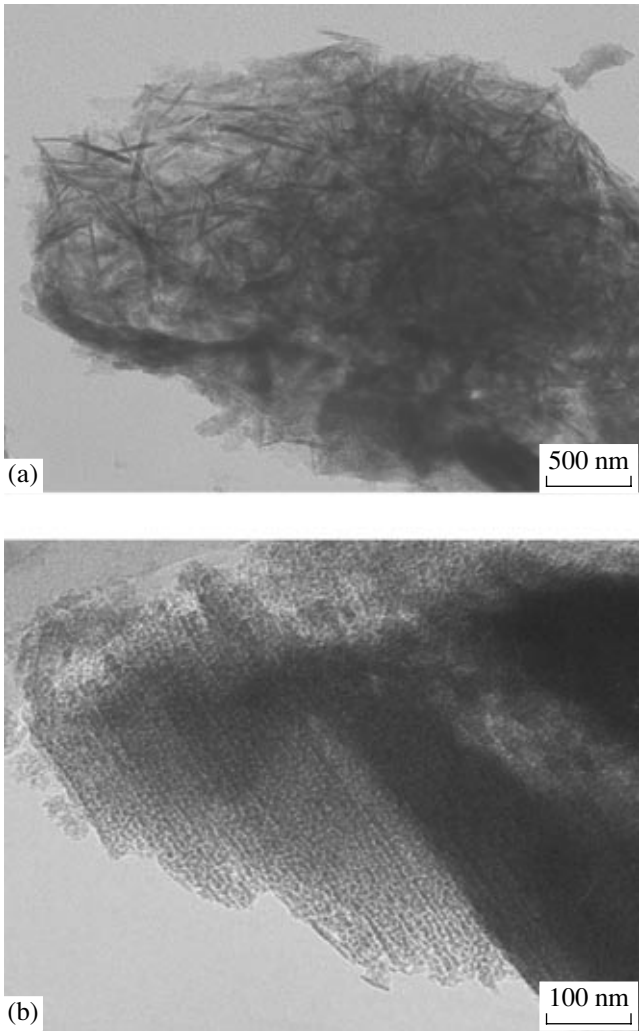

Fig. 10. Electron micrograph of the CTA HG acidic-hydration product obtained at T_{hydr} = 20°C. t_{hydr} = (a) 25 and (b) 200 h.

ever, the formation of pseudoboehmite fibrils and crystalline bayerite particles is governed by hydration conditions.

The resulting pure hydroxide phases or their mixtures (amorphous aluminum hydroxide, pseudoboehmite, and bayerite in various ratios) are usable in the production of alumina-based supports and catalysts with various phase compositions, specific surface areas, acid–base surface properties, and porous structures, including materials based on η-Al_2O_3, a low-temperature alumina modification possessing unique acid–base properties [1].

Properties of Low-Temperature Aluminum Oxides Obtained from the Rehydration Products

The products of CTA HG rehydration in the alkaline, neutral, and acidic media were calcined at 550°C for 4 h. The properties of the resulting oxides are presented in Table 2. According to X-ray diffraction data, the oxides obtained via alkaline and neutral rehydration are η-Al_2O_3 with a unit cell parameter of a = 7.913–7.920 Å, which is nearly equal to the standard value reported for this oxide (JCPDS, no. 4-0875). However, the resulting oxides are somewhat different in terms of the parameter B, which is defined as the intensity ratio of reflection 311 to reflection 222. The parameter B provides a means to evaluate the phase homogeneity of the oxides. For aluminum oxides obtained from precipitated bayerite and pseudoboehmite, B = 1.8–1.9 and 1.2–1.3, respectively [15, 16]. The B data listed in Table 2 suggest that the CTA product samples hydrated in the alkaline medium for 24 or 200 h at 15–20°C and the sample hydrated in water for 200 h at 15–20°C, which consist largely of bayerite, turn into η-Al_2O_3 with B = 1.56–1.70 upon calcination (Table 2, samples 1, 2, 4). For the oxide obtained from sample 3, which was rehydrated in water for 24 h and contained ~40% pseudoboehmite, B = 1.4, indicating that a considerable amount of γ-Al_2O_3 has resulted from calcination. The alumina resulting from the amorphous acidic-rehydration product (Table 2, sample 5) has a B value most similar to that of γ-Al_2O_3 and the smallest value of a. This is possible evidence that the alumina obtained is similar in properties to γ-Al_2O_3.

The oxides obtained are characterized by D = 40 ± 5 Å (Table 2, samples 1–4) and are, therefore, finely dispersed. This is confirmed by our adsorption data, according to which the specific surface area of the η-oxides is 300 ± 20 m^2/g (Table 2, samples 1, 2, 4). For alumina obtained by calcination of precipitated ("pure") bayerite at 550°C, D = 65 Å and S_{BET} = 380 ± 20 m^2/g (Table 2, samples 3, 5). For alumina obtained by calcination of precipitated pseudoboehmite at 550°C, D = 35 Å and S_{BET} = 320 ± 20 m^2/g [16, 17]. The fact that the oxides obtained in this study have a somewhat smaller specific surface area along with a small coherent-scattering domain size may be due to part of the surface being inaccessible to the adsorbate. This can be caused, for example, by the formation of microblock crystallites, which can affect the acid–base properties of the oxide surface.

Thus, the data reported here are evidence that the aluminum hydroxides synthesized by mild rehydration of the CTA products somewhat differ in their properties from the precipitated hydroxides. Accordingly, the aluminum oxides obtained from the rehydrated CTA products also differ in their properties from the oxides derived from the precipitated hydroxides.

CONCLUSIONS

The amorphous product resulting from the centrifugal thermal activation of hydrargillite is rehydratable in aqueous electrolytes at atmospheric pressure and moderate temperatures (15–35°C). The composition of the rehydration product depends on temperature, the pH of medium, and the hydration time.

Table 2. Properties of the aluminum oxides obtained by calcination of rehydrated CTA HG products at 550°C

Sample no.	Hydration conditions at T_{hydr} = 20°C	X-ray diffraction data for aluminum oxide					
		phase	amount*, wt %	a, Å	$B = I_{311}/I_{222}$	D, Å	S_{BET}, m^2/g
1	Alkaline medium, t_{hydr} = 24 h	η-Al_2O_3	40	7.911	1.6	40	285
2	Alkaline medium, t_{hydr} = 200 h	η-Al_2O_3	60	7.913	1.7	45	300
3	Water, t_{hydr} = 24 h	η- + γ-Al_2O_3	55	7.920	1.4	35	250
4	Water, t_{hydr} = 200 h	η-Al_2O_3	59	7.920	1.56	45	290
5	Acidic medium, t_{hydr} = 24 h	η- + γ-Al_2O_3	50	7.903	1.34	40	275
	For comparison						
6	Precipitated bayerite [15, 16]	η-Al_2O_3	100	7.913	1.8–1.9	65	380 ± 20
7	Precipitated pseudoboehmite [15, 16]	γ-Al_2O_3	100	7.911	1.2–1.3	35	320 ± 20

* The amount of η-Al_2O_3 was derived from calibration plots constructed for η-Al_2O_3 + amorphous Al_2O_3 mixtures.

Raising the temperature and pH of the medium and extending the hydration time lead to a product containing up to 80% bayerite with $D = 330 \pm 20$ Å. This product has a fairly large specific surface area of 130 ± 10 m^2/g.

In water and in the alkaline medium, short-time rehydration yields a product with a rather high pseudoboehmite content of ~50% and $D = 60 \pm 10$ Å. This product has a specific surface area of 170 ± 10 m^2/g.

Rehydration in the acid medium does not yield a crystalline phase, but an amorphous hydroxide phase of variable composition.

The results of this study enable one to control the crystalline and amorphous phase contents, the coherent-scattering domain size, and the specific surface area of the CTA HG rehydration products and, therefore, the properties of the oxides resulting from these hydroxides (η- and γ-phases with a large specific surface area). This provides a means of widely varying the physicochemical and mechanical properties of the alumina supports derived from the CTA HG product.

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