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A New Method of Synthesis of Nanosized Boehmite (AlOOH) Powders with a Low Impurity Content

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Abstract—A new method of synthesis of nanosized aluminum oxyhydroxide (AlOOH, boehmite) powders has been suggested through a hydrothermal treatment of nanosized γ-Al_2O_3 powder in water and a 1.5 wt % HCl solution at different temperatures. It has been found that hydrothermal treatment in a 1.5 wt % HCl solution leads to the purification of the starting material; different treatment durations allow one to obtain boehmite particles of different shape. It has been demonstrated that a nanosized boehmite powder is obtained upon the hydrothermal treatment of a nanosized γ-Al_2O_3 in water above 80°C. The nanosized boehmite powders synthesized at different temperatures have been studied by various methods.

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At present, modern industry has shown great interest in nanoscale materials, due to the possibility of their widespread use to increase the economic feasibility of production. In particular, nanosized boehmite powders (AlOOH) differ from micron-sized powders by high chemical activity, adsorption capacity, specific surface area, low specific pore volume, low thermal conductivity, etc. Owing to the high adsorption capacity, nanosized boehmite powder (AlOOH) is widely used as adsorbents, catalyst supports, and catalysts themselves [1]. In addition, nanosized boehmite powder can be used as a filler in composites, pains and varnishes, and decorative materials. Heat treatment of boehmite allows one to obtain different phases of aluminum oxide (χ-, η-, γ-, δ-, κ-, θ-, and α-Al_2O_3) with preservation of the shape and size of the original particles of boehmite. Nanoscale boehmite powder with the content of the basic substance 99.997 wt % after its thermal treatment and conversion to α-Al_2O_3 can be used as a raw material for the synthesis of single crystal sapphire and nonporous corundum ceramics with a density of 4.02 g/cm^3. According to [2], obtaining nanosized boehmite powder is currently of paramount importance for medicine, as modern developments indicate the possibility of its use as a sorbent for the extraction of viruses and bacteria.

According to [3], hydrothermal treatment in water of a widespread industrial raw material $Al(OH)_3$ (hydrargillite) at 200–250°C leads to boehmite with an average particle size of 2–3 μm. Boehmite can also been obtained by mechanical activation of aluminum particles in a planetary mill, followed by aging in distilled water at 90°C [4]. The boehmite particles synthesized in this way are fibers with a width of 10 nm and a length of about 150 nm. In addition, a method has been described for treating aluminum in water with pulsed electrical discharges [5], which results in aluminum erosion and cleaning of the surface from an oxide film, and aluminum interacts with water to form boehmite nanofibers. The simplest method presented in the literature is a method for producing boehmite particles (with sizes up to 100 nm) due to the interaction of aluminum nanopowders obtained by the electric explosion of a conductor with distilled water [6]. All considered methods for producing nanosized boehmite powders are characterized by the presence of nanosized aluminum compounds as a precursor and the complexity of the technologies developed, the implementation of which not only does not purify the starting material, but also causes contamination during processing, for example, in using ball mills.

This paper proposes a new method for the synthesis of nanosized boehmite (AlOOH) powders through hydrothermal treatment of nanosized γ-Al_2O_3 powder

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Table 1. Impurity content (wt %) in boehmite synthesized by hydrothermal treatment of γ-Al_2O_3 in a 1.5 wt % HCl solution at 200°C, $P = 1.6$ MPa, $\tau = 24$ h

Fe	Si	Ca	Mg	Na	K
4.5×10^{-4}	1×10^{-3}	2×10^{-3}	8×10^{-5}	1×10^{-3}	5×10^{-4}

(synthesized by the carbohydrate technology [7]) in water at temperatures above 80°C and in a 1.5 wt % HCl solution at 150 and 200°C, which provides boehmite powders with different habit and low impurity content.

The γ-Al_2O_3 hydrothermal treatment was carried out in laboratory autoclaves with a volume of 18 cm^3 in a Teflon liner in water at temperatures of 130 and 80°C and in a 1.5 wt % HCl solution at 200, 170, and 150°C. Sealed autoclaves were placed in a heated SNOL-M2U42 electric oven and kept in air at a given constant temperature for different periods of time. The treatment time was counted from the moment of autoclave reaching the specified temperature. The use of Teflon liner avoids contamination of the product due to erosion of the autoclave walls.

In the developed method, hydrothermal treatment in a 1.5 wt HCl solution leads to cleaning of the starting nanosized γ-Al_2O_3 powder, which makes it possible to obtain a nanosized boehmite powder with low impurity content and the basic substance content 99.97 wt % (Table 1).

According to X-ray powder diffraction and IR spectroscopy, the hydrothermal treatment of a nanosized γ-Al_2O_3 powder in a 1.5 wt % HCl solution at 200, 170, and 150°C for different periods of time, gives a nanosized boehmite powder in 2.5 h at 200°C, in 12 h at 170°C, and in 36 h at 150°C (Table 2, Fig. 1). According to SEM and PEM, irrespectively of the treatment temperature, the resulting boehmite powder consists of bulky agglomerates about 10–15 μm in size, which are composed of tiny particles 10 to 40 nm in size, with an average size of about 20 nm (Fig. 1). However, after a longer keeping in an autoclave, for example, after the hydrothermal treatment of a nanosized γ-Al_2O_3 powder in a 1.5 wt % HCl solution at 150°C for 96 h, agglomerates retain sizes of about 10–15 μm, but boehmite particles become needle-shaped: about 1–2 μm in length and no more than 20 nm in width (Fig. 2). This phenomenon is presumably associated with the redistribution of crystallites inside the boehmite structure during the hydrothermal treatment in a 1.5 wt % HCl solution. The change in the shape of a boehmite particle depending on the duration of hydrothermal treatment is also reflected in the change in the specific surface area of boehmite. In particular, the specific surface area of boehmite, obtained by hydrothermal treatment of a nanosized γ-Al_2O_3 at 150°C for 36 h, is 66.4 m^2/g (Table 2), which coincides with the specific surface areas of boehmites obtained at 200 and 170°C, which is evidence of the identity of the resulting powders. Further hydrothermal treatment of a nanosized boehmite powder at 150°C for 96 h leads to a change in the particle structure and shape; the specific surface area of this material is 138 m^2/g, which makes this boehmite a good candidate for use as a sorbent for the uptake of viruses and bacteria and as a sorbent for extraction of valuable metals from wastewater of industrial plants and so on.

According to X-ray powder diffraction and IR spectroscopy, the hydrothermal treatment of nanosized γ-Al_2O_3 in water at 130°C for 24 h and at 80°C for 80 h also leads to a nanosized boehmite powder with the properties identical to the boehmite synthesized through hydrothermal treatment of a nanosized γ-Al_2O_3 powder in a 1.5 wt % HCl solution at temperatures from 150 to 200°C (Table 2).

Thus, nanosized γ-Al_2O_3 powders are highly reactive, which indicates the economic feasibility of using them in the production of nanoscale boehmite powders.

A new method for the synthesis of nanosized boehmite (AlOOH) powders has been proposed. As a precursor, nanosized γ-Al_2O_3 powder was used, its hydrothermal treatment in water allows to obtain nanosized boehmite powders at temperatures above 80°C, and its processing at temperatures from 150 to 200°C in a

Table 2. Characteristics of the nanosized boehmite powder synthesized by hydrothermal treatment of a nanosized γ-Al_2O_3 in water and in 1.5 wt % HCl solution at different temperatures

Parametr	In a 1.5 wt % HCl solution			In water	
	200°C	170°C	150°C	130°C	80°C
Average particle size, nm	10–40	10–40	10–40	10–40	10–40
Thermal conductivity, W/(m K)	0.021	0.024	0.023	0.031	0.027
Specific surface area, m^2/g	65.32	67.45	66.34	68.57	67.13
Pore size, nm	1.87	1.9	1.9	1.87	1.87
Specific pore volume, cm^3/g	0.031	0.033	0.030	0.036	0.032
Total conversion time, h	2.5	12	36	24	80

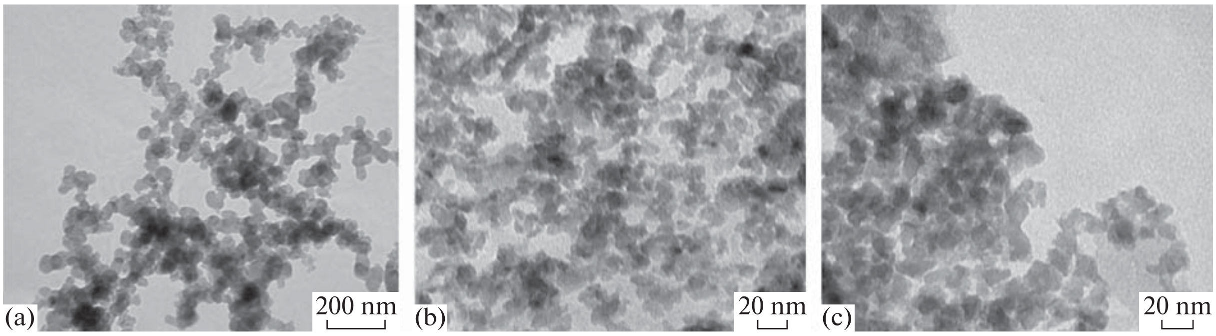

Fig. 1. TEM images of boehmite particles obtained by hydrothermal treatment of a nanosized γ-Al_2O_3 powder in a 1.5 wt % HCl solution (a) at 200°C for 2.5 h, (b) at 170°C for 12 h, and (c) at 150°C for 36 h.

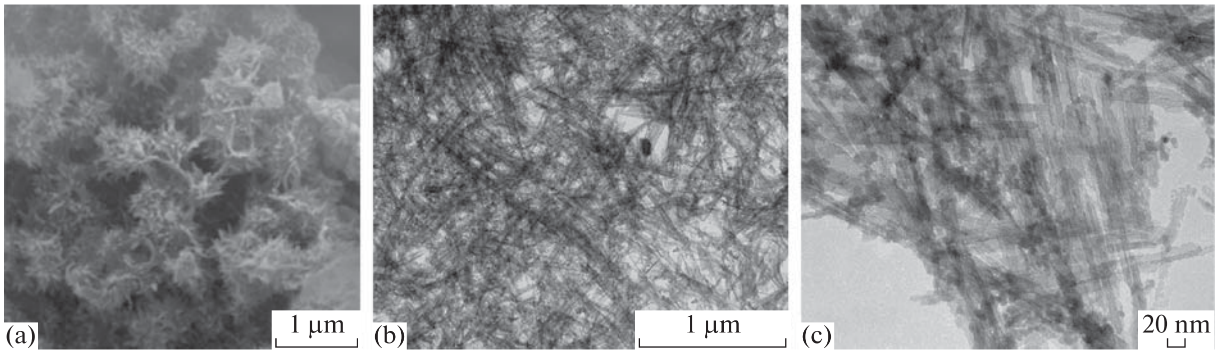

Fig. 2. (a) SEM and (b, c) TEM images of boehmite particles obtained by hydrothermal treatment of a nanosized γ-Al_2O_3 powder in a 1.5 wt % HCl solution at 150°C for 96 h.

1.5 wt % HCl solution allows to obtain nanosized boehmite powders of different habit with a low content of impurities.

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