Synthesis of nanocrystals of corundum

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Nanosized aluminium oxide is widely applied in the modern technics. It can be used in production of rare earth tri-band phosphor, luminescent phosphor with long afterglow, nano-meter grade lapping material, catalyst and support, inorganic membrane, alumina ceramic soleplate, filler and bending agent, high purity or high strength alumina ceramics, transparent alumina ceramic tube for pressure sodium vapor lamps, aluminium nitride, far infrared material and bioceramics. [1], nanostructured ceramics, cements and coatings that are being considered for use in medical applications. Therefore development of new methods of obtaining of nanosized aluminium oxide is enough actual problem. For obtaining of nanocrystals can be used the autoclave treatment of hydroxides, oxides and salts of metals in hydrothermal conditions (temperature 150 - 250ºC) and in water supercritical fluid (temperature above 375ºC) [2]. With use of autoclave treatment the next methods have been developed:

Decomposition of salts of aluminium via aqueous thermovapour treatment.

1.The aluminium lactate was autoclaved in vapour of water at temperature of 400°C during one hour. Commercial aluminium lactate of manufacture of the company of "Reahim" of mark "pure" was used. Treatment was carried out in autoclave of volume of 20 ml, coefficient of filling was 20%.Thus starting salt was decomposed with formation nanocrystals of boehmite (AlOOH) with the size of crystals 50 - 200 nm. Such crystals after the heating up at 1100 - 1200°C do not agglomerate and nor change its habit but pass into alpha aluminium oxide (Fig. 1).

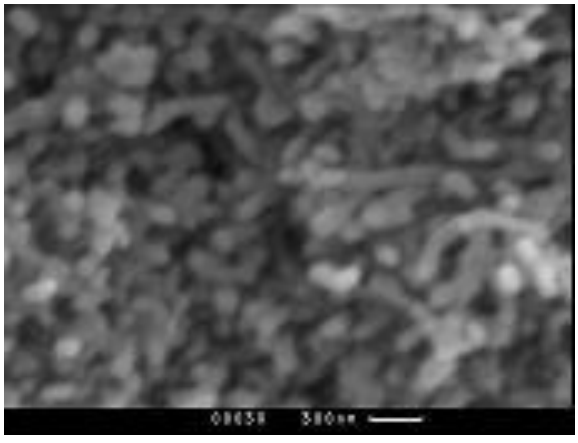

Fig.1. Alpha aluminium oxide obtained by autoclave treatment of aluminium lactate at 400ºC during 1 hour with the subsequent heating at 1100ºC within 5 hours.

2. Aluminium hydroxyacetate was autoclaved, in water vapour, at the temperature of 400°C during 5 hours.This treatment has allowed to obtain boehmite crystals with a thickness 300-500 nm and in length up to 10 microns (Fig. 2) which at heating at 800ºC pass in gamma aluminium oxide (Fig. 3) and at heating at 1200°C pass in an alpha aluminium oxide (Fig. 4). Crystals keep the size and the form at heating up to 1500°C and at temperature above 1500ºC

break up into corundum crystals (Fig. 5). This material can be utilized for manufacture of ceramics, for purification of water and in other areas of technique.

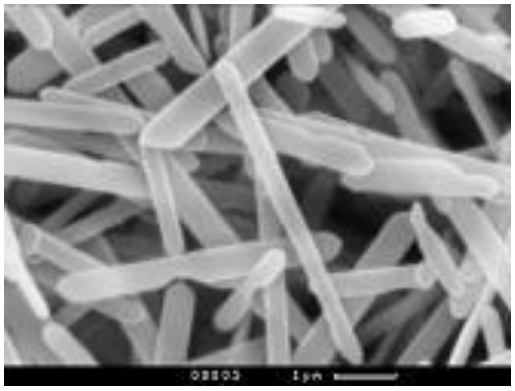

Fig. 2. Boehmite obtained by autoclave treatment of aluminium hydroxyacetate at 400ºC within 24 hours.

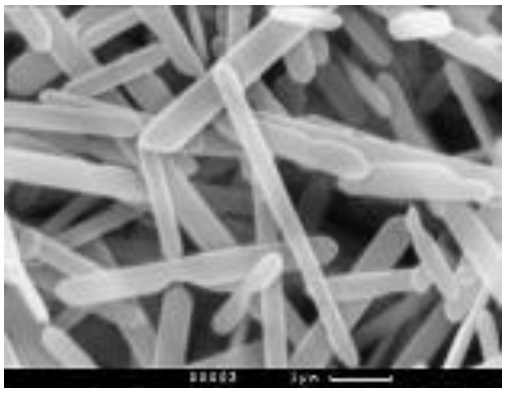

Fig. 3. Gamma aluminium oxide obtained by autoclave treatment of aluminium hydroxyacetate at 400ºC within 24 hour with the subsequent heating at 800ºC within 4 hours.

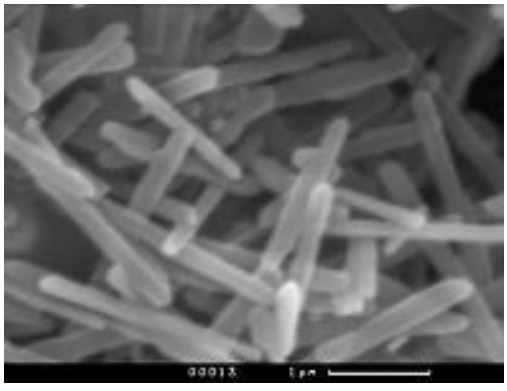

Fig. 4. Alpha aluminium oxide obtained by autoclave treatment of aluminium hydroxyacetate at 400ºC within 24 hour with the subsequent heating at 800ºC within 4 hours and 1200ºC within 1 hour.

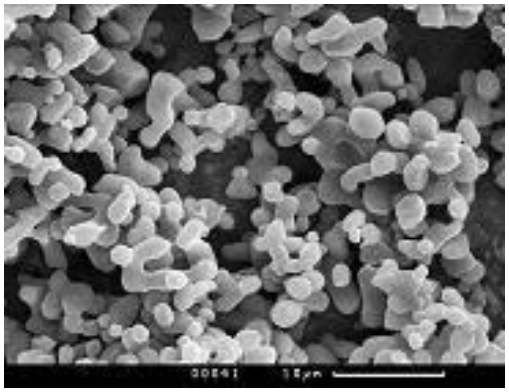

Fig. 5. Alpha aluminium oxide obtained by autoclave treatment of aluminium hydroxyacetate at 400ºC within 24 hours with the subsequent heating at 800ºC within 4 hours, at 1200ºC within 1 hour and at 1500ºC within 1 hour.

Hydrothermal treatment of aluminium hydroxide

Hydrothermal treatment of aluminium hydroxide (hydrargillite) was carried out at the temperature of 200°C in acidic medium. Thus has been obtained boehmite with the size of crystals 300 nm (Fig.6), which at 1250°C transfers in an alpha alumina without a sintering and change of a habit. (Fig.7). Heating at 1500°C leads to disintegration of initial crystals on smaller crystals of corundum which sinter at subsequent heating (Fig.8).

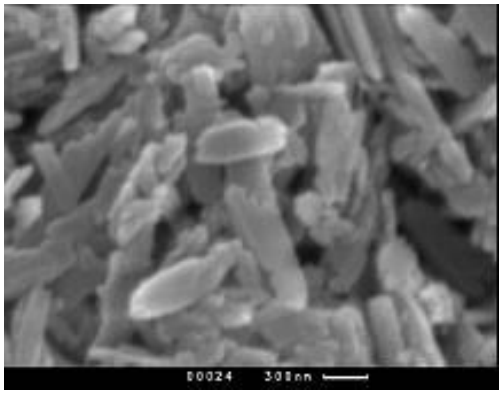

Fig 6. Hydrargillite after treatment in autoclave in 1.5% solution of HCl at 200ºC during 7 days.

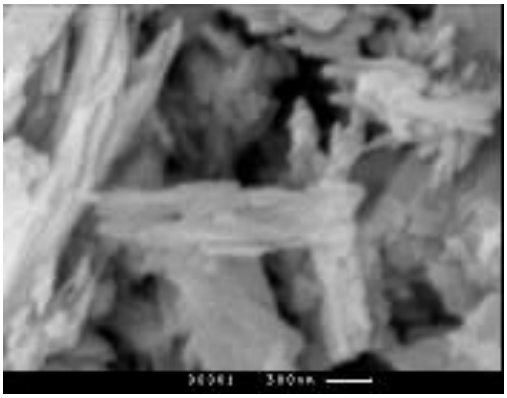

Fig 7.Hydrargillite after treatment in autoclave in 1.5% solution of HCl at 200ºC during 7 days and subsequent heating at 1200^{o}C within 5 hours.

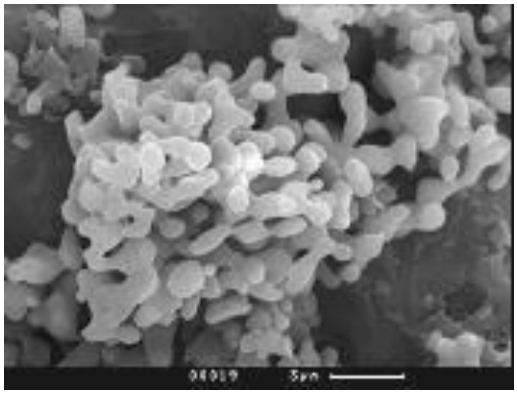

Fig. 8. Hydrargillite after treatment in autoclave in 1.5% solution of HCl at 200ºC during 7 days and subsequent heating at 1500°C within 1 hour.

Decomposition of supramolecular structures at thermal treatment

Decomposition of crystals of supramolecular structure: terephthalic acid - aluminium hydroxide was investigated. For obtaining of these crystals, solution of A1OC1 and terephtalate of ammonium were mixed up. The solid precipitate was washed out and dried at 100ºC. The synthesized crystals were decomposed at temperature of 700°C with formation of gamma aluminum oxide (Fig.9), which at 1250°C pass into nanocrystals of alpha aluminium oxide without agglomeration and change of the crystal habit.

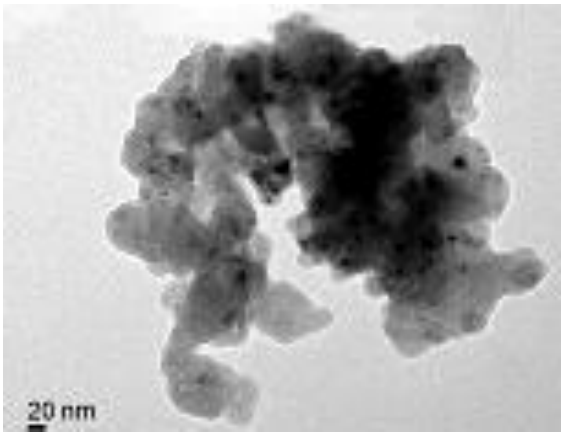

Fig. 9. Gamma aluminium oxide obtained from supramolecular structure a terephthalic acid – aluminium hydroxide after heating at 700ºC within 4 hours.

Obtaining of hydroxide aluminium from solutions ammonium and salts of organic acids

The crystallization of aluminium hydroxide from solutions of aluminium chloride and ammonium terephtalate. The crystallization from such solutions allows to obtain the fibrous aluminium hydroxide with the size of crystals about 50 nm..(Fig .10)

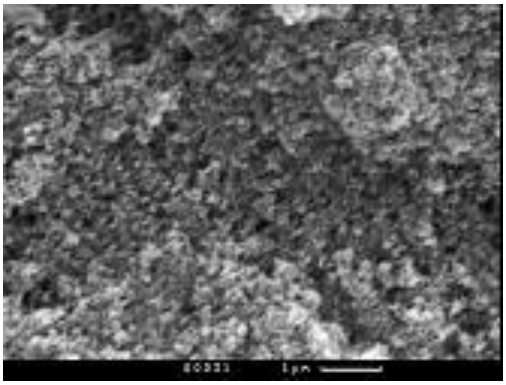

Fig. 10. Hydrargillite obtained by interaction of solutions of aluminium chloride and ammonium terephtalate.

References:

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