NEW WAYS OF OBTAINMENT OF NANOCRYSTALS ALUMINUM HYDROXIDES AND OXIDES

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Nano powders of aluminium oxides and hydroxides (corundum, γ- alumina, α- alumina, boehmite, hydrargillite) are used in particular for creating different kinds of ceramics, especially strong nano ceramics, ceramics with low temperature of sintering , transparent ceramics; for matrixre in forcement of organic or metallic layers, as fillers, as polishing powders, for the production of abrasives, as additives in paints and laminates, for creation of the superthin heat insulator in structure of heat-insulating paints, filtration membranes and catalyst substrates, water purification and in other areas of modern technics.

Ceramic materials may be created from specially synthesized corundum powder whose crystals have the definite shape The open porosity and the strength property for ceramic materials made from these powders depends on the sizes and habitus of the powder particles used. Therefore obtaining of nanosized aluminium hydroxides and oxides is enough actual problem.The production of the ultrafine alumina powders is effected either by chemical synthesis, mechanical comminution methods or a thermophysical methods.

In work some developed new methods of obtaining of nanosized aluminium hydroxides and oxides are presented.

Perspective method of obtaining of such materials is autoclave treatment For nano boehmite can be used the autoclave treatment of hydroxides, oxides and salts of aluminium in hydrothermal conditions /1/ (temperature 150 - 250ºC). For obtaining of nano corundum can be used autoclave treatment of aluminium hydroxide (hydrargillite or boehmite) in water supercritical fluid /2/ (temperature above 375ºC). For obtaining of nano gamma aluminium oxide can be used thermal treatment of nano boehmite/3/.

For this purpose Mutual transitions of aluminum hydroxides and oxides at hydrothermal treatment in water solutions, at thermovapour treatment in a supercritical water fluid and at thermal treatment was investigated. The general scheme of these transformations is presented in fig.1. Apparently from the presented scheme, the combination of methods of hydrothermal, thermovapour and thermal treatment allows to obtain nanosized boehmite , gamma and alpha aluminum oxides practically from any initial raw materials. In particular, in most cases as a result of hydrolysis of or a decomposition of salts of aluminium are formed aluminium hydroxide (amorphous, hydrargillite, gels) which as a result of the subsequent thermal or hydrothermal treatment can be transformed to a demanded phase state.The principal question of technology of obtaining of aluminium nano oxides is preservations of size and habitus of particles at thermal treatment. In this connection the change of the size and habitus of particles of of boehmite, obtained in various conditions, depending on warming up temperature has been investigated. It is shown that the size and habitus of particles of initial boehmite practically does not change at warming up to 1300°C. Decomposition of particles and their sintering begins only at 1400°C..

For example 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 (A) which at heating at 800ºC pass in gamma aluminium oxide (B) and at heating at 1200°C pass in an alpha aluminium oxide (C). Crystals keep the size and the form at heating up to 1500°C and at temperature above 1500ºC break up into corundum crystals (D). This material can be utilized for manufacture of ceramics, for purification of water and in other areas of technique.

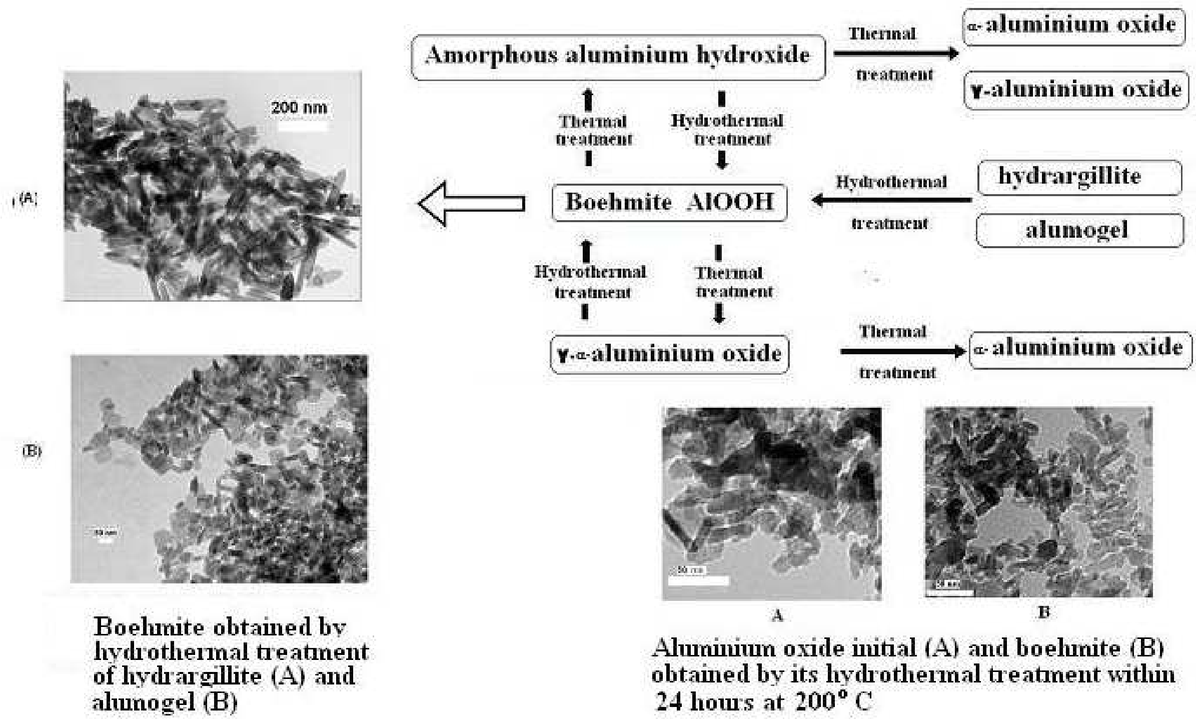

Fig.1 The general scheme of mutual transitions of aluminum hydroxides and oxides at hydrothermal and thermal treatment

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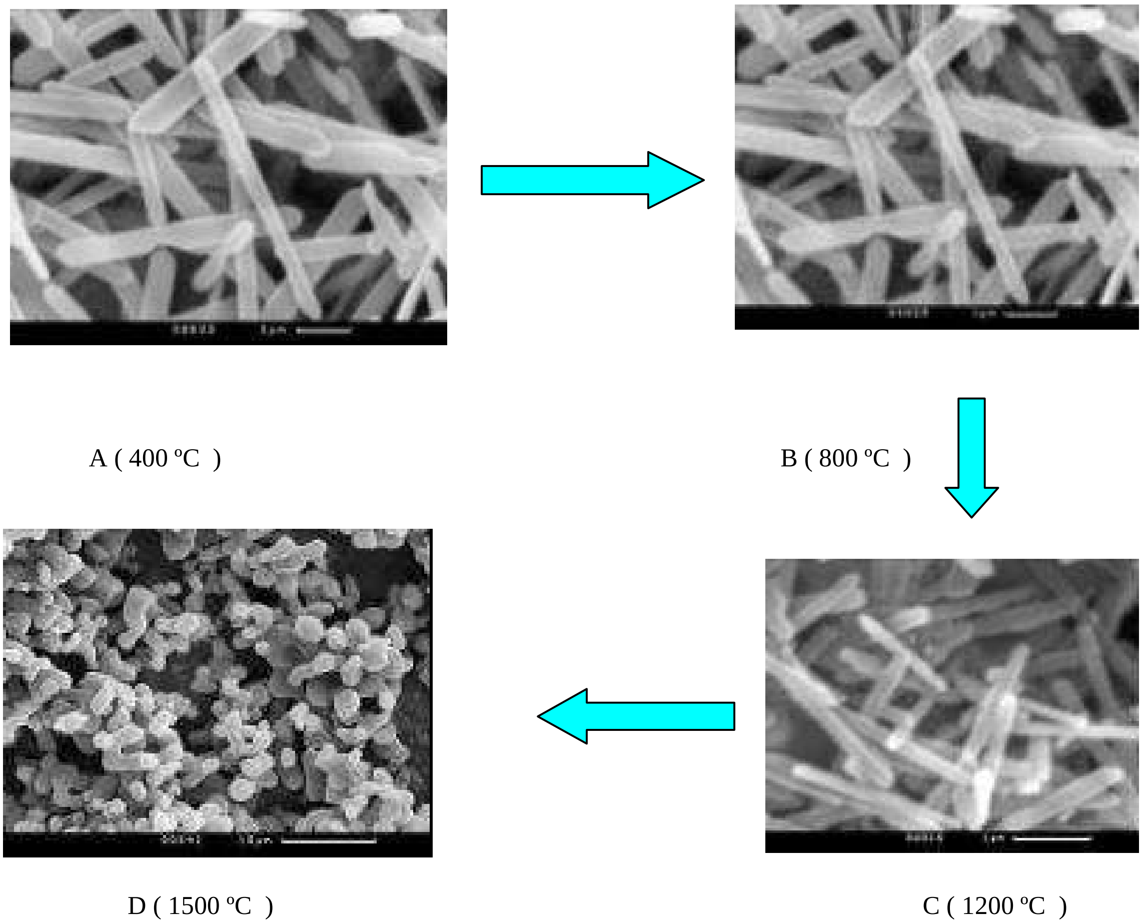

Fig 2 Preservation of size and habitus of crystals of aluminium oxides at thermal treatment

A)Boehmite obtained by autoclave treatment of aluminium hydroxyacetate at 400ºC within 24 hours

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

C) Alpha aluminium oxide obtained by autoclave treatment of aluminium hydroxyacetate at 400ºC within 24 hour with the subsequent heating at 800ºC within 4 hours and1200ºC within 1 hour.

D) Alpha aluminium oxidebtained byautoclave 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

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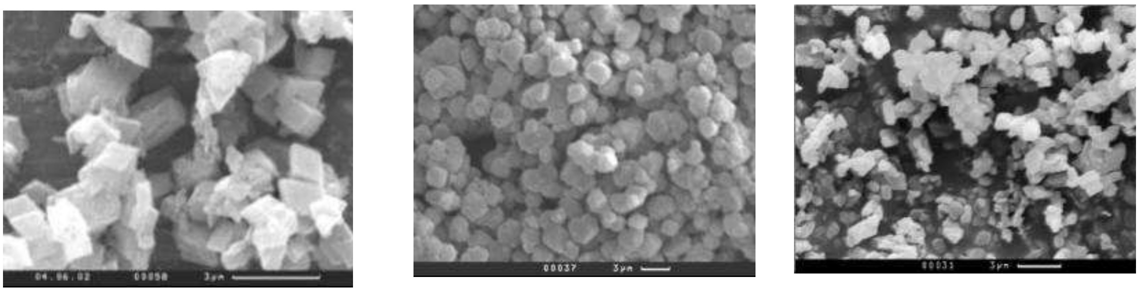
The developed ways of obtaining of nanosized aluminum hydroxides and oxides are presented lower

1. Autoclave treatment of raw materials

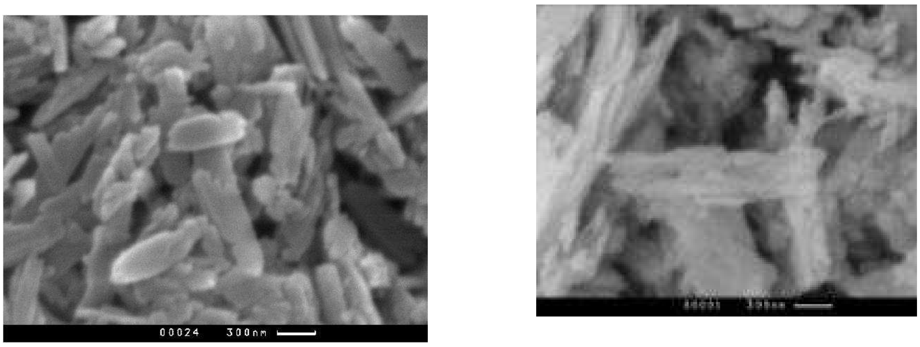
1) Obtaining of nanosized boehmite by hydrothermal treatment and its translation in alpha and gamma aluminum oxide by the subsequent thermal treatment



A B C

Boehmite Corundum α - aluminium oxide

Fig.3 Boehmite prepeared by autoclave treatment of hydrargillite MDGA in water at temperature 200°C during 3 days (A), and after that heating at 1300˚C in air during 5 hours (B) and in autoclave in water vapour at 400°C during 3 days (C),



A B

Fig 4 Boehmite prepeared from hydrargillite MDGA at 200 ˚C in 1.5% solution of hydrochloric acid during 3 days (A) and after that heating at 1300 ˚C during 5 hours (B) The average size of crystals of 0.7 microns

2).Obtaining of non aggregated of boehmite

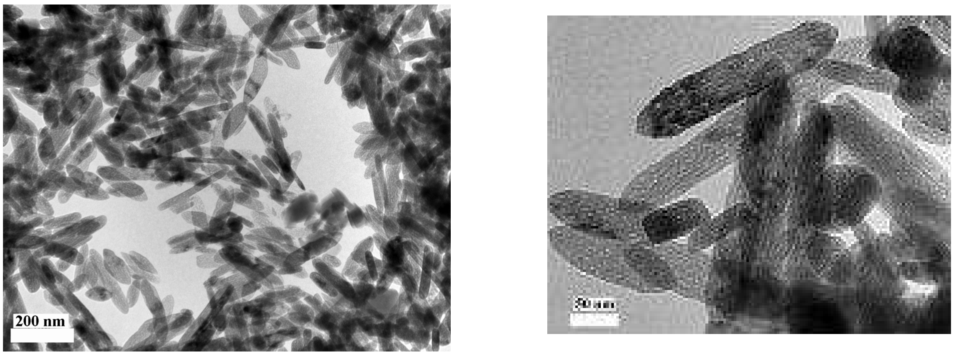


Fig 5. Boehmite prepeared from hydrargillite MDGA at 200 ˚C in 1.5% solution of hydrochloric acid during 3 days and treated after that by 10% solution of ammonium citrate

3). Reduction of the size of crystals of obtained boehmite

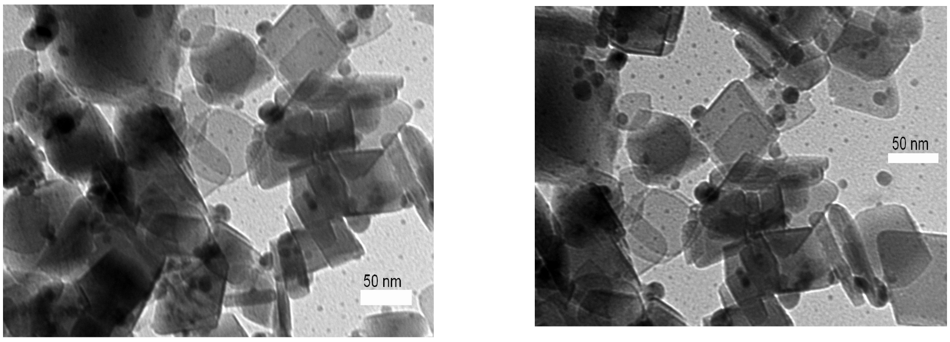
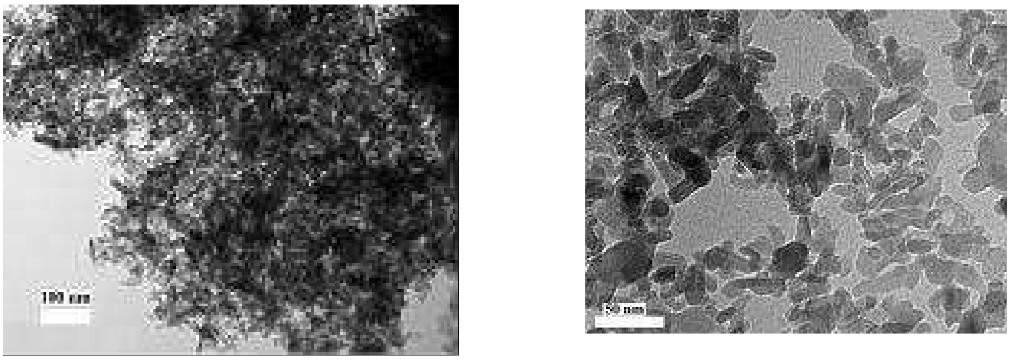


Fig 6 Reduction of the size of crystals of boehmite after treatment of CO_2

4) Conversion to boehmite of gamma aluminium oxide



A B

Fig 6 Gamma aluminium oxide prepeared from china abrasives corporation Hainan Branch (A) and boehmite obtained by autoclave treatment of this material at 200 ˚C in 1.5% solution of hydrochloric acid during 24 hour (B)

2. Obtaining of nanosized gamma aluminum oxide with low bulk density by thermal decomposition of the concentrated solutions of sugar containing aluminum salts.

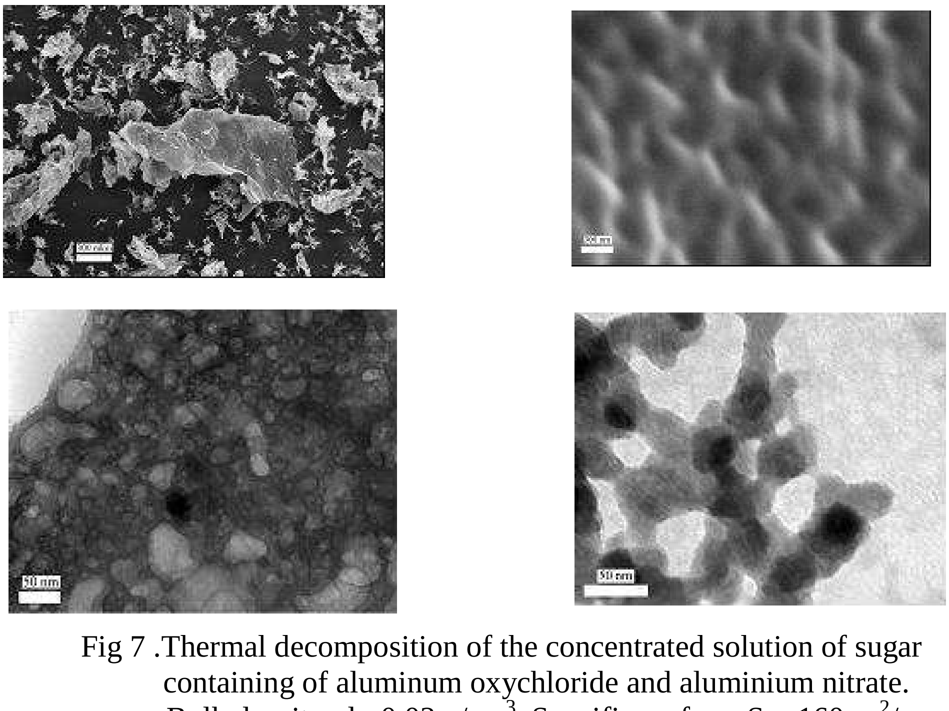


Fig 7 .Thermal decomposition of the concentrated solution of sugar containing of aluminum oxychloride and aluminium nitrate. Bulk density d =0.02 g/cm^3, Specific surface S = 160 $м^2$/g, Heat conductivity at room temperature of 0.03 Wt / (м-K).

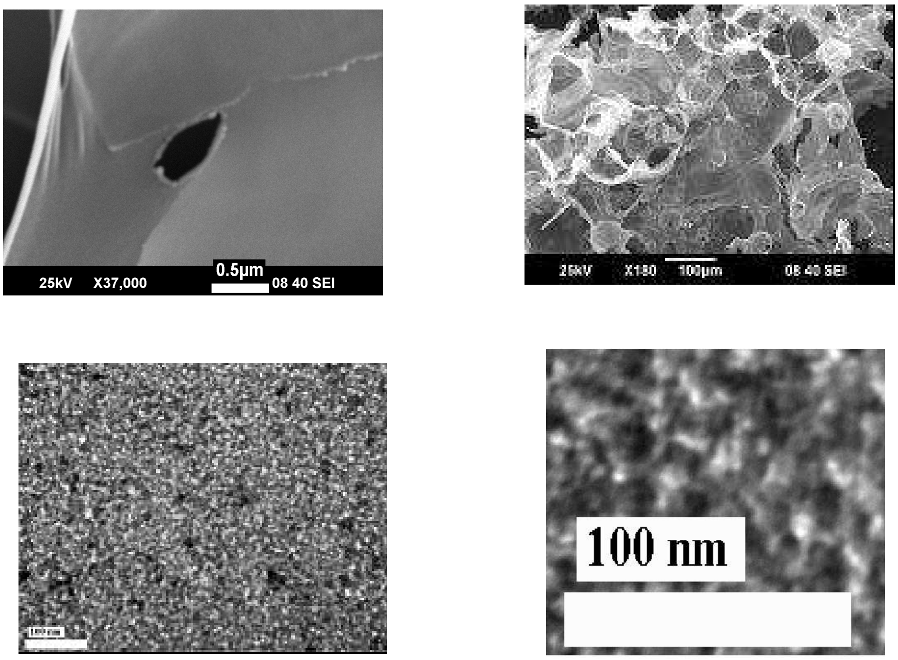


Fig 8 Thermal decomposition of the concentrated solution of sugar containing of aluminum oxychloride.
Bulk density d =0,011 g/cm^3. Specific surface S = 145 м^2/g,
Heat conductivity at room temperature of 0.02 Wt / (м-К),

Use of this method has allowed to obtain gamma and alpha aluminum oxides with following properties:
Bulk density 0.01 – 0,04 g/sm^3. Specific surface 160 – 180 m^2/g Heat conductivity at room temperature of 0.02-0.03 Wt / (м-К). Open porosity: to 99%. The average size of particles is 5-40 nanometers.

The obtained powders of gamma aluminum oxides with low values of bulk density and heat conductivity by some properties are identical to aerogels. In particular, value of bulk density same as at aerogels though value of a specific surface is more low.

There is a principle possibilityof application of the obtained material in following areas of technics:
1. A heat insulation .
Direct use of obtained powders:
Creation of heat insulation building panels with density
0.01 – 0.04 g/sm3, nonflammable, not absorber of moisture.
The cryogenic technics: heat conductivity in vacuum of 0.002-0.003 Wt / (м-К) at room temperature

2. A firefighting.
   Application in powder fire extinguishers
3. Improvement of properties of ceramics.
   Reduction of temperature of sintering and improvement of properties of ceramics.
4. Improvement of properties of metals. Additives to melt of metals
5. Usage for storage of certain substances, in particular, rocket fuel and an oxidizer that allows to replace liquid fuel on solid.

CONCLUSION

Mutual transitions of aluminum hydroxides and oxides at hydrothermal and thermal treatment are investigated. Ways of obtaining of nanosized boehmite , gamma and alpha aluminum oxides from any accessible initial raw materials are developed. Ways of obtaining of nanosized aluminum hydroxides and oxides with low values of bulk density and heat conductivity are developed. The properties of the obtained materials are identical to aerogels, that defines their wide technical application.

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