Microscopy of Nanoobjects

Structural and morphological features of disperse alumina synthesized using aluminum nitrate nonahydrate

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Transformation of $Al(NO_3)_3 \cdot 9H_2O$ (upon heating in the range of 20-1200°C) into blends of amorphous and crystalline boehmite (210-525°C), amorphous alumina and crystalline -Al_2O_3 (850°C), and crystalline -Al_2O_3 (1100°C) was analyzed using XRD, HRTEM, IR spectroscopy, thermogravimetry and low-temperature nitrogen adsorption. Investigations of calcination effects on transformations of aluminum nitrate nonahydrate into boehmite, -Al_2O_3 and -Al_2O_3 show that it occurs according to scheme

$$Al(NO_3)_3 \cdot 9H_2O\ (100\text{-}140°C) \rightarrow [Al(OH_2)_6]^{3+} \cdot 3NO_3^-\ (140\text{-}152°C) \rightarrow$$
$$[AlOH(OH_2)_5]^{2+} \cdot 2NO_3^-\ (152\text{-}210°C) \rightarrow AlONO_3 \cdot H_2O\ (210\text{-}525°C) \rightarrow$$
$$AlOOH\ (525\text{-}1000°C) \rightarrow \gamma\text{-}Al_2O_3\ (1100°C) \rightarrow \alpha\text{-}Al_2O_3.$$

Amorphous and crystalline phases of boehmite formed at $T > 210$°C represent nanoparticles of 6-10 nm in size strongly aggregated since $S_{BET} = 66$ m^2/g is smaller than it should be for unbound nanoparticles of the same sizes. In the amorphous phase, chains with – AlOH – O – AlOH – have the length of 1-5 nm. Heating at 350-525°C results in the formation of mesoporous aluminum hydroxide with decreasing size (2.5-5.0 nm) of nanoparticles. However, tight joining of these nanoglobules strongly reduces the specific surface area to 180 m^2/g instead of 350 m^2/g for individual spherical nanoparticles of similar sizes. Subsequent heating at 850°C leads to enhanced binding of γ-Al_2O_3 nanoparticles in strongly aggregated structures and S_{BET} drops down to 77 m^2/g. Corundum particles formed at 1100°C are characterized by additionally increased joining in the aggregates because the value of S_{BET} is minimal (14 m^2/g).