

## Effect of hydrothermal treatment of $\gamma$ -Al<sub>2</sub>O<sub>3</sub> on boehmite properties

© Svetlana R. Egorova,<sup>+</sup> Alia N. Muhamedyarova,  
Yuqing Zhang, and Alexander A. Lamberov\*

Physical Chemistry Division. A.M. Butlerov Institute of Chemistry. Kazan Federal University.  
Kremlevskaya, 18. Kazan, 420008. Tatarstan Republic. Russia.  
Phone: +7 (843) 233-73-46. E-mail: Segorova07@gmail.com

\*Supervising author; <sup>+</sup>Corresponding author

**Keywords:** alumina, boehmite, hydrothermal treatment, phase transformation, porous system.

### Abstract

The effect of conditions of  $\gamma$ -Al<sub>2</sub>O<sub>3</sub> hydrothermal treatment on the phase composition and porous system parameters of the products obtained at T = 150-200 °C, P = 0.5-1.5 MPa and pH = 4.0-9.5 was studied. The products of  $\gamma$ -Al<sub>2</sub>O<sub>3</sub> hydrothermal treatment in the aqueous suspension are phases of boehmite and bayerite with an admixture of gibbsite. Bayerite is formed in an amount of up to 4.9 % by weight. At T $\approx$ 129-172 °C and pH = 9.5 aluminum hydroxides crystallize at the same time parallel routes by the mechanism of the dissolution of aluminum oxide and boehmite (bayerite) precipitation. Platelike crystals of boehmite are formed at pH = 4.0-9.5. At pH = 6.0-9.5 and T = 180-200 °C three-dimensional particles of the boehmite type are formed as a result of the cross-linking of plates. Phase conversion of  $\gamma$ -Al<sub>2</sub>O<sub>3</sub> to boehmite in an amount up to 90.3-99.8 wt% in the hydrothermal condition is carried out at 190-200 °C and pH = 6.0-9.5 after 90-180 minutes and it is accompanied by a decrease in the SBET values from 207 to 26-30 m<sup>2</sup>/g and VBET from 0.64 to 0.27-0.46 cm<sup>3</sup>/g. Mesopores with a diameter of 3.1-9.5 nm are formed at T = 150-190 °C and pH = 4.0-7.3 due to close packing of the primary particles of boehmite with D (020) = 17.0-41.0 nm, D (120) = 12.7-31.8 nm with its concentration of ~40-60 wt %. Mesopores with a diameter of 10.2-37.0 nm are formed at T = 180-200 °C and pH = 4.0-9.5 by packing larger platelike crystals of boehmite with D (020) = 21.8-44.5 nm, D (120) = 23.1-38.4 at its concentration in the of ~60-90 wt% of mass. Pores with a diameter of 68.5-72.6 nm are formed at T = 180-200 °C and pH = 6.0-9.5 as a result of the formation of three-dimensional packets at a concentration of 90% in the samples.

### References

- [1] A.R. Auxilio, P.C. Andrews, P.C. Junk, L. Spiccia, D. Neumann, W. Raverty, N. Vanderhoek, J.M. Pringle. Functionalised pseudo-boehmite nanoparticles as an excellent adsorbent material for anionic dyes. *J. Mater. Chem.* **2008**. Vol.18. P.2466-2474. DOI:10.1039/b715545j
- [2] I.S. Park, M.S. Kwon, N. Kim, J.S. Lee, K.Y. Kang, J. Park. Rhodium nanoparticles entrapped in boehmite nanofibers: recyclable catalyst for arene hydrogenation under mild conditions. *Chem. Commun. (Camb)*. **2005**. P.5667-5669. DOI:10.1039/b511577a
- [3] A.V.H. Soares, G. Perez, F.B. Passos. Alumina supported bimetallic Pt-Fe catalysts applied to glycerol hydrogenolysis and aqueous phase reforming. *Appl. Catal. B Environ.* **2016**. Vol.18. P.77-87. DOI:10.1016/j.apcatb.2015.11.003
- [4] M. Teixeira, S.C. Rodrigues, M. Campo, D.A. Pacheco Tanaka, M.A. Llosa Tanco, L.M. Madeira, J.M. Sousa, A. Mendes. Boehmite-phenolic resin carbon molecular sieve membranes-Permeation and adsorption studies. *Chem. Eng. Res. Des.* **2014**. Vol.92. P.2668-2680. DOI:10.1016/j.cherd.2013.12.028
- [5] F. Granados-Correa, J. Jiménez-Becerril. Chromium (VI) adsorption on boehmite. *J. Hazard. Mater.* **2009**. Vol.162. P.1178-1184. DOI:10.1016/j.jhazmat.2008.06.002
- [6] G. Lee, C. Chen, S.T. Yang, W.S. Ahn. Enhanced adsorptive removal of fluoride using mesoporous alumina. *Microporous Mesoporous Mater.* **2010**. Vol.127. P.152-156. DOI:10.1016/j.micromeso.2009.07.007
- [7] V.G. Hill, K.G. Zimmerman. The hydrothermal growth and thermal decomposition of boehmite single crystal. *J. the Am. Mineral.* **1970**. Vol.55. P.285-288. URL: [http://www.minsocam.org/ammin/AM55/AM55\\_285.pdf](http://www.minsocam.org/ammin/AM55/AM55_285.pdf)

- [8] S.R. Egorova, A.N. Mukhamed'yarova, A.A. Lamberov. Specific features of the phase transition of gibbsite into boehmite under hydrothermal treatment of floccules in an aqueous suspension. *Rus. J. Appl. Chem.* **2015**. Vol.88. P.758-768. (russian)
- [9] I. Levin, D. Brandon. Metastable Alumina Polymorphs: Crystal Structures and Transition Sequences. *J. Am. Ceram. Soc.* **2005**. Vol.81. P.1995-2012. DOI:10.1111/j.1151-2916.1998.tb02581.x
- [10] S. Music, D. Dragcevic, S. Popovic. Hydrothermal crystallization of boehmite from freshly precipitated aluminum hydroxide. *Mater. Lett.* **1999**. Vol.40. P.269-274. DOI:10.1016/S0167-577X(99)00088-9
- [11] K.T. Hwang, H.S. Lee, S.H. Lee, K.C. Chung, S.S. Park, J.H. Lee. Synthesis of aluminium hydrates by a precipitation method and their use in coatings for ceramic membranes. *J. Eur. Ceram. Soc.* **2001**. Vol.21. P.375-380. DOI:10.1016/S0955-2219(00)00209-0
- [12] B.E. Yoldas. Hydrolysis of aluminium alkoxides and bayerite conversion. *J. Appl. Chem. Biotechnol.* **1973**. Vol.23. P.803-809. DOI:10.1002/jctb.5020231103
- [13] S.K. Mehta, A. Kalsotra, M. Murat. A new approach to phase transformations in gibbsite: the role of the crystallinity. *Thermochim. Acta.* **1992**. Vol.205. P.191-203. DOI:10.1016/0040-6031(92)85260-3
- [14] T. Tsuchida. Hydrothermal synthesis of submicrometer crystals of boehmite. *J. Eur. Ceram. Soc.* **2000**. Vol.20. P.1759-1764. DOI:10.1016/S0955-2219(00)00052-2
- [15] Absi-Halabi, A. Stanislaus, H. Al-Zaid. Effect of acidic and basic vapors on pore size distribution of alumina under hydrothermal conditions. *Appl. Catal. A. Gen.* **1993**. Vol.101. P.117-128. DOI:10.1016/0926-860X(93)80142-D
- [16] F. Roelofs, W. Vogelsberger. Dissolution kinetics of nanodispersed  $\gamma$ -alumina in aqueous solution at different pH: Unusual kinetic size effect and formation of a new phase. *J. Colloid Interface Sci.* **2006**. Vol.303. P.450-459. DOI:10.1016/j.jcis.2006.08.016
- [17] J. Madarasz, G. Pocol, C. Novak, F.T. Cobos, S. Gal. Studies on isothermal kinetics of some reactions of aluminum oxides and hydroxides. *J. Thermal Anal.* **1992**. Vol.38. P.445-454. DOI:10.1007/BF01915509
- [18] C. Novak, G. Pokol, V. Izvekov, T. Gal. Studies on the reactions of aluminium oxides and hydroxides. *J. Thermal Anal.* **1990**. Vol.36. No.10. P.1895-1909. DOI:10.1007/BF01913436
- [19] G. Lefevre, M. Duc, P. Lepeut, R. Gaplain, M. Fedoroff. Hydration of  $\gamma$ -alumina in water and its effects on surface reactivity. *Langmuir.* **2002**. Vol.18. P.7530-7537. DOI: 10.1021/la02565li
- [20] X. Carrier, E. Marceau, J-F. Lambert, M. Che. Transformations of  $\gamma$ -alumina in aqueous suspensions. 1. Alumina chemical weathering studied as a function of pH. *J. Colloid Interface Sci.* **2007**. Vol.308. P.429-437. DOI:10.1016/j.jcis.2006.12.074
- [21] Desset, O. Spalla, P. Lixon, B. Cabane. Variation of the surface state of  $\alpha$ -alumina through hydrothermal treatments. *Colloids Surf. A Physicochem Eng. Asp.* **2002**. Vol.196. P.1-10. DOI:10.1016/S0927-7757(01)00493-4
- [22] I.V. Kozerozhets. Razrabotka metoda polucheniia i issledovanie submikronnykh i nanorazmernykh chastits oksidov aluminiia s nizkim soderzhaniiem primesei: Dis. kand. khim. nauk: 02.00.04. Kozerozhets Irina Vladimirovna. *Moscow.* **2011**. 129p.
- [23] W. Ma, P.W. Brown. Mechanisms of reaction of hydratable aluminas. *J. Am. Ceram. Soc.* **1999**. Vol.82. No.2. P.453-456. DOI: 10.1111/j.1551-2916.1999.tb20085.x
- [24] M. Trueba, S.P. Trasatti.  $\gamma$ -alumina as support for catalysts: a review of fundamental aspects. *Eur. J. Inorg. Chem.* **2005**. Vol.2005. No.17. P.3393-3403. DOI: 10.1002/ejic.200500348
- [25] A. Stanislaus, K. Al-Dolma, M. Absi-Halabi. Preparation of a large pore alumina-based HDM catalyst by hydrothermal treatment and studies on pore enlargement mechanism. *Journal of Molecular Catalysis A: Chemical.* **2002**. Vol.181. P.33-39. DOI: 10.1016/S1381-1169(01)00353-3
- [26] J. Li, L. Xiang, X. Feng, Z. Wang. Influence of hydrothermally modified  $\gamma$ - $Al_2O_3$  on the properties of NiMo/ $\gamma$ - $Al_2O_3$  catalyst. *Appl. Surf. Sci.* **2008**. Vol.254. P.2077-2080. DOI: <http://dx.doi.org/10.1016/j.apsusc.2007.08.037>
- [27] V.A. Dzisko, A.P. Karnaukhov, D.V. Tarasova. Fiziko-khimicheskie osnovy sinteza okisnykh katalizatorov. *Novosibirsk: Nauka.* **1978**. 384p. (russian)