EFFECT OF SYNTHESIS CONDITIONS OF BOEHMITE ALOOH FROM $AL(OH)_3$ ON ITS PHYSICOCHEMICAL CHARACTERISTICS

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ABSTRACT

Boehmite AlOOH is an important industrial materials for utilizing in preparing advanced catalysts, adsorbents, etc. In this paper, With Al(OH)3 as precursor, the high surface area AlOOH powders were prepared using heating treatments. The AlOOH boehmite is synthesized at different temperatures of 300- 450°C. The structure and morphology of AlOOH were characterized by X-ray diffraction (XRD), scanning electron microscope (SEM), BET surface area and Fourier transformed infrared (FTIR). The synthesized AlOOH at 400°C has shown highest surface area (237 m2/g). The boehmite particles synthesized at temperature of 400ºC gave an XRD trace whose peak intensity order and d-spacing most closely matched with the standard pattern.

INTRODUCTION

The synthesis of inorganic micro- and nanostructure materials with special morphologies has been of great interest in recent years because the intrinsic properties of materials are mainly determined by their composition, structure, crystallinity, size and morphology [1]. Among the various inorganic materials, boehmite AlOOH and γ-Al_2O_3 are important industrial materials for use in advanced catalysts, adsorbents, abrasives, composite materials, ceramics, filters and separation [2-5]. Various methods have been reported on the synthesis of AlOOH and γ-Al_2O_3 [6]. Recently, Ren and et.al have reported microwave-assisted construction of hierarchical mesoporous-macroporous boehmite AlOOH and Al_2O_3 [5]. Buchold and Feldmann have synthesized nanoscale AlOOH hollow spheres using water-in-oil micro emulsion templates [7]. Wu and et al. reported the synthesis of AlOOH self-encapsulated and hollow architectures using the amphiphilic block copolymer, poly-styrene-block-poly-hydroxyl-ethyl-acrylate, as a structure-directing reagent [8]. Porous alumina has also been prepared from the precursors of diethyl aluminum amide, acetone, and water [9]. However, most of the previously reported mesoporous aluminas were comprised mainly amorphous framework walls, which would limit their hydrothermal stability and greatly compromise their usefulness in catalytic applications. The size and morphology of the final Al_2O_3 particle depend greatly on the precursor of AlOOH in the process of chemical decomposition.

In this work, we demonstrate a simple route to synthesize boehmite AlOOH by the heat treatment of $Al(OH)_3$ at different temperature of 300 - 450°C and characterization of synthesized AlOOH has been reported.

EXPERIMENTAL

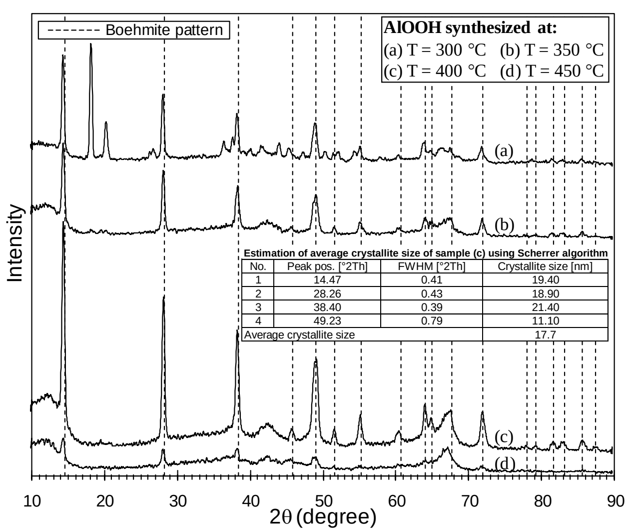
Boehmite AlOOH samples were prepared by heating of aluminium hydroxide ($Al(OH)_3$, 99%, Merck) as precursor. The $Al(OH)_3$ powder was heated with the rate of 1-2ºC/min under air flow for 4 hrs at the temperatures of 300, 350, 400 and 450°C. The boehmite samples are named a, b, c and d in the following, which, respectively, heating at 300, 350, 400 and 450°C.

Powder X-ray diffraction (XRD) patterns, The specific surface area (BET), Scanning electron microscopy (SEM) and Fourier transformed infrared (FT-IR) were used to observe the characteristics of the prepared AlOOH powders. The phase identification of as-produced powders was carried out using a computer controlled X-ray diffractometer (Siemens XRD Diffractometer D5000). The diffractometer scanned from 10 to 90 θ. The BET surface area was determined by a multipoint BET method (CHEMBET-3000, using the adsorption data in the relative pressure (P/P_0) range of 0.1-0.3. The adsorption branch of nitrogen adsorption-desorption isotherms was used to determine the surface area of synthesized AlOOH.

RESULTS AND DISCUSSIONS

XRD Analysis:

The XRD results for the boehmite obtained under different temperature are presented in Figure 1 indicating that a, b and c samples show boehmite as the only crystalline phase detected by X-ray diffraction. However, the powders produced using different temperature gave XRD traces in which the order of the relative peak intensities differed from the standard pattern (PDF NO. 01-083-2384). The elongated boehmite particles synthesized at temperature of 400ºC (Figure 1-c) gave an XRD trace whose peak intensity order and d-spacing most closely matched the standard pattern (PDF NO. 01-083-2384). In lower temperatures (samples a and b), the order of the relative peak intensities differed from the standard pattern. This is most likely related to the change in boehmite particle size producing varying degrees of preferred orientation.



No.	Peak pos. [°2Th]	FWHM [°2Th]	Crystallite size [nm]
1	14.47	0.41	19.40
2	28.26	0.43	18.90
3	38.40	0.39	21.40
4	49.23	0.79	11.10
Average crystallite size			17.7

Figure 1
Effect of temperature on XRD patterns of synthesized boehmite.

It can be seen from Figure1 that, during thermal treatment boehmite undergoes from AlOOH to γ-Al_2O_3. Furthermore, the intensity of XRD reflections depends on the degree of crystalline perfection; thus, apparent variations in temperature could just be due to an increase or decrease in the crystalline order. For example, the synthesis temperature increased from 300 to 400ºC, the median particle size increased from 19.40 to 21.40 nm. But with increased synthesis temperature from 400ºC to 450ºC, the median particle size decreased from 21.40 to 17.7 nm.

SEM Analysis:

The SEM microstructures and morphology of the produced AlOOH powders using heating of $Al(OH)_3$ at 400ºC are shown in Figure 2. As can be seen from the figure, the majority of the particles are in a highly agglomerated state, and there is a wide range of particle size distribution resulting in the presence of some very fine, 70 nm particles as well as larger 4 μm particles. However, it is believed that the heating conditions used in this work lead to the formation of a three-dimensional tabular morphology resulting from the $Al(OH)_3$ in different directions with different particles.

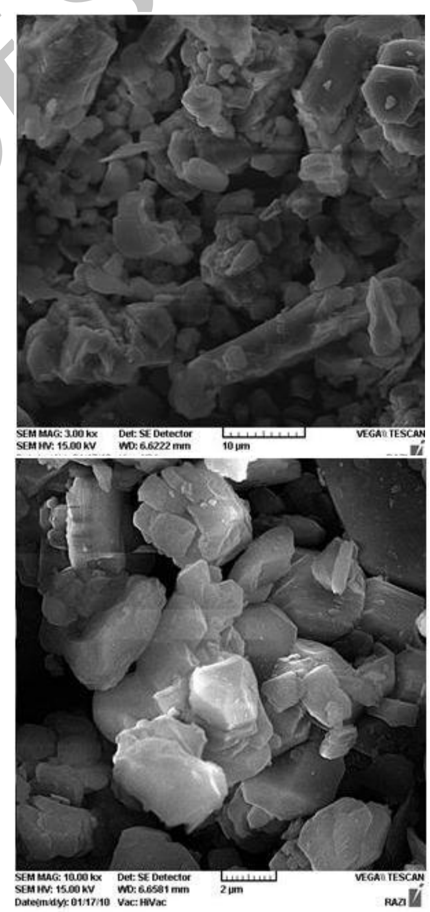


Figure 2
. SEM images of synthesized Boehmite at a temperature of 400°C.

BET Analysis:

Figure 3 shows the BET surface area of synthesized AlOOH at the four different temperatures. By increasing the treatment temperature from 300 to 400ºC, the surface area of synthesized AlOOH is increased (from 126 to 237 m^2/g), while if the temperature exceed 400ºC (400 to 450ºC) the surface area of synthesized AlOOH is decreased. Therefore, the temperature of 400ºC for synthesis of AlOOH from $Al(OH)_3$ is suitable

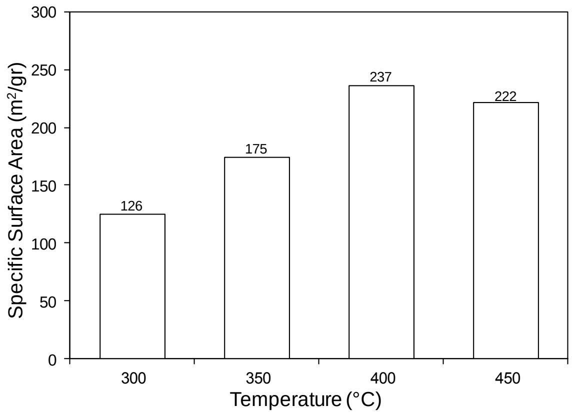


Figure 3
Effect of temperature on BET surface area of synthesized boehmite.

FTIR Analysis:

FTIR measurements (a Mattson 1000 FTIR spectrometer) were conducted on a sample containing AlOOH, as shown in Figure 4.

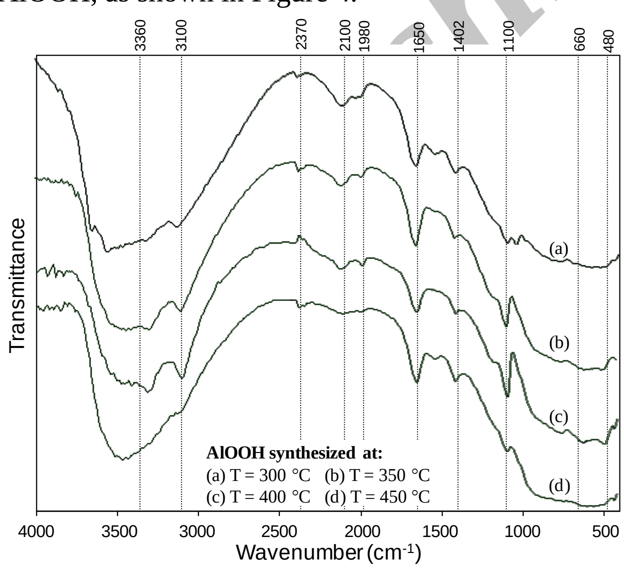


Figure 4
Effect of temperature on FTIR spectrums of synthesized boehmite.

For the boehmite AlOOH, 7 strong bands of 480, 660, 1100, 1650, 2100, 3100, and 3360 cm−1 can be observed indicating the asymmetric stretching. The adsorption edge of the hydroxyl bands on the surface was found at 1650 cm-1 and the intense bands at 3360 and 3100 cm−1 were assigned to the asymmetric and symmetric O-H stretching vibrations from (O)Al-OH. The band at 1100 cm−1 was ascribed to symmetric and asymmetric Al-O-H bending. The broad band around 2100 cm−1 was known to come from the combination band.

CONCLUSIONS

In the present work boehmite samples with high surface areas have been successfully synthesized by heat treatment of $Al(OH)_3$. The samples prepared at 400°C showed the highest surface area (237 m^2/g). The average crystallite size of AlOOH are determined by XRD which was in the order of 17.7 nm. The peak intensity order and d-spacing of this sample most closely matched with the standard patterns. The synthesized boehmite AlOOH is expected to have very good potential applications in preparing supported catalysts and adsorbents.

ACKNOWLEDGMENTS

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REFERENCES

[1] J T. He, L. Xiang, W. Zhu, Sh. Zhu, H2SO4-assisted hydrothermal preparation of γ-AlOOH nanorods, Materials Letters 62 (2008) 2939–2942.

[2] A. Ebadi, J.S. Soltan Mohammadzadeh, A. Khudiev, Adsorption of Methyl tert-butyl Ether on Perfluorooctyl Alumina Adsorbents – High Concentration Range, Chem. Eng. Technol. 30 (2007)1666–1673.

[3] R.D. Cortright, R.R. Davda, J.A. Dumesic, Hydrogen from catalytic reforming of biomass-derived hydrocarbons in liquid water, Nature 418(2002)964-967.

[4] G. Paglia, E.S. Bozˇin, S.J.L. Billinge, A novel fine-scale nanostructure in \gamma-Al2O3, Chem. Mater. 18 (2006) 3242-3248.

[5] T.Z. Ren, Z.Y. Yuan, B.L. Su, Microwave-assisted preparation of hierarchical mesoporous-macroporous boehmite AlOOH

and gamma-Al2O3, Langmuir 20 (2004)1531-1534.

[6] X. Wu, D. Wang., Z. Hu, G. Gu, Synthesis of γ-AlOOH (γ-Al2O3) self-encapsulated and hollow architectures, Materials Chemistry and Physics 109 (2008) 560–564.

[7] D.H.M. Buchold, C. Feldmann, Nanoscale γ-AlO(OH) Hollow Spheres: Synthesis and Container-Type Functionality, Nano.Lett.7 (2007) 3489-3492

[8] X.Wu, D.Wang, Z.Hu, G.Gu, Synthesis of γ-AlOOH (γ-Al2O3) self-encapsulated and hollow architectures, Mater.Chem.Phys.109 (2008) 560-564.

[9] L. Wilcox, G. Burnside, B. Kiranga, R. Shekhawat, M.K. Mazumder, R.M quist, Porous Alumina Prepared from Diethylaluminum Amide, Acetone, and Water, Chem.Mater. 15 (2003) 51-56.