



Comparative parametric study on development of porous structure of aluminium oxide in presence of anionic and cationic surfactants

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Abstract

Aluminium oxide was prepared from aluminium nitrate by precipitation in presence of cationic cetyl trimethyl ammonium bromide (CTAB) and anionic sodium dodecyl sulfate (SDS) surfactants. Effects of surfactant type and concentration, pH, aging temperature and aging time on development of porous structure of aluminium oxide were investigated and compared. Surface area of samples prepared with CTAB was higher compared to that prepared with SDS at all preparation conditions. Mainly mesopores were obtained with SDS, whereas in presence of CTAB micropores were also obtained, particularly at lower surfactant concentration. The bayerite and boehmite were predominant structures in presence of CTAB and SDS, respectively. Formation of mesopores was favored at higher pH in presence of SDS and at lower pH in presence of CTAB. Surface area and pore volume of aluminium oxide increased with aging time in presence of SDS, but the effect was not significant for CTAB. © 2014 Elsevier Ltd and Techna Group S.r.l. All rights reserved.

Keywords: Aluminium oxide; Porous structure; Precipitation; CTAB; SDS

1. Introduction

Aluminium oxide is widely used in chemical and petrochemical industries as catalyst, catalyst support and adsorbent material. Aluminium oxide has high porosity, surface area and thermal stability [1]. Mesoporous aluminium oxides are highly desirable in petrochemical industry involving extensive coke deposition and large feed molecules [2]. The pore blocking by coke deposition is minimized and diffusion of molecules is facilitated in mesoporous aluminium oxides. Preparation of mesoporous aluminium oxide in presence of different surfactants is reported by various authors [2–4]. Synthesis of aluminium oxide in presence of surfactant is based on hydrolysis and condensation of aluminium precursor in interaction with the self-assembled surfactants. Mesoporous aluminium oxide can be prepared from organic and inorganic precursors in presence of different anionic, cationic and non ionic surfactants [5–11]. Khalil et al. [5] investigated formation

of mesoporous aluminium oxide at room temperature by using aluminium isopropoxide, acetic acid and different molar concentration of CTAB in n-heptane medium. Aguado et al. [6] studied the effect of calcination temperature and time on properties of mesoporous aluminium oxide, prepared by sol-gel method in acidic medium using cationic surfactant. Surface area decreased with time and temperature of calcination. Ray et al. [7] reported superior textural property for aluminium oxide prepared with CTAB in organic solvent using alkoxide precursor. Lesiant et al. [8] reported the importance of temperature and length of autoclave phase but was unable to conclude the role of micelle aggregates in formation of mesopores. Valange and coworkers [9] reported that the nature of aluminium precursor and surfactant controlled the pore structure. Ruihong et al. [10] observed that aluminium oxide source and pH also affected the pore structure. Yue and coworkers [11,12] reported very low surface area and large pores for aluminium oxide prepared with CTAB surfactant. They suggested negligible interaction between surfactant and inorganic aluminium precursors. They reported increase in surface area and decrease in pore size in presence of organic

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modifiers such as citrate or tartarate. Kim et al. [13] obtained aluminium oxide with average pore size of 3 nm in presence of CTAB and urea. The aluminium oxide was prepared by spray pyrolysis of aluminium nitrate precursor. Literature on the mechanism of aluminium hydroxide formation in presence of surfactant is limited. Yada et al. [14] proposed that layered structure of mesoporous aluminium oxide transformed to hexagonal structure when prepared from aluminium nitrate, SDS and urea. Yue et al. [12] suggested formation of mesopores in presence of CTAB, with citrate or tartrate acting as bridging component between aluminium precursor and surfactant.

In this study, aluminium oxide was prepared from aluminium nitrate by precipitation process in presence of cationic (cetyl trimethyl ammonium bromide) and anionic (sodium dodecyl sulfate) surfactants. Effects of surfactant type and concentration, pH, aging temperature and aging time on development of porous structure of aluminium oxide were investigated and compared. To the best of our knowledge this kind of detailed comparative parametric study of porous structure of aluminium oxide in presence of different surfactants is not reported yet. The aluminium oxide samples were characterized by surface area and pore size analysis, Thermogravimetric Analysis (TGA), X-Ray Diffraction (XRD), Fourier Transform Infrared Spectroscopy (FTIR) and Field Emission Scanning Electron Microscopy (FESEM). Based on experimental observations the nature of interaction of aluminium hydroxide with SDS and CTAB has been proposed.

2. Experimental

2.1. Preparation procedure

Aluminium nitrate ($\text{Al}(\text{NO}_3)_3 \cdot 9 \text{H}_2\text{O}$, Merck) was used as precursor for aluminium oxide. Sodium dodecyl sulphate (SDS, $\text{CH}_3(\text{CH}_2)_{11}\text{OSO}_3\text{Na}$, Merck) and cetyl trimethyl ammonium bromide (CTAB, $\text{CH}_3(\text{CH}_2)_{15}\text{N}(\text{CH}_3)_3\text{Br}$, Loba chemie) were used for aluminium oxide preparation. Ammonium hydroxide (NH_4OH , Merck) was used for precipitation and nitric acid (HNO_3 , Merck) was used for controlling pH. All the chemicals were used as received.

Preparation scheme of aluminium oxide in presence of anionic and cationic surfactants by ammonical hydrolysis method is shown in Fig. 1. Precursor solution, 0.1 M and surfactant solution (0.025 to 0.2 M) were prepared by dissolving required amount of aluminium nitrate and surfactant (CTAB or SDS) respectively in distilled deionized water. The surfactant solution (200 ml) was added to the precursor solution (200 ml) and mixture solution was stirred for 15 min. Precipitation was carried out by gradual addition of 25% ammonia solution. Pale–yellow precipitate was obtained with SDS and white precipitate with CTAB. The mixture was stirred for 60 min in a round bottom flask at room temperature. Then the solution was aged under reflux at specified temperature for specified time.

The synthesis parameters that were studied include surfactant concentration, pH, aging temperature and aging time in presence

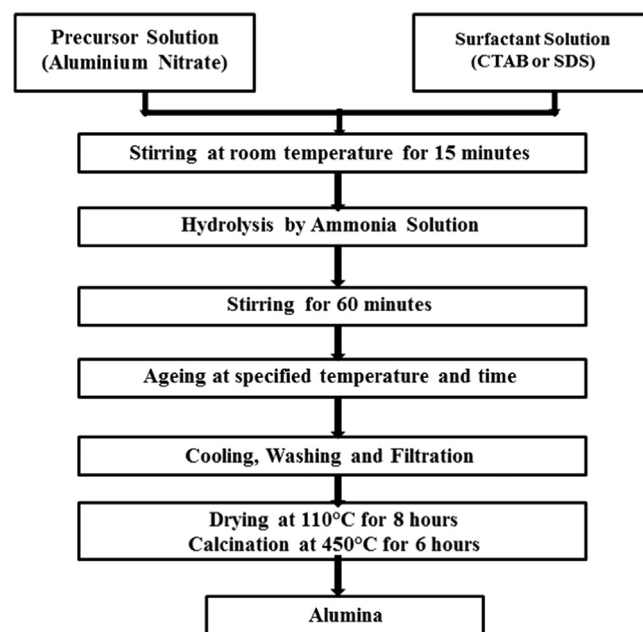


Fig. 1. Preparation scheme for aluminium oxides in presence of different surfactants.

of both SDS and CTAB surfactants. The molar ratio of aluminium precursor to surfactant was varied as 1:2, 1:1, 2:1 and 4:1. Aging time was varied from 2 to 24 h and aging temperature was varied in the range of 25 to 75 °C. The pH of the solution mixture was varied from 5 to 10.5 using of 25% ammonia and 70% nitric acid solutions. After aging, the mixture was cooled to room temperature. The resulting precipitate was filtered and washed repeatedly with water and acetone to remove the excess surfactant. The precipitate was dried at 110 °C for 8 h and calcined at 450 °C for 6 h in air. For reference aluminium oxide was also prepared without using any surfactant. Representation of the samples in text is as follows: Al–W represented aluminium oxide prepared without using surfactant; Al–CTAB and Al–SDS referred to aluminium oxides prepared in presence of CTAB and SDS respectively. The ‘P: S’ used in text is the molar ratio of precursor to surfactant.

2.2. Characterization

The surface area, pore volume and pore size distributions by Branauer Emmett Teller (BET) method were determined by N_2 physisorption at -196 °C using a Beckman Coulter SA 3100 surface area analyzer. TGA of aluminium hydroxides was performed using TGA/SDTA 851e/LF/1100 (Mettler Toledo). The samples were heated at rate of 10 °C/min from 25–800 °C under N_2 flow (30 ml/min). X–Ray diffraction pattern was measured using Bruker D8 model. Scanning was done in the range of 10–70° at speed of 0.005°/sec in continuous mode. XRD patterns were obtained using $\text{Cu K}\alpha$ radiation ($\lambda = 0.154$ nm). The X–ray source was operated at 40 KV and 25 mA. FESEM images were recorded by ZEISS (model 1430VP) instrument. Infrared spectra were recorded using IRAffinity–1 Fourier transform infrared spectrophotometer in the range of 400–4000 cm^{-1} with a resolution of 4 cm^{-1} .

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