SYNTHESIS AND PROPERTIES OF ALUMINA AEROGELS (II)

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Abstract

Synthesis of alumina aerogels was carried out by hydrolysis of aluminium isopropanolate and butanolate in benzene, methanol and isopropanol solutions as components of support precursors followed by gelation, drying under supercritical conditions and calcination. The influence of the type of precursor and solvent, synthesis temperature as well as drying and calcination temperature on bulk density, specific surface area and total pore volume was investigated.

Keywords: Alumina aerogel, synthesis, gelation, calcination

INTRODUCTION

Due to their very strongly developed porous structure, large pore volume (even larger than 5 cm³/g) and high specific surface area [1-7], aerogel materials are very interesting materials and can find many different applications [4]. They are mainly obtained by hydrolysis of organic metal compounds, gelation of sol and drying under supercritical conditions the solvent used. Both the hydrolysis and gelation reactions proceed under strictly controlled conditions, which allow to obtain gels of the claimed properties and do not allow aluminium hydroxide to precipitate. Drying of alumina gels under supercritical conditions ensures the porous aerogel structure to be maintained. It was stated in our first paper that the main parameters influencing the pore structure of alumina aerogels are the type of solvent and concentration of aerogel precursor [7]. However, the pore structure of alumina aerogels

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depends also on synthesis conditions, and drying and calcination conditions. In this paper we present data on the influence of synthesis parameters (temperature, type of precursor and solvent), drying and calcination conditions on the textural properties of alumina aerogels that may be applied as catalyst supports.

EXPERIMENTAL

Preparation of alumina aerogels

Alumina isopropanolate and butanolate (purity > 98 wt.%) were applied as aerogels support precursors, while benzene (critical temperature 289°C, critical pressure 4.8 MPa), anhydrous alcohols: methyl alcohol (critical temperature 240°C, critical pressure 8.0 MPa), isopropyl alcohol (critical temperature 236°C, critical pressure 4.7 MPa), and 2-butyl alcohol (critical temperature 261°C, critical pressure 4.14 MPa) as precursors solvents of p.a. quality. De-mineralized water was applied for hydrolysis alumina precursor.

The aerogel synthesis is a four-step process: (i) hydrolysis of precursor, (ii) gelation and ageing, (iii) drying, and (iv) calcination. The hydrolysis of precursor (16 or 30 wt. % in alcohol) and ageing of gels was carried out in a constant temperature chamber (20 or 60°C) in flat-bottomed 50 cm³ glass test tubes with different water quantities (3 or 9 mol of demineralized water per 1 mol precursor). Because of the readiness of alumina precursor hydrolysis at pH ca. 7 no catalyst was applied. A content of glass test tubes was continuously mixed in the course of the hydrolysis step (1 h) using a magnetic stirrer.

Gelation was done without catalyst, under the same parameters as hydrolysis. Total gelation and ageing time attained 18 h. The gels were dried in a typical stainless reactor (100 cm³) under supercritical conditions for methanol, isopropanol and sec-butanol (280°C and 14 MPa) and for benzene (310°C, 14 MPa). In order to prevent the gels from collapse at the heating-up stages, the reactor was filled with nitrogen to a pressure of ca. 7 MPa (close to the critical parameters of the alcohols and benzene). The excess pressure, a result of solvent vaporization, was carefully reduced during the heating stage. The rate of heating was ca. 50°C/h. The aerogel samples were kept in the reactor for one hour after reaching the predetermined conditions. Then the pressure was reduced and the reactor brought to room temperature. The aerogels were calcined at 500°C for 5 h in air atmosphere. All measurements of physicochemical properties were done for calcined samples. The characterization of the samples included measurements of bulk density, specific surface area by thermal desorption of nitrogen method and total pore volume by