THE SUMMARY OF Ph.D. DISSERTATION

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Title

Preparation, Characterization and Application of Porous Titania by Sol-Gel Method

Abstract

Recent advances in the surface chemistry of oxide materials are remarkable. Functional oxides with high porosity are attractive due to their interesting properties and many applications, e.g. TiO_2 gels as photocatlyst, electrode of solar cell, gas sensors, etc. Many important and interesting phenomena take place on the surfaces of the materials. An understanding of the surface controlling of oxides is a primary focus for environmental catalysis. The surface properties of the oxides can be improved by controlling their microstructure via surface modification. However, surface controlling of the functional oxides is not well known. The properties of meso- and nano-porous titania have generated increasing interest in its use as a photocatalyst or a catalyst-support. The purpose of this study is to produce porous titania with high surface area and controlled porosity using various organic materials as "templates" by the Sol-Gel method. The efforts to find new routes for control of the pore shape are emphasized.

Chapter 1 and Chapter 2 summarizes the backgoroud and motivation of this study and experimental procedures. Chapter 3 describes the preparation of porous titania films using surfactants as templates, and their application to photocatalyst. The porosity, surface morphology and the catalytic activity were examined. The porous films of anatase with rectangular and columnar structure were obtained by drying and annealing after immersion of the wet gel films in BTAC- and CTAC- solutions, respectively. The micelles or surfactant molecules are introduced into the the wet gels and adsorbed on the surface of the gels. The spacing between the columns or rectangular grains and the microstructure of the sol-gel derived films can be controlled by changing the type of surfactant species and the concentration of surfactant solutions. Photocatalytic activity of the surfactant-modified films was higher than that of the gel films without surfactant modification. Chapter 4 represents the preparation and characterization of mesoporous titania gels by the immersion method using surfactants and hydrophilic polymers as templates.

The wet gels were dried and annealed after immersion in surfactant (CTAC or BTAC) or polymer (PEG or Pluronic) solutions under an atmospheric pressure. The pore volume and specific surface area of the gels can be increased by the surfactant- and polymer-modification. The average pore radius was also increased with surfactant-immersion. The pore size and shape obviously depended on the size and shape of the surfactant micelles. Chap. 5 describes the preparation and characterization of mesoporous titania gels by the mixing method using surfactants and hydrophilic polymers as templates. The gels were prepared with addition of the templating agents in the alcoholic solution of titanium alkoxide. In the precursor solution of Ti-alkoxide with surfactants, the micelles are incorporated in the gels of titania during gelation. On the other hand, in the precursor solution of Ti-alkoxide with high concentration of polymers, the polymer molecules form entangled aggregates. During gelation, these polymer-aggregates are incorporated into the gels. Thus, the incorporated micelles of surfactants or polymer aggregates prevent the shrinkage of the gels during drying, and the pore size of the gels increases. High concentration of surfactants and polymers is favourable for the formation of micelles or polymer-aggregates, respectively. Thus the specific surface area and pore volume of the gels can be increased with addition of hydrophilic polymer and surfactant. Surfactants are effective to control the pore size and to increase the pore volume and surface area. The pore size and the pore volume of the gels prepared with addition of CTAC+PEG were also larger than that of the xerogels without additives. Micelles and polymer-aggregates may play an important role to increase the porosity of the gels. Chapter 6 represents the preparation and characterization of mesoporous titania gels using hydrophobic polymers as templates. Mesoporous titania with large surface area was obtained by hydrophobic polystyrene templating. The pore size of the gels can be increased by polystyre templating, and the pore size and pore shape of the gels depends on the concentration of polymer.

Mesoporous titania can be obtained by both of the methods, the immersion- and the mixing method. The microstructure and pore size of the gels can be controlled by the templating with surfactants, and hydrophilic- and hydrophobic polymers through an atmospheric pressure route. The newly developed methods can be widely applied to functional porous oxides.