CHARACTERIZATION OF ZrO₂ - Al₂O₃ MIXED OXIDES SUPPORT PREPARED BY UREA HYDROLYSIS

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Received September 24, 2003
In revised form May 29, 2003
Accepted June 11, 2003

Abstract

The characteristics of Al₂O₃, ZrO₂ and three binary mixtures of ZrO₂-Al₂O₃ were studied by determining their BET surface areas, micropore surface area, total pore volume, adsorption-desorption isotherms, the X-ray diffractogram, surface acidity and catalytic functionality for cumene cracking. The XRD results show that the incorporation of alumina into the zirconia from 50% and beyond renders it amorphous. Furthermore, the mixed oxide containing 50% alumina and 50% zirconia had the highest BET surface area of 199.9 m²/g whilst pure zirconia had the lowest BET surface area of 37.19 m²/g. The pores for all the mixed oxides were found to be monomodal and zirconia pores were more open. The results of the acidity measurements and cumene cracking functionality indicates that whilst pure zirconia has low total acidity, the incorporation of alumina increases its acidity through a synergistic effect.

Keywords: ZrO₂ - Al₂O₃, support

INTRODUCTION

Petroleum products hydrotreatment is undertaken for the removal of sulfur compounds that are sources of environmental polluting emissions and poisons of precious metal catalysts used in reforming processes. The reactions have been widely undertaken on supported molybdenum and tungsten catalysts.

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Although alumina is the most widely used support material [1-3], the use of other materials such as TiO$_2$ [4], ZrO$_2$ [5-6], MgO [7-10] for the production of catalysts of improved activity and selectivity have been proposed. In recent times promising results have been obtained with catalysts supported on mixed oxides [11-12]. One mixed oxide that has recently attracted attention is zirconia based mixed oxide. This is because it gives rise to a substantially different interaction between the active phase and the support, altering the activity and selectivity of the system [13-14].

The use of zirconia as a support has been limited in the past because it can exist in three crystallographic forms, namely, monoclinic, tetragonal and cubic, depending on the temperature. Its tetragonal structure has however been reported to produce a catalyst of higher hydrodesulfurization activity than alumina supported catalyst [13]. It has also been demonstrated that this tetragonal structure can be stabilized by constraining its particles into an alumina matrix which has a higher elastic modulus than zirconia [15]. In this regard it should be possible to take advantage of tuning the mixed ratio of zirconia to alumina to produce a support of higher activity and selectivity than either pure zirconia or pure alumina.

There are a number of methods for the production of mixed oxides, however, the urea hydrolysis method of precipitation has been reported to be cheap and permits the formation of a precipitate at a nearly ideal rate sought which are rarely attained by conventional methods [16].

In this communication we present the results of an investigation of the characterization of zirconia-alumina mixed oxides prepared by urea hydrolysis method. The mixed oxides were characterized by BET surface area, micropore surface area, pore size distribution, adsorption-desorption isotherm, X-ray diffraction, surface acidity and catalytic functionality for cumene cracking.

**EXPERIMENTAL**

**Materials**

Zirconia oxychloride (ZrOCl$_2$.8H$_2$O) of Analar grade, a product of Loba Chem., India was used as a precursor for zirconia. Extra pure aluminium nitrate (Al(NO$_3$)$_3$.9H$_2$O) a product of S.D. Chemicals Ltd. India., was used as a precursor of alumina. Extra pure urea ((NH$_2$)$_2$CO) a product of E. Merck (India) Ltd., was used for *in-situ* ammonia production.