

Synthesis of Camphene by α -Pinene Isomerization Using W_2O_3 – Al_2O_3 Catalysts

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Abstract W_2O_3 – Al_2O_3 catalysts prepared by “sol–gel” and impregnation methods were characterised by XRD, FTIR and nitrogen adsorption. Both catalysts were tested in the isomerization of α -pinene in a batch reactor at atmospheric pressure and 150 °C. Conversion of α -pinene and selectivity to camphene was superior for the “sol–gel” catalyst than that observed for the impregnated catalyst.

Keywords α -Pinene · Isomerization · Solid-acid catalyst · Camphene · Tungsten alumina catalysts

1 Introduction

α -Pinene is one of the most important monoterpenes, which are extendedly used as solvent and precursor for the production of camphene, which is an important component in the fragrance industries. Although some solid-acid catalysts as zeolites [1–5], modified montmorillonite [6], clays [7–9], functionalized MCM-41 [10] and sulfated zirconia [11] has been tested for the isomerization of α -pinene, the development of new solid-acid catalysts for this reaction is still a subject of continuous research in many laboratories.

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Therefore, the aim of this work is to synthesize W_2O_3 – Al_2O_3 catalysts by impregnation and “sol–gel” methods and test them for the isomerization of α -pinene.

2 Experimental

W_2O_3/Al_2O_3 –I catalyst was prepared by impregnation of the alumina support previously synthesized by “sol–gel” with a solution containing ammonium tungstate, in order to obtain a final W_2O_3 content of 20 wt%. W_2O_3 – Al_2O_3 –SG catalyst was prepared by co-gelling the aluminum alkoxide with the required amount of ammonium tungstate solution to obtain a 20 wt% of W_2O_3 in the final solid. Both catalysts were dried at 100 °C for 12 h and then calcined at 500 °C for 4 h prior to the catalytic test. The samples were labelled as “I” and “SG” for impregnation and “sol–gel” preparation, respectively. Alumina “sol–gel” partner was prepared in the same way described for the W_2O_3 – Al_2O_3 –SG catalyst but without adding ammonium tungstate.

XRD patterns of the samples were obtained by using a Siemens D-500 equipment and a graphite crystal as monochromator to select Cu K α radiation (1.5406 Å) with a step of 0.02° s^{–1}. The BET specific surface area and BJH pore size distribution were calculated by the nitrogen adsorption–desorption isotherms determined in an ASAP-2000 Micromeritics apparatus. The total acidity and the Lewis/Brønsted sites ratio were determined by FTIR–pyridine adsorption using a Nicolet 170 SX spectrometer.

α -Pinene isomerisation was carried out using the following procedure: 0.5 g of catalyst and 50 mL of α -pinene were loaded into a batch multiple reactor system (Parr Model 500). The experiments were performed at atmospheric pressure. The reaction products were analyzed by