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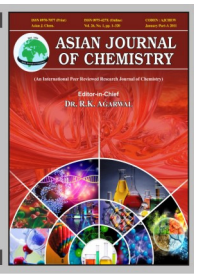

Synthesis of 1-Bromooctane Catalyzed by Solid Super Acid SO_4^{2-}/ZrO_2-ZnO

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The solid super acid catalyst SO_4^{2-}/ZrO_2-ZnO was prepared by impregnation and characterized by infrared spectroscopy, X-ray diffraction, different scanning calorimetric and thermogravimetric analysis. Their performances were evaluated by the synthesis of 1-bromooctane. Under the condition of *n*-octanol/hydrobromic acid molar ratio of 1:3, a catalyst amount of 8 wt. %, reaction time of 7 h, temperature of 130 °C, produce 1-bromooctane (yield 70.22 %).

Keywords: Solid super acid, Impregnation, 1-Bromooctane.

INTRODUCTION

1-Bromooctane is an important intermediate in organic synthesis. It is soluble in ethanol and ether, slightly toxic and narcotic[1]. The atom of bromine can be substituted by hydroxy, amino, alkoxy to generate corresponding alcohols, amines and ether. It is widely used for synthesis of pesticides, organic dyes, surfactants, ultraviolet absorbers, *etc.*[2-4]. Due to its broad spectrum of activity, the compound has received a great deal of attention in connection with their synthesis. Several methodologies of preparation of 1-bromooctane have been reported in the literatures[5-7].

The bromine-phosphorus method causes environmental pollution because of the volatility and toxicity of bromine and the corrosivity and flammability of phosphorus. Some by-products are produced by sodium bromide-sulfuric acid method because of the dehydration and oxidation of concentrated sulfuric acid. Another hydrobromic aqueous solution method has long reaction time, too much raw material consumption and low yield.

Recently considerable attention has been devoted to the super solid acids. Generally, these catalysts offer several advantages, such as strong acid sites, nontoxicity, noncorrosive-ness, easily handling, mild reaction conditions, high selectivity, low cost and easy to recover and recycling[8]. In this work, a kind of solid super catalyst sulfated zirconia and zinc oxide was prepared and its performance applying in synthesis of 1-bromooctane was investigated. The solid base catalyst showed a good performance during the synthesis process, with a high purity and yield of 1-bromooctane product in a relatively short time.

EXPERIMENTAL

Sulfuric acid (98 %), aqueous ammonia (25-28 %), hydrobromic acid (40%), sodium bicarbonate (A.R) were purchased from Kermel, Tianjin Chemical Reagent Co., Ltd. Zirconium(VI) oxychloride octahydrate ($ZrOCl_2 \cdot 8H_2O$), N-octyl alcohol (99 %) and Silver nitrate (A.R) were purchased from Sinopharm Chemical Reagent Co., Ltd., Sanmenxia AOKE Chemical Co., Ltd. and Beijing chemical plant respectively.

Preparation of catalyst: 2:1 of the molar ratio of $ZrOCl_2 \cdot 8H_2O$ and $Zn(NO_3)_2$ was dissolved in distilled water. To this solution, aqueous concentrated ammonia (25-28 %) was added drop-wise from a burette with vigorous stirring until the pH of the solution reached 10 and then left for 24 h. The obtained precipitate was washed several times with deionized water and filtrated by pump until a neutral filtrate and the absence of chlorine ion, detected by $AgNO_3$ (0.1 mol/L). The cake after filtration was dried at 110 °C for 16 h. The sample thus obtained was ground into powder under 120 mesh. To prepare the targeted catalyst, the powdered complex oxides were impregnating with sulfuric acid of 0.5 M for 6 h followed by filtration. The sample was oven dried at 110 °C for 12 h then calcined at 650 °C for 4 h to obtain SO_4^{2-}/ZrO_2-ZnO[9,10].

Characterization of catalyst: Infrared spectroscopy spectrogram of the sample showed the support of H_2SO_4 on ZrO_2-ZnO. X-ray diffraction (XRD) analysis revealed the presence of zirconia crystal phases and zincoxide crystal phases. DSC-TG measurements indicated temperature from the tetragonal phase to the monoclinic phase of zirconia rises.