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Materials and Product Engineering

A simple strategy to synthesize and characterization of zirconium modified PCs/γ-Al_2O_3

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Abstract Crystalline phase is the key factor for catalyst activity. The zirconium modified PCs/γ-Al_2O_3 samples were prepared through a simple step incipient-wetness impregnation method. The raw materials and samples were characterized by thermogravimetric-differential analysis (TG-DTA), X-ray diffraction (XRD), Fourier transform infrared spectroscopy (FT-IR), Temperature-programmed desorption of ammonia and carbon dioxide (NH_3- and CO_2- TPD). The effects of calcination temperature and zirconium content on structure, chemical transformation, and acidity-basicity were investigated. Calcination temperature exhibited the major effect on the crystalline phase of samples. The new phase of $Al0_{.1}Zr_{0.9}O_{1.95}$ was exhibited which was above 650°C. In addition, zirconium content was influenced by the acidic and basic properties of the surface. The acidity and basicity of the ZrPCs/γ-Al_2O_3 sample increased with the increasing of zirconium content.

Keywords: TG-DTA, XRD, FTIR, Acid-base properties, Zirconia, Aluminium-Zirconium oxide

1. Introduction

PCs/γ-Al_2O_3 is very popular catalyst applied in many reaction such as aldol condensation [1,2], and transesterification [3]. Due to aluminium oxide has gained significant importance in catalysis owing to their surface area which usually uses as a commercial supporter. The addition of alkali and alkaline earth such Li, Na, K, Cs were obtained basicity property [4]. Phosphorus was investigated as a resource for increasing acidic property and reducing the amount of coking of catalyst in many reactions [5,6]. Particularly, the combination of these components is well known as a bifunctional catalyst. Furthermore, zirconium represented as active material after added during synthesis can lead to the formation of Lewis and Brönsted acid sites along with basic sites [7]. Other than acidic and basic properties, its crystalline phase and physical properties is key factor for catalytic properties, synthesis controlling is very importance.

So far, many techniques including co-precipitation [6,7], sol-gel [8], hydrothermal [9], and impregnation [3] have been developed to synthesize catalysts. Previously, considerable attention was focused on the preparation method, through co-precipitation and sol-gel method. Meanwhile, there was no one study on the impregnation method. Impregnation has several advantages which including its relative simplicity, rapidity, and capability for depositing the precursor at high metal loadings. In addition, it's easier way and suitable for large scale preparation. Many attempts to prepare catalysts have been reported, while no study on experiment condition to obtain this catalyst was found. Impregnation method consist of three main steps, impregnating step, drying step, and calcination step. The calcination step is the important step. The major factor in this step is calcination temperature which effected to chemical transformation, crystalline structure and catalyst properties [10,11].

The aim of this work was a simple synthesis of zirconium modified PCs/γ-Al_2O_3 with fixed P and Cs content in various calcination temperature and Zr content through the step incipient-wetness impregnation method. TG-DTA, XRD, FT-IR, and NH_3- and CO_2- TPD were used for the thermal behavior study, phase identification of a crystalline material, functional group, and acidity-basicity on the sample surface, respectively.

2. Experimental

2.1 Materials

The γ-Al_2O_3 spheres were obtained from a commercial company with average particle diameter of 1 mm. Cesium carbonate [Cs_2CO_3; 99.0%], diammonium hydrogen phosphate [$(NH_4)_2HPO_4$, 99.0%], and zirconium nitrate pentahydrate [$Zr(NO_3)_4$•$5H_2O$, 99%] utilized in this work which were A.R. grade.

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