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Catalytic performance of a Ti added Pd/SiO_2 catalyst for acetylene hydrogenation

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ABSTRACT

A series of Pd/SiO_2 and Pd–Ti/SiO_2 catalysts were prepared by the incipient wetness impregnation method. The catalytic performance for selective hydrogenation of acetylene to ethylene was measured under "high concentration acetylene", "high space velocity" and "no dilution gas" conditions. The crystal structure and particle size of the catalysts were characterized by the X-ray diffraction (XRD), energy dispersive X-ray spectroscopy (EDS), nitrogen physisorption using the BET method and transmission electron microscope (TEM). The results showed that the titanium oxide in Pd–Ti/SiO_2 catalyst was amorphous and the addition of Ti reduced the particle size of Pd significantly. Comparing to the Pd/SiO_2 catalyst, the ethylene yield increased from 64.1% to 88.3% under Pd–Ti/SiO_2 catalytic system.

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1. Introduction

Ethylene is an important material and chemical production in modern industries and mainly obtained by the naphtha cracking method. But petroleum is a shortage and non-renewable strategic resource, seeking a new source and technology to prepare ethylene could ease the dependence on the petroleum industry.

Selective hydrogenation reactions have been investigated for several decades, it was concentrated on an ethylene stream from a naphtha cracker unit typically contains about 0.1–1% of acetylene as an impurity. This must be removed to a level of less than 5 ppm because it poisons the catalyst which was used in subsequent ethylene polymerization processes and eventually degrades the quality of the produced polyethylene [1–8]. The high concentration of acetylene as raw material produce ethylene is rarely reported. Supported Pd catalysts are most widely used for Selective hydrogenation of acetylene [6,8] and remain under intensive study with the aim of further improving catalyst stability and selectivity. Catalytic activity, selectivity and stability strongly depend on the nature of the support and the size of the Pd nanoparticles, because the hydrogenation of acetylene is a structure-sensitive reaction [9–12]. Therefore, we wish to provide a method for producing ethylene via acetylene by a new catalyst of Pd–Ti/SiO_2. We prepared a series of Pd/SiO_2 and Pd–Ti/SiO_2 catalysts by the incipient wetness impregnation method. For the consideration of industrialization, pure acetylene and hydrogen gas were chosen as raw materials under "high concentration" "space velocity" conditions without adding any dilution gas and the total space velocity is up to 15,000 h^{-1}. The catalytic performance for selective hydrogenation of acetylene to ethylene was measured. The catalysts were characterized by X-ray diffraction (XRD), energy dispersive X-ray spectroscopy (EDS), nitrogen physisorption using the BET method and transmission electron microscope (TEM).

2. Experimental details

2.1. Preparation of the catalyst

Pd/SiO_2 catalyst was prepared by the incipient wetness impregnation method using Pd $(OAc)_2$ as Pd precursor. Firstly, $Pd(OAc)_2$ was dissolved in MeCN, SiO_2 was used as a catalyst support and obtained from Jilinji Qing Technology Co., Ltd. (particle size 20–40 mesh, surface area = 280 m^2/g). The silica was impregnated with Pd, dried at 120 °C for 12 h and calcined at 400 °C for 2 h, a second impregnation was performed using $Ti(OC_4H_9)_4$ (aq) as the source of TiO_2. The impregnated catalyst was then dried at 120 °C for 12 h and calcined at 400 °C for 2 h. The Catalysts was reduced at 400 °C under H_2 atmosphere for 2 h and marked as Ti/SiO_2-T, Pd/SiO_2-T, and Pd–Ti/SiO_2-T respectively.

2.2. Catalyst characterization

The crystalline structure of the catalysts were determined the powder X-ray diffraction (XRD) of Germany Brooke D8 ADVANCE X-ray diffractometer with a Cu Kα radiation source and a Ni filter (λ = 1.54056 Å). The 2θ Bragg angles were scanned over a range of 30–90° at a rate of 5/min with a 0.02 angular resolution. The tube current was 40 mA and the tube voltage was 40 kV.

The size and morphology of palladium nanoparticles on the silicon dioxide support were analyzed by transmission electron

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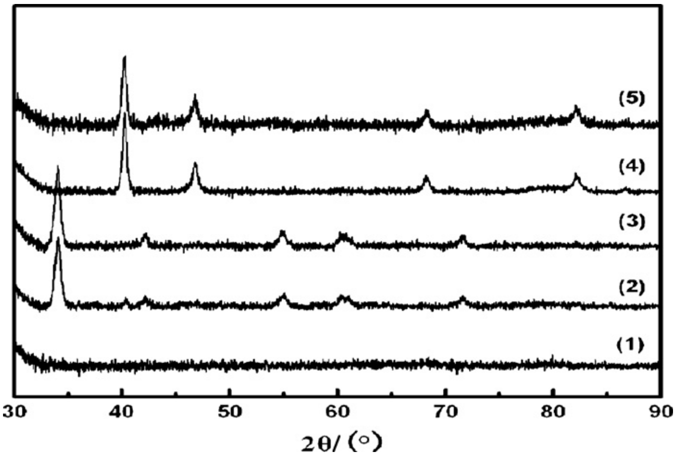

Fig. 1. XRD patterns of Ti/SiO_2-T (1), Pd–Ti/SiO_2 (2), Pd/SiO_2 (3), Pd–Ti/SiO_2-T (4) and Pd/SiO_2-T (5).

microscope (TEM). A Hitachi H600 transmission electron microscope was operated at 100 kV, magnified 100 nm and selected the 70 particles Photoshop software to calculate the average diameter and the particle size distribution.

Energy dispersive X-ray spectroscopy (EDS) of Oxford-7420 was employed to study the elemental composition of the catalyst. It was operated at 8000 V.

The specific surface areas of the catalysts were determined by nitrogen physic-sorption using the BET method in ASAP 2020C instruments (Micromeritics Instruments, USA)

2.3. Catalytic test

Acetylene hydrogenation was carried out in QGF2-type small-scale fixed-bed catalytic reactor (Tianjin University, the Process of Industrial Technology and Equipment Research). The products were analyzed by GC7890 II FID thermal conductivity gas chromatography detector, porapak N (2 m × 3 mm) packed column (Shanghai Tian Mei Scientific Instrument Co., Ltd.). The catalyst was pre-treated under H_2 atmosphere at 400 °C for 2 h with a flow rate of 100 mL/min. The feed gas was C_2H_2/H_2

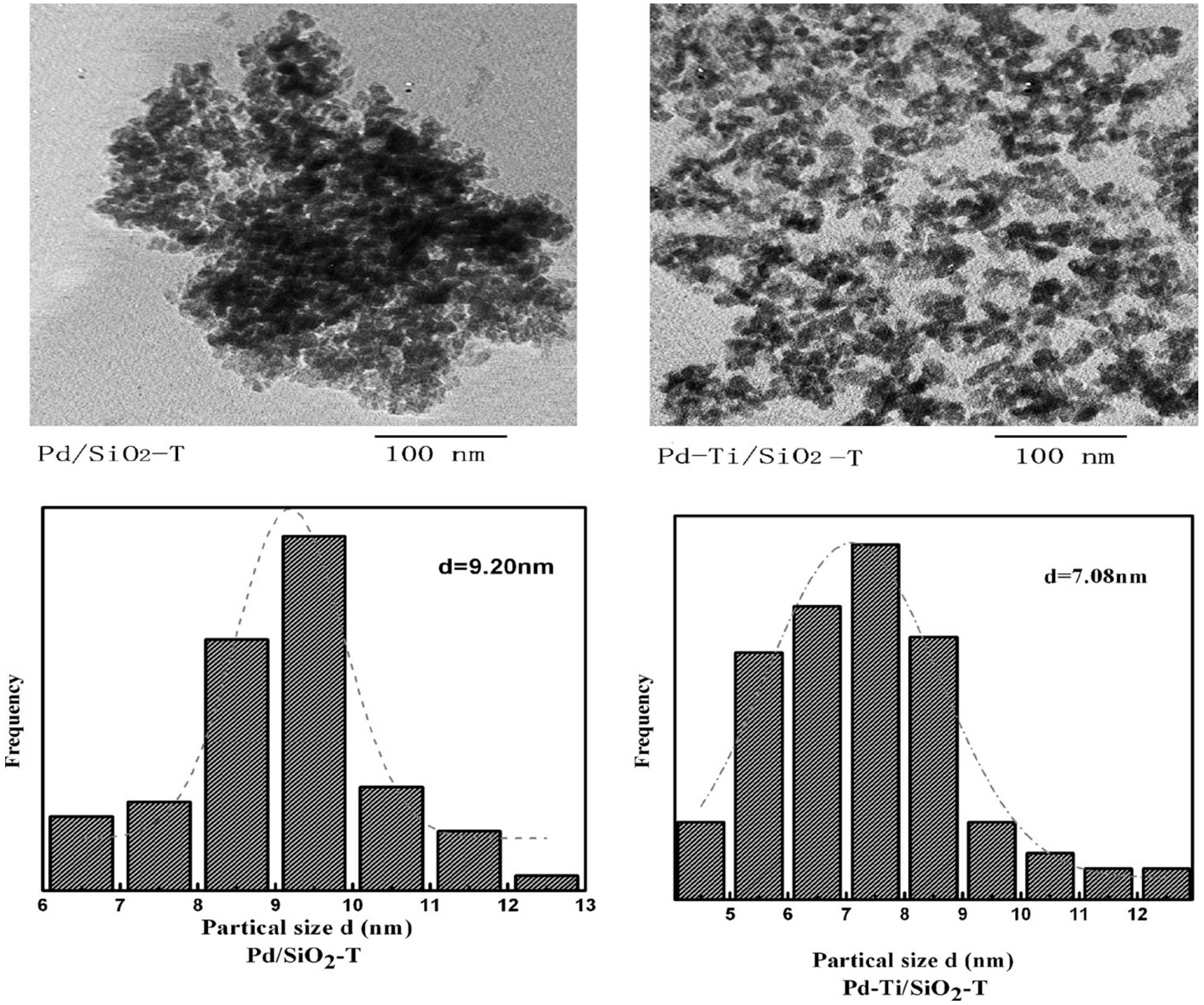

Fig. 2. TEM images and particle distribution of Pd/SiO_2-T (1) and Pd–Ti/SiO_2-T (n_{Ti}:n_{Pd} = 1:1) (2).

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