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Article

Carbonylative Suzuki coupling reactions of aryl iodides with arylboronic acids over Pd/SiC

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ABSTRACT

High surface area SiC has been used to prepare a Pd/SiC catalyst using the liquid reduction method, and the resulting catalyst was used for the carbonylative Suzuki coupling reaction of aryl iodides with arylboronic acids. The catalyst was also characterized by X-ray diffraction, inductively coupled plasma-mass spectroscopy and high-resolution transmission electron microscopy. The results of these analyses showed that homogeneous Pd nanoparticles with a mean diameter of 2.8 nm were uniformly dispersed on the SiC surface. Optimization of the reaction conditions for the carbonylative Suzuki coupling reaction, including the solvent, base, pressure, temperature and reaction time, revealed that the model reaction of iodobenzene (1.0 mmol) with phenylboronic acid (1.5 mmol) could reach 90% conversion with a selectivity of 99% towards the diphenyl ketone using 3 wt% Pd/SiC under 1.0 MPa of CO pressure at 100 °C for 8 h with K₂CO₃ (3.0 mmol) as the base and anisole as the solvent. The Pd/SiC catalyst exhibited broad substrate scope towards the carbonylative Suzuki coupling reaction of aryl iodides with arylboronic acids bearing a variety of different substituents. Furthermore, the Pd/SiC catalyst exhibited good recyclability properties and could be recovered and reused up to five times with the conversion of iodobenzene decreasing only slightly from 90% to 76%. The decrease in the catalytic activity after five rounds was attributed to the loss of active Pd during the organic reaction.

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1. Introduction

Aromatic ketones are important building blocks in organic chemistry, and compounds belonging to this structural class can be found in a wide variety of biologically active natural products and pharmaceutical molecules [1,2]. In light of their importance and general utility, various methods have been reported for the preparation of aromatic ketones. Traditional synthetic routes for the construction of aromatic ketones include the Friedel-Crafts acylation of aromatic compounds with

acyl halides [3] and the transition-metal-catalyzed cross-coupling reactions of aryl metal reagents such as C₆H₅MgBr [4] and C₆H₅SiF_nMe_{3-n} [5] with carbon monoxide and a suitable electrophile [6]. However, the former of these two reactions requires the addition of a large excess of Lewis acid and is incompatible with many functional groups [3,7,8], whereas the latter usually results in the formation of biphenyl byproducts.

In 1993, Suzuki's group [9] reported a direct method (i.e., the carbonylative Suzuki coupling reaction) for the synthesis of aromatic ketones by the reaction of CO with an aryl halide and

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arylboronic acid in the presence of PdCl₂ and Pd(dba)₂ (bis(dibenzylideneacetone)palladium). This reaction provides a versatile platform for the direct synthesis of aromatic ketones using boronic acids, which are generally non-toxic and stable to air and moisture. Ishiyama *et al.* [10] used a variety of different catalysts to affect this carbonylative Suzuki reaction, including PdCl₂(PPh₃)₂ and PdCl₂(dppf)[[1,1'-bis(diphenylphosphino)ferrocene]dichloropalladium(II)]. However, the conversion and selectivity of these reactions are low and require the presence of a ligand to proceed effectively. Khedkar *et al.* [11] reported that immobilized palladium ion-containing ionic liquids exhibited excellent catalytic activity towards the carbonylative Suzuki coupling reaction of aryl halides with arylboronic acids, with the coupled products being isolated in high yields. However, the overall utility of this process is limited by its requirement for the use of toxic organic compounds, which are harmful to the environment. Niu *et al.* [12] reported the stabilization of Pd on hollow magnetic mesoporous spheres and the use of this material as a heterogeneous catalysis for the carbonylative Suzuki coupling reaction. However, the preparation of this catalyst is complex. Therefore, despite good progress during the last 20 years towards the development of robust methods for the preparation of aromatic ketones, the high costs associated with the preparation of complex catalysts and ligands, and the difficulties involved in the separation of catalysts from the reaction mixture have greatly limited the application and development of the carbonylative Suzuki coupling reaction. Based on these limitations, there is still considerable scope for exploring the development of new stable and efficient heterogeneous catalysts for the carbonylative Suzuki coupling reaction.

SiC has excellent chemical stability and thermal conductivity properties, and has been successfully used as catalyst support in various catalytic reactions, including the oxidation of CO [13], reforming of CH₄ and CO₂ [14,15], photocatalytic water splitting [16], methanation [17] and the photocatalytic reduction of CO₂ [18]. We recently reported that SiC-supported Pd nanoparticles significantly enhanced the hydrogenation of furan derivatives under low temperature (25 °C) and low pressure (1.0 MPa of H₂) conditions by visible light irradiation [19]. Notably, the yield of tetrahydrofuran from the hydrogenation of furan under these conditions was 99% with a turnover frequency of 70 h⁻¹. In this study, a Pd/SiC catalyst has been prepared by a liquid reduction method using high surface area SiC as a support, and investigated its catalytic performance towards the carbonylative Suzuki coupling reaction under heterogeneous conditions.

2. Experimental

2.1. Preparation and characterization of Pd/SiC

The Pd/SiC catalyst (3 wt%) was prepared using a liquid phase reduction method. Palladium nitrate (132.6 mg, 0.564 mmol) and SiC powder (1.94 g, S_{BET} = 50 m²/g) were dispersed in absolute ethanol (50 mL) under sonication conditions, and the resulting suspension was magnetically stirred to get a homogeneous mixture of palladium nitrate and SiC. The mixture was dispersed in 80 mL of diethylene glycol (DEG), and the

resulting suspension was heated to 140 °C for 30 min. Water (2.5 mL) was then added to the reaction, and the resulting mixture was reduced for 2 h at 180 °C to give the 3 wt% Pd/SiC catalyst.

The Pd content of the catalyst was determined by inductively coupled plasma (ICP) atomic emission spectrometry using an Atomscan 16 system (Thermo Fisher Scientific, Shanghai, China). The phase characterization of the catalyst was determined using a Rigaku D-Max/RB X-ray diffraction (XRD) system (Tokyo, Japan) using Cu-K_α radiation. The structure and morphology of the catalyst were characterized by transmission electron microscopy (TEM) using a JEM-2100F system (JEOL, Tokyo, Japan). X-ray photoelectron spectroscopy (XPS) measurements were conducted on an ESCALAB 3 MKII de VG spectrometer (VG Scientific, East Sussex, United Kingdom) using Mg K_α (15 kV, 20 mA) as an X-ray source.

2.2. Carbonylative Suzuki coupling reactions

All of the carbonylative Suzuki coupling reactions were conducted in a sealed stainless steel reaction kettle. Arylboronic acid (1.5 mmol), aryl iodide (1.0 mmol), base (3.0 mmol), 3 wt% Pd/SiC and anisole (10 mL) were added to the reaction kettle, and the resulting mixture was placed under a certain pressure of CO. All of the reactions were conducted over several hours at certain temperatures. The products of the reactions were analyzed by GC-MS on a Bruker SCION SQ 456 GC-MS system (Karlsruhe, Germany).

3. Results and discussion

3.1. Morphology and structure of the Pd/SiC catalyst

The Pd loading of the Pd/SiC catalyst was determined to be 2.91 wt% by ICP-MS analysis, which is similar to the calculated value. BET analysis showed that the surface area of the Pd/SiC catalyst was 56 m²/g. The TEM images of the Pd/SiC catalyst (Fig. 1) showed that homogeneous metallic Pd nanoparticles were uniformly dispersed on the SiC surface with a narrow size distribution of 1–5 nm and a mean diameter of 2.8 nm. The lattice fringe of the nanoparticles had an interplanar spacing of 0.19 nm, which was in agreement with the (200) plane of Pd. The XRD pattern of the Pd/SiC catalyst (Fig. 2) showed that all of the strong diffraction peaks could be indexed to β-SiC. The

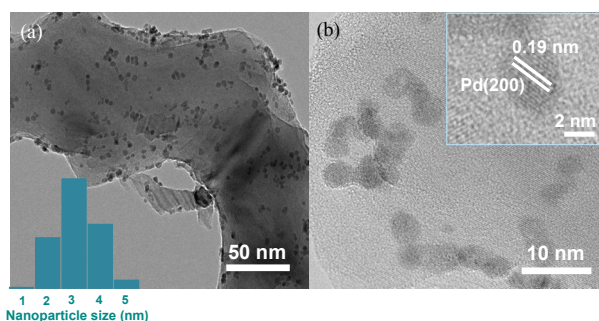


Fig. 1. TEM and HRTEM images (inset of (b)) of Pd/SiC catalyst, and the size distribution of Pd nanoparticles (inset of (a)).

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