



A 1D palladium coordination polymer and its catalytic activity in microwave-assisted Sonogashira reactions

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ABSTRACT

A palladium coordination polymer, $[\text{Pd}(\text{dppp})(\text{L})](\text{OTf})_2 \cdot (\text{H}_2\text{O})$ (**1**) (dppp = 1,3-bis(diphenylphosphino)propane), was prepared from a bipyridine-type ligand ($\text{L} = (4\text{-py})\text{-CH} = \text{N-C}_{10}\text{H}_6\text{-N} = \text{CH-(4-py)}$) and $[\text{Pd}(\text{dppp})](\text{OTf})_2$. An X-ray diffraction study showed that it has a one-dimensional zigzag chain structure and the triflate ions act as counterions. Compound **1** exhibited Sonogashira coupling reactivity in air under microwave conditions.

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1. Introduction

Coordination polymers (CPs) and metal-organic frameworks (MOFs) of transition metals have received continuous attention due to their desirable properties and potential application in catalysis, gas storage and photoluminescence [1–10]. According to IUPAC's definitions in 2013, CPs are coordination compounds with repeating coordination entities extending in 1, 2 or 3 dimensions, and MOFs are coordination networks with potential voids [11]. However, the crystal structures of such species with heavy metals, except cadmium, are relatively rare.

Palladium-catalyzed Sonogashira coupling reactions are of considerable importance in organic chemistry for the preparation of pharmaceuticals, natural products, fine chemicals and polymers. Recently, microwave irradiation has been utilized in organic synthesis, including the Sonogashira coupling reaction, because it is an efficient heating method [12–15]. A wide range of molecular palladium compounds and palladium nanoparticles have been extensively employed in catalytic carbon-carbon coupling reactions, with industrial and academic interest. On the other hand, studies of heterogeneous catalysis using the palladium CPs and MOFs, especially for carbon-carbon coupling, are rather limited [16–26]. The limited use of palladium CPs and MOFs is related to the small number of crystal structures [16–18]. In this paper, we report the preparation and structure of a one-dimensional palladium coordination polymer, as well as its catalytic activity under microwave irradiation in Sonogashira coupling reactions. We also

attempted to investigate the catalytic activity in the Suzuki coupling reaction, but our palladium polymer did not exhibit any activity at all.

2. Experimental

2.1. Materials and measurements

All solid chemicals were purified by recrystallization and solvents were vacuum-distilled and stored over 4Å molecular sieves under argon. Triethylamine was distilled over calcium hydride and stored under argon. Activation of molecular sieves was performed under vacuum by heating and then purging with nitrogen gas [27]. The ligand (**L**) and $[\text{Pd}(\text{dppp})(\text{OTf})_2]$ (dppp = 1,3-bis(diphenylphosphino)propane, $\text{OTf} = \text{CF}_3\text{SO}_3^-$) were prepared by the literature methods [28,29]. The microwave heating experiments were conducted with a Monowave 300 (Anton Paar, 300 W). IR spectra were obtained in the range 400–4000 cm^{-1} on a Nicolet 320 FT-IR spectrophotometer. ^1H and ^{13}C NMR spectra in the supporting information were obtained with a Bruker 500 MHz spectrometer. Melting points were measured with a 1201D MEL-TEMP apparatus. Elemental analyses were carried out by the Cooperative Center for Research Facilities (CCRF) in Sungkyunkwan University.

2.2. Preparation of the Pd coordination polymer (**1**)

A mixture of $[\text{Pd}(\text{dppp})(\text{OTf})_2]$ (0.0082 g, 0.010 mmol), the ligand (0.0034 g, 0.010 mmol) and dichloromethane (20 mL) was stirred at room temperature for 6 h. The resulting solution was fil-

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