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High Catalytic Activity of C₆₀Pd_n Encapsulated in Metal-Organic Framework, UiO-67 for Tandem Hydrogenation Reaction

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Abstract: Metal nanoparticles (NPs) stabilized by MOFs are very promising for catalysis, while introduction of C_{60} into MOFs has been very rarely used and there was no report for their cooperative catalysis on organic syntheses. In this work, we synthesized C_{60} @UiO-67 by one pot method so that C_{60} is uniformly distributed on UiO-67 in molecular form. Pd NPs coordinating with C_{60} have been successfully embedded into the framework. The obtained multifunctional $C_{60}Pd_n$ @UiO-67 catalyst exhibits remarkable synergistic catalytic activity in cascade reactions under mild conditions, where UiO-67 affords Lewis acidity and $C_{60}Pd_n$ offers higher hydrogenation activity relative to sole Pd NPs.

Fullerene (C₆₀) and its derivatives have been widely used in many fields owing to their unique physical and chemical properties.^[1] Furthermore, it is one of elemental forms of carbon which has great potential for being high-capacity hydrogen-storage material and possible catalyst for hydrogenation reactions with special catalytic activity due to its unique molecular structure.^[2,3,4] In addition, C₆₀ molecules can act as a support for metal nanoparticles (NPs) through stronger metal- π interactions and collaborate with NPs to synergistically improve catalytical activity.^[5-11] Zheng Xu et al. demonstrated that C₆₀ can activate molecular hydrogen and is a novel nonmetal hydrogenation catalyst.^[8] The hydrogenation of aromatic nitro compounds to amino aromatics was achieved on this catalyst with high yield and selectivity under 1 atmospheric pressure of H₂ and light irradiation at room temperature. Metal complexes C₆₀Ptn^[9] and C₆₀Pdn^[10,11] have been reported to present good catalytical activity in the reaction of hydrogenation of acetylene derivatives such as diphenylacetylene and nitro compounds in mild reaction conditions. However, these catalysts are difficult to work on multistep catalytic reactions for which multiple catalytic functional sites are needed. It is still a challenge to develop new type of composites behaving high synergistic catalytical activity and working on a complex reaction.

Metal organic frameworks (MOFs) are a new class of porous materials with multifunctional sites assembled by metal/metal cluster and organic ligand. Due to their high surface area, tunable pore size and well-ordered crystalline structure, MOFs could be promising materials for applications in heterogeneous catalysis. ^[12,13] The presence of defects in the MOF can have significant

come simply from an imperfect crystallization of the MOF, or be created in a controlled manner by the introduction of auxiliary ligands with missing functional group(s).[14,15] Furthermore, the cages or channels with tunable sizes in MOFs allow them to host or support metal nanoparticles (NPs) for catalysis over a broader scope.^[16,17] Recently, Hai-Long Jiang et al. have reported several composites of NPs@MOF which exhibit superior catalytic activity in selective hydrogenation reaction.^[18] Yu-Zhen Chen et al. have reported Pd-Ag alloy NPs encapsulated in MIL-101 as catalyst for the selective synthesis of secondary arylamine.^[19] Bifunctional catalysts Pd/MIL-101 and Pt/MIL-101 have been used for the synthesis of quinolines, pyrroles and 3-arylpyrrolidines via tandem hydrogenation-reductive amination reactions.^[20] However, these catalytical reactions still need high hydrogen pressure, long reaction time and/or high temperature. Therefore, it is necessary to develop new catalysts for hydrogenation reactions, especially tandem ones in milder conditions.

effects on the catalytic activity of the material. These defects can

In this work, we wish to design a catalyst with multi-functional catalytical sites to complete a tandem hydrogenation reaction in one-step under mild conditions. In other words, we would make use of the advantages of three components, MOFs, NPs, and C₆₀ to attain this goal through integrating them into one composite. UiO-67, composed of 4,4'-biphenyl dicarboxylic acid (BPDC) and Zr₆O₄(OH)₄ clusters, one of the most popular MOFs in recent reports on composite catalysts was chosen to load C₆₀Pd_n NPs onto the surface because of its large pore sizes (ca. 12 to 23 Å), surface area (BET surface area ca. 1575 m² g⁻¹), as well as excellent stability.^[21] For the zirconium dicarboxylate MOFs, modulators like acetic acid were introduced by Schaatte et al. in the synthesis of UiO-66 and UiO-67.[22] Thermal activation of the material leads not only to dehydroxylation of the hexanuclear Zr cluster but also to post-synthetic removal of the modulator groups such as trifluoroacetate and benzoic acid resulting in a more open framework with a large number of active sites.^[14,23] We have succeeded in synthesizing novel composite materials C60@UiO-67 and C60Pdn@UiO-67 by one-pot and soaking method sequentially (Scheme 1). C60Pdn@UiO-67 has shown high catalytic efficiency for the tandem nitrobenzene hydrogenation reaction because of the presence of multiple functional sites in the composite material.

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Scheme 1. Schematic illustration for the sequential synthesis of C_{60} @UiO-67 and C_{60} Pd_n@UiO-67.

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High resolution transmission electron microscope (HRTEM) images of C₆₀@UiO-67 composite show that there is no lattice dispersion of C₆₀ in UiO-67 (Figure 1a-b). Comparing the Raman spectrum of the C₆₀@UiO-67 composite and UiO-67 (Figure S1), the increased number of peaks of composite are attributed to pentagonal pinch mode $A_g(2)$ (1447 cm⁻¹) and degenerate mode $H_g(8)$ (1564 cm⁻¹) for C₆₀.^[24] C₆₀ encapsulated in UiO-67 is also testified by UV-vis absorption spectra (Figure S2). Moreover, C₆₀@UiO-67 presents better thermal stability than the pristine framework based on X-ray thermodiffractometry and TGA analysis (as shown in Figure S3). C₆₀@UiO-67 can be stable up to 550 °C which is much higher than that of UiO-67, 450 °C, revealing that the guest C₆₀ molecules may support the MOF framework.^[25,26]



Figure 1. HRTEM images of C_{60} @UiO-67 NPs (a, b) and C_{60} Pd_n@UiO-67 NPs (c, d) and corresponding EDX elemental (C, O, Pd and Zr) mapping of C_{60} Pd_n@UiO-67 (e).

According to the HRTEM of $C_{60}Pd_n@UiO-67$ (Figure 1c, d), the $C_{60}Pd_n$ NPs are highly dispersed with mean diameters of 5 ± 2 nm (Figure S4). To obtain more direct evidence, energy-dispersive X-ray (EDX) was used to map the distribution of different elements (C, O, Pd and Zr, as shown in Figure 1e) for $C_{60}Pd_n@UiO-67$. EDX elemental mapping indicates that the carbon content far exceeds that of other elements, and meanwhile, Pd is well-distributed throughout the whole composite, suggesting the uniform distribution of $C_{60}Pd_n$ throughout the particles of $C_{60}Pd_n@UiO-67$. The above results are well matched with our hypothesis.

Powder X-ray diffraction (PXRD) will give the crystal lattice information and phase purity of crystalline materials. PXRD patterns of UiO-67, C_{60} @UiO-67 and $C_{60}Pd_n$ @UiO-67 are shown in Figure 2a. The diffraction peaks of one-pot synthesized C_{60} @UiO-67 are in good agreement with reported ones of UiO-67.^[27] Moreover, no diffraction peaks of C_{60} are appeared which represents that C_{60} @UiO-67, the diffraction pattern is also in accordance with C_{60} @UiO-67, the diffraction pattern is also in accordance with C_{60} @UiO-67, and no peak related to Pd is detected implying that Pd is coordinated with C_{60} to form nanoparticle instead of formation over the support UiO-67 due to direct soaking and synthetic approaches during the preparation of this composite. The metal- π interactions between Pd and C_{60} allow Pd to form a shell on the surface of C_{60} , other than crystalline phase that can be detect by PXRD.

Porosity of the materials can be determined by N_2 adsorptiondesorption isotherms. Figure 2b shows the N_2 isotherms of all prepared samples, and porous properties are presented in Table S1. N₂ adsorption-desorption isotherms of all samples are completely merged without any hysteresis loop. It indicates that most of the adsorbed N₂ has been desorbed, and high amount of N₂ was adsorbed below the partial pressure of 0.1. The isotherm patterns for all samples come to category of type-I by IUPAC classification which indicates that these materials are micropores. Specific surface area and total pore volume were 1739 m²·g⁻¹, 0.99 cm³·g⁻¹ for UiO-67, 1488 m²·g⁻¹, 0.58 cm³·g⁻¹ for C₆₀@UiO-67 and 506 m²·g⁻¹, 0.35 cm³·g⁻¹ for C₆₀Pd_n@UiO-67 respectively. Successive decrease in surface area and pore volume indicated that the cavities of UiO-67 were occupied by C₆₀ and C₆₀Pd_n NPs respectively.



Figure 2. PXRD patterns of UiO-67, C_{60} @UiO-67, C_{60} Pd_n@UiO-67 (a). N₂ sorption isotherms at 77 K of as-synthesized UiO-67, C_{60} @UiO-67 and C_{60} Pd_n@UiO-67 catalysts before reaction (b).

The X-ray photoelectron spectroscopy (XPS) results of the C_{60} @UiO-67 show that the peak at 284.8 eV is ascribed to C 1s spectrum for the fullerene carbon atoms;^[28] the peak at 284.6 eV is assigned to sp²-hybridized carbon atoms in ligands (Figure 3a). For C_{60} Pd_n NPs, Pd@UiO-67 and C_{60} Pd_n@UiO-67, all of them present two peaks assigned to Pd 3d_{5/2} and Pd 3d_{3/2} (Figure 3b), however, Pd@UiO-67 exhibits lower binding energies with shifting ~0.6 and ~0.8 eV as compared to C_{60} Pd_n@UiO-67 and C_{60} Pd_n@UiO-67 and C_{60} Pd_n respectively

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(Table S2), indicating that the state of Pd in $C_{60}Pd_n@UiO-67$ is similar to it in $C_{60}Pd_n$. Furthermore, the data also implies that palladium atoms coordinate with C_{60} resulting in a small shift of binding energies toward higher values relative to $Pd(0)^{[29,30]}$, which may be related to the partial charge transfer between palladium and fullerene.^[31]



Figure 3. XPS spectra of C 1s for C_{60} @UiO-67 composite (a) and Pd 3d for Pd@UiO-67, C_{60} Pd_n and C_{60} Pd_n@UiO-67 (b).

The mass ratio of C₆₀ and UiO-67 was determined by measuring the carbon content of UiO-67 and C₆₀@UiO-67 using elemental analysis as shown in Table S1. The molar amount of C₆₀ was calculated to be *ca*. 0.027 mmol for 100 mg UiO-67 host matrix.

The coexistence of Lewis acid sites in UiO-67 and $C_{60}Pd_n$ species may render $C_{60}Pd_n$ @UiO-67 a multifunctional catalyst that is suitable for multistep cascade reactions. In order to prove that $C_{60}Pd_n$ @UiO-67 has good tandem hydrogenation catalysis activity, a reaction route involving acid catalysis and catalytic hydrogenation steps was chosen for the overall three-step process including nitroarene hydrogenation, reductive amination of aldehydes and selective hydrogenation to secondary arylamine.

For comparison, the reaction of synthesis of benzyl aniline (product **A**) by benzaldehyde and nitrobenzene was performed using different catalysts including the target composite $C_{60}Pd_n@UiO-67$ and Pd@UiO-67, $C_{60}Pd_n$, $C_{60}@UiO-67$ as well as

commercial Pd/C (as shown in Table 1). The results indicated that C₆₀Pd_n@UiO-67 gave the best catalytic activity with 100% conversion rate and highest selectivity for A (76%). Among three UiO-67 supported catalysts, Pd@UiO-67 presented lower conversion (80%) and selectivity (60% for A) than the target one, whereas C₆₀@UiO-67 without Pd showed very weak activity with less than 10% conversion rate at the similar conditions (the reaction time extended to 6 h). This comparison implies that Pd NPs have intrinsic hydrogenation activity [32,33], and when combined with C₆₀, the catalytic activity (including conversion and selectivity) will be significantly enhanced. In addition, the other two catalysts without being supported with MOF but with Pd particles also gave poorer performance wherein C₆₀Pd_n showed 90% conversion rate and 5% selectivity for A during an extended reaction time, and commercial Pd/C just presented 50% conversion rate and 38% selectivity for A under same conditions. The result of the former should be ascribed to its poor dispersion and lack of sufficient Lewis acidity, whereas the result of the latter could reflect the critical role of acidity in the reaction between nitrobenzene and benzaldehyde. In addition, the lower catalytic activity (40%) and selectivity (3%) over a physical mixture of C₆₀Pd_n and UiO-67 should be ascribed to the absence of synergistic effect.





	Catalyst ^[a]	Time (h)	Conversion (%)				
				Α	В	С	
	C60Pdn@UiO-67	3	100	76	0	24	
	Pd@UiO-67	3	80	60	15	25	
	Pd/C	3	50	38	0	62	
ν.	$C_{60}Pd_n$	6	90	5	0	95	
	C60@UiO-67	6	< 10	-	-	-	
	$C_{60}Pd_n + UiO-67^{[b]}$	6	40	3	24	73	
	UiO-67						
	no						

[a] Reaction conditions: substrates benzaldehyde (107 mg, 1.0 mmol) and nitrobenzene (124 mg, 1.0 mmol), 20 mg catalyst in 3 mL of ethanol at room temperature, 1 atmospheric pressure of H₂. [b] The catalytic result was based on a physical mixture of C₆₀Pd_n and UiO-67, in which the content of Pd is the same as those in C₆₀Pd_n@UiO-67.

Furthermore, the reaction was also conducted with UiO-67 and without catalyst, but no products were detected. If taking metal (Pd) to benzaldehyde molar ratio used in the reactions into account, the superior hydrogenation ability of $C_{60}Pd_n@UiO-67$ compared to Pd@UiO-67 could also be verified by the increasing turnover frequency (TOF) values (to 180 h⁻¹ from 142 h⁻¹). These results strongly prove that the presence of Lewis acid active sites in UiO-67 and $C_{60}Pd_n$ NPs cooperate with each other, greatly improving the catalytic activity of the reaction.

The recyclability of C₆₀Pd_n@UiO-67 was also studied as well. After five recycles, the conversion of secondary amine was 96% which was maintained throughout each cycle almost without any

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loss of activity (Figure S5a). The structural characteristics of recycled catalysts were studied and presented in Figure S6. No significant loss of crystallinity and no identifiable peaks for $C_{60}Pd_n$ NPs in the PXRD patterns was observed (Figure S6b). The results revealed retained integrity of the UiO-67 framework and the nonexistence of $C_{60}Pd_n$ NP agglomeration, which is further evident in HRTEM images and N₂ adsorption-desorption isotherms (Figure S5b), showing that $C_{60}Pd_n$ NPs have well retained sizes after five cycles and demonstrating good recyclability and durability of the catalyst (Figure S6a).

Among other reports on this tandem reaction with MOFsupported Pd catalysts, there are two works that can be compared with the present work, as shown in Table S3. For PdAg@MIL-101, the introduction of Ag raised the selectivity (90%) but suffurred from extended time (28 h) and higher H₂ pressure (*ca.* 2 atmospheric pressure).^[19] For MIL-101-SI-Pd, the reaction could be completed in 6 h with higher selectivity (90%) but with higher temperature and H₂ pressure (110 °C and *ca.* 5 atmospheric pressure).^[20] From both synthetic and industrial points of view, the synthesis of our target composite catalyst is more convenient, practical and energy-consuming (one-pot synthesis of C₆₀@UiO-67 and simple soaking method to obtain C₆₀Pd_n@UiO-67, see Figure S7).

In conclusion, a facile and efficient one-pot method has been developed for the encapsulation of C₆₀ molecule in UiO-67, and subsequent direct soaking and stirring synthetic approach were used to rationally introduce Pd NPs over C₆₀@UiO-67. In comparison to monometallic Pd NPs, this new composite C60Pdn NPs confined in UiO-67 have greatly improved hydrogenation activity due to the synergistic effect of Pd coordinated with C₆₀ and Lewis acid sites afforded by UiO-67. More importantly, C60Pdn@UiO-67 requires shorter time and milder reaction conditions to achieve the same conversion compared with literature works. The facile and rational preparation approach for the synthesis of C₆₀Pd_n@UiO-67 opens a new route for the design of MOF based multifunctional material with high catalytic activity. The present study may bring light to the construction of functional materials combining MOFs and fullerene materials, which has great potential for broad applications in optical, electrical, magnetic, catalysis and other fields in the future.

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Conflicts of interest

The authors declare no competing financial interest.

Keywords: metal-organic frameworks • fullerene $(C_{60}) \cdot C_{60}Pd_n$ NPs • concerted catalysis • tandem hydrogenation reaction

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A new composite consisting of Pd, C_{60} and MOF was designed and synthesized showing high catalytical activity in a tandem hydrogenation reaction.



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