一种高效可循环的有机介孔树脂负载的 *N*-杂卡宾络合钯催化剂催化的 Sonogashira 反应

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摘要: 新型有机介孔材料 FDU-15 负载卡宾配体络合醋酸钯催化剂 FDU-NHC/Pd(II) 的制备方法简单, 通过三步反应即可制得, 采用 X 射线衍射、N₂ 吸附-脱附和透射电镜等手段对催化剂进行了表征, 并考察了它在 Sonogashira 偶联反应中的催化性能. 结果表明, 功能化不影响 FDU-15 的有序介孔结构, 仅其孔径、孔体积和 BET 比表面积有所减小. 另外, 该催化剂在 Sonogashira 偶联反应中表现出较高的催化活性, 底物普适性好, 无论是带有吸电子取代基还是供电子取代基的碘苯都能够得到较高的分离产率, 且该催化剂分离简单, 重复使用 10 次以上时, 其催化性能基本保持不变.

关键词: 介孔聚合物; *N*-卡宾; Sonogashira 反应; 钯催化剂; 固载; 多相催化剂

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An Efficient and Recyclable Mesostructured Polymer-Supported *N*-Heterocyclic Carbene-Palladium Catalyst for Sonogashira Reactions

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Abstract: Palladium catalyst systems are the most widely used for the formation of C–C and C–N bonds. Environmentally friendly heterogeneous palladium catalysts have strongly stimulated growth in this research area. Herein, a FDU (Fudan University)-15 mesopolymer with *p6mm* mesostructures that combine the advantages of organic polymers and mesoporous materials was investigated as a new solid support for Pd complexes. The FDU-15 mesopolymer-supported *N*-heterocyclic carbene (NHC) precursor was developed by a simple chemical functionalization approach. It has been used as a phosphine-free ligand to prepare the novel heterogeneous palladium catalyst FDU-NHC/Pd(II), which efficiently catalyzes Sonogashira reactions between aryl halides and terminal alkynes to afford the corresponding products in good yields. Various techniques such as X-ray diffraction, N₂ adsorption-desorption isotherms, high resolution transmission electron microscopy, and X-ray photoelectron spectroscopy have been used to characterize the catalyst. The results indicate that the mesoporous structure of FDU-15 was not affected by immobilization, however, the pore diameter, pore volume, and BET surface area decrease. The stability of the heterogeneous catalyst was also studied. The FDU-NHC/Pd(II) complex catalyst was easily separated from the reaction system by a simple filtration of the reaction mixture and it has been shown to be robust without a significant loss of catalytic activity after recycling and reuse in 10 times.

Key words: mesostructured polymer; *N*-heterocyclic carbene; Sonogashira reaction; palladium catalyst; immobilization; heterogeneous catalyst

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Palladium-catalyzed coupling reactions between terminal alkynes and aryl or alkenyl halides, known as the Sonogashira reaction, are recognized as one of the most powerful and versatile methods to construct C–C bonds in organic synthesis. These reactions have been widely used in the synthesis of fine chemicals, agrochemicals, pharmaceutical intermediates, and precursors of natural products [1–3]. The reactions are mostly carried out using a homogeneous Pd complex catalyst in conjunction with a copper salt CuX (X = Cl, Br, I) as a co-catalyst. Although homogeneous palladium catalysts often offer higher activities for these transformations, they suffer from drawbacks such as difficulties in handling sensitive ligands, product separation and catalyst recovery, reuse of expensive ligands and Pd complexes, and the contamination of the desirable product by heavy metals. An alternative method to overcome these problems is to develop environmentally friendly heterogeneous catalyst systems.

Heterogeneous catalysts possess significant advantages from a practical point of view and in terms of sustainable green chemistry [4–6]. The catalysts can be easily recovered from the reaction mixture by simple filtration and they can then be reused. Therefore, an increasing number of heterogeneously catalyzed Sonogashira reactions have been developed. Pd catalysts have been immobilized on various support materials such as silica [7–16], polymers [17–27], chitosan [28,29], metal oxide nanoparticles [30,31], and ionic liquids [32] etc. However, alternative immobilization strategies are still needed. It has been demonstrated that the supporting matrix crucially influences the performance of the Pd catalysts [33]. Thus, research into the development of more efficient catalyst systems with high activity and high reusability is extremely important.

Mesoporous polymers that comprise pure organic frameworks or pore walls possess the characteristics of both mesoporous materials (high surface area, large pore volume, and tunable mesostructure) and organic polymers (high hydrophobicity and availability for multi-functionalization). Hence, they are considered to be potential supports for the preparation of efficient Pd-based catalysts for Sonogashira

reactions. Among them, FDU-type mesoporous phenolic resins appear to be amenable to either acidic or basic reaction conditions and they also exhibit a unique hydrophobic property toward organic reactants or solvents, which is quite different to silica-based inorganic analogues [34,35]. The abundant aromatic rings in their polymeric networks provide FDU-mesopolymers with abilities useful for functionalization. Continuous research efforts in our laboratory have been devoted to the modification of these materials. A series of FDU-type mesoporous phenolic resin-based catalysts have been developed by the chemical modification of organic framework walls, i.e. sulfonic acid groups, amino groups or ligand functionalized mesopolymers [36–40]. The resultant materials exhibit good catalytic activity and good reuse properties. On the other hand, *N*-heterocyclic carbene groups are highly effective ligands for transition metals and have attracted great attention in homogeneous catalysis. These ligands provide much stronger binding to transition metals and are chemically and thermally more inert than their corresponding phosphines [41,42].

In this work, we used the FDU-15 mesoporous phenolic resin with *p6mm* symmetry as a new solid support for the Pd complexes. Based on previous studies, we introduced commercially available and cheap *N*-methylimidazole as a *N*-heterocyclic carbene (NHC) precursor into the mesopolymer and then developed a new recyclable supported heterogeneous Pd catalyst (Scheme 1). This was carried out by the chloromethylation of the mesopolymer, the quaternary and then the immobilization of the NHC precursor and finally simple ligand exchange with Pd(OAc)₂ to obtain the NHC-Pd complexes. Herein, we report the preparation of this kind of phosphine-free, stable and active catalyst, and its catalytic activity as well as recyclability in the Sonogashira reaction.

1 Experimental

1.1 Preparation of the FDU-NHC/Pd(II) catalyst

All reagents were commercially available (Aldrich, Alfa

Scheme 1. Preparation of the FDU-NHC/Pd(II) catalyst.

Aesar) and directly used without further purification. The FDU-15 mesopolymer was prepared according to previously reported procedures [35]. FDU-15 was chloromethylated with chloromethyl methyl ether to obtain FDU-CH₂Cl [40]. The FDU-CH₂Cl was further functionalized with *N*-methylimidazole under mild conditions. The reaction was carried out by stirring 1 g of FDU-CH₂Cl with 1 g of *N*-methylimidazole (3.3 mmol) in 5 ml anhydrous toluene at 353 K for 24 h. The obtained material was washed with acetone to remove physisorbed *N*-methylimidazole and finally dried under vacuum at 323 K for 8 h. The *N*-heterocyclic carbene ligand precursor functionalized mesopolymer was thus obtained, which is denoted FDU-NHC. FDU-NHC (1 g) was subsequently mixed with 0.044 g (0.2 mmol) Pd(OAc)₂ in 5 ml dimethyl sulfoxide (DMSO). The mixture was stirred at 323 K for 6 h and then at 373 K for 3 h to bring about the deprotonation of imidazolium groups to form the Pd-NHC complexes [19]. The resulting suspension was filtered. The residue was washed with acetone and finally dried under vacuum to give FDU-NHC/Pd(II).

1.2 Characterization methods

X-ray diffraction (XRD) patterns were collected on a Bruker D8 ADVANCE instrument using Cu *K*_α radiation (λ = 0.15418 nm) at 40 kV and 40 mA. Using CDCl₃ as solvent and TMS as the internal standard, ¹H and ¹³C NMR spectra were acquired on a Bruker DRX500 spectrometer at 500 MHz and 125 MHz, respectively. Flash column chromatography was performed using silica gel 60 (230–400 mesh). Nitrogen adsorption-desorption isotherms were recorded on a Quantachrome Autosorb-3B instrument after the samples were evacuated at 573 K for 5 h. The specific surface area was evaluated using the Brunauer-Emmett-Teller (BET) method. The high resolution transmission electron microscopy (HRTEM) images were recorded using a JEOL JEM-2100 microscope. Cl elemental analyses were performed by the Volhard method. The nitrogen content was quantified on an Elementar VarioEL III CHN elemental analyzer. The bulk loading amount of Pd was determined by inductively coupled plasma (ICP, Thermo Electron Corporation IRIS Intrepid II XSP). X-ray photoelectron spectroscopy (XPS) was performed on a Kratos Axis Ultra DLD spectrometer with a single anode (Al) apparatus using Al *K*_α radiation.

1.3 General procedures for the Sonogashira reaction

The reactions were carried out in an Argonaut advantage seriesTM 2410 personal screening synthesizer. In a typical experiment aryl halide (0.5 mmol), substituted

phenylacetylene (0.75 mmol), K₂CO₃ (1.0 mmol), direct memory access (4.0 ml), and FDU-NHC/Pd(II) (1 mol %) were mixed in a reaction tube, which was heated to 373 K. The reaction progress was monitored by thin-layer chromatography (TLC). After reaction completion the catalyst was separated by simple filtration and washed with acetone for further reaction. The filtrate was diluted with water and the product was extracted with ethyl acetate (3×10 ml). The combined extracts were dried over anhydrous Na₂SO₄ and concentrated under vacuum. The residue was purified by column chromatography on silica gel (petroleum ether) affording the product. The structures of the known products were characterized by comparison with ¹H NMR and ¹³C NMR data from the literature [43].

2 Results and discussion

2.1 Catalyst characterization

The Pd catalyst was synthesized according to the procedures described in Scheme 1. The chloromethylation of the benzene rings in FDU-15 was realized effectively by catalysis with anhydrous AlCl₃, which resulted in the –CH₂Cl group-grafted mesopolymer, FDU-CH₂Cl. Elemental analysis by the Volhard method indicated that FDU-CH₂Cl contained 1.2 mmol/g of –CH₂Cl groups. FDU-CH₂Cl was reacted with excess *N*-methylimidazole to afford FDU-NHC. FDU-NHC/Pd(II) was obtained by the chemical coordination of the *N*-heterocyclic carbene ligand with Pd(OAc)₂. The loading of the NHC ligand was determined from the nitrogen content by elemental analysis. A nitrogen content of 0.84 mmol/g implied the presence of 0.42 mmol/g of NHC ligand in FDU-NHC/Pd(II). In addition, the amount of Pd was 0.18 mmol/g as determined by ICP. To coordinate one Pd ion requires a pair of carbene ligands that are in its neighborhood and as close as possible. Some NHC ligands may be present on the mesopolymer walls in a random or an isolated manner. Thus, it is reasonable that the maximum Pd loading is less than that determined by the NHC ligand method.

XPS was applied to trace the above-mentioned functionalization procedures qualitatively. Chloromethylation was responsible for the Cl 2*p* peaks centered at 200.3 eV (Fig. 1(a) curves (1) and (2)). The quaternary with *N*-methylimidazole caused the Cl 2*p* peaks to shift to the lower binding energy region (Fig. 1(a) curve (3)), during which chlorine became negatively charged from a covalent state. This eases electron constraints in the outer orbitals, which lowers the binding energy. The N atoms in FDU-NHC became positively charged ions and donated electrons to Pd(II) in FDU-NHC/Pd(II). As a result, the N 1s peaks in the catalyst are present at 401.8 and 401.6 eV,

Fig. 1. XPS spectra of Cl 2p (a), N 1s (b), and Pd 3d (c) for FDU-15 (1), FDU-CH₂Cl (2), FDU-NHC (3), and FDU-NHC/Pd(II) (4).

respectively (Fig. 1(b) curves (3) and (4)). The carbene ligands captured the Pd species successfully. The XPS spectrum shows peaks at 337.5 and 342.8 eV (Fig. 1(c) curve (4)), which are assigned to Pd 3d_{5/2} and Pd 3d_{3/2}, respectively. By comparison with the Pd 3d_{5/2} peak at 335 eV for Pd⁰, the higher binding energy value found for FDU-NHC/Pd(II) indicated that the incorporated Pd species is in the +2 oxidation state.

Figure 2 shows the powder XRD patterns of the FDU-15 mesopolymers before and after functionalization. FDU-15 was a typical mesostructured material that possessed a two-dimensional (2D) hexagonal structure with a *p6mm* space group as the XRD pattern revealed three diffraction peaks indexed as the (100), (110), and (200) planes (Fig. 2(1)). Although the intensity of the diffraction peaks decreased after multi-step modification, well-resolved XRD patterns confirmed that the *P6mm* symmetry of the mesostructure was reserved even after chemical coordination with Pd(II) ions (Fig. 2(2) and (3)).

HRTEM indicated that the array of mesopores in FDU-NHC/Pd(II) was typical of a 2D hexagonal

mesostructure even after multi-functionalization (Fig. 3). Thus, in agreement with above XRD patterns the HRTEM images also verified that the well-ordered mesostructure remained intact after chemical modification.

Fig. 3. HRTEM images of FDU-NHC/Pd(II) as viewed in the [11] (a) and [10] (b) directions.

Fig. 2. Powder XRD patterns of FDU-15 (1), FDU-NHC (2), and FDU-NHC/Pd(II) (3).

Figure 4 shows the nitrogen adsorption-desorption isotherms of FDU-NHC/Pd(II) and its precursors. These samples exhibited a typical type IV isotherm, which is characteristic of mesoporous materials. Furthermore, as summarized in Table 1, the surface area of the parent FDU-15 was 489 m²/g while FDU-NHC/Pd(II) had a lower specific surface area of 271 m²/g, and the pore diameter decreased from 3.5 to 3.0 nm. Changes in the N₂ adsorption-desorption isotherms, the surface area, and the pore volume values can be attributed to the incorporation of organic groups with relatively large molecular dimensions. Nevertheless, based on XRD, HRTEM, and N₂ adsorption characterization, we conclude that the Pd(II) supported mesopolymer catalyst FDU-NHC/Pd(II) with an ordered hexagonal structure was prepared successfully.

Fig. 4. N_2 adsorption-desorption isotherms of FDU-15 (1), FDU-NHC (2), and FDU-NHC/Pd(II) (3).

Table 1 Textural properties of the parent FDU-15 and the functionalized samples

Sample	A_{BET} (m^2/g)	Pore volume (cm^3/g)	Pore size (nm)
FDU-15	489	0.38	3.5
FDU-NHC	389	0.27	3.0
FDU-NHC/Pd(II)	271	0.20	3.0

2.2 Performance of the catalysts

The Sonogashira reaction between iodobenzene and phenylacetylene over FDU-NHC/Pd(II) was studied as a model reaction to optimize the reaction conditions. The results listed in Table 2 show the effect of solvent, reaction temperature, and catalyst dose. When DMA was used as the solvent, a high yield of product **1a** was obtained (Table 2, entry 1). DMF and DMSO gave lower yields (Table 2, entries 2 and 3). Other solvents such as toluene, acetonitrile, and ethanol were also evaluated and the results were not satisfactory (Table 2, entries 4–6). The effect of reaction temperature and catalyst dose on the Sonogashira coupling was also investigated. When the reaction proceeded at a lower temperature of 353 or 363 K, a prolonged reaction time was needed to obtain a considerably higher product yield (Table 2, entries 7 and 8). Furthermore, when the amount of FDU-NHC/Pd(II) catalyst was reduced to 0.5 mol% relative to the substrate the product was obtained in only 84% yield.

After optimizing the reaction conditions, we investigated the performance of FDU-NHC/Pd(II) for the transformation of various aryl halides with substituted phenylacetylene. The results are summarized in Table 3. The reactions between aryl iodides bearing electron deficient groups such as nitro and fluoro all gave satisfactory yields for the corresponding products within 6 h (Table 3, entries 1–6). On the

Table 2 Influence of solvents, reaction temperature, and catalyst dose on the model reaction of iodobenzene and phenyl acetylene

Entry	Solvent	Temperature (K)	Catalyst dose (mol %)	Time ^a (h)	Yield ^b (%)
1	DMA	373	1	3	94
2	DMF	373	1	3	90
3	DMSO	373	1	3	92
4	CH_3CN	355	1	28	84
5	Toluene	373	1	5	28
6	$\text{CH}_3\text{CH}_2\text{OH}$	343	1	3	10
7	DMA	363	1	5	81
8	DMA	353	1	6	61
9	DMA	373	0.5	3	84

Reaction conditions: iodobenzene 0.5 mmol, phenyl acetylene 0.75 mmol, K_2CO_3 1 mmol, catalyst 1 mol%, CuI 10 mol %, solvent 5.0 ml.

^aMonitored by TLC.

^bIsolated yield.

other hand, the catalyst offered moderate yields at a lower reaction rate for the substrates with electron-donating groups such as methyl (Table 3, entries 7–9). The reactions of various substituted phenylacetylene and iodobenzene also resulted in moderate product yields of 70%–84% (Table 3, entries 10–12). Furthermore, the reaction of bromobenzene also gave the product with a moderate yield (85%) whereas the reactions of 4-bromobenzonitrile and 1-bromo-4-nitro-

Table 3 Sonogashira reactions of various aryl halides and terminal alkynes catalyzed by FDU-NHC/Pd(II)

Entry	R	R'	X	Time ^a (h)	Product	Yield ^b (%)
1	4- NO_2	H	I	1	1b	92
2	2-F	H	I	6	1c	93
3	4-F	H	I	3	1d	90
4	3- CF_3	H	I	4	1e	91
5	4- COCH_3	H	I	4	1f	98
6	4- COOEt	H	I	2	1g	95
7	4- CH_3	H	I	24	1h	80
8	4- OCH_3	H	I	10	1i	51
9	1,3- CH_3	H	I	16	1j	45
10	H	3- CH_3	I	23	1k	83
11	H	4- CH_3	I	11	1h	70
12	H	4-CN	I	3	1l	84
13	H	H	Br	17	1a	85
14	4-CN	H	Br	13	1l	93
15	4- NO_2	H	Br	24	1m	87
16	4- NO_2	H	Cl	24	1m	Trace

Reaction conditions: aryl halides 0.5 mmol, substituted phenyl acetylene 0.75 mmol, K_2CO_3 1 mmol, catalyst 1 mol%, CuI 10 mol %, DMA 5.0 ml, 373 K.

^aMonitored by TLC. ^bIsolated yield.

benzene both gave good product yields (Table 3, entries 13–15). For 1-chloro-4-nitrobenzene, only a trace amount of corresponding product was detected (Table 3, entry 16).

The reusability of FDU-NHC/Pd(II) in the model reaction between iodobenzene and phenylacetylene was also investigated. The catalyst was recovered by simple filtration after the reactions, washed with acetone, dried and reused without any further activation. As shown in Fig. 5, the catalyst did not show noticeable deactivation after 10 times. The high reusability of the supported catalyst in the Sonogashira cross-couplings further demonstrates that the Pd ions are effectively stabilized by the *N*-Heterocyclic carbene ligands in the catalyst. Moreover, TEM characterization indicated that no Pd nanoparticles were present in the used FDU-NHC/Pd(II) catalyst and its Pd 3d XPS spectra were essentially the same as the fresh one.

Fig. 5. Recoverability of FDU-NHC/Pd(II) in the model reaction.

3 Conclusions

We developed a novel phosphine-free NHC-Pd(II) complex supported mesopolymer catalyst, FDU-NHC/Pd(II), which is efficient and reusable in Sonogashira coupling reactions. The catalyst can be easily separated from liquid reaction mixtures and reused more than 10 times without significant loss of activity. The optimal coordination modes of the *N*-heterocyclic carbene ligand with the palladium complex and the application of this catalyst to other palladium-catalyzed reactions will be further investigated.

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