

**Original Research Article**

# Schiff-base Post-Synthetic Modification of IRMOF-3 to Encapsulate Pd Nanoparticles: It's Application in C-C Bond Formation Cross-Coupling Suzuki Reaction

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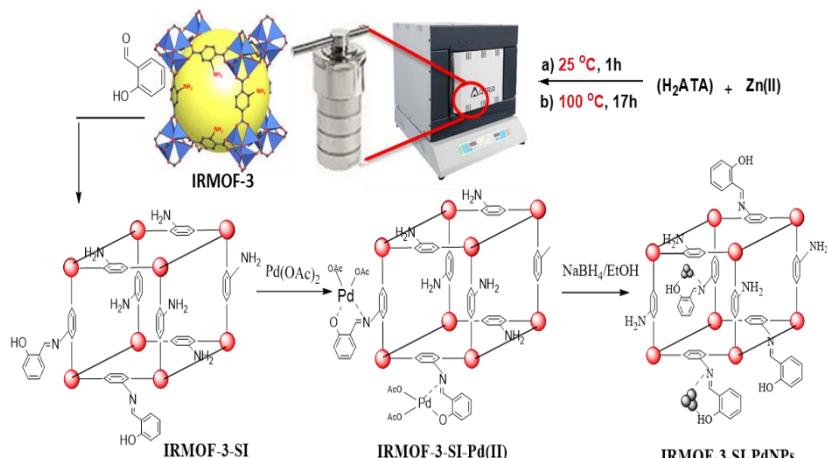
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## ABSTRACT

In this report, an IRMOF-3 was prepared via a solvothermal method, and a post-synthesis modification approach was employed for its Schiff-base functionalization with salicylaldehyde (SI). Next, Pd(II) ions were encapsulated into the pores of the Schiff-base modified MOF by an impregnation method. Reduction of Pd(II) to Pd NPs was performed by two distinct methods, namely reduction by NaBH<sub>4</sub> and reduction in ethanol. This study showed that the reduction of Pd NPs in ethanol solution resulted in larger NPs with better dispersity between the layers of the catalyst. In comparison, the reduction of Pd NPs with the NaBH<sub>4</sub> resulted in smaller NPs, positioning into the pores of the IRMOF-3 structure, which reduced the leaching of the catalyst. IRMOF-3-SI-Pd NPs was characterized with FT-IR, XRD, SEM, and TEM techniques. The results showed that the Schiff-base functionality of MOF helped stabilization of the Pd entities, and the catalyst successfully synthesized and preserved its structure during the synthesis and application steps. The synthesized IRMOF-3-SI-Pd NPs was used as a catalyst for the Suzuki cross-coupling reaction, which exhibited promising results.

## GRAPHICAL ABSTRACT



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## Introduction

Organic synthesis has long been a fascinating area of study in chemistry [1, 2]. In particular, carbon-carbon synthesis strategies that yield more robust and complex molecules are a hot topic in this area [3]. Akira Suzuki, the Nobel prize laureate who discovered Suzuki cross-coupling reactions, developed one of the most widely used protocols to form carbon-carbon bonds [4, 5]. Availability, stability, and sustainability of precursors, along with a mild condition and applicability for a wide range of substrates possessing various functional groups, are the main reasons for the popularity of Suzuki cross-coupling reactions [6].

In organic solvents and, more recently, in aqueous environments, soluble palladium (Pd) complexes have been used to promote these reactions [7, 8]. Although homogeneous catalysts have proved great activity and selectivity, their laborious separation procedure has driven researchers to employ heterogeneous catalysts [9]. Heterogenization of homogeneous catalysts on solid supports with high surface area seems to be a reasonable choice to overcome such obstacles [10]. To date, various heterogeneous solid supports, including graphene, graphene oxide, perovskites, metal-organic frameworks, etc., have been employed for the heterogenization of soluble metal nanoparticles (NPs) [11–13].

Metal-organic frameworks (MOFs) are porous crystalline reticular materials with ultrahigh surface area, tunable structure, and permanent porosity [14, 15]. These materials can be constructed from various inorganic nodes and almost infinite organic linkers, which cause their exceptional structural diversity and chemical tunability. Such features lead to their broad application in catalysis, electrocatalysis, photocatalysis, separation, drug delivery, organic transformations, etc. [16, 17]. Among them IRMOF-3 (isoreticular MOF-3 as an isoreticular structure of MOF-5), constructed from the amino-functionalized terephthalic acid (H<sub>2</sub>ATA), linker and octahedral [Zn<sub>4</sub>O]<sup>6+</sup> inorganic nodes have attracted a great deal of attention due to their high porosity and increased stability [18–20]. Amine functionality of organic linker in IRMOF-3

structure heightens its water resistance and provides the opportunity for the post-synthesis modification (PSM) of MOF structure [21–23]. Here, we prepared IRMOF-3 through a solvothermal synthesis method and employed the post-synthesis modification strategy for the Schiff-base functionalization of MOF with salicylaldehyde ligand (IRMOF-3-SI). Finally, an impregnation method was hired to encapsulate palladium ions (Pd<sup>2+</sup>) into the cavities of MOF, followed by a reduction to Pd NPs with NaBH<sub>4</sub> reductant and also a reduction in ethanol. Structural studies with FT-IR spectroscopies showed that the Schiff-base pendants helped stabilize Pd(II) ions. Synthesized IRMOF-3-SI-Pd<sub>NPs</sub> was successfully employed to promote Suzuki cross-coupling of various aromatic compounds, which showed promising results.

## Materials and Methods

### *Chemicals and apparatus*

Zinc(II) nitrate hexahydrate (Zn(NO<sub>3</sub>)<sub>2</sub>), amino-functionalized terephthalic acid (H<sub>2</sub>ATA), salicylaldehyde, palladium(II) acetate (Pd(OAc)<sub>2</sub>), sodium borohydride (NaBH<sub>4</sub>), ethanol, *N,N*-dimethyl formamide (DMF), CHCl<sub>3</sub>, and CH<sub>2</sub>Cl<sub>2</sub> were purchased from Merck.

### *Synthesis of IRMOF-3*

To prepare IRMOF-3, 12.5 mmol of Zn(NO<sub>3</sub>)<sub>2</sub> (2.36 g) and 4.1 mmol H<sub>2</sub>ATA (0.74 g) were dissolved in 100 mL dry DMF. A Teflon-lined autoclave was used to seal the solution and keep it at 100 °C for 17 hours after being stirred for 60 minutes at room temperature. Following the formation of brown solids, DMF and CHCl<sub>3</sub> were used to wash the synthesized IRMOF-3, and then 60 °C vacuum drying was used to dry the solids.

### *Synthesis of IRMOF-3-SI*

First, 1 g IRMOF-3 was dispersed in a 15 mL CH<sub>2</sub>Cl<sub>2</sub> solution. In another vessel, 107 μL of salicylaldehyde (1 mmol) was dissolved in a 15 mL CH<sub>2</sub>Cl<sub>2</sub> solution and added to the dispersed IRMOF-3 solution. Finally, the yellow solid was collected by centrifugation and dried at 60 °C in a vacuum.

### Synthesis of IRMOF-3-SI-Pd(II) and IRMOF-3-SI-Pd NPs

First, 0.5 g of synthesized IRMOF-3-SI and 20 mg Pd(OAc)<sub>2</sub> were added to a 6 mL acetonitrile solution. The mixture was vigorously stirred for six hours to stabilize Pd(II) ions in the MOF cavities. Then, the IRMOF-3-SI-Pd(II) was collected by centrifugation and dried at 60 °C in a vacuum.

Two different methods were employed for the preparation of IRMOF-3-SI-PdNPs. In the first method, IRMOF-3-SI-Pd<sub>NPs</sub> were prepared by refluxing IRMOF-3-SI-Pd(II) in an ethanol solution for 24 h. In the second method, 0.5 g IRMOF-3-SI-Pd(II) was dispersed in 5 mL of solvent, and then 0.3 g of NaBH<sub>4</sub> solution was added to this mixture. To ensure complete reduction of Pd(II) to Pd(0), this solution was vigorously stirred for 2 h. Finally, IRMOF-3-SI-Pd NPs were collected by centrifugation, washed, and dried at 60 °C in a vacuum.

#### General Suzuki cross-coupling procedure

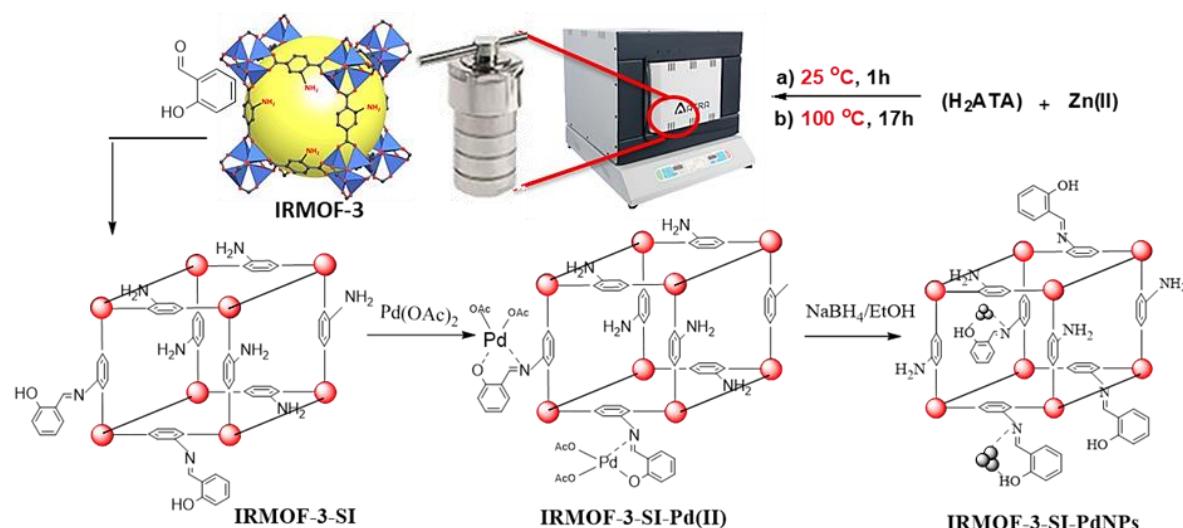
Aryl halide (1 mmol), phenylboronic acid (1 mmol), and potassium carbonate (2 mmol) were dissolved in a 3 mL DMF/H<sub>2</sub>O solution. Then, 0.5 mol% Pd encapsulated IRMOF-3 was added to this solution as a catalyst, and the solution was refluxed at 80 °C. Solid products were separated from the solution using CH<sub>2</sub>Cl<sub>2</sub> and distilled water. Column chromatography was used for the purification of the products. The concentrated

products were added to the silica gel column and extracted by EtOAc in an *n*-hexane (3:10) solution.

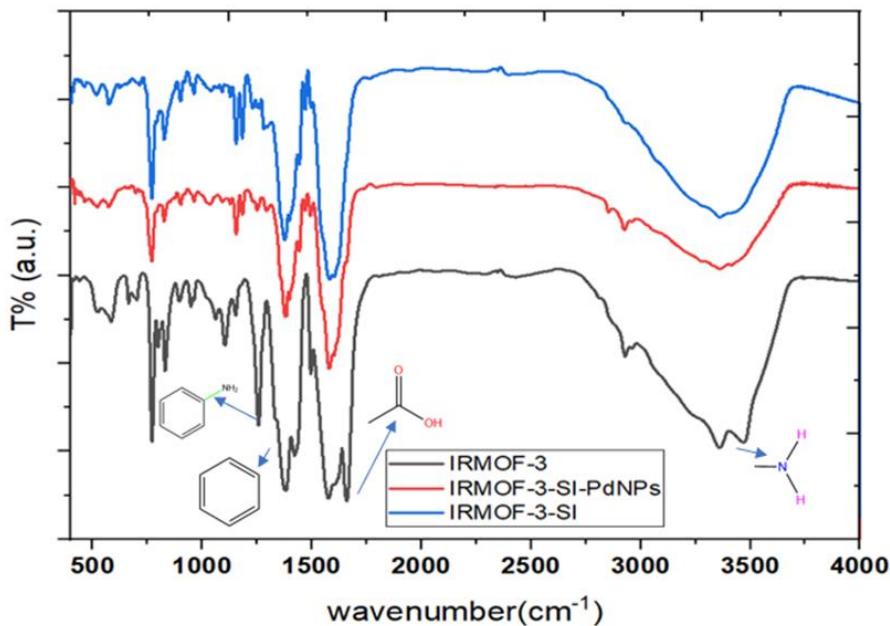
### Results and Discussions

**Scheme 1** illustrates preparation steps of IRMOF-3, IRMOF-3-SI, IRMOF-3-SI-Pd(II), IRMOF-3-SI-Pd NPs prepared by the reduction in ethanol and IRMOF-3-SI-Pd<sub>NPs</sub> prepared via reduction by NaBH<sub>4</sub>.

The FT-IR spectra of a series of IRMOF-3 PSM are presented in **Figure 1**. In the case of IRMOF-3, the peaks that appeared at 3359 and 3470 cm<sup>-1</sup> are related to the amino group of the organic linker. The peaks at 1385 and 1575 cm<sup>-1</sup> represent the C=O group of carboxylic acid. The sharp peaks at 1421, 1496, and 1656 cm<sup>-1</sup> show the C=C bonds of the aromatic ring. The peak at 1257 cm<sup>-1</sup> is related to the C-N bond between the aromatic ring and the amino group. FT-IR spectra of IRMOF-3-SI show a decrease in the intensity of -NH<sub>2</sub> peaks that appeared at 3359 and 3470 cm<sup>-1</sup>. Also, the peak related to the C-N bond red-shifted to 1250 cm<sup>-1</sup>. As can be seen from these two differences, post-synthesis modification was a success. Furthermore, the weak peak appeared at 1490 cm<sup>-1</sup>, showing the formation of an azomethine group. The FT-IR spectra of IRMOF-3-SI-Pd NPs show a decrease in the intensity of the peaks related to the -NH<sub>2</sub> group and the azomethine group, which prove the coordination of Pd NPs to these functional groups.



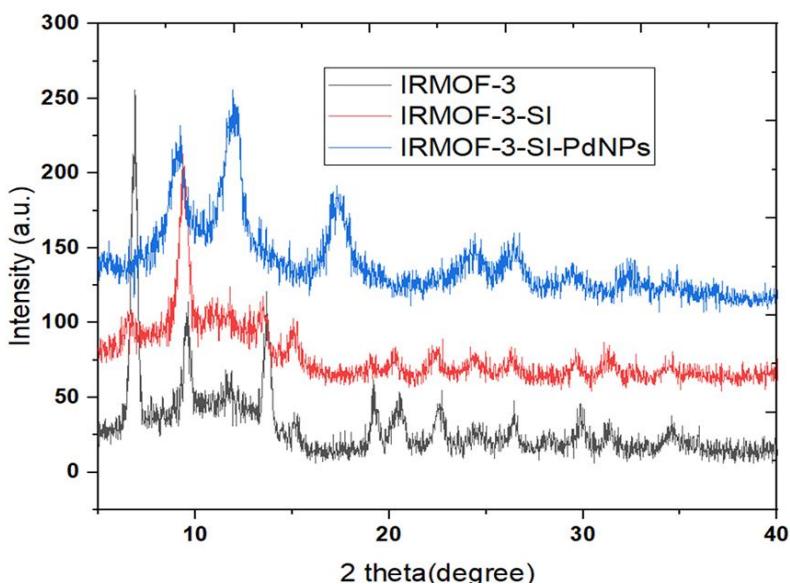
**Scheme 1:** Schematic synthesis of IRMOF-3-SI-PdNPs



**Figure 1:** FT-IR spectra of IRMOF-3, IRMOF-3-SI, and IRMOF-3-SI-Pd<sub>NPs</sub>

The crystallinity and the phase purity of the synthesized IRMOF-3, IRMOF-3-SI, and IRMOF-3-SI-Pd NPs were investigated by X-ray diffraction analysis (XRD) (Figure 2). The XRD pattern of the IRMOF-3 shows sharp peaks, which proves the crystallinity of the synthesized MOF. Also, the position of the peaks are in good coordination with reported examples, which proves the successful formation of the structure. The ZnO

lattice parameters were compatible with the JCPDS-36-1451 dataset and may be used to index a hexagonal wurtzite structure. The XRD patterns of modified MOF show that even after the post-synthesis modification of IRMOF-3 with salicylaldehyde ligand and reduction of Pd NPs, the crystallinity of IRMOF-3 was preserved.



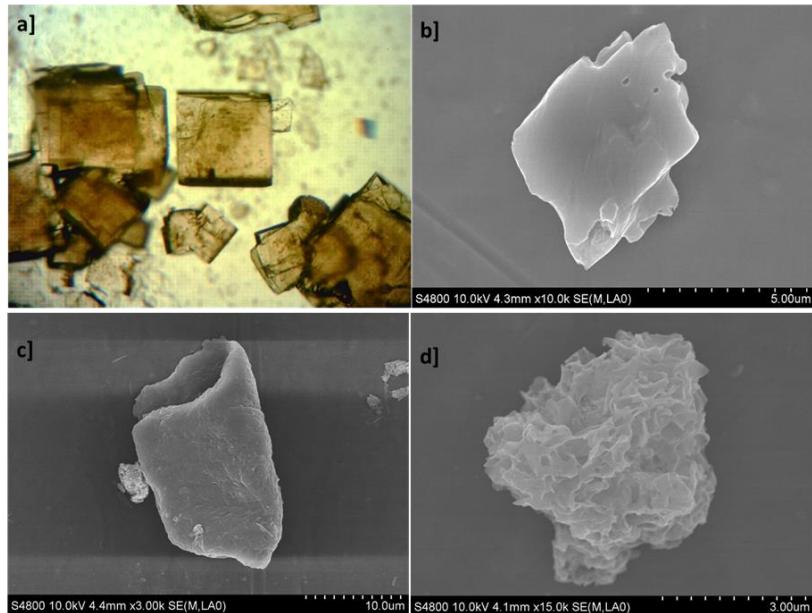
**Figure 2:** XRD pattern of IRMOF-3, IRMOF-3-SI, and IRMOF-3-SI-PdNPs

The morphology of as-synthesized MOFs was investigated by scanning electron microscope (SEM) images. Figure 3a and 3b show the sheet-

like morphology of bare IRMOF-3 by studies of optical photomicrograph and SEM analysis of MOF. In this structure, noncovalent interactions

such as hydrogen bondings,  $\pi$ - $\pi$  interactions, and aromatic-metal interactions between the IRMOF-3 layers led to the aggregation of the MOF. The SEM image of IRMOF-3-SI shows that even after its post-synthesis modification with salicylaldehyde,

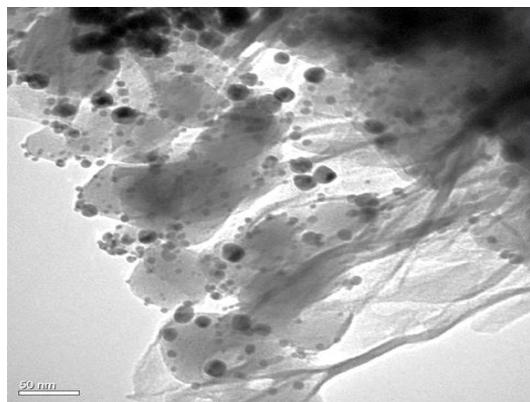
the sheet-like morphology of IRMOF is preserved (**Figure 3c**). **Figure 3d** images show the IRMOF-3-SI-Pd<sub>NPs</sub> surface. This image indicates that after the reduction of Pd NPs, the relatively smooth surface of IRMOF-3 becomes extremely rough.



**Figure 3:** (a, b) Optical photomicrograph and SEM images of IRMOF-3 [18], (c) SEM image of IRMOF-3-SI (d) SEM image of IRMOF-3-SI-Pd NPs

The transmission electron microscopy (TEM) image of IRMOF-3-SI-Pd NPs is represented in **Figure 4**. This image demonstrates that Pd NPs with a size distribution between 4-12 nm were obtained, which indicates that the Pd NPs are

formed between the layers of MOF on its outer surface. This image also indicates that the proposed support perfectly prevented the aggregation of the NPs, and Pd NPs are perfectly dispersed through the entire IRMOF-3-SI support.



**Figure 4:** TEM images of IRMOF-3-SI-Pd<sub>NPs</sub> prepared by the reduction in ethanol

The reaction between 4-bromoacetophenone and phenylboronic acid was chosen as the model reaction to test the efficiency of three catalysts (IRMOF-3-SI-Pd(II), IRMOF-3-SI-Pd NPs reduced by NaBH<sub>4</sub> and alcohol) in the Suzuki coupling reaction (**Scheme 2**). There were no discernible

differences in the catalytic activity of these three catalysts. The efficiencies of the products produced from all three catalysts were quite similar, and the only difference was in the time of the reaction.

**Scheme 2:** The fragmentation pattern of ligand (LH)

Various conditions, including the base, solvent, mol% of loaded Pd on the MOF, and the reusability of the catalyst, were tested to obtain the optimized condition for the Suzuki cross-coupling reaction. We initiate our study by selecting the best base for this reaction. In this regard, several bases, including  $K_2CO_3$ ,  $NaOAc$ ,  $Na_2CO_3$ , and  $Cs_2CO_3$ , were used as a base in the model reaction, in which  $K_2CO_3$  and  $Cs_2CO_3$  showed the most promising results. Next, the progress of the reaction in various solvents, including DMF, toluene, MeOH, EtOH, and a mixture of DMF/H<sub>2</sub>O (2:1), was monitored to select the best solvent for this reaction. This study showed that a mixture of DMF/H<sub>2</sub>O (2:1) is the best solvent. Still, due to the insignificant difference between the yield of the

reaction in this solvent and EtOH and considering the fact that EtOH is a green solvent; EtOH was chosen as the optimum solvent (**Table 1**). Next, we investigate the effect of mol% of loaded Pd on the solid support. To reach this goal, the progress of the reaction in the presence of solid MOF support containing 0.1, 0.3, 0.5, and 1 mol% of the Pd NPs were investigated. As shown in Table 1, 0.1 mol% of Pd NPs loaded on the solid support showed the least catalytic activity, and the 0.5 mol% of Pd NPs loaded on the solid support showed the highest catalytic activity. Further increase in mol% of loaded Pd NPs did not increase the catalytic activity, and hence 0.5 mol% was selected as the optimum amount.

**Table 1:** Optimization of the conditions for Suzuki cross-coupling reaction

Entry	Catalyst (mol%)	T (°C)	T (h)	Base	Solvent	Catalyst, Base Solvent	<chem>O=C(c1ccc(Br)cc1)c2ccccc2Br + c3ccccc3B(O)2 -&gt; [IRMOF-3-SI-Pd] Solvent, Base c4ccccc4-c5ccccc5C=O</chem>	Yield %
1	1	80	4	$K_2CO_3$	DMF/H <sub>2</sub> O (2:1)	[IRMOF-3-SI-Pd] DMF/H <sub>2</sub> O (2:1)	<chem>c1ccccc1-c2ccccc2C=O</chem>	100
2	0.5	80	4	$K_2CO_3$	DMF/H <sub>2</sub> O (2:1)	[IRMOF-3-SI-Pd] DMF/H <sub>2</sub> O (2:1)	<chem>c1ccccc1-c2ccccc2C=O</chem>	100
3	0.1	80	4	$K_2CO_3$	DMF/H <sub>2</sub> O (2:1)	[IRMOF-3-SI-Pd] DMF/H <sub>2</sub> O (2:1)	<chem>c1ccccc1-c2ccccc2C=O</chem>	80
4	1	80	4	$K_2CO_3$	DMF/H <sub>2</sub> O (2:1)	[IRMOF-3-SI-Pd] DMF/H <sub>2</sub> O (2:1)	<chem>c1ccccc1-c2ccccc2C=O</chem>	56
5	0.5	60	4	$K_2CO_3$	DMF/H <sub>2</sub> O (2:1)	[IRMOF-3-SI-Pd] DMF/H <sub>2</sub> O (2:1)	<chem>c1ccccc1-c2ccccc2C=O</chem>	92
6	0.5	50	4	$K_2CO_3$	DMF/H <sub>2</sub> O (2:1)	[IRMOF-3-SI-Pd] DMF/H <sub>2</sub> O (2:1)	<chem>c1ccccc1-c2ccccc2C=O</chem>	75
7	0.5	25	4	$K_2CO_3$	DMF/H <sub>2</sub> O (2:1)	[IRMOF-3-SI-Pd] DMF/H <sub>2</sub> O (2:1)	<chem>c1ccccc1-c2ccccc2C=O</chem>	64
8	0.5	80	4	$NaOAc$	DMF/H <sub>2</sub> O (2:1)	[IRMOF-3-SI-Pd] DMF/H <sub>2</sub> O (2:1)	<chem>c1ccccc1-c2ccccc2C=O</chem>	60
9	0.5	80	4	$Na_2CO_3$	DMF/H <sub>2</sub> O (2:1)	[IRMOF-3-SI-Pd] DMF/H <sub>2</sub> O (2:1)	<chem>c1ccccc1-c2ccccc2C=O</chem>	95
10	0.5	80	4	$K_2CO_3$	DMF	[IRMOF-3-SI-Pd] DMF	<chem>c1ccccc1-c2ccccc2C=O</chem>	94
11	0.5	80	4	$K_2CO_3$	Toluene	[IRMOF-3-SI-Pd] Toluene	<chem>c1ccccc1-c2ccccc2C=O</chem>	76
12	0.5	80	4	$K_2CO_3$	MeOH	[IRMOF-3-SI-Pd] MeOH	<chem>c1ccccc1-c2ccccc2C=O</chem>	68
13	0.5	80	4	$Cs_2CO_3$	EtOH	[IRMOF-3-SI-Pd] EtOH	<chem>c1ccccc1-c2ccccc2C=O</chem>	95

To test the generality of the proposed catalyst, we studied the Suzuki cross-coupling reaction of a variety of aryl halides and phenylboronic acids in the presence of three synthesized catalysts. The fact that all three catalysts show superior catalytic performance even at low concentrations of Pd ions or NPs proves the constructive interactions between the pores of the IRMOF-3-SI and the Pd entities. This synergic effect between Schiff-base functionalized IRMOF-3 and Pd entities result in

the facile progress of the Suzuki cross-coupling reaction for a variety of organic precursors possessing various functional groups. Such structure results in a high dispersity of Pd entities, preventing the homocoupling of aryl halides. **Table 2** summarizes the product of this reaction in the presence of IRMOF-3-SI-Pd NPs reduced by  $NaBH_4$  (Cat. a) and alcohol (Cat. b). Studies on the leaching of Pd NPs over the surface of IRMOF-3-SI was performed by the ICP

technique. This study shows that the leaching of IRMOF-3-SI-Pd NPs reduced by NaBH<sub>4</sub> is less than 0.6 % of all the Pd NPs in the catalyst (4.69 ppm), and the leaching of the sample reduced in ethanol

is 1.2 % of all the Pd NPs in the catalyst (8.27 ppm). The results could be due to the fact that the Pd NPs reduced by NaBH<sub>4</sub> are smaller in size and are positioned into the pores of the MOF.

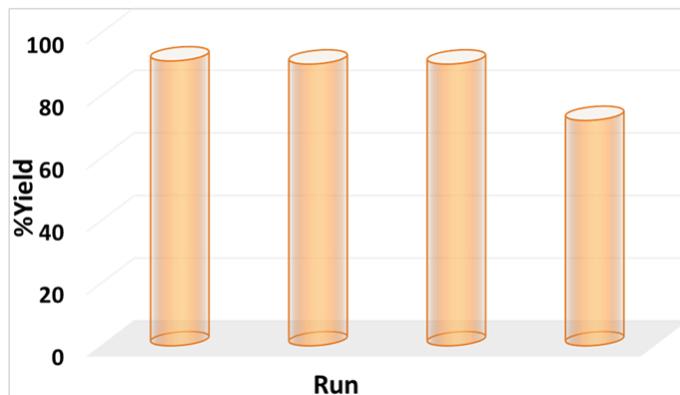
**Table 2:** Generality of proposed catalyst for the Suzuki cross-coupling reaction

Entry	product	Time (h) cat. a/cat. b	Yield (%) cat. a/cat. b
1 <sup>a'</sup>		5/3	100/95
2		6/4	96/96
3		24/24	82/85
4		4/3	95/97
5		4/2.5	98/98
6		24/18	85/85
7		24/18	90/93
8		24/24	88/80
9		4/3	98/95
10 <sup>b'</sup>		4/2	100/95
11		24/22	95/90
12		24/22	95/91
13 <sup>c</sup>		3/1.5	100/100
14 <sup>d</sup>		18/16	85/85

<sup>a'</sup> product of the reaction of bromobenzene and phenol, <sup>b'</sup> product of the reaction of 1-iodo-3-nitrobenzene and phenol, <sup>c</sup> product of the reaction of bromobenzene and phenol, <sup>d</sup> product of the reaction of 1-Bromo-3-nitrobenzene and phenol

One of the most important features of any catalyst is its reusability. To test the reusability of the proposed catalyst, we separated the catalyst from the reaction media and washed it with dichloromethane several times. IRMOF-3-SI-Pd

(II) for four cycles, IRMOF-3-SI-Pd NPs reduced by NaBH<sub>4</sub> for three cycles, and IRMOF-3-SI-Pd<sub>NPs</sub> reduced in ethanol for four cycles showed excellent recyclability (**Figure 5**).



**Figure 5:** Reusability of IRMOF-3-SI-Pd<sub>NPs</sub> for Suzuki reaction

## Conclusion

In conclusion, we synthesized a Schiff-base modified IRMOF-3 as a support for Pd NPs. We used synthesized IRMOF-3-SI-Pd<sub>NPs</sub> as a catalyst for the Suzuki cross-coupling of various organic linkers, in which the catalyst showed excellent performance. This study demonstrates that the Schiff-base functionality of manufactured catalyst helps stabilize Pd entities. In this method, we used reduction by NaBH<sub>4</sub> and reduction in ethanol solution to prepare Pd NPs. Results showed that the obtained catalyst with reduction of NPs in ethanol solution benefits high dispersity of the Pd NPs while the obtained catalyst by reducing NPs in the presence of NaBH<sub>4</sub> as reductant agent benefits from low leaching.

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## Authors' contributions

All authors contributed toward data analysis, drafting and revising the paper and agreed to be responsible for all the aspects of this work.

## Conflict of Interest

We have no conflicts of interest to disclose.

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