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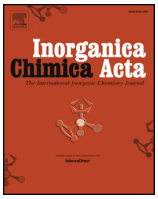

Research paper

One-pot synthesis of benzoazoles *via* dehydrogenative coupling of aromatic 1,2-diamines/2-aminothiophenol and alcohols using Pd/Cu-MOF as a recyclable heterogeneous catalyst

Javad Mokhtari[a,*], Abolfazl Hasani Bozcheloei[a]

[a] *Department of Chemistry, Science and Research Branch, Islamic Azad University, P.O. Box 14515/775, Tehran, Iran*

A R T I C L E I N F O

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Benzothiazoles
Pd-NPs/MOF
Nanoporous compound
$Cu_2(BDC)_2DABCO$
Heterogeneous catalyst

A B S T R A C T

In this paper we report an efficient synthetic approach for the preparation of the widespread numbers of benzoazoles *via* dehydrogenative coupling of 1,2-phenylenediamine or 2-aminothiophenol and benzyl alcohols by Pd/$Cu_2(BDC)_2$(DABCO)-MOF as new heterogeneous catalyst under solvent-free condition. The catalyst recycled and reused for four times without loss of catalytic activity. The structures of benzoazoles were corroborated spectroscopically (^{1}H- and ^{13}C NMR, and elemental analysis) and were confirmed by comparison with reference compounds. A plausible mechanism for this type of reaction is proposed.

1. Introduction

Benzoazoles and their derivatives are important building blocks found in miscellaneous series of biologically active compounds, agricultural chemicals, pharmaceutical chemicals, and organic materials [1–5] Furthermore, they are the basis of special chemicals for industrial applications such as pigments [6], optical brighteners for coatings [7] and etc.

The classical methods for the synthesis of these skeletons are the reaction between 1,2-phenylenediamines or 2-aminothiophenols and carboxylic acid, formic acid and aldehydes [8–12], hence experiencing disadvantages with regard to green chemistry points of view. Recently, different strategies for these structures have been developed and mentioned in the literature. Among the methods used for the synthesis of benzoazoles, [11–13] dehydrogenative coupling between alcohols and 1,2-phenylenediamine or 2-aminothiophenols are one of the most effective synthetic pathway by using homogeneous and heterogeneous catalyst such as Ir(2.0 wt%)/CeO_2, (PNNH)Co^ICl, Pt/TiO_2, Pt/Al_2O_3, $Ru_2Cl_4(CO)_6$ and etc. [14] that require stoichiometric amounts of hydrogen acceptors, strong bases, high temperature, unsafe solvent and in the case of homogeneous catalyst, failure to recover the catalyst [14–16]. So, the development of novel heterogeneous catalysts that can realize environmentally-benign synthesis of benzoazoles is an important topic. Application of supported palladium nanoparticles (PdNPs/support) as green and efficient heterogeneous catalysts has attracted a considerable attention [17–19]. Metal–organic frameworks (MOFs), as a new class of organic inorganic hybrid porous materials, have attracted considerable attention for stabilization and support for the metal nanoparticles due to their high surface areas, tunable pore sizes, and thermal stability [20]. Encapsulation of metal nanoparticles (NPs) in MOF structure generates new collaborative effects, by which the catalytic activity, selectivity and stability of the metal NPs can be enhanced effectively [21]. Recently, the application of MOFs as the supports for Pd nanoparticle has attracted considerable interest [22]. For catalytic applications Pd NPs have been successfully deposited at the outer surface of the MOF supports (Pd/MOFs) [23–28] or loaded inside the cavities of the MOFs (Pd@MOFs) via various methods such as impregnation and chemical vapor deposition [29–35].

We previously uncovered synthesis of Pd-NPs/$Cu_2(BDC)_2$DABCO-MOF using *via* temperature controlling program and its role as reusable heterogeneous catalyst in Suzuki cross coupling and oxidation of benzyl alcohols [36], Herein, in continues of our work and importance of the introduction and application of new catalysts in the synthesis of organic compounds and drawback of the reported methods in the literature as mentioned above, we report the development of this heterogeneous catalyst that are effective for the selective synthesis of benzimidazoles and benzothiazoles *via* a dehydrogenative coupling of 1,2-aryldiamines or 2-aminothiophenols with alcohols under mild conditions (Scheme 1).

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* Corresponding author.
E-mail address: j.mokhtari@srbiau.ac.ir (J. Mokhtari).

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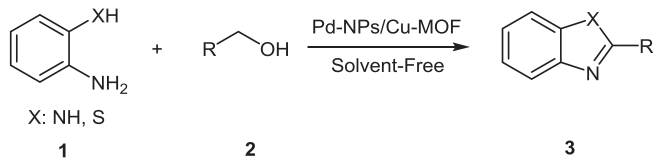
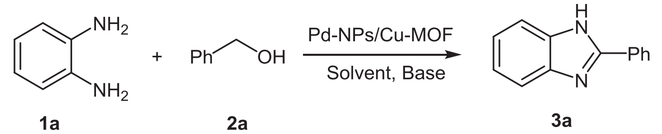


Scheme 1. Synthesis of benzoazoles catalyzed by Pd/Cu-MOF.

Table 1
Optimization of reaction conditions.[a]



Entry	Catalyst (mg)	Solvent	Base (20 mol%)	T (°C)	Yield (%)[b]
1	10	CH_3CN	Na_2CO_3	80	20
2	20	CH_3CN	Na_2CO_3	80	30
3	20	Toluene	Na_2CO_3	110	60
4	20	DMF	Na_2CO_3	120	65
5	**20**	–	**Na_2CO_3**	**120**	**88**
6	30	–	Na_2CO_3	120	86
7	40	–	Na_2CO_3	120	84
8	–	–	Na_2CO_3	120	0
9	20	–	–	120	25
10	20	–	Et_3N	120	82
11	Cu-MOF	–	Na_2CO_3	120	25

[a] Isolated yields.
[b] Reaction Conditions: benzene-1,2-diamine (1.3 mmol), benzyl alcohol (1.0 mmol), catalyst (20 mg), base (1.0 mmol).

2. Experimental section

2.1. Materials and instruments

All reagents including organic linker H_2BDC, metal salt Cu $(OAc)_2{\cdot}H_2O$, 1,4-benzenedicarboxylate (BDC, 99%), 1,4-diazabicyclo [2.2.2]octane (DABCO), Palladium (II) chloride ($PdCl_2$), 1,2-pheneylenediamines, 2-aminothiophenol, benzyl alcohols, sodium carbonate (Na_2CO_3) and ethyl acetate were obtained from commercially available sources such as Sigma–Aldrich and Merck without any purification. X-ray powder diffraction (XRD) measurements were performed using an X'pert MPD. Philips diffractometer with Cu radiation source (λ = 1.54050 Å) at 40 Kv voltage and 40 mA current. Transmission electron microscopy (TEM) was carried out using an EM10C-100 kV series microscope from the Zeiss Company, Germany and the actual loading of palladium was determined by Inductively Coupled Plasma (ICP) analysis on sequential plasma spectrometer, Shimadzu (ICPS-7000 ver. 2). BET (Brunauer-Emmett-Teller) surface area of the samples was determined from N_2 adsorption–desorption isotherms using a micromeritics ASAP 2020 analyzer. ^1H NMR and ^{13}C NMR spectra were measured ($CDCl_3$) with a Bruker DRX-500 AVANCE spectrometer at 500.13 and 125 MHz. Melting points were measured on an Electrothermal 9100 apparatus.

2.2. Supporting of palladium nanoparticle in MOF (Pd-NPs/$Cu_2(BDC)_2(DABCO)$)

Pd/$Cu_2(BDC)_2$DABCO was synthesized and characterized in the manner previously reported in our previous work [36]. A mixture of Cu $(OAc)_2{\cdot}H_2O$ (0.6 mmol), H_2BDC (0.6 mmol) and DABCO (0.3 mmol) with molar ratio of 2:2:1 were ball-milled at 28 Hz at room temperature for 2 h. The obtained green powder was washed with DMF (3 × 10 mL), and then with methanol (3 × 10 mL). Resulting green powder dried under vacuum at 130 °C for 12 h to yield 1.6 g of $Cu_2(BDC)_2$DABCO (94%).

200 mg of the synthesized $Cu_2(BDC)_2$(DABCO) was dissolved in 2 cc of DMF and then 5 mg of $PdCl_2$ with purity of 99.9% were added to the mixture. The solution was sonicated for 20 min, stirred at 80 °C for 20 h and finally stirred at 130 °C with the purpose of reduction of Pd(II) to Pd (0). The product (Pd-NPs/$Cu_2(BDC)_2$(DABCO) was centrifuged, washed with DMF and methanol and dried in vacuum at 120 °C for 12 h.

2.3. Catalyst usage for the dehydrogenative coupling

Typically, *o*-phenylenediamine (1.3 mmol) or 2-aminothiophenol (1 mmol), benzyl alcohols (1 mmol), Na_2CO_3 (20 mol%), and Pd-NPs/$Cu_2(BDC)_2$(DABCO) (20 mg, 0.01 mol%) were added to a round-bottom flask. The reaction mixture was heated to 120 °C and stirred at for the appropriate time in air (TLC monitoring). Ethyl acetate was added to the reaction mixture and catalyst was filtered. For the purification of impure products, chromatography on silica gel was performed (EtOAc:Hep. (1:6)). The entire products characterized by melting point, CHN, ^1H NMR and^{13}C-NMR spectroscopy.

3. Selected spectral data

3.1. 2-Phenyl-1H-benzo[d]imidazole (**3a**)

White crystals. mp 294–295 °C, ^1H NMR (500 MHz, DMSO-d_6, ppm): δ 12.91 (s, NH), 8.17 (d, 2H, ^3J = 7.6 Hz, 2CH of Ar), 7.66 (bs, 2CH of Ar), 7.47–7.56 (m, 4CH of Ar), 7.20 (bs, 2CH of Ar). ^{13}C NMR (125 MHz, DMSO-d_6, ppm): δ 151.2, 143.8, 134.9, 130.15, 129.8, 128.9, 126.4, 122.5, 121.6, 118.8, 111.3. Anal. Calcd. for $C_{13}H_{10}N_2$ (194.24): C, 80.39; H, 5.19; N, 14.42. Found: C, 80.35; H, 5.16; N, 14.38.

3.2. 2-Phenylbenzo[d]thiazole (**3g**)

White solid. m.p. = 113–114 °C. ^1H NMR (500 MHz, $CDCl_3$) δ 8.07–8.12 (m, 3CH of Ar), 7.91 (d, ^3J = 8.0 Hz, CH of Ar), 7.48–7.52 (m, 4CH of Ar), 7.26–7.42 (m, CH of Ar); ^{13}C NMR (125 MHz, $CDCl_3$) δ 167.9, 154.0, 134.9, 133.5, 130.8, 128.9, 127.4, 126.2, 125.0, 123.1, 121.5. Anal. Calcd. for $C_{13}H_{10}N_2$ (211.28): C, 73.90; H, 4.29; N, 6.63. Found: C, 73.86; H, 4.28; N, 6.61.

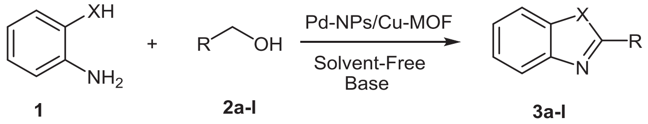
4. Result and discussion

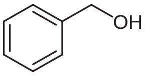
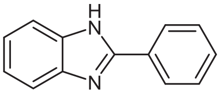
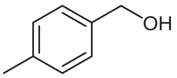
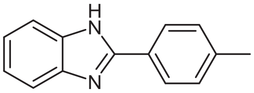
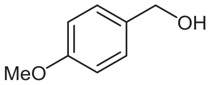
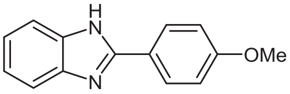
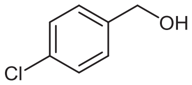
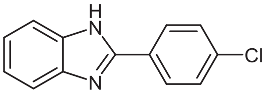
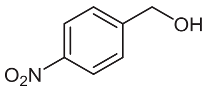
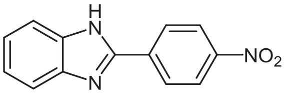
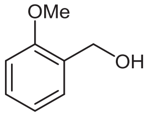
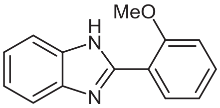
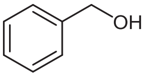
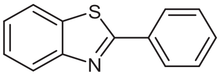
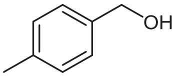
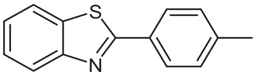
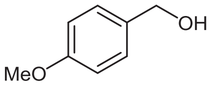
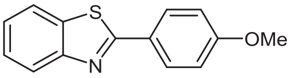
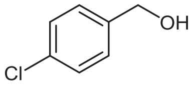
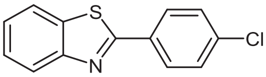
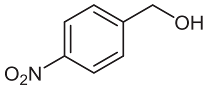
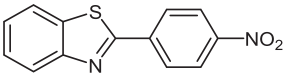
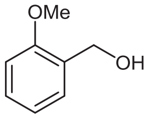
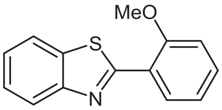
Pd/$Cu_2(BDC)_2$(DABCO) was synthesized and characterized in the manner previously reported in our previous work [36]. The XRD pattern of Pd@$Cu_2(BDC)_2$DABCO shows that the crystalline structure of $Cu_2(BDC)_2$DABCO is maintained after deposition of Pd NPs. The absence of a Pd diffraction pattern relates to the low Pd contents in the materials [36] (Fig. S1). Nitrogen adsorption-desorption and surface areas of Pd-NPs@$Cu_2(BDC)_2$(DABCO) samples were noticeably reduced compared to $Cu_2(BDC)_2$(DABCO), indicating that the cavities of $Cu_2(BDC)_2$(DABCO) may be occupied by highly dispersed Pd nanoparticles (Fig. S2). TEM micrograph (Fig. 2), confirmed that a crystalline and nano-scaled material was produced. It is also to emphasize that Pd nanoparticle has resulted in producing uniformly distributed nanoscale and with a mean particle diameter of 6.5 ± 0.2 nm as estimated from particle size distribution (Fig. 2). The actual loading of Pd (0) was characterized by ICP analysis and found to be 0.9%.

After full characterization of the synthetic PdNPs/$Cu_2(BDC)_2$DABCO, it was employed as a heterogeneous catalyst in the dehydrogenative coupling of 1,2-phenylenediamine and benzyl alcohols as model reaction. For this purpose, 1,2-phenylenediamine (1.0 equiv), benzyl alcohol (1.0 equiv), Na_2CO_3 (20 mol%) and catalyst (20 mg, 0.01 mol %) in CH_3CN under open air atmosphere were used as starting parameters. A yield of 30% within 24 h at a temperature of

Table 2

Synthesis of benzimidazoles and benzothiazoles *via* dehydrogenative of alcohols.



Entry	Benzyl alcohols	X	Products[a]	Yield (%)[b]	mp (°C) [ref.]
1		NH	 **3a**	88	294–295 [37]
2		NH	 **3b**	84	275–276 [37]
3		NH	 **3c**	86	226–227 [37]
4		NH	 **3d**	85	290–291 [37]
5		NH	 **3e**	81	324–326 [38]
6		NH	 **3f**	80	151–153 [39]
7		S	 **3g**	86	113–114 [40]
8		S	 **3h**	79	78–81 [40]
9		S	 **3i**	76	123–124 [40]
10		S	 **3j**	78	115–116 [41]
11		S	 **3k**	80	227–228 [40]
12		S	 **3l**	74	155–158 [40]

[a] Reaction Conditions: benzene-1,2-diamine (1.3 mmol) or 2-aminothiophenol (1.0 mmol), alcohol (1.0 mmol), Catalyst (20 mg), Na_2CO_3 (20 mol%).

[b] Isolated yields.

80 °C was observed. To investigate the effect of temperature and solvent on the yield, above reaction was examined in different solvent including toluene and DMF in reflux condition. The result summarized in Table 1. As shown in Table 1, from the acetonitrile to the DMF, the yield increased with increasing of temperature. On the other hand, and considering our experience in the oxidation of benzyl alcohol to aldehyde with this catalyst under solvent-free condition, [36] the reaction also was performed under solvent-free condition in 120 °C and interestingly, it was observed that the yield was very good and without the formation of impurities (Table 1, Entry 5). In order to prevent dialkylation of the diamine, the diamine to alcohol ratio was changed and an excess of diamine (1.3 eq) was used and as a result yield increased to 88%.

For the optimization of catalyst loading, 10, 20, 30 and 40 mg

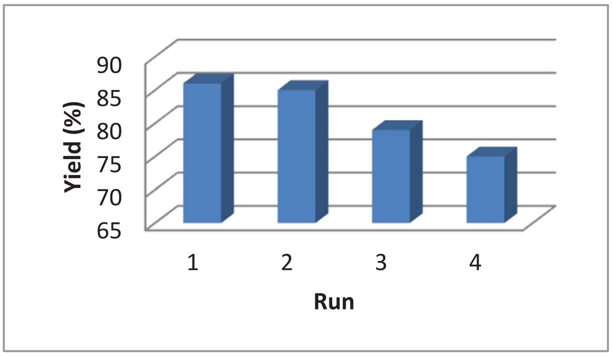


Fig. 1. Reusability test of the catalyst for the four times.

catalyst was used. The results in Table 1 shown that 20 mg (0.01 mol% base on Pd) (Entry 5) of catalyst is the best value and the increase over this amount does not have a noticeable effect on the yield (Entry 6, 7). On the other hand, when the reaction was examined without the catalyst, no product was formed (Table 1, Entry 8). To study the role of Pd nanoparticle in this reaction, a reaction with Cu-MOF alone was examined, the result shown that yield decreased significantly (Entry 11) and palladium plays an undeniable role in this reaction. The base, which accelerates the alcohol oxidation and mediates the condensation step, was optimized towards an efficient benzimidazole formation. Two different type of base such as Na_2CO_3 and Et_3N was used, results shown that both base led to good yield, however, due to the availability and easy work up of the product, sodium carbonate was used as base. In the absence of base, a yield of only 25% of **3a** was observed. We next investigated the scope of the synthesis of benzimidazoles and benzothiazoles derivatives (Table 2). The results summarized in Table 2 and shown that all benzylic alcohol with both electrons-withdrawing and electron-donating groups gave excellent conversions. Encouraged by this success, in the next step, we investigated the scope of the synthesis of benzothiazoles derivatives in the same manner as shown in Table 2.

Since the synthesized compounds are known compounds, therefore the structures of all products were elucidated from the comparison of melting point of them by literature reports (Table 2) and structure of some of them also characterized by ^{1}H NMR and CHN analysis (please see the supporting information).

For the study of reusability of Pd/Cu-MOF after completion of the reaction, ethyl acetate was added to the reaction mixture and catalyst filtered and washed by methanol and dried and activated under vacuum at 120 °C. The resulting recycled catalyst was used for dehydrogenative coupling of 1,2-phenylenediamines and benzyl alcohol. The reaction was completed after 12 h and isolated yield was 86%. This reusability experiment was repeated for more four times and the results summarized in Fig. 1. ICP analysis of the recycled Pd@Cu-MOF showed an overall loss of 1 wt% Pd after 4 runs.

As shown in Fig. 1, the yield decreased after 4 runs. The reason is probably due to the leaching of Pd nanoparticle and agglomeration. TEM Image of recycled catalyst confirmed crystalline structure of the palladium nanoparticles and a very small accumulation of palladium nanoparticles.

A comparison with other catalytic systems in the dehydrogenative coupling of diamines and alcohols was shown in Table 3. As shown in Table 3 the most of the catalyst needed harsh conditions such as difficult work up, homogenous catalyst and consequently the non-reusability of the catalyst, using of unsafe solvent such as toluene and expensive base including *t*BuOK. While, our present Pd@Cu-MOF catalyst exhibited a heterogeneous system, simple work up, solvent-free condition and reusability of the catalyst. To study the role of palladium in this reaction, under the same reaction conditions, three experiments with $Cu_2(BDC)_2$DABCO and CuO NPs were examined. The results shown in Table 3 indicate the importance of the presence of palladium NPs in this reaction.

The proposed reaction mechanism is shown in the scheme 2. At the first benzyl alcohol oxidized to benzaldehyde in presence of Pd (0) and O_2 [33]. In the next step, condensation of aldehyde and amine function of 1,2-phenylenediamine or 2-aminothiophenol led to intermediate **4**. In the next step Cu-MOF as a Lewis acid accelerated cyclization and formation of compound **6**. In the last step, hydrogen peroxide, which is obtained from the oxidation of benzyl alcohol in first step or O_2, leads to the oxidation of the compound **6** and the formation of compound **3**.

5. Conclusion

In conclusion, dehydrogenative coupling of aromatic 1,2-diamines or 2-aminothiophenol and benzyl alcohols for the formation of benzimidazoles and benzothiazoles under solvent-free conditions by a reusable heterogeneous Pd-NPs@Cu-MOF catalyst was discovered. The

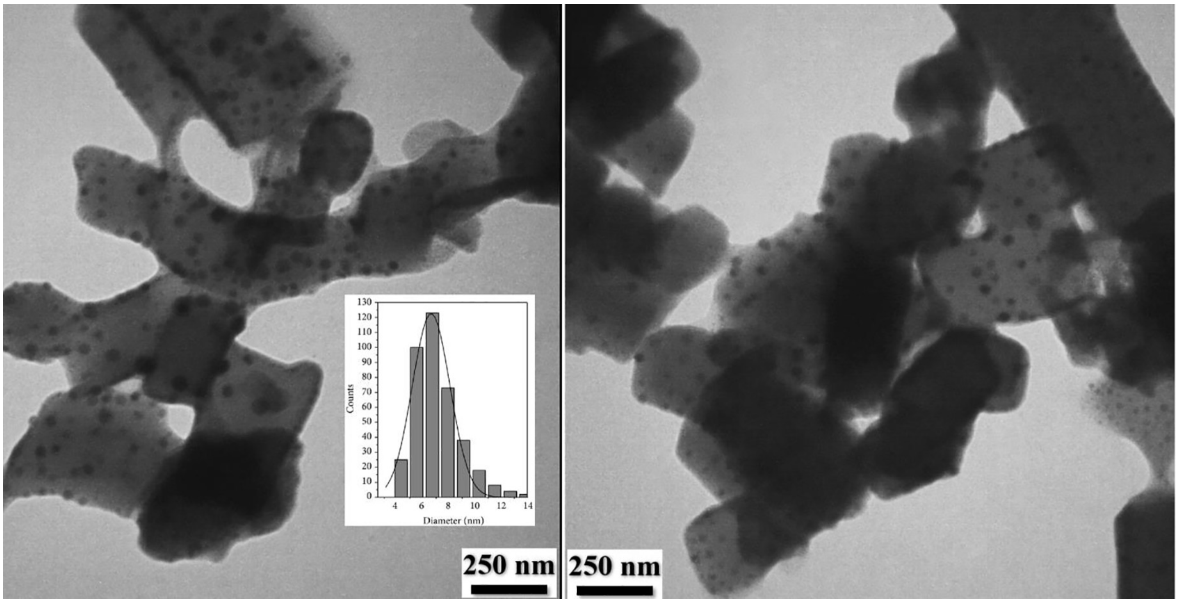
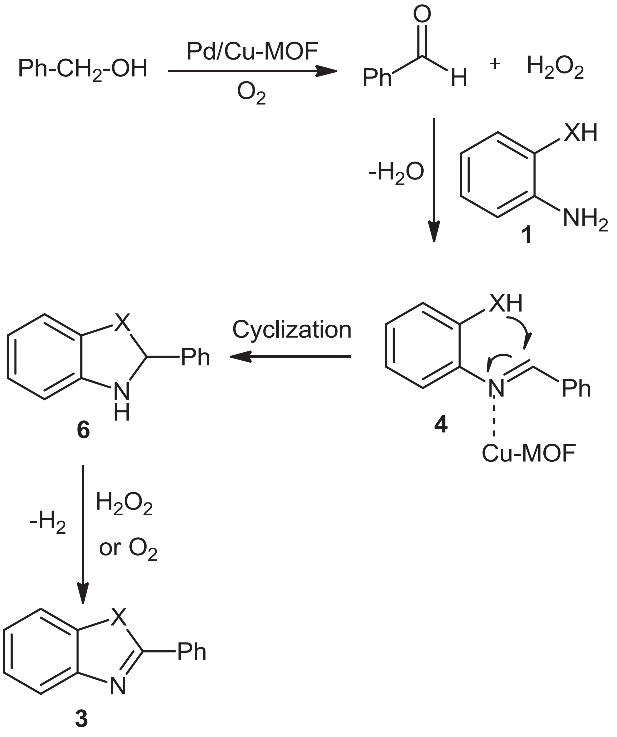


Fig. 2. TEM image of as-synthesized Pd@$Cu_2(BDC)_2$DABCO (left) TEM image of recycled Pd/$Cu_2(BDC)_2$DABCO (right).

Table 3
Comparison of different catalytic systems in the dehydrogenative coupling of *o*-phenylenediamines and benzyl alcohol.

Entry	catalyst	Reaction condition	Yield	Ref No.
1	Ir(2.0 wt%)/TiO_2 – 500	Mesitylene, 120 °C, 18 h, Ar atmosphere	91%	[37]
2	TAP-Cu 2 mol%	Toluene, reflux, *t*BuOK, 24 h	92%	[38]
3	10 mg Pt(0.2)@TiO_2	Ethanol, 80 °C, 303 k, 4 h, N_2 atmosphere	89%	[39]
4	$[IrCl(cod)]_2$ (1.4 mol%)	Diglyme, 110 °C, 24 h	85%	[40]
5	$Co_3O_4@Al_2O_3/SiO_2$ (5 mol %)	Solvent-free, 120 °C, 6 h, Air	92%	[41]
6	$Cu_2(BDC)_2DABCO$	Solvent-free/Na_2CO_3/120 °C, Air	31%	Present work
7	CuO NPs	Solvent-free/Na_2CO_3/120 °C, Air	15%	Present work
8	Pd@$Cu_2(BDC)_2DABCO$ (0.01 mol%)	Solvent-free/Na_2CO_3/120 °C, Air	88%	Present work



Scheme 2. The proposed mechanism for the synthesis of benzoazoles *via* dehydrogenative coupling.

reaction is applicable to a wide range of benzyl alcohols with good yields. We believe that this transformation provides an attractive benzimidazole and benzothiazole synthesis, being environmentally benign and atom efficient and forming H_2O and H_2 as the safe by-products. Further studies on encapsulation of other useful metal-NPs in MOFs and development of its catalytic scope are in progress and would be presented in the future.

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Appendix A. Supplementary data

Supplementary data associated with this article can be found, in the online version, at https://doi.org/10.1016/j.ica.2018.07.017.

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