Journal of Catalysis 320 (2014) 9–15

Contents lists available at ScienceDirect

Journal of Catalysis

journal homepage: www.elsevier.com/locate/jcat

Removable bidentate directing group assisted-recyclable metal–organic frameworks-catalyzed direct oxidative amination of Sp^2 C–H bonds

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ARTICLE INFO

Article history:
Received 18 June 2014
Revised 16 September 2014
Accepted 18 September 2014

Keywords:
Metal–organic frameworks
C–H activation
Copper-catalyzed
Direct amination
8-Aminoquinoline

ABSTRACT

Several Cu-MOFs were showed to be efficient heterogeneous catalysts for *ortho*-amination of benzoic acid derivative C–H bonds by N–H amines using 8-aminoquinoline as bidentate directing group. The optimal reaction conditions involve the use of Cu-MOFs (25%), *N*-methylmorpholine oxide (NMO) as an oxidant, secondary or primary amine coupling partner, DMF, DMA, or NMP solvent at 90–100 °C. Furthermore, the Cu-MOFs catalyst could be facilely isolated from the reaction mixture and reused several times without remarkable degradation in catalytic reactivity. Contribution from homogeneous leached active copper species, if any, is negligible. To the best of our knowledge, C–H activation reactions using bidentate directing groups, which are increasingly gaining importance, under heterogeneous catalytic systems were not previously mentioned in the literature.

1. Introduction

Transition metal-catalyzed carbon–nitrogen coupling reactions have offered widespread applications in the synthesis of many valuable substrates including pharmaceutical compounds, functional materials, and organic sensors [1–3]. Pre-functionalization of starting materials either in the aminated substrates or in the amines is required when conventional methods are employed either in homogeneous or heterogeneous manners [4,5]. Consequently, this adds up more chemical steps in the synthetic sequences which can significantly lengthen the procedures. From synthetic point of views, the direct coupling reactions from C–H and N–H are highly desired (Scheme 1) [6–8]. Since organic molecules often contain a wide range of chemically similar C–H bonds, selective transformations for non-activated C–H bonds are generally difficult to achieve [9,10]. Several approaches have been employed to address this challenge, the most common involves the use of directing groups, which bind to the metal center and bring catalysts to the proximal C–H bonds. Over last several years, a wide variety of functional groups have been evaluated as directing groups in the transformation of C–H bonds [11,12]. Homogeneous palladium, iridium, and ruthenium based catalysts have been employed for ligand-assisted direct amination of C–H bonds of [13–15]. Recently, reactivity enabled by cheap and abundant copper catalysts has attracted great interest. In particular, the first copper-mediated directed amination of C–H arene bonds was described by Yu group [16]. However, stoichiometric amount of copper is required and only 2-phenylpyridine derivatives were active [17–19]. Recently, with the use of 8-aminoquinoline (8-AQ) directing group [20,21], Daugulis has firstly reported that *ortho*-amination of sp^2 C–H bonds is possible under copper catalysis [22]. The utilization of an inexpensive copper catalyst and removable directing group allows for a favorable comparison with previous direct amination methodologies. Thus, the heterogeneous catalytic system for this transformation should be targeted for practical chemical industry as well as simplifying the product purification [23,24].

Metal–organic frameworks (MOFs) have recently emerged as an incredible class of crystalline porous materials with potential applications in different fields [25–28]. Although the utilization of MOFs in catalysis is a young research area, many MOFs have been investigated as heterogeneous catalysts or catalyst supports for a variety of organic transformations [29–32]. Recently, the use of MOFs and other heterogeneous systems with transition metal clusters for organic transformations, especially C–H functionalization reactions have increasingly gained attention [33,34]. With respect to MOFs catalysis, Sanford and Swartzers have pioneered using Pd-MOFs for arylation of naphthalene by arene C–H bonds [34a]. Bipyridyl-containing metal–organic frameworks of palladium and iridium were demonstrated as efficient catalysts for borylation of aromatic C–H bonds [34b]. However, regioisomers were obtained in many cases where steric effect is not predominant. Our group has described the deprotonative arylation of

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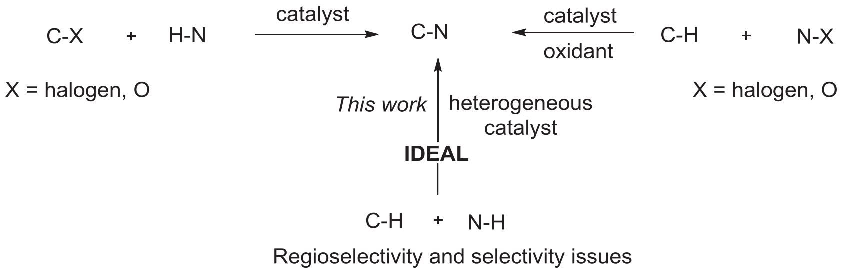
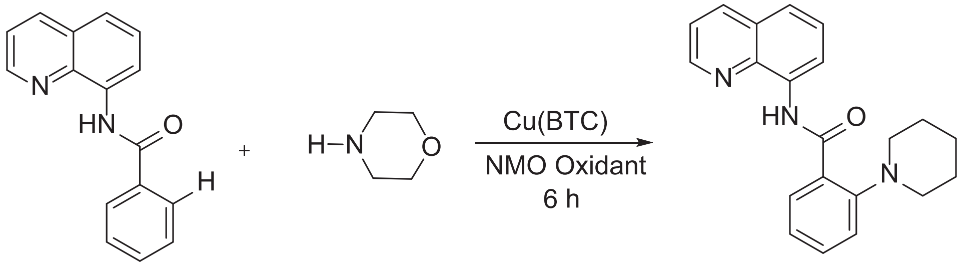
http://dx.doi.org/10.1016/j.jcat.2014.09.015



Scheme 1. Methods for transition metal-catalyzed C–N bond formation.

Table 1
Reaction condition development .[a]



Entry	Temperature (°C)	Amount of catalyst (%)	Solvent	Ratio	GC yield (%)
1	100	25	NMP	2	73
2	110	25	NMP	2	59
3	**90**	**25**	**NMP**	**2**	**73 (65)**
4	90	20	NMP	2	55
5	90	25	DMA	2	71
6	90	25	DMF	2	62
7	90	25	Dioxane	2	9
8	90	25	*p*-xylene	2	<2
9	90	25	NMP	1	66
10	90	25	NMP	3	71
11	90	25	NMP	0.5	44
12[b]	90	25	NMP	2	81 (71)
13[c]	**90**	**25**	**NMP**	**2**	**99 (87)**

Bold texts indicated the optimal conditions. These optimal conditions were further used in next studies.

[a] Volume of solvent 5 mL, 1.0 mmol scale, 6 h. Conversion by GC analysis.

[b] Reaction for 12 h.

[c] With AgOAc (20%) additive. Numbers in parentheses indicate the isolated yields. Kinetic studies of entries were placed in Supporting Information.

Table 2
Effect of silver salt additives to reaction yields.

Entry	Additive	Oxidant (2 equiv.)	GC yield
1	AgOAc	NMO	99
2[a]	AgOAc	NMO	92
3[b]	AgOAc	NMO	95
4	Ag_2CO_3	NMO	99
5	AgF	NMO	92
6	$AgNO_3$	NMO	73
7	Silica-supported silver	NMO	94
8[c]	AgOAc	–	14
9[d]	AgOAc	–	23
10	O_2	NMO	73

Reaction conditions: amide (0.5 mmol), amine (1 mmol), MOF-199 (25 mol%, 25.2 mg), AgOAc (25 mol%), NMO (2 equiv., 117 mg) in 5 mL NMP at 90 °C for 6 h.

[a] 10 mol% AgOAc was used.

[b] 50 mol% AgOAc was used.

[c] 1 equiv. AgOAc was used.

[d] 2 equiv. AgOAc was used.

variety of heterocycles using Cu-MOFs [35–37]. Despite the importance of ligand-assisted C–H activation reactions, especially in controlling regioselectivity, conditions using MOFs for these types of reactions have not been reported in the literature. Herein, we present the amination of *ortho*-aromatic C–H bonds using bidentate 8-aminoquinoline directing group under Cu-MOF catalysis. Similar isolated yields were obtained as compared to reports using homogeneous catalysts. Additionally, catalysts were able to be recycled and reused several times by simple filtration or centrifugation without significant degradation in catalytic activity.

2. Experimental

All Cu-MOFs were synthesized according reported literature (See Supporting Information for details) [38–43]. In addition, procedures for making all *N*-arenoyl-8-aminoquinoline were obtained from previous studies and placed in Supporting Information [21,44].

In a typical procedure, a mixture of *N*-benzoyl-8-aminoquinoline (0.124 g, 0.5 mmol), morpholine (0.087 g, 1 mmol) and diphenyl ether (0.055 mL) as an internal standard in *N*-methyl-2-pyrrolidone (NMP, 5 mL) were added into 25-mL flask containing the predetermined amount of Cu-MOF catalyst and *N*-methylmorpholine oxide (NMO, 0.087 g, 1 mmol) as an oxidant. The catalyst

Table 3
Effect of various oxidant.[a]

Entry	Oxidant	GC yield (%)
1	$K_2S_2O_8$	<5
2	Dicumyl peroxide	28
3	*Tert*-butylbenzoyl peroxide	25
4	H_2O_2	<2
5	O_2	22
6	Air	31

[a] Volume of solvent 5 mL, 1.0 mmol scale, 6 h. Kinetic studies of entries were placed in Supporting Information.

loading was calculated with respect to the copper/*N*-benzoyl-8-aminoquinoline molar ratio. The reaction mixture was stirred at 90 °C for 6 h. In kinetic studies, reaction conversion was monitored by withdrawing aliquots from the reaction mixture at different time intervals and quenching with an aqueous KOH solution (5%, 1 mL). The organic components were then extracted into ethyl acetate (2 mL), dried over anhydrous Na_2SO_4, and analyzed by GC with reference to diphenyl ether. The product characterization was confirmed by GC–MS and NMR.

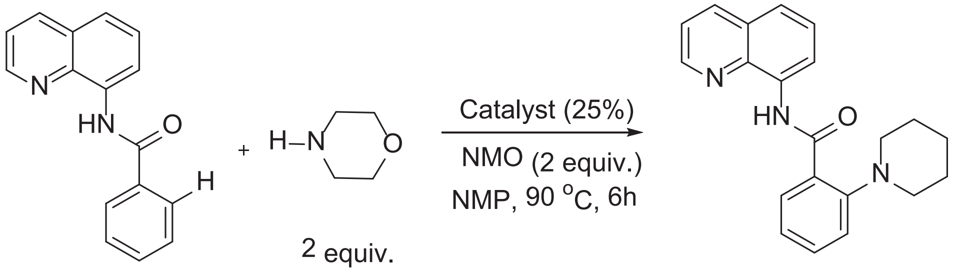
To investigate the recyclability of Cu-MOFs, the catalyst was separated from the reaction mixture by simple centrifugation and decantation, washed thoroughly with DMF followed by ethanol, dried under vacuum at 140 °C for 6 h, and reused. For the leaching test, a catalytic reaction was stopped after 30 min, analyzed by GC, and hot filtrated to remove the solid catalyst. The reaction solution was then stirred for a further 330 min. Reaction progress, if any, was monitored by GC as previously described. In addition, reactions using Poly(vinylpyridine) resin (20% mol) were carried out to further confirm the heterogeneity of catalysts. Three phase test with mercury (0) was also conducted to clarify the catalytic active species in reaction process.

3. Results and discussion

$Cu_3(BTC)_2$, (BTC = 1,3,5-benzenetricarboxylic acid), MOF-199, which was mostly used in this work, were fully characterized by several techniques including X-ray powder diffraction (XRD), scanning electron microscope (SEM), thermogravimetric analysis (TGA), Fourier transform infrared (FT-IR), inductively coupled plasma (ICP), and nitrogen physisorption measurements (Figs. S11–S17). The microscopic results are in agreement with the previous reports [36]. In particular, Cu-MOFs were highly crystalline in the cubic shape. TGA showed that the reduced mass at reaction temperature (90–110 °C) was negligible. All other MOFs synthesized according to previous reports were characterized by XRD and FT-IR to confirm the catalyst structure (Figs. S1–S10) [38–43].

In optimization screening, commercially available MOF-199 was chosen as catalyst for the reactions between *N*-benzoyl-8-amino-quinoline and morpholine using previously used NMO oxidant (Table 1). Kinetic studies of entries were placed in Supporting Information. With respect to reaction temperature, similar results were obtained at 100 °C and 90 °C (entries 1, 3). Increasing temperature resulted in the moderately drop in reaction conversion (entry 2). Catalyst loading of 20% gave only 55% conversion (entry 4). Similar to NMP solvent, acyclic amide solvent such as DMA or DMF afforded about 70% product (entries 5, 6). Other solvents such as dioxane and *p*-xylene which were showed to be insufficient with less than 10% of product were obtained (entries 7, 8). Furthermore, increasing or decreasing molar ratio of amine/arene C–H bonds slightly slowed down the reaction (entries 9–11). Interestingly, reaction at longer time, 12 h, gave 81% GC yield (entry 12). Similar to reported optimal conditions with homogeneous catalyst in which 25% silver salt additive was required [22], quantitative conversion was achieved when 20% AgOAc was added (entry 13). High reaction selectivity was confirmed by the closed numbers of GC yields and the isolated yields (entries 3, 12, 13).

Table 4
Reactivity of other catalysts.[a]

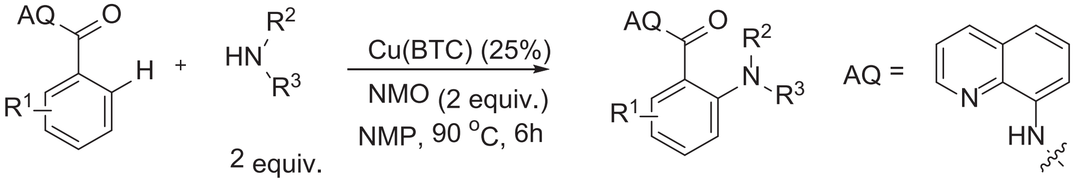


Entry	Type	Catalyst	GC yield (%)
1	Cu-MOFs	MOF-199	73 (99) (TOF[b] = 34)
2		CuBDC	78 (97)
3		$Cu_2(BDC)(DABCO)$	79 (93)
4		$Cu_2(BPDC)(BPY)$	50 (92)
5		$Cu_2(PDA)(BPY)$	68 (88)
6	Other MOFs	$Ni_3(BTC)_2$	5 (6)
7		Co-MOF-74	<2
8		$Mn_2(BDC)_2(DMF)_2$	<2
10	Common salts	$Cu(OCOPh)_2$	62 (84) (TOF[b] = 4.8)
11		$Cu(NO_3)_2$	65 (86)
12		$CuCl_2$	47 (79)

[a] Volume of solvent 5 mL, 1.0 mmol scale, 6 h. Kinetic studies of entries were placed in Supporting Information.

[b] Calculated at initial reaction rate (without AgOAc) as moles of product formed per hour and per mole of Lewis acidic active site (h^{-1}). Numbers in parentheses indicated the GC yields with 20% added AgOAc.

Table 5
Reactions scope of coupling components.



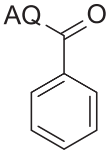
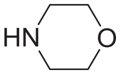
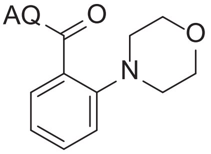
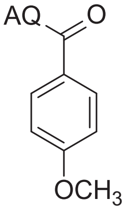
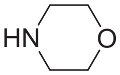
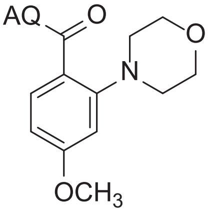
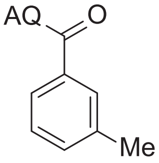
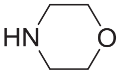
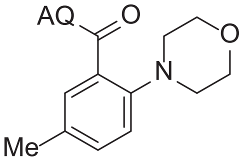
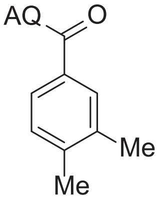
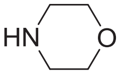
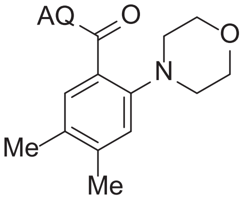
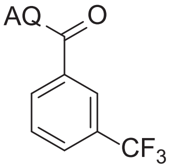
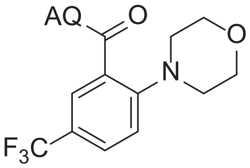
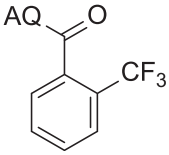
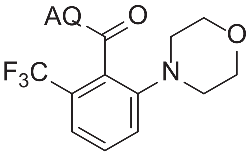
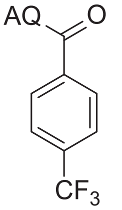
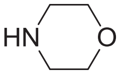
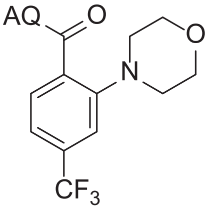
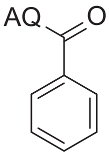
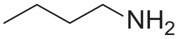
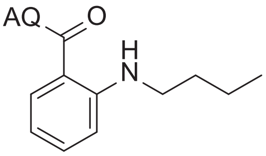
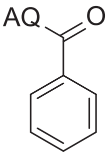
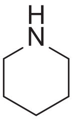
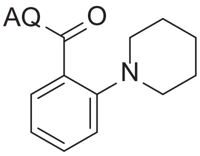
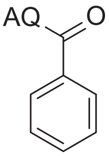
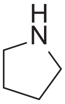
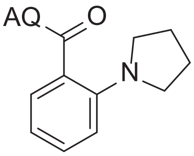
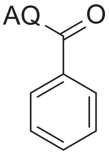
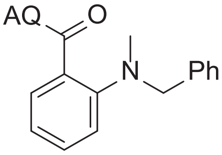
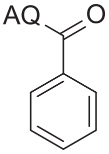
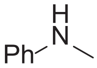
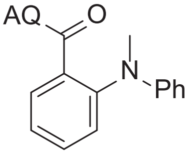
Entry	Benzoic derivative	Amine	Product	Yields (%)
1				65 82[a] 78[a,b]
2				61 85[a]
3				50
4				57
5				67
6				40
7				60 78[a]
8				41[a]

Table 5 (*continued*)

Entry	Benzoic derivative	Amine	Product	Yields (%)
9				72[a]
10				81[a]
11				88[a]
12				40

Conditions: DMA (4 mL), amide (0.5 mmol).

[a] Reactions with added AgOAc (20%).

[b] 5 mmol scale.

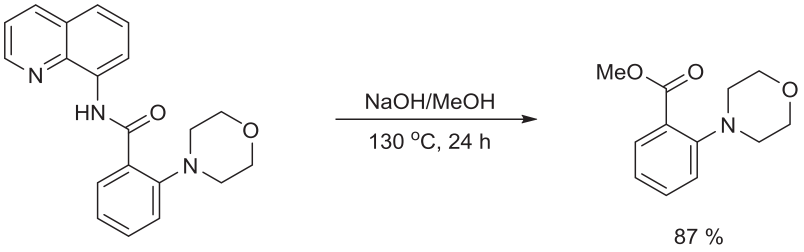
To clarify the role of additive AgOAc on the reaction process, several control experiments were conducted (Table 2). Particularly, reaction with 50% AgOAc afforded slightly lower yield than reaction with 20% (entries 1, 3). Similar results were obtained when only 10% AgOAc was employed (entry 2). Reactions with different silver salts including Ag_2CO_3, AgF, and $AgNO_3$ were also performed (entries 4–6). Interestingly, silver immobilized on silica gel was active and 94% GC yield was achieved (entry 7). When AgOAc was employed in stoichiometric amount and in the absence of NMO, significant drop on reaction rate was observed (entries 8, 9). Reaction under oxygen atmosphere afforded similar yield with reaction under argon without silver salt (entry 10). In pioneering work, Daugulis proposed silver as cocatalyst for above transformation [22]. Additionally, silver salts were frequently used as cocatalyst in organic reactions under palladium catalysis [9,10]. Based on previous reports and the aforementioned experimental results, under our optimal conditions, silver salt is likely to play as cocatalyst in reaction process.

Effect of various oxidants on reaction yields was then investigated (Table 3). In particular, inorganic oxidant $K_2S_2O_8$ was not suitable and no trace amount of product was detected (entry 1). Additionally, peroxides such as *tert*-butylbenzoyl peroxide or dicumyl peroxide gave similar results of about 25% conversion (entries 2,3). However, simple hydrogen peroxide is not effective (entry 4). Reaction under oxygen atmosphere did not afford reasonable yield (entry 5). Similarly, low reaction yield was observed when air was employed as reaction oxidant (entry 6). In pioneering work from Daugulis group, NMO was also chosen as optimal oxidant. The use of *N*-oxide oxidant is expected to promote the formation of Cu(III)-oxo complex intermediate [45].

We then decided to test the catalytic activity of several open metal sites Cu-MOFs which have been frequently used as catalysts for organic transformations (Table 4) [46–48]. Gratifyingly, several Cu-MOFs such as CuBDC and Cu_2(BDC)(DABCO) offered good activity (entries 2,3). Slightly lower conversions were obtained with Cu_2(BPDC)(BPY) and Cu_2(PDA)(BPY) with 50% and 68%, respectively (entries 4,5). It is worth mentioning that other transition metal-based organic frameworks such as $Ni_3(BTC)_2$, $Mn_2(BDC)_2(DMF)_2$ or Co-MOF-74 are not effective and unappreciable amount of product was observed even AgOAc was employed (entries 6–8). Interestingly, reaction using homogeneous copper (II) benzoate, which is the “monomer” of Cu-MOFs, afforded lower yield, with only 62%. Similar results were achieved when other common copper salts, $CuCl_2$, $Cu(NO_3)_2$, were employed. As expected, added AgOAc remarkably enhances the reaction rate under copper catalysis.

The generality of optimal conditions using Cu(BTC) on other derivatives of coupling components is described in Table 5. Functionality occurs at *ortho* position and mono-aminated products were obtained in all cases. With respect to benzoic components, amination of substituted C–H bonds with electron-withdrawing, trifluoromethyl, or electron-donating groups, methyl and methoxy, are possible and products were obtained in good conversions (entries 2–7). In details, amination reaction of 8-AQ-benzamides with substituents at *meta*-position afforded products in reasonable conversions (entries 3–5). Reaction of morpholine with *ortho*-substituted sp^2 C–H bonds was also conducted and 40% conversion was obtained (entry 6). In agreement with homogeneous work, addition of silver salt significantly enhanced the reaction yields. Cross-coupling reactions of cyclic secondary amine, piperidine and pyrrolidine, resulted in aminated products in reasonable yields (entries 9, 10). In addition, acyclic secondary N–H is active and product was afforded in excellent yield (entry 11). Optimal condition is also applicable for mono-amination by primary amine and 41% product yield was achieved (entry 8). Interestingly, aromatic amine, which was not active under homogeneous conditions, was cross-coupled in moderate yield (entry 12).

In terms of practical viewpoint, the reactions can be scaled up to 10-fold without significant loss of yield (Table 4, entry 1). Furthermore, 8-aminoquinoline auxiliary can be removed by base hydro-



Scheme 2. Bidentate directing group cleavage.

lysis. In particular, heating amide **1** with NaOH in methanol at elevated temperature for 24 h afforded high yield of *ortho*-aminated benzoic acid (Scheme 2).

In leaching test, a control experiment was carried out using a simple hot filtration during the course of the reaction. The direct C–H/N–H coupling reaction was then conducted under optimal conditions. After 10 min with a conversion of 35% being detected, the Cu-MOF catalyst was removed from the reaction mixture by hot filtration. The liquid phase was then transferred to a new reactor vessel, magnetically stirred for an additional 5.5 h at 90 °C with aliquots being sampled at different time intervals, and analyzed by GC. It was observed that no further conversion was detected in the reaction mixture after the Cu-MOFs catalyst was separated from the reaction mixture (Fig. 1). In addition, ICP-MS revealed less than 30 ppm of copper in the filtrate from reaction mixture. To further clarify the actual active catalyst species, a control reaction with added insoluble poly(vinylpyridine) (PVPy) resin was carried out using MOF-199 catalyst. Poly(vinylpyridine) is well known to remove free copper species from solution. Also, it is clearly that PVPy is too big to enter the pore channel of Cu-MOFs catalyst. The experimental results revealed that similar reaction conversion was still achieved with 71% after 6 h in the presence of PVPy. Additionally, there was no significant change in reaction rate when pyridine was added. Furthermore, under homogeneous CuI catalysis and identical conditions, the catalysis was completely shut down when PVPy resin was added. These data conclusively indicated that the catalytic activity associated with the immobilized sites. Therefore, it is unlikely that there was contribution from leached active copper species. In additional, a three phase test in the presence of mercury (0) was also conducted. Under optimal condition, a conversion of 68% was obtained when 20% Hg (0) was used. This suggested that a Cu(0)/Cu(II) catalytic cycle is unlikely.

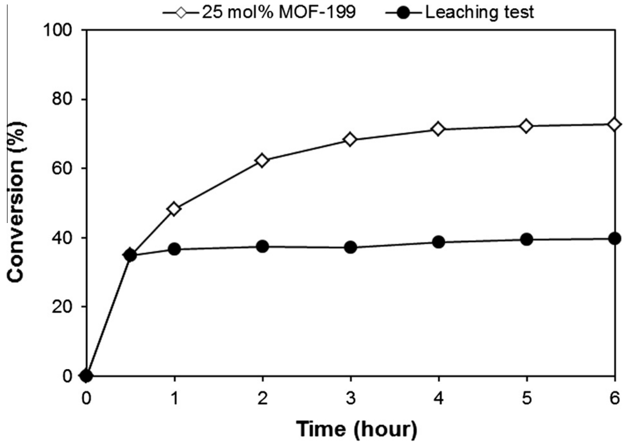


Fig. 1. Leaching test.

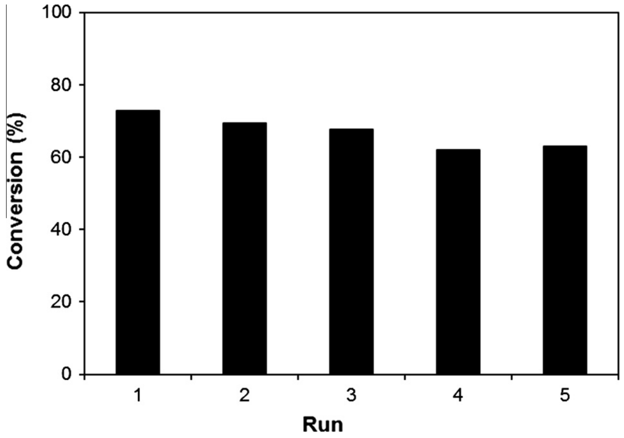


Fig. 2. Catalyst recycling studies.

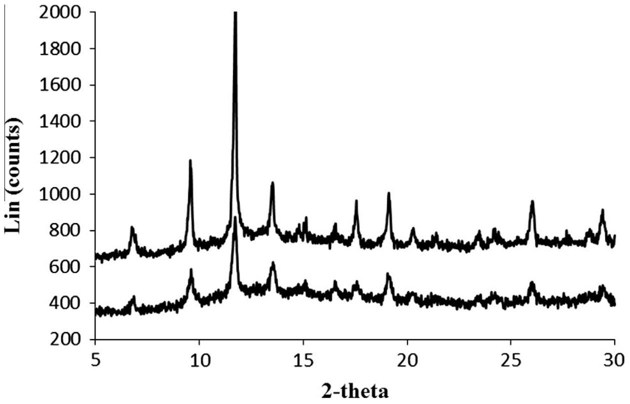


Fig. 3. PXRD patterns of fresh (a) and reused (b) MOF-199.

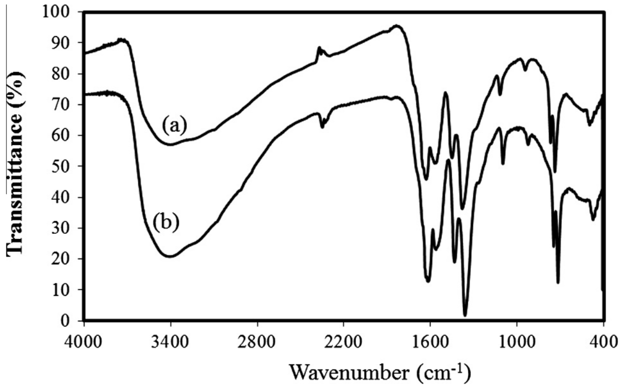


Fig. 4. FT-IR spectra of MOF-199 before (a) and after reused (b).

The recoverability and reusability of the Cu-MOFs catalyst were also targeted. After the experiment, the Cu-MOF catalyst was filtered from the reaction mixture, washed with copious amounts of DMF to remove any physisorbed reagents, dried at 140 °C under vacuum in 6 h, and then reused in further reactions under identical conditions to those of the previous runs. The results exhibited that a conversion of 63% was still achieved after 180 min in the 5th run (Fig. 2). Furthermore, PXRD and FT-IR of the reused catalyst after the 5th run indicated that the change in catalyst crystallinity is trivial, and the reused Cu-MOFs were still highly crystalline (Figs. 3 and 4).

4. Conclusions

In conclusion, we have described a method for auxiliary-assisted direct amination of β-sp^2 C–H bonds of benzoic acid derivatives under several Cu-MOFs catalysis. The optimal reaction conditions involve the use of NMO as an oxidant, secondary or primary amine coupling partner, DMF, DMA, or NMP solvent at 90–100 °C. Broad substrate scope is compatible under reported conditions. The method is advantageous compared to the existing routes due to utilization of heterogeneous copper catalyst and a removable bidentate directing group, which has not been previously described under heterogeneous catalytic systems. In addition, Cu-MOFs were showed to be recovered and reused several times without a significant degradation in catalytic activity and contribution from homogeneous leached active copper species, if any, is negligible.

Acknowledgments

The Viet Nam National Foundation for Science and Technology Development - is acknowledged for financial support.

Appendix A. Supplementary material

Supplementary data associated with this article can be found, in the online version, at http://dx.doi.org/10.1016/j.jcat.2014.09.015.

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