



Metal-Organic Framework $\text{Fe}_3\text{O}(\text{BPDC})_3$ as an efficient catalyst for the oxidative coupling reaction of benzaldehyde and (*E*)-1-phenylethan-1-one *O*-acetyl oxime

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ABSTRACT

A porous crystalline metal-organic framework $\text{Fe}_3\text{O}(\text{BPDC})_3$ was synthesized, and its properties were characterized by various techniques, including X-ray powder diffraction (PXRD), Fourier transform infrared (FT-IR), scanning electron microscopy (SEM), transmission electron microscopy (TEM), and nitrogen physisorption measurements. The $\text{Fe}_3\text{O}(\text{BPDC})_3$ was used as an efficient catalyst for the oxidative coupling reaction of benzaldehyde and (*E*)-1-phenylethan-1-one *O*-acetyl oxime to form 2,4,6-triphenylpyridine as desired product. The reaction could proceed readily, with more than 83 % reaction yield being achieved after 360 min at 140 °C in the presence of 10 mol% $\text{Fe}_3\text{O}(\text{BPDC})_3$ catalyst and Di-*tert*-butyl peroxide as an oxidant. This Fe-MOF exhibited higher activity than other MOFs and traditional homogeneous catalysts in the oxidative coupling reaction. The transformation could only proceed to obtain main product in the presence of $\text{Fe}_3\text{O}(\text{BPDC})_3$.

Introduction

Arylpyridines have attracted many scientists for their various applications such as photosensitizers [1], chemosensors [2], and important intermediates in the synthesis of surfactants, insecticides, herbicides, and therapeutic drugs [3]. For the synthesis of 2,4,6-triarylpyridines, several methods have been developed based on Chichibaban reaction, Mannich reaction, Michael reaction, Vilsmeier-Haack reaction or other synthetic procedures [4]. These synthetic ways usually occurred in the presence of various kinds of catalysts [5-11], or without a catalyst [11] in the organic reactions. For example, Montazeri and co-workers previously reported that pentafluorophenylammonium triflate (PFPAT) was found to be a recyclable catalyst for the preparation of 2,4,6-triarylpyridines from the reaction

of acetophenone derivatives, aromatic aldehydes and ammonium acetate [5]. Similarly, Satasia group demonstrated that cellulose supported ionic liquid catalysts exhibited high activity for the solvent-free synthesis of hydroxylated trisubstituted pyridines [6]. Under solvent-free conditions, Ren group also mentioned that the PEG₁₀₀₀-based dicationic acidic ionic liquid (PEC₁₀₀₀-DAIL) could be used as an efficient heterogeneous catalysts for the synthesis of substituted 2,4,6-triarylpyridines by the reaction of chalcones and ammonium acetate or via one-pot, three-component condensation of aromatic aldehydes with acetophenones and ammonium acetate [7]. Despite the efficiency of ionic liquids, there are some limitations such as tedious workup, hard conditions or the difficulty in separating catalysts for recycling. Consequently, catalysts containing metals were used to

overcome these drawbacks. The magnetic MIL-101-SO₃H was prepared and successfully employed as a highly active nanocatalyst for the synthesis of 1,3,5-triarylbenzenes and 2,4,6-triaryl pyridines with fairly good yields [8]. Elham group synthesized a highly efficient, eco-friendly and recyclable heterogeneous nano titania-supported sulfonic acid (n-TSA) catalyst for the synthesis of 2,4,6-triarylpyridines through one-pot three-component reaction of acetophenones, aryl aldehydes and ammonium acetate [9]. By using Fe₃O₄@TiO₂@O₂PO₂(CH₂)₂NHSO₃H as a sulfonic acid-functionalized titania-coated magnetic nanoparticle catalyst, Mohammad group developed green, efficient and powerful protocols for the preparation of 2,4,6-triarylpyridines and 1,8-dioxodecahydroacridines via an anomeric-based oxidation under mild and solvent-free reaction conditions [10]. Recently, a simple and efficient protocol developed for one-pot three-component synthesis of 2,4,6-triarylpyridines from aromatic aldehydes, substituted acetophenones and ammonium acetate using triflimide (HNTf₂) catalyst was reported [11]. From a viewpoint of green chemistry, developing an efficient catalyst system for this synthesis still remains to be explored.

Metal-organic frameworks (MOFs) containing metal sites with potential coordinative unsaturation and organic linkers are attractive due to their topological structures and special physical properties [12]. They possess high surface areas, tunable pore sizes, and the ease of processability, flexibility, and structural diversity [12]. In recent years, MOFs have great attention because they could be promising materials in many fields, particularly in the area of catalysis [13,14]. In details, MOFs have been proved to be remarkable heterogeneous catalysts in various organic reactions [13,14]. Owing to the nature of MOFs, iron containing metal-organic frameworks (Fe-MOFs) exhibited high catalytic activities in many organic transformations as compared to several popular MOFs [15-17]. Herein, we would like to describe the synthesis of 2,4,6-triphenylpyridine from benzaldehyde and (*E*)-1-phenylethan-1-one *O*-acetyl oxime utilizing the metal-organic framework Fe₃O(BPDC)₃ as an efficient catalyst.

Materials and Methods

Materials and instrumentation

All reagents and starting materials were purchased from Sigma-Aldrich and Acros, and used as received

without further purification. X-ray powder diffraction (XRD) patterns were recorded using a Cu K α radiation source on a D8 Advance Bruker powder diffractometer. Fourier transform infrared (FT-IR) spectra were obtained on a Nicolet 6700 instrument. Scanning electron microscopy studies were conducted on a S4800 Scanning Electron Microscope (SEM). Transmission electron microscopy studies were performed using a JEOL JEM 1010 Transmission Electron Microscope (TEM) at 80 kV. Nitrogen physisorption measurements were conducted using a Micromeritics 2020 volumetric adsorption analyzer system. Samples were pretreated by heating under vacuum at 150 °C for 3 h.

Gas chromatographic (GC) analyses were performed using a Shimadzu GC 2010-Plus equipped with a flame ionization detector (FID) and an SPB-5 column (length = 30 m, inner diameter = 0.25 mm, and film thickness = 0.25 μ m). The temperature program for GC analysis held samples at 100 °C for 1 min; heated them from 100 to 280 °C at 10 °C/min; held them at 280 °C for 8 min. Inlet and detector temperatures were set constant at 280 °C. Diphenyl ether was used as an internal standard to calculate reaction conversions. GC-MS analyses were performed using a Hewlett Packard GC-MS 5972 with a RTX-5MS column (length = 30 m, inner diameter = 0.25 mm, and film thickness = 0.5 μ m). The temperature program for GC-MS analysis heated samples from 60 to 280 °C at 10 °C/min and held them at 280 °C for 10 min. Inlet temperature was set constant at 280 °C. MS spectra were compared with the spectra gathered in the NIST library.

Synthesis of Fe₃O(BPDC)₃

In a typical preparation, a solution of FeCl₃·6H₂O (0.333 g, 1.24 mmol) in DMF (DMF = *N,N'*-dimethylformamide; 20 mL) was added in a mixture solution of DMF (20 mL) and acetic acid (0.8 mL, 14 mmol) containing H₂BPDC (H₂BPDC = 4,4'-biphenyldicarboxylic acid; 0.16 g, 0.66 mmol). After adding 40 mL DMF, the suspension was stirred to achieve a clear solution. The resulting solution was then distributed to 10 mL vials. The vials were then heated at 120 °C in an isothermal oven for 24 h. After cooling the vials to room temperature, the solid product was removed by decanting with mother liquor and washed in DMF (3 x 20 mL) for 3 days. Solvent exchange was carried out with dichloromethane (3 x 20 mL) at room temperature for 3 days. The material was then evacuated under vacuum at 150 °C for 6 h, yielding 0.15 g of Fe₃O(BPDC)₃ in the form of orange crystals (73 % based on H₂BPDC).

Catalytic studies

In a representative experiment, a mixture of (*E*)-1-phenylethan-1-one *O*-acetyl oxime (0.0708 g, 0.4 mmol), benzaldehyde (0.0106 g, 0.1 mmol), and diphenyl ether (0.017 g, 0.1 mmol) as an internal standard in chlorobenzene (1 mL) was added into a pressurized vial containing a predetermined amount of the $\text{Fe}_3\text{O}(\text{BPDC})_3$. The reaction mixture was magnetically stirred for 3 min to disperse the Fe-MOF catalyst in the reaction solution. Di-*tert*-butylperoxide (DTBP; 0.037 ml g, 0.30 mmol) as oxidant was then added. The resulting mixture was magnetically stirred at 140°C for 6 hours. Reaction yield was recorded by withdrawing samples from the reaction mixture at different time periods, analyzed by GC concerning diphenylether. The major product, 2,4,6-triphenylpyridine, was purified by column chromatography on silica gel. The product specification was additionally verified by GC-MS, ^1H NMR, and ^{13}C NMR.

Results and discussion

In this study, the $\text{Fe}_3\text{O}(\text{BPDC})_3$ was prepared from the reaction of iron chloride hexahydrate, and 4,4'-biphenyldicarboxylic acid via a solvothermal method, following a literature protocol. The Fe-MOF was then characterized by using several analysis techniques.

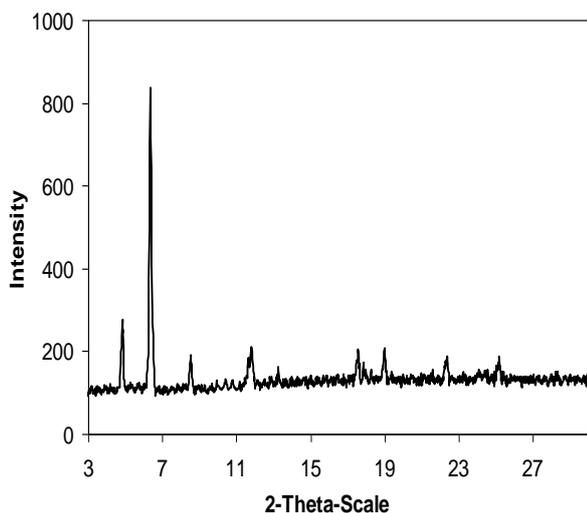


Figure 1: X-ray powder diffractograms of the $\text{Fe}_3\text{O}(\text{BPDC})_3$

As seen in Figure 1, a highly sharp peak with 2θ of approximately 6.8 was demonstrated on the X-ray diffraction pattern, revealing that a crystalline framework was generated. FT-IR indicated that the

carboxylate ion, formed by the deprotonation of $-\text{COOH}$ groups in H_2BPDC upon the reaction with iron (III) ions, exhibited a lower value for $\text{C}=\text{O}$ stretching vibration when compared with the free carboxylic acid (Figure 2). Besides, SEM micrograph showed that Fe-MOF exhibited highly polygonal-shaped morphology (Figure 3), and TEM image indicated a porous structure (Figure 4). The Langmuir surface areas were achieved approximately $1800 \text{ m}^2/\text{g}$, as calculated from nitrogen adsorption/desorption isotherm data.

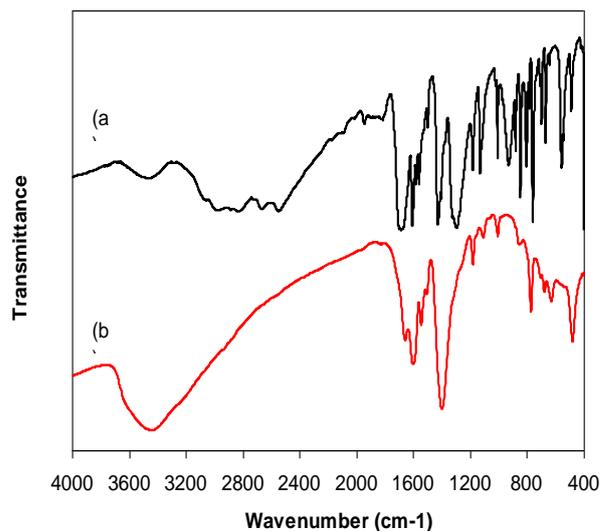


Figure 2: FT-IR spectra of biphenyl-4,4'-dicarboxylic acid (a), and the $\text{Fe}_3\text{O}(\text{BPDC})_3$ (b)

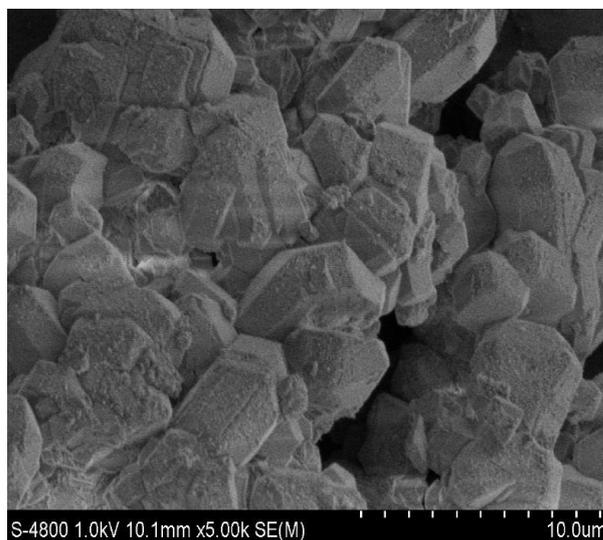


Figure 3: SEM micrograph of the $\text{Fe}_3\text{O}(\text{BPDC})_3$

In optimization studies, the $\text{Fe}_3\text{O}(\text{BPDC})_3$ was used as an efficient catalyst for the oxidative coupling reaction of benzaldehyde with 1(*E*)-1-phenylethan-1-one *O*-acetyl oxime to form 2,4,6-triphenylpyridine as the principal product (Scheme 1). Its structures were confirmed by ^1H NMR, and ^{13}C NMR.

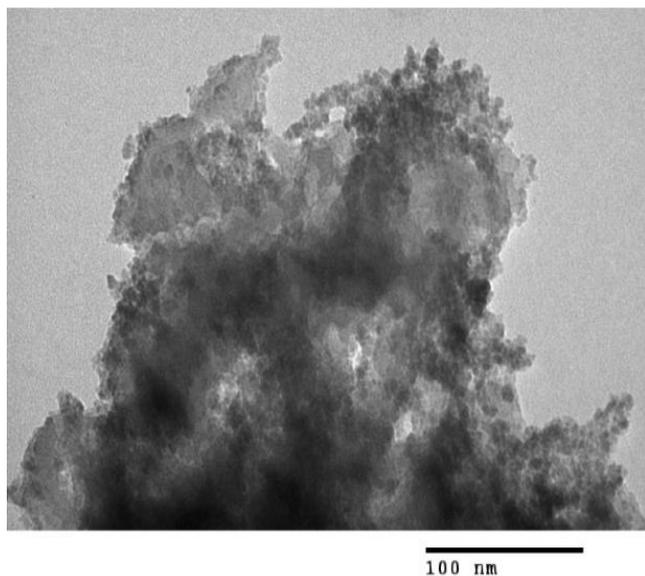
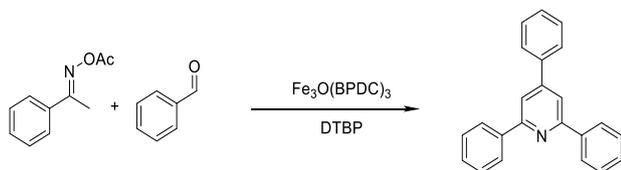
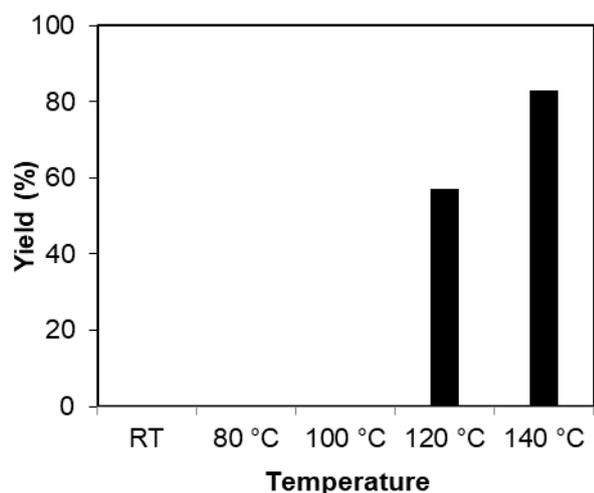
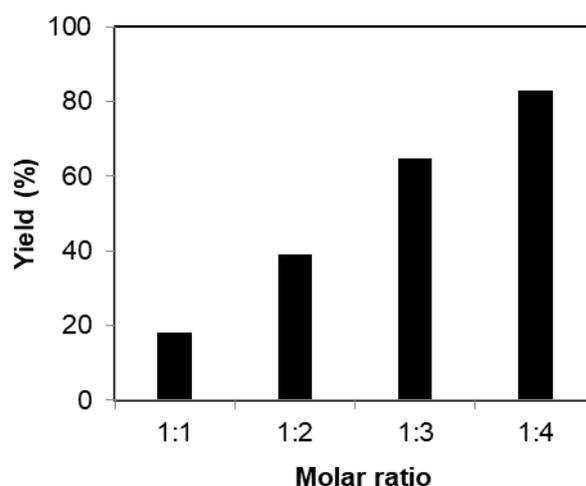
Figure 4: TEM of the $\text{Fe}_3\text{O}(\text{BPDC})_3$ Scheme 1: The coupling reaction of benzaldehyde and (*E*)-1-phenylethan-1-one *O*-acetyl oxime using $\text{Fe}_3\text{O}(\text{BPDC})_3$ catalyst

Figure 5: Effect of temperature on reaction yield

Initial studies addressed the effect of reaction temperature on reaction yield. The oxidative coupling reaction was carried out in 1 mL chlorobenzene at 10 mol% $\text{Fe}_3\text{O}(\text{BPDC})_3$ catalyst for 360 min, using the benzaldehyde and (*E*)-1-phenylethan-1-one *O*-acetyl oxime molar ratio of 1:4, in the presence of three

equivalents of *Di-tert*-butyl peroxide (DTBP) as an oxidant, at room temperature, 80 °C, 100 °C, 120 °C and 140 °C, respectively. The results showed that no desired product was detected in the transformation at room temperature, 80 °C, and 100 °C after 360 min. Interestingly, increasing the reaction temperature to 120 °C and 140 °C resulted in a significant drop in reaction yield, with 57% and 83% yields being observed after 360 min (Figure 5).

With this result in mind, the impact of the benzaldehyde and (*E*)-1-phenylethan-1-one *O*-acetyl oxime molar ratio on the reaction yield should be observed.

Figure 6: Effect of the molar ratio of benzaldehyde and (*E*)-1-phenylethan-1-one *O*-acetyl oxime on reaction yield

The reaction was carried out at 140 °C in 1 mL chlorobenzene, 10 mol% $\text{Fe}_3\text{O}(\text{BPDC})_3$ catalyst for 360 min, in the presence of three equivalents of *Di-tert*-butyl peroxide (DTBP) as an oxidant, using the benzaldehyde and (*E*)-1-phenylethan-1-one *O*-acetyl oxime molar ratio of 1:1, 1:2, 1:3, and 1:4, respectively. The obtained results revealed that the molar ratio exhibited a profound effect on reaction yield. Indeed, it was observed that the reaction proceeded difficultly when using the benzaldehyde and (*E*)-1-phenylethan-1-one *O*-acetyl oxime molar of 1:1, affording a reaction yield of only 18% after 360 min. Increasing the molar ratio to 1:2 and 1:3 led to a dramatic enhancement in reaction yield, with 39% and 65% yields, respectively, being achieved after 360 min. As expected, the reaction yield was obtained 83% after 360 min in the presence of the benzaldehyde and (*E*)-1-phenylethan-1-one *O*-acetyl oxime molar of 1:4 (Figure 6).

One important factor that should be addressed in an investigation of the oxidative coupling reaction

between benzaldehyde and 1(*E*)-1-phenylethan-1-one *O*-acetyl oxime with $\text{Fe}_3\text{O}(\text{BPDC})_3$ as the catalyst is the influence of different concentration of *Di-tert*-butyl peroxide (DTBP). The coupling reaction was carried out at 140 °C in 1 mL chlorobenzene, 10 mol % $\text{Fe}_3\text{O}(\text{BPDC})_3$, in the presence of the benzaldehyde and (*E*)-1-phenylethan-1-one *O*-acetyl oxime molar ratio of 1:4, using 0, 1, 2, 3, 4 equivalents of *Di-tert*-butyl peroxide (DTBP) as the oxidant, respectively. Without the presence of DTBP, it was found that almost no reaction yield was observed for the coupling reaction. When using 1 equivalent and 2 equivalents of DTBP, the reaction yields increased to 68% and 83% after 360 min, respectively. However, the principle product only obtained 85% after 360 min in the presence of 5 equivalents of DTBP. Therefore, increasing DTBP more than 3 equivalents was not necessary in this reaction (Figure 7).

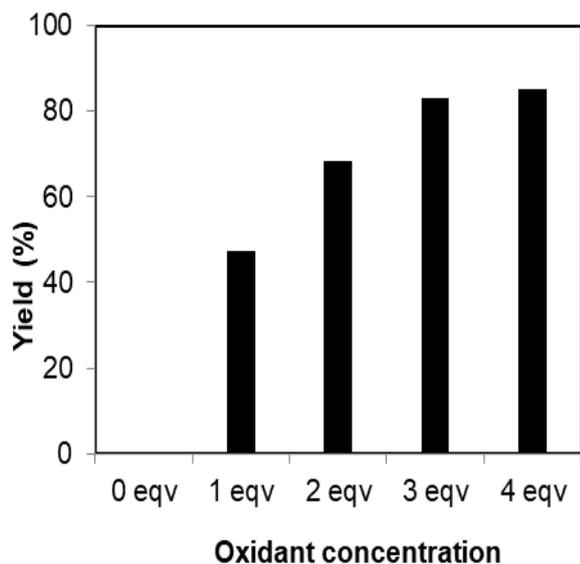


Figure 7: Effect of oxidant concentration on reaction yield

In the presence of 3 equivalents of DTBP as oxidant, another factor that must be observed for the reaction between benzaldehyde and 1(*E*)-1-phenylethan-1-one *O*-acetyl oxime is the catalyst amount of $\text{Fe}_3\text{O}(\text{BPDC})_3$. The catalyst amount was studied in the range of 0 mol% - 12 mol% relative to benzaldehyde at 140 °C.

Experimental results showed that the reaction yield was only obtained 17% after 360 min in the absence of $\text{Fe}_3\text{O}(\text{BPDC})_3$ catalyst. As expected, the reaction yield should be improved to 62% and 74% after 360 min when increasing the catalyst amount from 5 mol% and 7 mol%, respectively.

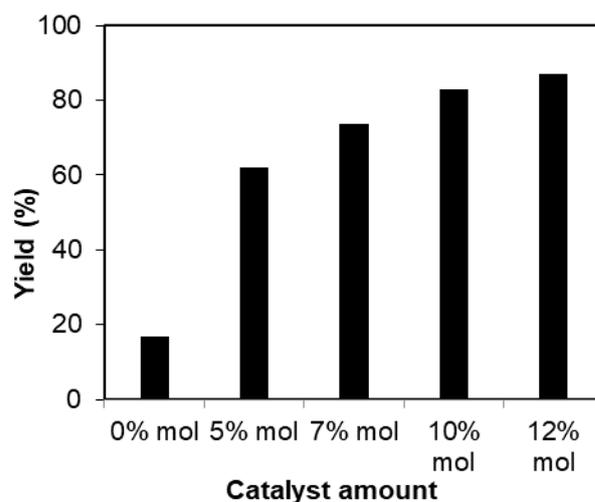


Figure 8: The effect of the catalyst amount on reaction yield

The reaction performed could lead to 83% yield after 360 min when using 10 mol % $\text{Fe}_3\text{O}(\text{BPDC})_3$ catalyst. Interestingly, it should be noted that 87% reaction yield was only achieved after 360 min in the presence of 12 mol% $\text{Fe}_3\text{O}(\text{BPDC})_3$ catalyst (Figure 8).

In several cases, the solvent could significantly accelerate or slow down the transformation, depending on the nature of the solid catalyst [18]. It was therefore decided to investigate the effect of different solvents on the reaction yield, having carried out the reaction at 140 °C for 240 min 10 mol% $\text{Fe}_3\text{O}(\text{BPDC})_3$ catalyst in the presence of 3 equivalents of DTBP as oxidant, using the benzaldehyde and (*E*)-1-phenylethan-1-one *O*-acetyl oxime molar ratio of 1:4, in different solvents including toluene, *p*-xylene, 1,4-dioxane, tetrahydrofuran (THF), *N,N'*-dimethylformamide (DMF), dimethylsulfoxide (DMSO), and chlorobenzene, respectively (Figure 9). The results showed that the reaction could occur well in non-polar solvents than polar solvents. Indeed, it was observed that there was no product formed in DMSO. Interestingly, *p*-xylene, 1,4-dioxane, THF, DMF were found to be unsuitable for the transformation, with the reaction yields of 25%, 17%, 19%, and 6%, respectively, being detected after 360 min. The yield should be improved to 70% when using toluene as solvent. As expected, chlorobenzene was found to be the best solvent, with 83% yield of 2,4,6-triphenylpyridine being obtained after 360 min for the transformation.

For a liquid-phase organic transformation in the presence of a solid catalyst, an important issue that should be taken into account is the possibility that some of catalytically active sites could dissolve into the reaction solution during the course of the reaction [19].

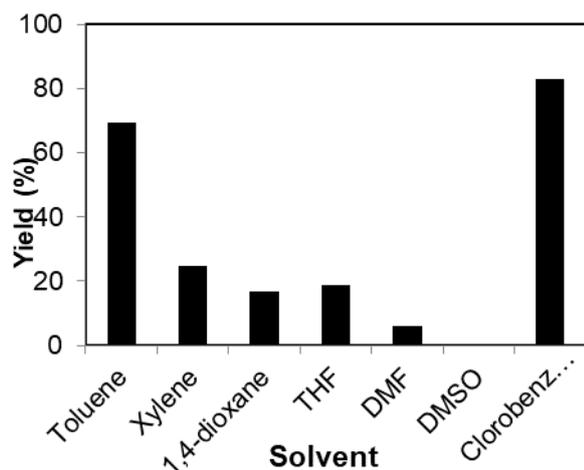


Figure 9: The effect of solvent on reaction yield

In order to determine if active iron species dissolved from the solid $\text{Fe}_3\text{O}(\text{BPDC})_3$ catalyst contributed to major product, the leaching test was carried out (Figure 10). The reaction was conducted in chlorobenzene at 140 °C for 360 min, using the benzaldehyde/ (*E*)-1-phenylethan-1-one *O*-acetyl oxime molar ratio of 1:4 and 3 equivalent of DTBP as the oxidant, in the presence 10 mol% $\text{Fe}_3\text{O}(\text{BPDC})_3$ catalyst. The solid catalyst was removed from the reaction mixture by simple centrifugation after the first 120 min reaction time with 44% yield being achieved. The liquid phase was then transferred to a new vial, and stirred for an additional 240 min at 140 °C with aliquots being samples at different time intervals, and analyzed by GC. The results showed that no further desired product was detected. It was obviously confirmed that the transformation could only proceed in the presence of the solid catalyst. Moreover, it was negligible if the active iron species was dissolved in the liquid phase.

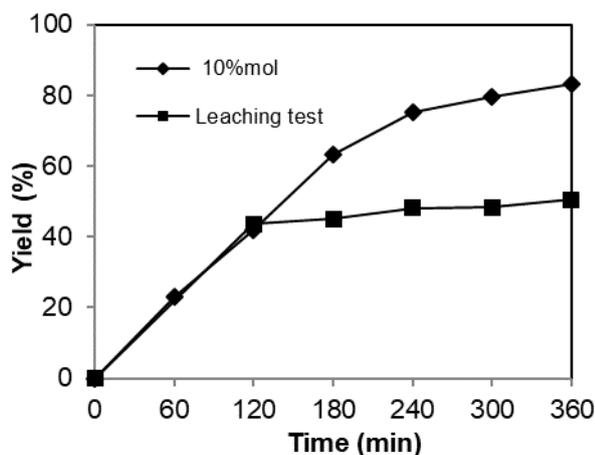


Figure 10: No donation from homogeneous catalysis to the generation of 2,4,6-triphenylpyridine was detected

To highlight the significant point of using $\text{Fe}_3\text{O}(\text{BPDC})_3$ as catalyst, the activity of $\text{Fe}_3\text{O}(\text{BPDC})_3$ in the oxidative coupling reaction of benzaldehyde and (*E*)-1-phenylethan-1-one *O*-acetyl oxime was compared with that of other homogeneous catalysts, including FeCl_3 , FeCl_2 , $\text{Fe}(\text{NO}_3)_3$, $\text{Fe}_2(\text{SO}_4)_3$, CuSO_4 , and $\text{Cu}(\text{NO}_3)_2$ (Table 1). The reaction was performed in chlorobenzene at 140 °C for 360 min, using the benzaldehyde/ (*E*)-1-phenylethan-1-one *O*-acetyl oxime molar ratio of 1:4 and 3 equivalent of DTBP as the oxidant, in the presence 10 mol% solid catalyst. It was noted that all homogeneous catalysts exhibited lower catalytic activity than $\text{Fe}_3\text{O}(\text{BPDC})_3$. In details, the reaction using FeCl_3 , FeCl_2 , $\text{Fe}(\text{NO}_3)_3$, and $\text{Fe}_2(\text{SO}_4)_3$ could proceed only 31%, 51%, 33%, and 20% yields after 360 min. Similarly, CuSO_4 and $\text{Cu}(\text{NO}_3)_2$ were found to be unsuitable as catalysts for the reaction, with the reaction yields of 46% and 18%, respectively, being detected after 360 min. Interestingly, 83% yield of 2,4,6-triphenylpyridine was achieved when using $\text{Fe}_3\text{O}(\text{BPDC})_3$ as catalyst.

Table 1: Different homogeneous catalysts for the coupling reaction

Entry	Catalyst	GC yield (%)
1	$\text{Fe}_3\text{O}(\text{BPDC})_3$	83
2	FeCl_3	31
3	FeCl_2	51
4	$\text{Fe}(\text{NO}_3)_3$	33
5	$\text{Fe}_2(\text{SO}_4)_3$	20
6	CuSO_4	46
7	$\text{Cu}(\text{NO}_3)_2$	18

In the next study, the catalytic performance of some heterogeneous was also investigated (Table 2). The oxidative coupling reaction was carried out in chlorobenzene at 140 °C for 360 min, using the benzaldehyde/ (*E*)-1-phenylethan-1-one *O*-acetyl oxime molar ratio of 1:4 and 3 equivalent of DTBP as the oxidant, in the presence 10 mol% catalyst. It was found that Fe-MOFs demonstrated greater catalytic activity over Cu-MOFs. Indeed, the reaction using CuBDC , $\text{Cu}_2(\text{BDC})_2(\text{DABCO})$ and $\text{Cu}_2(\text{OBA})_2(\text{BPY})$ as the catalysts proceeded with significantly more difficulty, with the reaction yields of 25%, 19%, and 23%, respectively, being detected after 360 min. Experimental results revealed that the $\text{Fe}_3\text{O}(\text{BDC})_3$ offered lower catalytic activity for the coupling reaction

as compared to the $\text{Fe}_3\text{O}(\text{BPDC})_3$, with only 67% yield being achieved after 360 min. As expected, the best yield of desired product was obtained with 83% in the presence of the $\text{Fe}_3\text{O}(\text{BPDC})_3$ catalyst.

Table 2: Different heterogeneous catalysts for the coupling reaction

Entry	Catalyst	GC yield (%)
1	$\text{Fe}_3\text{O}(\text{BPDC})_3$	83
2	$\text{Fe}_3\text{O}(\text{BDC})_3$	67
3	CuBDC	25
4	$\text{Cu}_2(\text{BDC})_2(\text{DABCO})$	19
5	$\text{Cu}_2(\text{OBA})_2(\text{BPY})$	23

Conclusions

In summary, the $\text{Fe}_3\text{O}(\text{BPDC})_3$ was successfully prepared by solvothermal method, and characterized by several techniques. This material could be employed as an efficient catalyst for the reaction between benzaldehyde and 1 *E*-1-phenylethan-1-one *O*-acetyl oxime to form 2,4,6-triphenylpyridine. The reaction afforded 83% yield of 2,4,6-triphenylpyridine in chlorobenzene at 140 °C after 360 min, utilizing the benzaldehyde/ (*E*)-1-phenylethan-1-one *O*-acetyl oxime molar ratio of 1:4 in the presence of 10 mol% $\text{Fe}_3\text{O}(\text{BPDC})_3$ catalyst and Di-*tert*-butyl peroxide as the oxidant. The $\text{Fe}_3\text{O}(\text{BPDC})_3$ exhibited higher activity to other MOFs such as $\text{Fe}_3\text{O}(\text{BDC})_3$, Cu(BDC), $\text{Cu}_2(\text{BDC})_2(\text{DABCO})$, $\text{Cu}_2(\text{OBA})_2(\text{BPY})$, and homogeneous catalysts including FeCl_3 , FeCl_2 , $\text{Fe}(\text{NO}_3)_3$, $\text{Fe}_2(\text{SO}_4)_3$, CuSO_4 , and $\text{Cu}(\text{NO}_3)_2$. No the contribution of leached iron species was detected in liquid-phase organic transformation when using $\text{Fe}_3\text{O}(\text{BPDC})_3$ as catalyst.

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