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Highly selective and efficient oxidation of benzylic alcohols with sulfate radical over metal-organic frameworks

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ABSTRACT

phosphonate-based metal-organic frameworks known as STA-12(M) (M = Mn, Fe, Co, Ni), show excellent catalytic activity towards oxidation of aromatic alcohols to aldehydes with $Na_2S_2O_4$/TBHP (*tert*-Butyl hydroperoxide) mixture. The STA-12(Fe) exhibited higher oxidation activity than other MOFs used in this work. Aldehydes were produced selectively in high yields under mild conditions at desired times. Recyclability for fourteen runs, without any noticeable decline in the conversion and selectivity of the product, demonstrates the high recoverability and the remarkable stability of title metal-organic frameworks. Selective radical quenching studies were performed and sulfate radicals was found to be the major oxidizing species in this catalytic system.

1. Introduction

The main advantage of metal-organic frameworks (MOFs) compared with other porous materials, relates to their substantially developed metal loading, which brings the possibility to considerably decrease the total amount of catalyst, provided that the interior metal sites are available to the reactants. In addition, MOFs are highly tunable in the structure of linker and nature of metal ion that can participate in their structure [1]. MOFs usually have high specific surface areas and porosity that make them suitable materials for catalytic applications both as supports and as main catalysts. From a catalytic viewpoint, metal-organic frameworks are noteworthy for utilization in the liquid phase, since they have single-site active center property of homogenous catalysts, accompanied with the privileges of easy separation and reusing characteristics of heterogeneous catalysts [2]. Considering that MOFs can be synthesized from transition metals usually used as oxidation catalyst, it is expected that oxidation reactions are amongst the important process categories that have been accelerated by MOFs [3]. Different kinds of oxidation reactions have been promoted by metal-organic frameworks in the liquid phase [4,5]. Accordingly, the preparation of various MOFs and evaluation of their catalytic performances in oxidation reaction demonstrate an attractive research field [6].

Selective synthesis of aldehydes from oxidation of alcohols and controlling its over-oxidation to acids is a significant problem [7]. Many organic and inorganic oxidants have been studied to achieve this target. These materials are not only hazardous but also produce large amount of waste. Also, they are not appropriate for acid sensitive organic substrate. To resolve these problems research has been conducted to the catalytic oxidation reactions. Peroxides and molecular oxygen were reported as oxidants over many homogenous catalytic agents with distinct active sites that provide comparatively high efficiency and selectivity [8,9]. Main drawbacks of homogenous catalysts are their inconvenient recovering, stability, and usage in industrial procedures. In contrast, heterogeneous catalysts can simply resolve these difficulties. Therefore, the sensible designing a very active and selective heterogeneous catalyst from MOFs are currently required and significant [10].

The use of phosphonate-based MOFs as catalyst for oxidation of alcohols are very rare. The only report is our previous work that explained the over oxidation of benzyl alcohols to acid in the presence of STA-12(Co) and TBHP (*tert*-Butyl hydroperoxide) as oxidant [11]. As a continuation of our research on selective method for oxidation of aromatic alcohols to aldehydes, we found that TBHP/$Na_2S_2O_4$ mixture in non-catalytic conditions can oxidize aromatic alcohols to corresponding aldehydes without any acid

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formation [12]. In this paper, a novel heterogeneous catalytic system is investigated for controlled oxidation of aromatic alcohols in the presence of the metal-organic frameworks of the STA-12 family. In this method, a mixture of sodium dithionite/TBHP was used to produce sulfate radical anion ($SO_4^{\bullet-}$) as main oxidizing agent in the oxidation reaction (Scheme 1 To the best of our knowledge, this is first report that catalytic produced sulfate radical utilized for selective synthesis of various aromatic aldehydes from the corresponding alcohols in good yields and short time under mild conditions without any acid formation.

2. Experimental

2.1. Materials

All materials used in this work are used up without any purification. Cobalt (II) nitrate hexahydrate ($Co(NO_3)_2{\cdot}6H_2O$), Ferrous chloride tetrahydrate ($FeCl_2{\cdot}4H_2O$), Manganese (II)acetatetetrahydrate ($Mn(OAc)_2.4H_2O$), Nickel (II) acetate tetrahydrate ($Ni(OAc)_2.4H_2O$), Ethyl acetate (EtOAc), Sodium dithionite ($Na_2S_2O_4$), Sodium persulfate ($Na_2S_2O_8$), Nitrobenzene, Phenol, Piperazine, Formaldehyde, TBHP (70%, v/v) and HCl (37%, v/v), were purchased from Merck Company.

2.2. Synthesis of phosphonic acid ligand (H_4L)

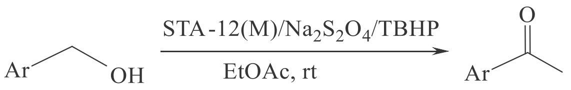
The organic linker [1,4-piperazinediylbis (methylene) phosphonic acid, $H_2O_3P\text{-}CH_2\text{-}NC_4H_8N\text{-}CH_2\text{-}PO_3H_2$], was synthesized based on the previous research [11] and characterized by IR, ^{31}P NMR, ^{1}H NMR,^{13}C NMR and chemical analysis (supporting information, Figs. S1–S4).

2.3. Preparation of catalysts

Metal-organic frameworks of the STA-12(M) family, (M = Co, Fe, Mn, Ni) were prepared based on to the reported procedures (supporting information, Figs. S5–S8) [13,14].

2.4. Instrumentation

Inductively coupled plasma (ICP-AES, PerkinElmer Optima 7300DV) and elemental analyzer (Heraeus CHN-O-RAPID) were used for elemental analyses. A Shimadzu model 800 FT-IR spectrometer and X'Pert PRO diffractometer (Cu-Kα radiation) were utilized for recording of the IR spectra and powder X-ray diffraction (PXRD) data, respectively. A Bruker (Avance DRS)500 spectrometer was used for recording of ^{1}H, ^{13}C, and ^{31}P NMR spectra. Avantes spectrometer (Avaspec-2048-TEC model) was utilized for recording diffuse reflectance UV/Vis spectra. The textural properties and porosity of catalysts were examined with a Belsorp Mini II instrument. The samples were degassed at 100 °C in vacuum for 5 h, the N_2 adsorption/desorption isotherms were recorded at 77 K. The stability of MOFs was assessed by TGA (NETZSCH TG 209 F1 Iris) with heating rate of 10 °C/min from 27 to 900 °C.



Scheme 1. Oxidation of aromatic alcohols with STA-12(M)/TBHP/$Na_2S_2O_4$ catalytic system.

2.5. Typical procedure for alcohol oxidation

27.4 µl TBHP (70%, v/v) (0.5 mmol) and 0.2 mmol $Na_2S_2O_4$ were added to a mixture of EtOAc (1 mL), 0.1 mmol alcohol and 2 mg MOF. The mixture was stirred for specified time at room temperature, which was examined by TLC or GC to generate the corresponding aldehydes. The conversions were determined by GC. The catalyst was easily isolated after adding EtOAc to the final reaction mixture followed by centrifugation.

2.6. Reusability test for catalyst

The recoverability of MOF was evaluated for the oxidation of 4-chlorobenzyl alcohol. After completion of the reaction, EtOAc was poured to the reaction mixture, centrifuged. So the catalyst was isolated and dried at 60 °C. The collected catalyst was reused for the subsequent run with fresh alcohol.

3. Results and discussion

3.1. Catalytic properties of the MOFs

We initially investigated the reaction between 4-chlorobenzyl alcohol as a typical substrate with TBHP/$Na_2S_2O_4$ mixture in the presence of 2 mg of as-prepared catalysts (see characterization data in the supplementary information and [13]) in ethyl acetate at room temperature for 2 h (Table 1, entries 1–4). Fortunately, the STA-12(M)/$Na_2S_2O_4$/TBHP oxidation system showed excellent catalytic activity for oxidation of 4-chlorobenzyl alcohol. The 4-chlorobenzyl alcohol was oxidized with STA-12(Fe) faster than other prepared MOFs (Table 1, entry 2). STA-12(Fe) exhibited low intensity and broad peaks in its PXRD pattern (Fig. S2 of Supplementary Information) that can be ascribed to some structural defects in its structure and a lower crystallinity, which can improve the catalyst activity. Interestingly, replacing the MOF catalyst by free bisphosphonic acid ligand and ferrous chloride tetrahydrate as catalysts, reduced considerably the oxidation efficiency (Table 1, entries 5, 6) revealing that the adsorption of substrates into the micropores of MOF [15] have a vital effect on the reaction efficiency. Given these

Table 1
Catalytic Oxidation of 4-chlorobenzyl alcohol with synthesized MOFs under different conditions.[a]

Entry	Catalyst	Oxidizing additive	Conversion (%)[b]
1	STA-12(Mn)	$Na_2S_2O_4$/TBHP	70
2	STA-12(Fe)	$Na_2S_2O_4$/TBHP	100
3	STA-12(Co)	$Na_2S_2O_4$/TBHP	70
4	STA-12(Ni)	$Na_2S_2O_4$/TBHP	60
4	H_4L	$Na_2S_2O_4$/TBHP	35
5	$FeCl_2,4H_2O$	$Na_2S_2O_4$/TBHP	10
6	-	$Na_2S_2O_4$/TBHP	10
7	STA-12(Fe)	$Na_2S_2O_4/H_2O_2$	20
8	STA-12(Fe)	$Na_2S_2O_4$/Oxone	15
9	STA-12(Fe)	$Na_2S_2O_4$/UHP	10
10	STA-12(Fe)	$Na_2S_2O_4$/Air	nr[c]
11	STA-12(Fe)	$Na_2S_2O_4$/TBHP[d]	83
12	STA-12(Fe)	$Na_2S_2O_4$	nr
13	STA-12(Fe)	TBHP	trace
14	STA-12(Fe)	$Na_2S_2O_3$/TBHP	nr
15	STA-12(Fe)	Na_2SO_4/TBHP	nr
16	STA-12(Fe)	$Na_2S_2O_8$/TBHP	trace

[a] Oxidation conditions: 4-chlorobenzyl alcohol (0.1 mmol), solvent (EtOAc, 0.5 mL), alcohol:$Na_2S_2O_4$:oxidant ratio (1:2:4), catalyst (2 mg), room temperature, time (2 h).

[b] Confirmed with GC and chlorobenzene as standard.

[c] No reaction.

[d] Reaction with anhydrous TBHP. The selectivity of product was always 100%.

promising results, screening of the reaction conditions for oxidation of 4-chlorobenzyl alcohol as a typical substrate with $Na_2S_2O_4$/TBHP over STA-12(Fe) as catalyst, were intended with a number of experiments. The results are presented in Table 1 and Fig. S9. Systematic investigation on solvent nature and amount showed well 1 ml ethyl acetate as an environmentally friendly solvent is the best media for this oxidation system (Figs. S9a and b). Further control tests were performed to assess the influence of other common oxidants over STA-12(Fe) catalyst (Table 1, entries 7–10) and the yields were diminished effectively compared with TBHP. The results showed that the molar ratio of 1:2:5 of alcohol/$Na_2S_2O_4$/TBHP is the best for the reaction progression and a further increase in TBHP did not did not improve the reaction yield (Fig. S9c). The blank experiments confirmed that the presence of both TBHP and $Na_2S_2O_4$ were crucial for the effective oxidation of alcohol Table 1, entries 12, 13 and the use of anhydrous TBHP did not change the oxidation performance (Table 1, entry 11). Also, the results show that reducing the amount of MOF to 6.5 mol% diminished the conversion of substrate, while enhancing the catalyst loading to 10 mol% did not improve the reaction yield (Fig. S9d).

3.2. *Heterogeneity, recyclability and stability of MOF*

To confirm the heterogeneity of the catalytic oxidation, the STA-12(Fe) was separated from reaction media when the conversion reached to 40% after 30 min (Fig. 1). After removal of catalyst, the substrate conversion increased eventually to 70% after 12 h, in consistent with the result obtained under catalyst-free condition [12]. Moreover, analysis of the filtrate with ICP-AES showed no Fe ions. Therefore, it can be say that the reaction in filtrate has proceeded in the catalyst-free state and under effect of stoichiometric $Na_2S_2O_4$/TBHP oxidant system. The selectivity was also 100% at all times, and only the aldehyde product was obtained (Fig. 1).

In order to evaluate the catalyst recyclability, fourteen runs of the oxidation of 4-Chlorobenzyl alcohol were accomplished using the 8.6 mol% of catalyst. After each run, the MOF was removed by centrifugation, washed with Ethyl acetate, dried at 60 °C and used in subsequent reaction. No decrease in the substrate conversion was observed through fourteen runs, indicating the high recoverability and the remarkable stability of metal-organic frameworks derived from phosphonate ligands (Fig. S10). More confirmation for this claim was attained by characterization of separated catalyst. Verified by PXRD and IR, STA-12(Fe) presented high crystallinity and stable structure (Fig. S11).

3.3. *Assessment of the catalytic system in the oxidation of various aromatic alcohols*

In order to evaluate the performance of the catalytic system, the oxidation of diverse aromatic alcohols was investigated at the optimized conditions (Table 2). The primary and secondary benzylic alcohols with electron-donating substitutes were converted to correspondent aldehyde in excellent yields at desired reaction times (Table 2, entries 1, 2, 3, 5–7). Nevertheless, electron-poor molecules exhibited less reactivity and the reaction yields were diminished significantly (Table 2, entries 4, 8, 14). Moreover, hindered alcohols were inferior in this catalytic system (Table 2, entries 9, 10), featuring the significant steric effect around the catalyst site, an evidence for occurring the reactions inside the catalytically active cavity of porous STA-12(Fe). It should be noted that, heterocyclic aromatic alcohols such as furfuryl alcohol, pyridin-2-ylmethanol are actually inactive substrates for this catalytic system (Table 2, entries 11, 12). A competitive reaction between alcohol and alkene led to selective oxidation of alcohol highlighting the chemoselectivity of title catalytic system. (Table 2, entry 14). Performing a control reaction using 20 mmol alcohol was very effective featuring the methodology is amenable to scalability. (Table 2, entry 3).

3.4. *Study of radical species in catalytic system*

To elucidate the active species involving in the catalytic oxidation reaction several control experiments were carried out. No change in the oxidation efficiency of $Na_2S_2O_4$/TBHP was observed under Ar atmosphere, providing an evidence for critical role of TBHP as main oxidizing agent in the catalytic oxidation system. On the other hand, significant decreases in product yields were detected when radical trapping agents such as butylated hydroxytoluene (BHT) and 2,2,6,6-tetramethylpiperidin-1-yloxyl (TEMPO) were added to reaction mixture in excess, confirming that active radicals are responsible for the oxidation process (Fig. 2).

MOF catalyzed activation of $Na_2S_2O_4$/TBHP can generates two main kinds of radicals, namely sulfate ($SO_4^{\bullet -}$) and hydroxyl ($^{\bullet}OH$) radicals [16]. To identify which species is prevailing in STA-12(Fe)/$Na_2S_2O_4$/TBHP system, selective radical trapping studies were conducted. phenol and nitrobenzene (NB) were selected as trapping agents since phenol is a well-known scavenger for both sulfate and hydroxyl radicals but nitrobenzene predominantly reacts with hydroxyl radical [17]. Addition of 600 mM phenol to the reaction

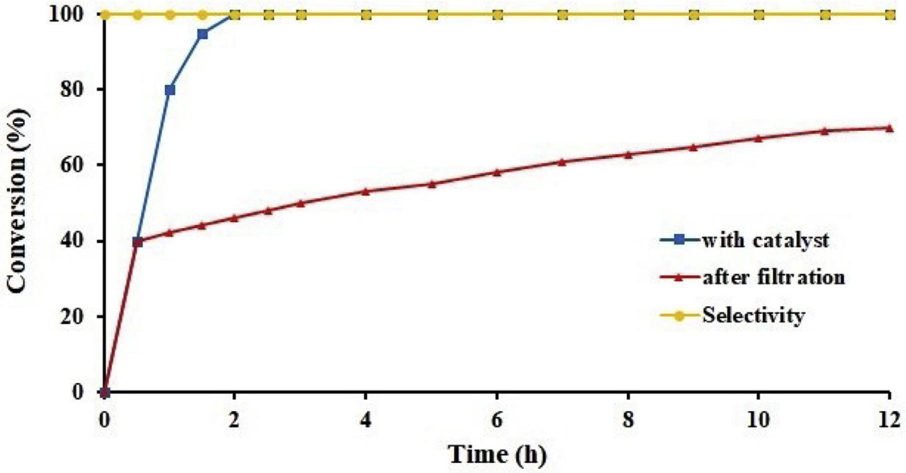
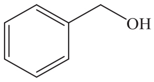
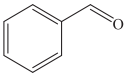
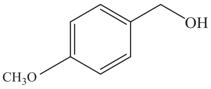
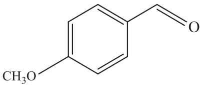
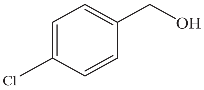
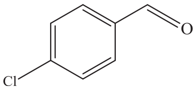
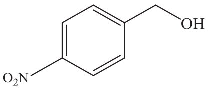
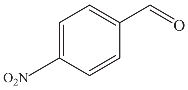
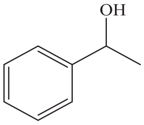
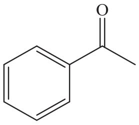
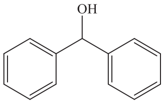
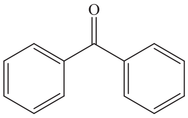
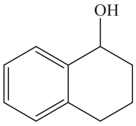
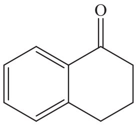
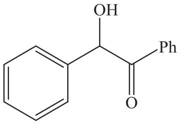
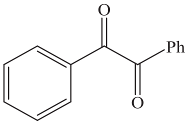
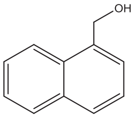
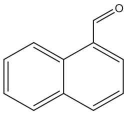
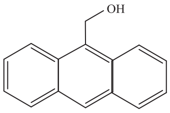
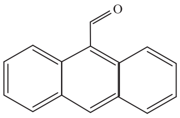
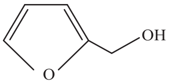
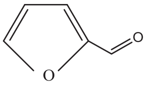
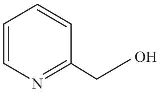
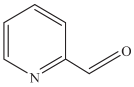
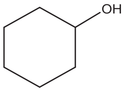
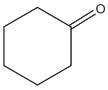
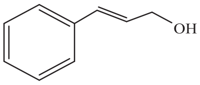
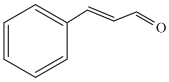


Fig. 1. Time conversion plot and leaching test as evidence for heterogeneity in the oxidation of 4-chlorobenzyl alcohol catalyzed by STA-12(Fe) under optimal reaction conditions.

Table 2
Oxidation of aromatic alcohols by STA-12(Fe)/$Na_2S_2O_4$/TBHP catalytic system.[a]

Entry	Substrate	Product	conversion (%)[b]	TOF[c]
1			95 (80)[d]	9.26
2			98 (80)	9.26
3			100 (80) (95)[e] (100)[f]	9.26
4			30 (30)	3.47
5			100 (100)	11.57
6			90 (80)	9.26
7			75 (60)	6.94
8			10 (10)	1.16
9			40 (30)	3.47
10			5 (5)	0.58
11			Trace	-
12			Trace	-
13			20 (20)	2.31
14			40 (10)	1.16

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[a] Oxidation conditions: alcohol (0.1 mmol), $Na_2S_2O_4$ (0.2 mmol), TBHP (0.5 mmol), STA-12(Fe) (4 mg, 8.6 mol%), EtOAc (1 mL), time (2 h), 25 °C.
[b] Confirmed with GC based on internal standard.
[c] TOF calculated as mol (product)/[mol (catalyst) × (1 h)].
[d] Yield after 1 h
[e] Yield for 20 mmol alcohol.
[f] Reaction under Ar atmosphere.

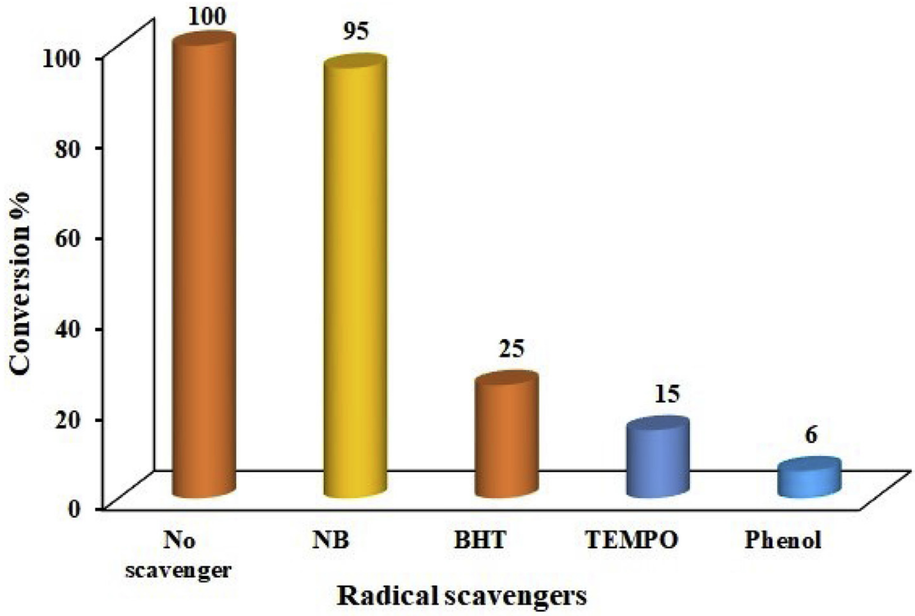

Fig. 2. Oxidation of 4-chlorobenzyl alcohol with STA-12(Fe)/$Na_2S_2O_4$/TBHP catalytic system in the presence of radical scavenger agents (scavenger amount: 600 mM).

mixture of 4-chlorobenzyl alcohol, reduced significantly the reaction rate and 6% of pertinent aldehyde was obtained. Nevertheless, NB was actually ineffective and slight reduce in oxidation efficiency was observed compared to phenol showing the involvement of sulfate anion radicals as dominant oxidizing species (Fig. 2). For oxidation of many organic compounds, $SO_4^{\bullet-}$ is more appropriate than hydroxyl radical, since it is more selective through electron transfer reaction and hydrogen abstraction and have longer lifetime (due to its stability) compared to $^{\bullet}OH$ [18].

When sodium dithionate ($Na_2S_2O_4$) was replaced by sodium thiosulfate ($Na_2S_2O_3$) and sodium sulfate (Na_2SO_4) no oxidation product was observed (Table 1, entries 14, 15). Also, the desired aldehyde was not obtained by using $Na_2S_2O_8$/TBHP mixture as oxidant (Table 1, entry 16) and extensive over-oxidation of alcohol to the corresponding acid was observed. These results confirm that sulfate radical ($SO_4^{\bullet-}$) cannot be produced from the above salts under these conditions.

Giving this evidences, we presented the plausible mechanism for the oxidation reaction with $Na_2S_2O_4$/TBHP mixture over STA-12(Fe) catalyst (Eqs. (1)–(3)). Initially, sulfate radicals are generated from reaction of dithionite ion and TBHP. Sulfate radical ($SO_4^{\bullet-}$) readily reacts with alcohol in two step and the desired aldehyde is produced [19].

$$4\ {}^{t}\text{Bu-O-OH} + S_2O_4^{2-} \rightarrow 4\ {}^{t}\text{Bu-OH} + 2SO_4^{\bullet-} \quad (1)$$

$$SO_4^{\bullet-} + \text{R—CH}_2\text{-OH} \rightarrow HSO_4^{-} + \text{R—CH}_2\text{-O}^{\bullet} \quad (2)$$

$$SO_4^{\bullet-} + \text{R—CH}_2\text{-O}^{\bullet} \rightarrow HSO_4^{-} + \text{R—CH=O} \quad (3)$$

To show the merit of this catalytic system, the results attained for oxidation of 4-methoxybenzyl alcohol were compared with the reported data for other MOFs [20–25] (Table 3). Examination of the data shows the supremacy of presented method for oxidation of aromatic alcohols in terms of the excellent selectivity, high yields of the products, short reaction time, catalyst loading, and mild reaction conditions. Also, STA-12 and MOFs having phosphonate ligand can be introduced as stable and efficient catalysts for the oxidation of benzylic alcohols.

4. Conclusions

In summary, a series of phosphonate-based metal-organic frameworks known as STA-12(M), has been utilized as the highly efficient and selective heterogeneous catalysts for oxidation of a variety of aromatic alcohols with $Na_2S_2O_4$/TBHP mixture. Aldehydes produced selectively in high yields under mild conditions within a short time. The catalyst has high recoverability and notable stability in successive runs without change in the process conversion and selectivity. The outstanding aspects of our methodology are the excellent selectivity and durability of the catalyst. In addition, the catalytic procedure employs a small amount of catalyst for activate a relatively cheap and commercially available oxidant mixture ($Na_2S_2O_4$/TBHP) in ethyl acetate as a green solvent. These advantageous possess high generality and environmentally benign

Table 3
The evaluation of catalytic performance of STA-12(Fe) with recently reported MOFs for oxidation of 4-methoxybenzyl alcohol.

Entry	Oxidation System	Catalyst amount	Conditions	Time (h)	Conv. (%)	Sel.(%)	Ref.
1	AuNi/MIL-101-1	20 mg	THF/O_2/80 °C	4	44.9	99.1	[20]
2	Hf-MOF-808-V/triethylamine	66 mg	Toluene/O_2/105 °C	8	75	˃99	[21]
3	V-Zr-NU-1200/triethylamine	47 mg	Toluene/1,2-dichlorobenzne/O_2/105 °C	11	100	˃99	[22]
4	CuNi/MIL-101	40 mg	THF/O_2/100 °C	4	87.9	23.8	[23]
5	Cu-MOF/TEMPO	113 mg	MeCN/Na_2CO_3/75 °C	16	100	˃99	[24]
6	$Cu_3(BTC)_2$/TEMPO	15 mg	MeCN/NMI/O_2/70 °C	9	99	˃99	[25]
7	STA-12(Co)	2 mg	EtOAc/TBHP/60 °C	1.5	65	˃99	[11]
8	STA-12(Fe)	4 mg	EtOAc/TBHP/$Na_2S_2O_4$/25 °C	1	100	˃99	This work

which makes our methodology proper for applied goals.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.jorganchem.2019.120995.

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