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Synthesis, characterizations and catalytic studies of a new two-dimensional metal–organic framework based on Co–carboxylate secondary building units

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ABSTRACT

A metal–organic framework $[Co_3(BDC)_3(DMF)_2(H_2O)_2]$ was synthesized and structurally characterized. X-ray single crystal analysis revealed that the framework contains a 2D polymeric chain through coordination of 1,4-benzenedicarboxylic acid linker ligand to cobalt centers. The polymer crystallize in monoclinic $P2_1/n$ space group with $a=13.989(3)$ Å, $b=9.6728(17)$ Å, $c=16.707(3)$ Å, and $Z=2$. The polymer features a framework based on the perfect octahedral Co–O6 secondary building units. The catalytic activities of $[Co_3(BDC)_3(DMF)_2(H_2O)_2]_n$ for olefins oxidation was conducted. The heterogeneous catalyst could be facilely separated from the reaction mixture, and reused three times without significant degradation in catalytic activity. Furthermore, no contribution from homogeneous catalysis of active species leaching into reaction solution was detected.

1. Introduction

Metal–organic frameworks (MOFs) have received a great deal of interest and represented an active area of coordination chemistry not only by their multiple potential applications in gas storage, separation, magnet, optoelectronics and catalysis, but also by their enormous variety of intriguing structural topologies [1–5]. The most powerful strategy for the architecture of diverse structural topologies in MOFs as coordination polymers is the selection of appropriate multidentate ligand as a linker in connecting metal ions to achieve one-, two-, or three-dimensional crystalline network structures [6–9]. Thus, there has been considerable amount of research on multicarboxylate ligands to design metal-containing polymers [10–13].

Among transition metal in MOFs, there has been a great attention in coordination chemistry of cobalt complexes with carboxylate ligands, due to the fact that polynuclear cobalt carboxylates are suitable candidates for structural assembly of new coordination solids with interesting network architectures [14–16]. As multicarboxylate ligand, 1,4-benzenedicarboxylic acid (H_2BDC) with its derivetives and 1,3,5-benzenetriacetic acid have been used in the synthetic systems because they can exhibit a short bridge via one carboxylic group or a long one via the benzene ring which leads to varieties of multi-dimensional MOFs with different kinds of topologies [17–19].

To enrich this field of research, we used H_2BDC as the rigid multicarboxylate ligand to synthesize new cobalt MOF. The rigid aromatic unit in H_2BDC ligand as spacer seemed to be promising to control and adjust stable framework. Here, we report hydrothermal synthesis, characterizations and catalytic studies of MOF of $[Co_3(BDC)_3(DMF)_2(H_2O)_2]$ formed between Co-centers and H_2BDC ligands. Particular attention is focused on the catalytic efficiency of the MOF in olefin oxidation reaction.

2. Experimental section

2.1. Materials and instruments

All reagents for synthesis and analysis were obtained commercially with analytical grade and used without further purification. The elemental analysis (CHN) of compound was obtained from a Carlo ERBA Model EA 1108 analyzer. Fourier transform infrared (FT-IR) spectrum was obtained by utilizing a Unicam Matson 1000 FT-IR spectrophotometer using KBr disks at room temperature. Thermogravimetric analysis (TGA) was performed on a Perkin Elmer TGA7 analyzer in N_2 atmosphere with a heating rate of 5 °C min^{-1}. Powder X-ray diffraction (XRD) patterns were recorded by a Rigaku D-max C III, X-ray diffractometer using Ni-filtered Cu$K\alpha$ radiation. Inductively coupled plasma (ICP) was performed by ICP–MS HP 4500. The products of olefins oxidation reaction were determined and

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analyzed by a HP Agilent 6890 gas chromatograph equipped with a HP-5 capillary column (phenylmethylsiloxane 30 m × 320 μm × 0.25 μm) and a flame-ionization detector.

2.2. Synthesis of $[Co_3(BDC)_3(DMF)_2(H_2O)_2]_n$

A DMF solution (5 mL) of $CoCl_2 \cdot 6H_2O$ (291 mg, 1 mmol) was added to a solution of 1,4-benzenedicarboxylic acid (H_2BDC) (166 mg, 1 mmol) in 5 mL DMF in the small sample vessel. The solution was heated for 48 h at 80 °C without any stirring or shaking and then the temperature was gradually decreased to 60 °C (2 °C h^{-1}). After 2 days that solution was kept in 60 °C, purple crystals of $[Co_3(BDC)_3(DMF)_2(H_2O)_2]_n$ (85% yield based on H_2BDC) were obtained. The crystals were washed with DMF (3 mL, two times) and dried in air to yield pure product. Anal. Cal: for $C_{30}H_{24}Co_3N_2O_{16}$: C, 42.42; H, 3.32; N, 3.30. Found: C, 42.63; H, 2.86; N, 3.28. FT-IR (KBr 4000–400 cm^{-1}): 3491 (br), 2931 (w), 1663 (s), 1619 (s), 1559 (s), 1418 (s), 1254 (w), 1115 (m), 855 (m), 813 (m), 760 (s), 693 (s), 655 (m), 468 (m).

2.3. X-ray crystallographic studies

Single crystal of the complex was mounted in a random orientation on a glass fiber. Data collection was carried out at 296 K on an Oxford Diffraction X calibur four-circle kappa geometry single-crystal diffractometer with a Sapphire 3 CCD detector, using a graphite monochromated MoKα (λ=0.71073 Å) radiation, and applying the CrysAlis Software system. The crystal–detector distance was 50 mm. Data reduction, including absorption correction, was done by the CrysAlice RED program. The structure was solved by direct methods implemented in the SHELXS-97 program [20]. The coordinates and the anisotropic displacement parameters for all non-hydrogen atoms were refined by full-matrix least-squares methods based on F^2 values using the SHELXL-97 program. All hydrogen atoms were placed in geometrically idealized positions and constrained to ride on their parent C atoms with C–H=0.93 and 0.96 Å for CH and CH_3, respectively, and with U_{iso} (H)=$k \times U_{eq}$ (C), where k=1.2 for CH H atoms and k=1.5 for CH_3 H atoms [21]. Graphical work was performed via OLEX2 [22]. The termal ellipsoids were drawn at the 50% probability level.

2.4. General process for catalytic oxidation reaction

For the heterogeneous olefins oxidation, the reactions was done in a two-necked round-bottom flask fitted with a condenser and placed in a temperature controlled oil bath. Typically, 1 mmol of the substrate was taken in 2 mL solvent, followed by the addition of 3 mg catalyst (0.01 mmol to Co center) and then, the mixture was heated at 75 °C under stirring. The reaction began with the addition of *tert*-butyl hydroperoxide (TBHP) (equimolar with respect to substrate). The products from the reaction mixture were analyzed by Gas Chromatography in the presence of chlorobenzene as an internal standard and were identified through being compared with known standards.

2.5. General procedure for recyclability of $[Co_3(BDC)_3(DMF)_2(H_2O)_2]_n$ catalysts

The recyclability of the $[Co_3(BDC)_3(DMF)_2(H_2O)_2]_n$ catalyst was investigated in oxidation reaction of styrene in the presence of TBHP in 1,2-dichloroethan at 75 °C for 7 h. Then, after the first catalytic reaction, the solid catalyst was easily isolated by centrifuge and recovered by being washed with solvent and dried at 70 °C. Afterwards, catalyst was used for the next run under the same reaction condition as the first run.

3. Result and discussion

3.1. Crystal structure description

Single-crystal X-ray structure analysis revealed that $[Co_3(BDC)_3(DMF)_2(H_2O)_2]_n$ crystallizes in the monoclinic $P2_1/n$ space group (Fig. 1). The MOF structure is composed of Co–O_6 octahedral linked by 1,4-benzenedicarboxylate linkers (Fig. 2). The structure of $[Co_3(BDC)_3(DMF)_2(H_2O)_2]$ coordination polymer is a 2D-periodic framework made of discrete secondary building units (SBUs) each of which is constructed from $Co^{II}O_6$ corner shared octahedral (Fig. 3). The crystal data is shown in Table 1. The Co–carboxylate 2D coordination polymer of $[Co_3(BDC)_3(DMF)_2(H_2O)_2]$ is formed by two-dimensional infinite straightforward chains through covalent interactions (Fig. 4). The MOF structure is composed of Co–O–C units in which the carboxylate links are coordinated to the metal centers in at least one of the following modes as shown in Fig. 5 that helps to form a 2D-polymeric chain.

There are two distinct symmetrically independent Co centers in the crystal structure which are further mutually linked by benzene units from 1,4-benzendicarboxylate links furnishing 2D framework in the (1 0 1) plane. The Co(II) ions located in general positions, Co1 and Co2, are surrounded by six oxygens in the octahedral

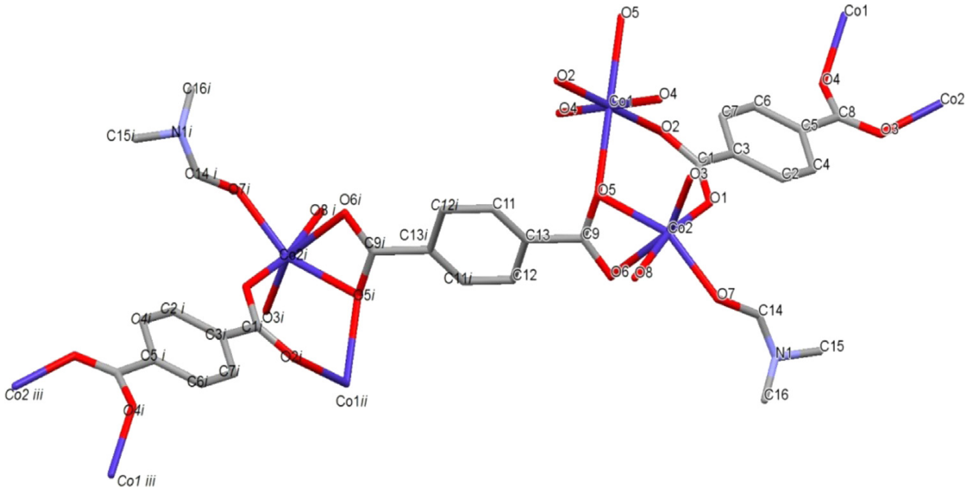

Fig. 1. Coordination environment of Co(II) in $[Co_3(BDC)_3(DMF)_2(H_2O)_2]_n$. The hydrogen atoms are omitted for clarity. (*i*) 1−*x*, −*y*, −*z*; (*ii*) *x*, −1+*y*, *z*; (iii) 1/2−*x*, −1.5+*y*, −1/2−*z*.

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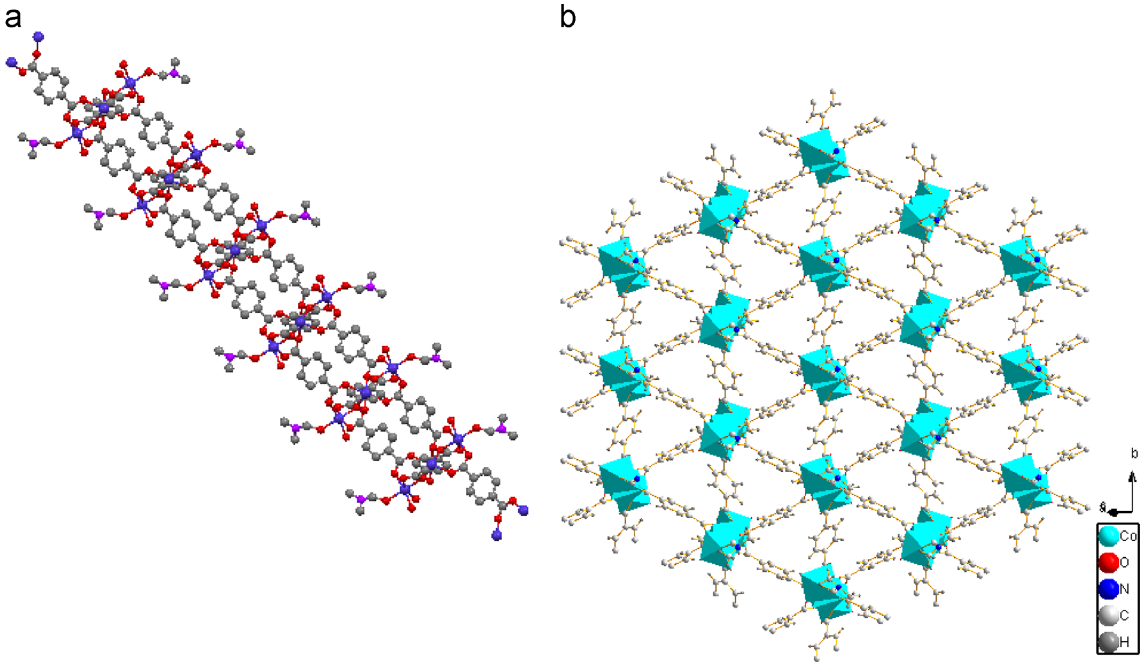

Fig. 2. Representation of the SBUs along the [1 0 1] direction (a); crystalline framework in the (1 0 1) plane with Co shown as polyhedral and SBUs linked together via benzene rings of 1,4-benzenedicarboxylate ligand (b).

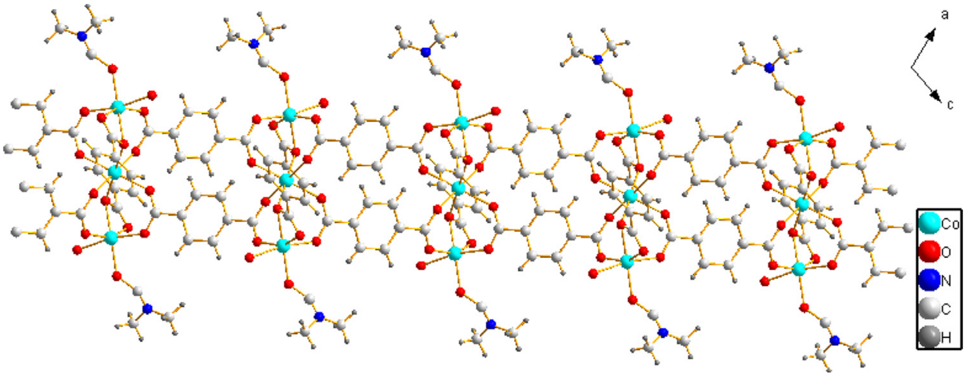

Fig. 3. View of the corner sharing octahedral $[Co_3(BDC)_3(DMF)_2(H_2O)_2]_n$ along *b* axis.

Table 1
Crystal data and details of the structure determination for $[Co_3(BDC)_3(DMF)_2(H_2O)_2]_n$.

Coordination polymer	$\mathbf{[Co_3(BDC)_3(DMF)_2(H_2O)_2]}$
Empirical formula	$C_{30}H_{24}Co_3N_2O_{16}$
Formula weight	845
Color and habit	Purple, prism
Crystal system, space group	Monoclinic, $P2_1/n$
Unit cell parameters:	
a (Å)	13.989(3)
b (Å)	9.6728(17)
c (Å)	16.707(3)
α (°)	90
β (°)	108.66(2)
γ (°)	90
V (Å^3)	2141.84
Z	2
D_{calc} (g cm^{-3})	1.31
$F(0\,0\,0)$	974
R^a, wR^b $[I \geq 2\sigma(I)]$	0.0650, 0.0416
Goodness of fit on F^2, S^d	1.021

$R = \sum||F_o| - |F_c||/\sum|F_o| wR = [\sum(F_o^2 - F_c^2)^2/\sum w(F_o^2)^2]^{1/2}$, $w = 1/[\sigma^2(F_o^2) + [(g_1 P)^2 + g_2 P]]$ where $P = (F_o^2 + 2F_c^2)/3$, $S = \sum[w(F_o^2 - F_c^2)^2/(N_{obs} - N_{param})]^{1/2}$.

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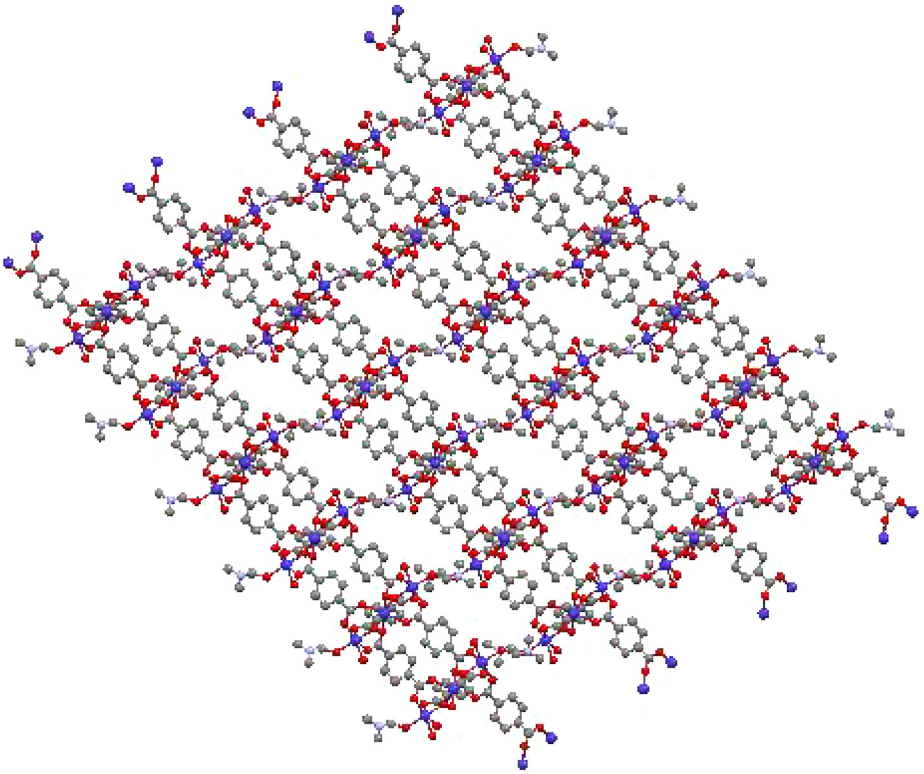
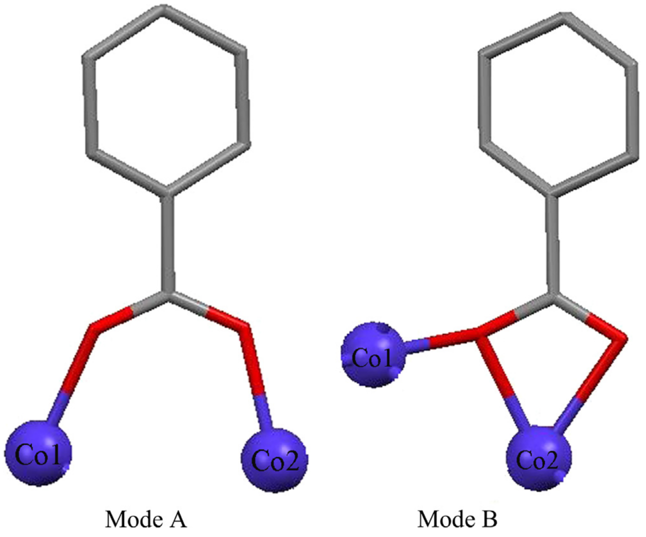


Fig. 4. A view infinite straightforward tow-dimensional chain in $[Co_3(BDC)_3(DMF)_2(H_2O)_2]_n$.



Mode A Mode B

Fig. 5. Different coordination mode of 1,4-benzendicarboxylate linker observed in $[Co_3(BDC)_3(DMF)_2(H_2O)_2]_n$.

Table 2
Selected bond lengths (Å) and angels (°) for $[Co_3(BDC)_3(DMF)_2(H_2O)_2]_n$.

Bond length (Å)		**Bond angles (°)**			
Co1–O2	2.10 (1)	O2–Co1–O2	180.0 (6)	O1–Co2–O5	102.7 (7)
Co1–O5	2.17 (2)	O2–Co1–O4	84.1 (6)	O1–Co2–O6	95.4 (6)
Co1–O4	2.04 (1)	O2–Co1–O4	95.9 (6)	O1–Co2–O7	89.8 (8)
		O2–Co1–O5	88.8 (6)	O1–Co2–O8	169.6 (7)
Co2–O1	2.05 (1)	O2–Co1–O5	91.2 (6)	O1–Co2–O3	101.0 (6)
Co2–O5	2.13 (2)	O5–Co1–O4	92.0 (6)	O5–Co2–O6	59.1 (6)
Co2–O6	2.24 (1)	O5–Co1–O4	88.0 (6)	O5–Co2–O7	156.0 (8)
Co2–O7	2.03 (3)	O5–Co1–O5	180.0 (7)	O5–Co2–O8	87.1 (7)
Co2–O8	2.18 (2)	O4–Co1–O4	180.0 (6)	O5–Co2–O6	95.6 (7)
Co2–O3	2.02 (1)	O4–Co1–O5	88.0 (6)	O6–Co2–O7	99.8 (7)
		O4–Co1–O5	92.0 (6)	O6–Co2–O8	86.5 (7)
				O6–Co2–O3	152.5 (6)
				O7–Co2–O8	79.8 (8)

(*i*) $-x-1, 1-y, -z-1$; (*ii*) $-x, -y, -z$.

position. Co1 is surrounded by six oxygens originating from three different 1,4-benzendicarboxylate (O2, O4 and O5) linkers and Co2 is surrounded by one DMF oxygen in the equatorial site (O7) and five oxygens originating from 1,4-benzendicarboxylate (O1, O3, O5, O6 and O8) completing the octahedral coordination sphere.

Selected bond distance and bond angles are listed in Table 2. The Co2–O bond distances (coordinated carboxylate oxygens) and Co2–O7 bond distance (coordinated DMF molecule) are 2.02–2.24 Å and 2.03 Å respectively. The bond angles for O8–Co2–O1 in axial position, O6–Co2–O5 and O5–Co2–O3 in equatorial position are 169.6°, 59.1° and 95.6° respectively. So, coordination spherical around Co2 is a disturbed octahedral. Co1–O bond distances in axial and equatorial position are 2.17 and 2.10 Å respectively and bond angles for O5–Co1–O5 and O5–Co1–O4 are 180.0° and 92.9° respectively. Consequently, Co1–O_6 has slightly distorted octahedral coordination sphere.

3.2. *Thermal investigation of $[Co_3(BDC)_3(DMF)_2(H_2O)_2]_n$*

The TGA curve shows that the $[Co_3(BDC)_3(DMF)_2(H_2O)_2]_n$ exhibits the first weight loss of 26.06% in 197 °C corresponding to the loss of DMF and H_2O molecules in the cluster (Fig. 6). The $[Co_3(BDC)_3(DMF)_2(H_2O)_2]_n$ shows final weight loss in a temperature range from 320 °C to 340 °C due to the polymer final collapse that occurred at higher temperature.

3.3. *Oxidation of olefins catalyzed by $[Co_3(BDC)_3(DMF)_2(H_2O)_2]_n$*

The catalytic potential of $[Co_3(BDC)_3(DMF)_2(H_2O)_2]_n$ was examined for the olefins oxidation. Prior to carrying the detailed study on the catalytic oxidation of various olefins, we first optimized the

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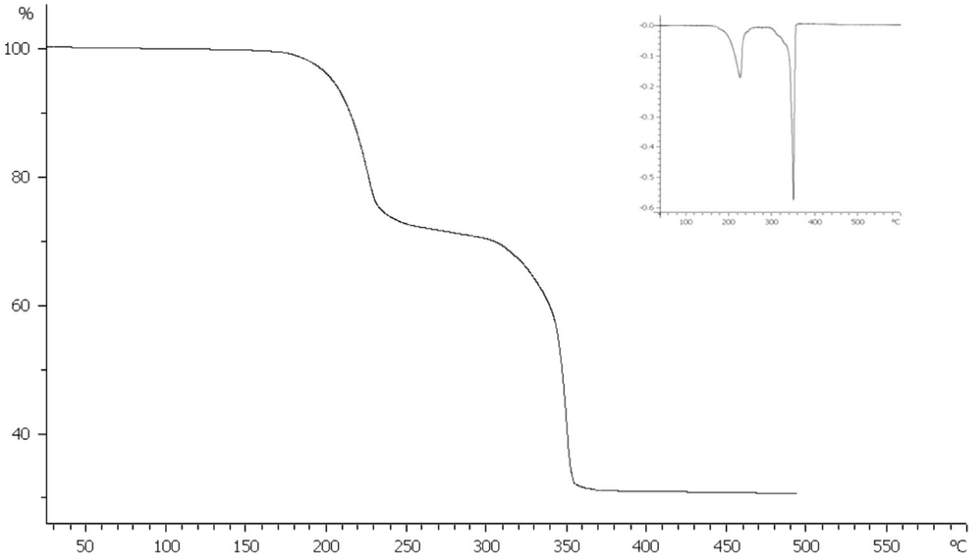

Fig. 6. TGA and DSC curves of $[Co_3(BDC)_3(DMF)_2(H_2O)_2]$ MOF.

reaction conditions by choosing styrene as a model substrate. The styrene oxidation reaction catalyzed by $[Co_3(BDC)_3(DMF)_2(H_2O)_2]_n$ was carried out in the presence of TBHP (70% in water) as oxidant. The efficiency of the catalysts in different solvents decreases in the order: 1,2-dichloroethane > acetonitrile > *N*,*N*-dimethylformamide > methanol (conversion 96, 90, 84, 10%, respectively). The result indicates the significant role of a suitable solvent for the oxidation reactions.

Aromatic and aliphatic alkenes react with TBHP in the presence of $[Co_3(BDC)_3(DMF)_2(H_2O)_2]_n$ catalyst to produce the corresponding epoxides and/or oxides in good yield, with moderate selectivity (Table 3).

Oxidation of styrene by TBHP/$[Co_3(BDC)_3(DMF)_2(H_2O)_2]_n$ in 1,2-dichloroethane involves C=C bond breaking, yielding styrene oxide (45%) and benzaldehyde (55%) within 7 h at 75 °C. Epoxidation of *trans*-stilbene provided solely *trans*-epoxide, while epoxidation of *cis*-stilbene produces *trans*-stilbene oxide (86%) and minor amounts of *cis*-stilbene oxide (13%) under similar conditions. Cyclohexene oxidation as substrate that is particularly prone to allylic oxidation, provided cyclohexene-1-one (95%) by 90% conversion. These oxidation products strongly suggest that oxidation reaction is taken place throughout the radical pathway, as it was expected on the basis of earlier reports [2,23]. This was verified by the use of ionol (2,6-di-tertbuthyl-*p*-crosol) as radical scavenger. Oxidation was completely inhibited in the presence of ionol (conversion 5% for styrene oxidation and 2% for cyclohexene oxidation). The impressive inhibition of oxidation by ionol confirmed the formation of the radical intermediate in the reaction pathway. The mechanism has been proposed for oxidation of olefins by cobalt/*t*-BuOOH oxidation system. Oxidation of olefins is assumed by this mechanism to occur Co–O–O–*t*-Bu intermediates, which are produced by the homolytic cleavage of the O–O bond of *t*-BuOOH and which then react with the olefin.

The amount of leached-out cobalt in the reaction solution after catalysis was also determined by ICP analysis. Since cobalt, in the reaction solution, could catalyze the olefin epoxidation and the amount of cobalt was found to be less than 2%, this suggests that there is almost no cobalt in the liquid phase from this catalyst and the nature of catalytic reaction is heterogeneous.

3.4. Reusability investigation of heterogeneous catalysts

A further set of experiment was carried out to check the reusability of the catalysts for the styrene oxidation. The catalyst was separated after each run by filtration, washed 2–3 times by using 1,2-dichloroethan and dried at 70 °C. The $[Co_3(BDC)_3(DMF)_2(H_2O)_2]_n$ catalyst could be recycled three times with no significant loss in activity (Fig. 7). During reusability studies third run provided 85% conversion and selectivity in favor of benzaldehyde. The crystalline phase stability and purity of the catalyst was confirmed by powder XRD analysis after catalytic run (Fig. 8). The results showed that the catalysts have nearly identical diffraction peaks, indicating that the basic lattice structures of heterogeneous catalyst was well maintained after catalysis.

Table 3
Oxidation of olefins catalyzed by $[Co_3(BDC)_3(DMF)_2(H_2O)_2]_n$/TBHP[a].

Substrate	% Conversion[b]	% Selectivity (to Epoxide)
1-Octene	75	100
Cyclooctene	67	100
Cyclohexene	90	95[c]
Styrene	96 (3)[d]	45
a-Methylstyrene	85	75
cis-Stylbene	30	100 (86)[e]
trans-Stylbene	62	100 (1 0 0)[e]
Indene	75	100

[a] Reaction condition: The reactions were run in 1,2-dichloroethane (2 mL) at 75 °C for 7 h by amount of 3 mg catalyst, 1 mmol chlorobenzene, 1 mmol substrate and 1 mmol TBHP. The molar ratio for catalyst: substrate: TBHP is 1: 100: 100.

[b] Conversion determined by GC using chlorobenzene as internal standard.

[c] Selectivity (%) to cyclohexene-1-one.

[d] The blank test without catalyst.

[e] Selectivity (%) to *trans*-product.

4. Conclusion

A 2D MOF of $[Co_3(BDC)_3(DMF)_2(H_2O)_2]_n$ was synthesized by a hydrothermal method. Single crystal structure analysis of the compound revealed that the MOF crystallizes in the monoclinic $P2_1/n$ space group with infinite network composed of Co–O–C units. The TGA study emphasized the stability of this MOF. The catalytic oxidation of various olefins with TBHP is effectively carried out with the $[Co_3(BDC)_3(DMF)_2(H_2O)_2]_n$. The results of catalytic study exhibited that the heterogeneous complex of $[Co_3(BDC)_3(DMF)_2(H_2O)_2]_n$ is an active and selective catalyst for olefins oxidation with the TBHP oxidizing agent in 1,2-dichloroethane solvent. The MOF catalyst was also found

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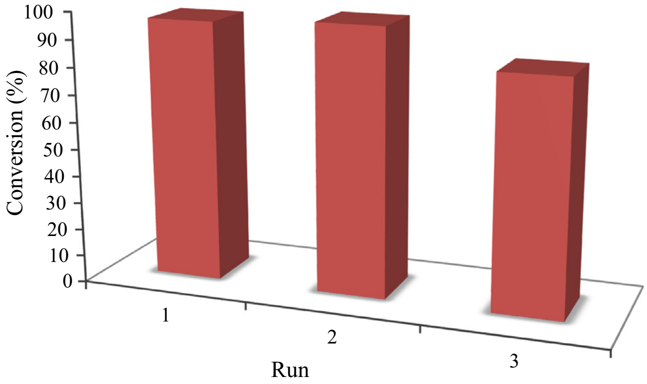

Fig. 7. Reusability studies $[Co_3(BDC)_3(DMF)_2(H_2O)_2]_n$ catalyst in oxidation of styrene by TBHP.

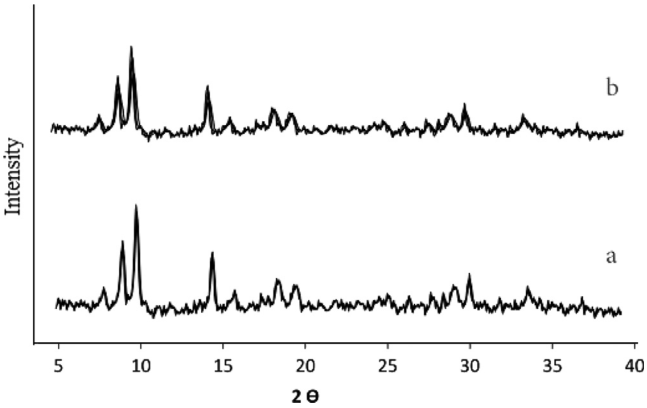

Fig. 8. Powder diffraction patterns of the MOF before and after the catalytic reactions. (a) $[Co_3(BDC)_3(DMF)_2(H_2O)_2]_n$ fresh catalyst, (b) $[Co_3(BDC)_3(DMF)_2(H_2O)_2]_n$ after three catalytic runs.

to be reusable for the oxidation of styrene with a conversion ranging from 96% to 85% at third run and the framework of the complex has not undergone important structural change during catalysis.

Appendix A. Supplementary data

CCDC 884088 contains the supplementary crystallographic data for this paper. This data can be obtained free of charge via www.ccdc.cam.ac.uk/data_request/cif or Cambridge Crystallographic Data Centre (CCDC), 12 Union Road, Cambridge CB2 1EZ, UK; Fax: +44 1223 336033; email: data_request@ccdc.cam.ac.uk.

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Appendix A. Supporting information

Supplementary data associated with this article can be found in the online version at http://dx.doi.org/10.1016/j.jssc.2014.05.011.

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