Lanthanide Carboxylate Frameworks: Efficient Heterogeneous Catalytic System for Epoxidation of Olefins

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Abstract Two lanthanide-based three dimensional metal–organic frameworks (MOF) viz. $[Nd(HCOO)_3]_n$ (1) and $[Pr(HCOO)_3]_n$ (2) have been synthesized and characterized. Both the compounds have similar structure. In this study we have demonstrated that the compounds are highly efficient in catalyzing epoxidation of various cyclic and linear olefinic substrates. MOF compounds are stable and recyclable under the reaction conditions. Notably, MOF systems are remarkably more active and selective than the corresponding lanthanide oxide in epoxidation reaction of olefins.

Keywords Metal–organic framework · Lanthanide · Epoxidation · Olefin · Heterogeneous catalysis

1 Introduction

Porous framework solids are increasingly attracting attention in recent time because of their fascinating and tunable porosity and molecular topologies. These materials have attracted a major share of attention of the chemists,

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Present Address: R. Sen Department of Chemistry, CICECO, University of Aveiro, 3810-193 Aveiro, Portugal material scientists, and physical scientists for their application potential in the field of catalysis [1], gas adsorption and separation [2], storage [3], artificial nucleases for the hydrolytic cleavage of DNA and RNA [4], contrast agents for magnetic resonance imaging [5] as well as magnetic materials [6]. Apart from naturally occurring porous materials and recently developed micro/mesoporous materials [7, 8] (SBA-15, MCM-41 etc.) or phosphate systems [9], hydrothermally synthesized tunable microporous MOFs are of great interest for their structural flexibility, diversity and spatial control. Microporous MOFs with tunable channel structures can have great potentials for the direct incorporation of catalytic sites with desirable activity [10]. Therefore, rational designing of microporous MOFs for selective catalytic activity has remained a challenge.

A variety of reactions have been studied using MOF catalyst. Metal centers have direct relevance in showing catalytic activity by MOFs. The active center might be either isolated metal ions or clusters [11, 12] (dimers [13], trimers [14], tetramers [15] etc.), chains [16], or sheets [17] connected through the organic linkers. Vitorino et al. [18] showed the successful use of lanthanide metal-organic frameworks as Ziegler-Natta Catalysts for the Selective Polymerization of Isoprene. Hupp and co-workers [19] obtained oxidation of olefins catalyzed by a bimetallic mixed ligand MOF in heterogeneous condition using substituted iodosylbenzene oxidant. In this compound the active center is a Schiff base Mn(III) moiety which is stacked in paddle-wheel carboxylate Zn₂ dimers and 4,4'-biphenyldicarboxylate ligands forming square planar layers. Iron(III)-based MOF containing benzene 1,3,5-tricarboxylate, Fe(III)-MIL100, with zeotype architecture has been synthesized hydrothermally [20]. Friedel-Crafts reaction of benzene with benzyl chloride was catalyzed by Fe(III)-MIL100 at 343 K and showed an excellent selectivity of diphenylmethane. Ravon et al. [21] have successfully used zinc dicarboxylate MOF solids as heterogeneous catalysts for the alkylation of aromatics. Alkylation of toluene with *tert*-butyl-chloride affords the corresponding *para*-substituted product selectively. The same reaction catalyzed by AlCl₃ or the acidic zeolite beta gives mixtures of *ortho*- and *para*-substituted compounds and dialkylated products. Recently, Garcia and co-workers [22] added a new example of aerobic oxidation of benzylic alcohol catalyzed by Cu₃(BTC)₂ (where, BTC: 1,3,5-benzenetricarboxylate) MOF.

Oxidative transformations [23] especially epoxidation of alkenes are the key chemical processes in biology [24], synthetic organic chemistry, and in the chemical industry [25]. In recent years, considerable advances have been made in the development of atom-efficient catalytic oxidation employing *tert*-butyl hydroperoxide (*tert*-BuOOH) [26]. The by-product, tert-BuOH generated from tert-butylhydroperoxide can be separated by distillation or recycled for other industrial production, for example, methyl tertbutyl ether (MTBE). There is an ever-growing interest in the application of reusable catalysis for the synthesis of fine chemicals, including enantioselective reactions [27], which could reduce the large amounts of waste products often formed in non-catalytic organic synthesis. Recently we have succeeded to use layered metal carboxylates and hydrogenphosphate to catalyze olefin epoxidation reaction in heterogeneous condition [28, 29]. Two dimensional layered metal-carboxylates having a space between layers of about 3 Å, can be used as heterogeneous catalyst in olefin epoxidation reaction instead of intercalating metal complex into layers of the clay or LDH type materials [28]. Layered V-P-O compound is also found to be useful in olefin epoxidation reaction [29]. Further, Lanthanide-based MOFs are being used in catalytic oxidation of organic sulfur compounds as well as alkanes [30]. However, reports of using lanthanide carboxylate in heterogeneous catalytic epoxidation of olefins are still very scarce [31].

Here, we wish to report the synthesis and use as heterogeneous epoxidation catalysts of two lanthanide carboxylate framework $[Ln(HCOO)_3]_n$ (where Ln = Nd/Pr).

2 Experimental

2.1 Materials and Methods

Neodymium nitrate hexahydrate, praseodymium nitrate hexahydrate, formic acid, cyclopentene, cyclooctene, cycloodecene, 1-hexene, 1-octene, 1-decene, styrene, 3-Me styrene, 4-Me styrene, *trans*-stilbene and *tert*-butyl-hydroperoxide (70 wt% aqueous) were purchased from

Aldrich and were used as received and solvents were purchased from Merck (India). The solvents were distilled and dried before use.

Fourier transform infrared spectra (in KBr) were measured on a Perkin–Elmer RX I FT-IR spectrometer. Elemental analyses (CHN) were performed using a Perkin– Elmer 240 elemental analyzer. The powder X-ray diffraction (XRD) patterns of the samples were recorded with a Scintag XDS-2000 diffractometer using CuK α radiation. The products of the catalytic reactions were identified and quantified by a Varian CP-3800 gas chromatograph using a CP-Sil 8 CB capillary column.

2.2 Synthesis and Characterization

Although the complexes were reported earlier [32], we have synthesized the materials with some modification. Previously Gao and co-workers [32] prepared these compounds hydrothermally by mixing LaCl₃ and lactic acid in aqueous solution using (Et)₃N as base, but in our case we prepared the compounds $[Nd(HCOO)_3]_n$ (1) and $[Pr(HCOO)_3]_n$ (2) solvothermally. For digestion, Nd(NO₃)₃ and formic acid were added in the molar ratio of 1:2 in 10 ml of DMF and kept at 180 °C for 3 days followed by slow cooling at the rate of 5 °C/h to room temperature. The crystals thus formed were filtered off, washed first with water and then with a small amount of ethyl alcohol and dried in air. Compound 2 was prepared in a similar manner by using Pr(NO₃)₃ instead of Nd(NO₃)₃ with the same molecular ratio of reactants as that of neodymium analogue. Yield ca. 65 and 68% (based on metal) for 1 and 2, respectively. For preliminary characterization of the compounds, elemental analysis and IR spectroscopic study were undertaken. Anal. Calcd. for $[Nd(HCOO)_3]_n$ 1; C = 12.9, H = 1.0, found C = 12.5, H = 1.1%; Selected IR peaks (KBr disk, cm⁻¹): 1579 $[v_{as} (CO_2^{-})]$, 1433, 1401 $[v_s$ (CO_2^{-})], 1338 [v_s (C–O)], and 3500–3200 [v(O–H); s. br]. Anal. Calcd. for $[Pr(HCOO)_3]$ **2**; C = 12.8, H = 1.0, found C = 12.5, H = 1.1%; Selected IR peaks (KBr disk, cm⁻¹): 1610 [v_{as} (CO₂⁻)], 1442, 1398 [v_{s} (CO₂⁻)], 1321 $[v_{s} (C-O)]$, and 3500–3200 [v(O-H); s. br].

2.3 General Procedure for Epoxidation Reaction

The catalytic reactions were carried out in a glass batch reactor. Substrate, solvent and finely powdered catalysts were first mixed. The mixture was then equilibrated to 70 °C in an oil bath. After addition of *tert*-BuOOH, the reaction mixture was stirred continuously for 24 h. The products of the epoxidation reactions were collected at different time intervals and were identified and quantified by gas chromatography. Compounds were dried under vacuum (~ 100 °C, 12 h) prior to use it as catalyst.

3 Results and Discussion

3.1 Structure of $[Nd(HCOO)_3]_n$ (1) and $[Pr(HCOO)_3]_n$ (2)

Although the structures of the compounds have been reported previously [32], we redetermined the crystal structures of compounds 1 and 2 only to verify the integrity of the compounds. X-ray studies reveal that compounds 1 and 2 have identical structure. Each Ln^{3+} ion possesses a distorted tricapped trigonal prismatic environment surrounded by nine oxygen atoms from nine different formate anions. Each formate anion acts as $\mu_{1,1,3}$ bridge and coordinates to three different Ln³⁺ ions. The distance between the $\mu_{1,1}$ -bridged metal ions is in the range of around 4.0 Å, where distance between the other metal ions are in the range of 6 Å. Packing diagram of the compounds 1 and 2 demonstrates that three-dimensional arrangement afforded equilateral triangular pores in compounds in the solid state (Fig. 1). The dimension of pores is $\sim 5.30 \times 5.30 \times$ 5.30 Å for both the compounds.

3.2 Nitrogen Sorption Studies

The nitrogen sorption measurements of the compounds were undertaken to verify porous nature of the compounds as well as to calculate the surface area of the catalyst. BET (Brunauer–Emmett–Teller) surface area of **1** was calculated from the N₂ adsorption isotherms. The adsorption and desorption isotherms of **1** are shown in Fig. 2. The adsorption of N₂ follows a type III isotherm with a surface area (S_{BET}) equal to $11 \text{ m}^2\text{g}^{-1}$. Nitrogen sorption measurement also afforded the pore size distribution of the porous compound. This corroborates the information obtained from X-ray crystallographic analysis as regards the porous nature of the compounds. As the structure of the two compounds is identical only compound **1** was subjected for nitrogen sorption study.



Fig. 1 Packing diagram showing porous structure of the compound 1



Fig. 2 *Plot* showing nitrogen sorption isotherm of compound 1. *Inset* shows the pore-size distribution of compound 1

3.3 Catalytic Epoxidation Reactions

Olefin epoxidation reactions catalyzed by lanthanide containing homogeneous catalysts are well documented [33]. Homogeneous catalysts, however, suffer from the problems of recycling of the catalyst as the separation of catalyst from reaction mixture is often impossible. Recycling of catalysts is a task of great economic and environmental advantages in chemical and pharmaceutical industry, especially when expensive and/or toxic heavy metal complexes are employed. To this end many attempts have been made, such as intercalating or encapsulating the metal complex into the layered compounds or within the cavities of a porous solid (e.g., zeolites) [34], binding the metal complex into the polymeric matrix [35], and employing the steric hindrance [36] to design recyclable catalysts. Polymer supported lanthanide-binol systems and $Ln(O-iPr)_3$ systems have been extensively used in catalytic oxidation of olefinic substrates [37]. Nevertheless, in this study porous lanthanide carboxylates have been used as such, instead of using any support, as recyclable heterogeneous catalyst in olefin epoxidation. The results of the catalytic epoxidation of different substrates are summarized in Table 1. The kinetic profiles of the progress of the reactions using the catalysts 1 and 2 are shown in Figs. 3 and 4, respectively. The overall catalytic efficacy of 1 and 2 in epoxidation reactions were complete for less bulky olefinic substrates (Table 1). The epoxidation of cyclopentene proceeded smoothly, showing good conversion to their epoxides with 100% selectivity. The oxidation of cyclooctene also goes smoothly, showing conversion (78 and 86%, for 1 and 2, respectively) to form cyclooctene oxide with 100% selectivity, while the conversion of bulky cyclododecene was not complete with the conversion up to 69% simultaneously producing the side product. In case of

Entry	Substrate	Reaction time/h	Conversion [wt%]		% Yield of products		TON
					Epoxide	Others	
1		9	a)	98	98	_	2,014
			b)	99	99	-	2,008
2	\sim	24	a)	78	78	-	976
			b)	86	86	-	1,066
3		24	a)	61	30	31 ^a	506
			b)	69	34	35 ^a	566
4		24	a)	89	51	38 ^b	1,180
			b)	91	54	37 ^b	1,192
5		24	a)	82	58	24 ^c	958
			b)	86	63	23 ^c	994
		24	-)	70	55	aad	010
0		24	a) b)	78 83	55 57	23 ^d	910 958
7		24	a)	53	53	_	406
			b)	51	51	_	386
8	\sim \sim //	24	a)	99	99	-	1,616
			b)	98	98	_	1,606
9		24	a)	48	48	-	664
			b)	42	42	-	622
10	$\land \land \land \land \land \land$	24	a)	42	22	20 ^e	410
	/ V V V V		b)	38	25	13 ^e	482

 Table 1 Epoxidation of olefins catalyzed by 1 and 2

(a), (b) corresponds to the catalytic performance of compounds **1** and **2**, respectively. Reaction conditions: alkenes (1 g); catalyst (2 mg); *tert*-BuOOH (2 mL); acetonitrile (8 mL); Temperature 65–68 °C. The products of the epoxidation reactions were collected at different time intervals and were identified and quantified by Varian CP-3800 gas chromatograph equipped with an FID detector and a CP-Sil 8 CB capillary column *Turn over number (TON)* moles converted/moles of active site

^a Cyclododecanol; ^b Benzaldehyde; ^c 3-Me-benzaldehyde; ^d 4-Me-benzaldehyde; ^e Decanone

linear alkenes, 1-hexene shows almost complete conversion and producing epoxide as the sole product, but in case of long chain alkenes the conversion was not complete like the earlier linear alkenes; the conversion only limited to 38–42% for 1-octene and 1-decene (Table 1). On the other hand, oxidation of styrene showed 89–91% conversion while epoxide yields were 51–54%. Along with styrene oxide, benzaldehyde is also formed. In fact, in the epoxidation of styrene with *tert*-BuOOH over transition metal immobilized zeolite or molecular-sieves catalysts, epoxide yield seldom exceeds 40% [38]. In our recent attempt we have succeeded to improve the yield of styrene epoxide up to \sim 70% using layered transition metal carboxylate catalysts [28]. The substituted styrene was also



Fig. 3 Kinetic profile for epoxidation of olefins catalyzed by compound ${\bf 1}$



Fig. 4 Kinetic profile for epoxidation of olefins catalyzed by compound ${\bf 2}$

converted 86% successfully with oxide selectivity up to 63% and the other products were found as their corresponding aldehyde. Here, trans-stilbene was also subjected for epoxidation, but the conversion was not inspiring, although oxide was the unique product. Evidently, as the bulkiness of the cyclic olefins and chain length of the linear alkenes is increasing the conversion and turnover numbers are gradually diminishing in the epoxidation reactions catalyzed by catalysts 1 and 2. Selectivity and conversion becomes less when the bulkiness or chain length of the substrates is increased which may be explained on the basis of steric hindrance causes by the substrates. When the substrate is bulky steric hindrance prevents itself in approaching towards active site with ease. Consequently, the conversion is lowered in catalytic reactions.

The catalytic reactions were performed employing varieties of oxidants to study the catalytic efficacy of 1

and 2. The best performance of the catalysts is observed in case of tert-BuOOH in the acetonitrile medium. Hydrogen peroxide and sodium hypochlorite solution were less effective for the conversion (Table 2). Alkyl-hydroperoxides are used on a large scale in industrial epoxidation, for example, in Halcon-Arco and Sumitomo processes [39, 40]. The recycling of co-products, e.g., tert-BuOH has been realized in the Sumitomo process. Besides, to ascertain the catalytic efficacy of compounds 1 and 2 we have undertaken few control experiments. Temperature dependence of catalytic performance of compounds 1 and 2 has been studied. While catalysts showed almost no conversion at room temperature they exhibit the desired conversion at 70 °C demonstrating the vital role of temperature on the activation of the catalyst. Further, comparison of catalytic efficiency of 1 and 2 with the corresponding heterogeneous non-porous lanthanide oxide catalysts clearly indicates that the lanthanide carboxylate MOFs perform as much superior catalyst than the metal oxides (Table 3).

3.4 Separation, Catalyst Reuse and Heterogeneity Test

The major advantage of the use of heterogeneous catalysts is to recover the catalyst from the reaction mixtures by simple filtration and recycle. To test if metal is leached out from the solid catalyst during reaction, the liquid phase of the reaction mixture is collected by filtration at the reaction temperature after 30% of the epoxidation reaction is completed and the residual activity of the supernatant solution after separation of the catalysts was studied. The impending leaching was premeditated as, the organic phase of a first run was separated from the catalyst and new reagents were added to the clear filtrate, and the composition of the reaction mixture was determined by GC. This homogeneous reaction mixture was treated as a standard catalytic experiment. After 6 h, the composition was determined, and no reaction was observed, which excludes the presence of active species in solution. In order to check the stability of the catalysts, we have characterized the solids after the completion of reactions. Besides, to test if metal ions are leaching out of the catalyst, the reaction mixture was filtered out after the reaction and was subjected to atomic absorption spectroscopic analysis. The analysis showed the absence of metal ions in the filtrate. These experiments clearly demonstrate that metal is not leaching out from the solid catalyst during epoxidation reactions. After the catalytic reactions are over, solid catalyst was recovered by centrifugation and washed with fresh acetonitrile several times and dried in air oven. The recovered catalyst was then subjected for X-ray powder diffraction analysis. Comparison of X-ray diffraction patterns of the virgin catalysts and recovered catalysts

Substrate	Oxidant	Reaction time/h Conversion [wt%] ^a		% Yield of products	
				Epoxide	Others
	H ₂ O ₂ NaOCl	9 9	55 (58) 16 (19)	39 (32) 6 (5)	16 (26) ^b 10 (14) ^b
	H ₂ O ₂ NaOCl	24 24	48 (41) 14 (15)	35 (31) 14 (15)	13 (10) ^c

Table 2 Epoxidation of alkenes using variety of oxidants catalyzed by 1 and 2

Reaction conditions were the same as given in footnote of Table 1

^a Values in parenthesis show the results obtained for complex 2; ^b Cyclopentanone; ^c 1-hexanone

Table 3 Epoxidation of alkenes catalyzed by metal oxides

Substrate	Reaction time/h	Conversion	% Yield of products			
		[wt%]	Epoxide	Others		
Cyclopente	ene					
Nd_2O_3	9	17	2	15 ^a		
Pr_2O_3	9	21	21	_		
Cyclododecene						
Nd_2O_3	9	15	6	9 ^b		
Pr ₂ O ₃	9	14	5	9 ^b		

Reaction conditions were the same as given in footnote of Table 1

^a Cyclopentanone; ^b Cyclododecanol

convincingly demonstrate that the structural integrity of the compounds **1** and **2** is remained unaltered after the epoxidation reactions (see Supplementary Data Figs. S1, S2). Notably the recovered catalyst can be reused in epoxidation reactions for several times with no considerable loss of activity (Table 4). The kinetic profiles of the epoxidation reaction of cyclopentene using virgin and recovered catalysts are virtually remained the same (see Supplementary Data Figs. S3, S4).

4 Conclusion

To summarize, we have demonstrated that the lanthanide carboxylate frameworks can be used as selective catalyst for the epoxidation of a variety of olefins using *tert*-butylhydroperoxide. The frameworks behaved as the heterogeneous catalyst, can be easily recovered after reaction by filtration and washing and subsequently be used without having significant deactivation of catalyst due to either leaching of active species or degradation of the structure. The catalyst shows excellent efficiency towards epoxidation reactions as reflected in high selectivity in epoxides and high turnover number of the reactions. Further investigations employing this type of catalyst in other reactions are currently in progress in our laboratory.

 Table 4 Catalytic efficacy of the recovered compounds 1 and 2 in successive runs in cyclopentene epoxidation

Compounds	Cycles	Conversion (wt%)	Yield of product (wt%)	TON
1	First reuse	99	99	2,014
	Second reuse	98	97	1,974
	Third reuse	97	97	1,965
2	First reuse	99	98	1,988
	Second reuse	99	98	1,988
	Third reuse	97	97	1,975

Reaction conditions were the same as given in footnote of Table 1

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