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Cooperative activation promoted on flexible porous organic frameworks

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Abstract: Developing solid catalysts with multi active sites working in a cooperation way, the mimicking enzyme's function is desirable for efficient chemical transformations. However, most solid catalysts are rigid and impede the cooperation between their spatially isolated active sites. Herein, two flexible porous organic frameworks (POFs) with integrated Co(salen) as active sites have been successfully synthesized for mimicking the cooperative modes of enzymes. The POFs exhibit second-order rate dependence on Co(salen) concentration in the network and afford much higher TOF (3300 versus 2670 h⁻¹) than homogeneous counterpart in the hydration of propylene epoxide. This demonstrates that POFs with flexible network not only facilitate but also enhance the cooperation of nearby Co(Salen). Moreover, POFs could catalyze oversized substrates, have a wide substrate scope and exhibit high stability.

Nowadays, the catalysis is facing challenges for producing chemicals with energy and raw material saving mode due to global environment problems and rapid consumption of fossil feedstock. Consequently, the catalysts should possess both high activity and selectivity under mild reaction conditions. Great efforts have been devoted for the synthesis of biomimetic catalysts due to the high activity and high selectivity of enzymes under mild conditions.^[1] Enzymes are macromolecules that work in cooperative modes and have complex internal protein dynamic motions. Internal dynamics and flexibility are highly relevant to the enzymatic reactions, where subtle structural changes induced at the reactive site enhance the reactivity and selectivity of chemical reactions. It is thus important to create reaction fields that can promote chemical reactions in a cooperative and responsive manner. Considering that enzymes are macromolecules and have three-dimensional structure, the biomimetic catalysts are designed mainly based on soft materials, such as micelles,^[2] vesicles,^[3] macromolecules^[4] and supramolecular gels^[5] and so on.

The porous solid materials, e.g. zeolites,^[6] mesoporous silicas,^[7] porous organic polymers^[8] and metal-organic frameworks (MOFs),^[9] with encapsulated/immobilized/integrated active sites, tunable affinity for substrates and products and the advantages of recycling and reusing have also been used as a platform for artificial enzymes though the examples are far less in comparison with soft materials. The representative examples are

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given below. Fe-ZSM5 with so-called surface "α-oxygen" species mimics unique catalytic performance of enzymes monooxygenases in selective oxidation reactions, including oxidizing methane to methanol and benzene to phenol.[6b,10] Zirconium(IV)-cluster-containing MOF with the combination of the strong Lewis-acidic Zr(IV) and bridging hydroxide anions for mimicking phosphotriesterase enzyme shows high activity for the methanolysis and hydrolysis of phosphate-basednerve agent simulants.^[9a] He and co-workers reported the encapsulation of Mn(TPP)OAc in the interlayer region of layered double hydroxides to mimic the bilayer structure of nature phospholipids.[11] For most reports, the mimicking cooperative activation mode, the classical character of enzymes, is not very successful. This is mainly due to the rigidity nature of solid porous materials. The active sites are spatially isolated in porous materials, consequently, the close proximity of active sites, required for the cooperative activation, is hard to achieve.

Herein, we report a new concept for the synthesis of solid catalyst with cooperative activation effect by combination of flexible network and high concentration of active sites. As a proof of concept, Co(salen) was chosen as active site considering its cooperative activation pathway in epoxide hydration reaction and POF is selected as solid material due to its chemical and physical tunability. POFs with the flexibility and enriched Co(Salen) exhibit enhanced cooperative activation effect in the epoxide hydration reaction, an important reaction in industry for the production of glycols and an energy consuming process due to high H₂O/epoxide ratio used for obtaining high glycol selectivity.^[12]



Scheme 1. Schematic illustration for the synthesis of POFs.

1,3,5-tris(3'-tert-butyl-4'-hydroxy-5'-The reaction of formylphenyl)benzene (TBHFPB) and cyclohexanediamine/ethanediamine produces threedimensionally linked salen network with rich porosity, imine and C-C bonds (Scheme 1). The existence of imine and C-C bonds allows the free rotation of the polymer leading to a flexible network. POF-1 and POF-2 were prepared respectively with cyclohexanediamine and ethanediamine. The formation of salenriched POFs was confirmed by Fourier-transform infra-red (FT-IR) spectroscopy and solid-state ¹³C-NMR technique (Figure S1).

POFs are insoluble in water and common organic solvents such as CH_2Cl_2 , toluene, tetrahydrofuran, propylene oxide and N,N-dimethylformamide (DMF). POF-1 and POF-2 have N

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content of 4 and 5 mmol/g, which is almost identical to theoretical value calculated based on the molecular structure. This indicates almost complete condensation between TBHFPB and diamine during the synthesis process. POF-Coll was synthesized via a straightforward coordination of Co(OAc)₂·4H₂O with POF (Scheme 1). The red shift of C=N vibration and the appearance of Co-O vibration peak at 580 cm⁻¹ in the FT-IR spectra indicate the successful coordination of Co^{II} with the salens in the porous network (Figure S1A, line b and e). N/Co molar ratio of POF-1-Coll and POF-2-Coll is respectively of 3.2 to 3.6 mmol/g, indicating that a large amount of salen units in POFs are accessible and could be coordinated to Co. The oxidation of POF-Co^{II} with air in the presence of p-toluene sulphonic acid results in the formation of POF-Co^{III}. The new vibrational peaks at 1120 cm⁻¹ and 1000 cm⁻¹ assigned respectively to asymmetric stretching vibration and symmetric vibration peak of sulfonyl group in the FT-IR spectra of POF-1-Co^{III} and POF-2-Co^{III} confirm the incorporation of ptoluenesulfonic anion in the porous polymer (Figure S1A, line c and f).



Figure 1. Simulated (A) POF-1, (B) POF-1-Co^{III} and (C) POF-1-Co^{III} network in an amorphous periodic cell, dimension of simulation box (the "amorphous cell") = 3.5 nm. Carbon atom was gray, hydrogen atom was white, nitrogen atom was blue, oxygen atom was red, cobalt atom was green and sulfur atom was yellow. (Below are the corresponding geometry structure).

X-ray photoelectron spectroscopy (XPS) survey results of POF-1-Co^{III} and Co^{III}(salen)-1 show the feature peaks of C 1s, O 1s, N 1s, S 2p and Co 2p (Figure S2). The fine structure of Co 2p core level shows one doublet corresponding to Co 2p3/2 and Co 2p1/2 spin-orbit split. The BE for Co 2p3/2 of POF-1-Co^{III} is about 781.5 eV, which is only slightly higher than that of Co^{III}(salen)-1 (781.2 eV). Similar tendency was observed for BE of Co 2p1/2 (796.5 eV versus 796.2 eV). The almost identical binding energy for POF-1-Co^{III} and Co^{III}(salen)-1 suggests that Co^{III}(salen) integrated in POF has similar coordination environment to homogeneous counterpart.

The N_2 sorption isotherms of all POFs afford type I sorption isotherm pattern with H3 hysteresis loop, showing that POFs have hierarchical micro/mesoporous structure (Figure S3 and Table S1). It is noteworthy to mention that BET surface area and pore volume sharply increased after coordination with Co (Table S1). This is possibly due to the change in local geometric structure of POFs before and after coordination with Co based on the computer-modeling results (Figure 1 and Figure S4). This also suggests that the polymer network has a soft nature and may distort according to coordination environment. This character may benefit the proper arrangement of molecular catalysts during the catalytic process. In comparision to POF-Co^{III}, the BET surface area and pore volume of POF-Co^{III} are not very high, possibly due to the occupation of *p*-toluenesulfonic anion in the micropore of POF-Co^{III}.

Unexpectedly, all POFs with microporous structure have high benzene adsorption capacity ranging from 429 to 858 mg/g (Table S1, Figure 2A and S5A). This suggests that POFs could accommodate guest molecules with large molecular size. Moreover, POF-Co^{II} gives type VI isotherm pattern with distinct multi adsorption steps which were often observed on flexible MOFs and COFs.^[13] The above results show that pore size expansion occurs during the adsorption, further confirming the N2 sorption results that the POFs may have flexible networks. POF-2-Coll gives a sigmoidal adsorption curve at relative pressure in the range of 0.01 to 0.5 and the distinctive multi adsorption steps at high relative pressure ($P/P_0 = 0.5-1.0$). The sigmoidal adsorption curve reflects the coordination adsorption behavior.^[14] Benzene adsorption experiment for a Coll(salen) integrated in conjugated microporous polymer^[16], CMP-Co^{II} was performed too (Figure S6). However, no stepped benzene uptake could be observed comparing to POF-1-Coll or POF-2-Coll. This suggests that CMP-Co^{II} is not as flexible as POF-Co^{II}.



Figure 2. (A) Benzene adsorption isotherms of POF-2 and POF-2-Co^{II}, (B) ³¹P static NMR of POF-2-Co^{III}-PF₆ under static conditions in its solid state and in the suspension of CHCl₃ and propylene epoxide (NS = 380 scans), SEM images of (C) POF-2, (D) POF-2-Co^{III} and (E) POF-2-Co^{III}.

The dynamic motions of POFs were investigated by ³¹P solid static NMR technique with POF-Co^{III}-PF₆ as model (Figure 2B). The ³¹P static NMR spectrum of POF-2-Co^{III}-PF₆ exhibits a broad signal at -116 ppm due to chemical-shift anisotropic of the nucleus under the solid state.^[15] The intensity of this signal increases and peak width decreases obviously with the addition of propylene epoxide as solvent. POF-1-Co^{III}-PF₆ shows the similar tendency (Figure S5B). This is the evidence that POFs framework do have some flexibility and mobility in the presence of solvent.

Interestingly, the morphology of POF-2 gradually transforms from nanofiber to microspheres (500 nm in size) after coordination

COMMUNICATION

(Co) and oxidation (Figure 2C-2E), suggesting that POFs have changeable morphology corresponding to outer environment. This is possibly due to different distortion of polymer network induced by coordination with metal cations.

For amorphous materials, it is difficult to characterize the accurate flexibility. In our case, we clearly observed the variation in BET surface area, pore diameter and morphology by coordinating POFs with cobalt. Moreover, POFs show stepped uptakes of benzene. All above characterizations strongly support that the POFs are flexible. Such a cooperative adsorption behavior is difficult to achieve with conventional rigid porous materials, such as zeolites, mesoporous silicas, porous carbons and so on.

Table 1. The catalytic performance of POF-Co^{III} in propylene oxide hydration reaction^[a].

Cat.	Conv. (%)	Sel. (%)	TOF (h ⁻¹)	
Co ^{III} (salen)-1	99	99	2670	
Co ^{III} (salen)-2	17	99	-	
POF-1	<1	-	-	
POF-1- Co ^{III}	97	99	3300	
POF-2- Co ^{III}	97	99	1470	
CMP-Co ^{III[16]}	6	99	-	

[a] S/C = 500, T = 40 °C, 3 h, H₂O/PO molar ratio = 2, TOF was calculated at conversion < 30%. [b] Co^{III} (salen)-1 is derived from cyclohexanediamine. [c] Co^{III} (salen)-2 is derived from ethanediamine.



Figure 3. The relationship of initial reaction rate with Co(salen) in the network of POF-1-Co^{III} for PO hydration reaction (S/C = 1000, H₂O/PO molar ratio = 2, 40 °C). The experimental values were fitted using the equation: $r = K(Co \text{ content})^2$.

The catalytic performance of POF-Co^{III} was tested in the propylene epoxide hydration reactions (Table 1). No product could be detected for the blank reaction or with POF-1/POF-2 as a catalyst using propylene oxide as substrate. Encouragingly,

POF-1-Co^{III} and POF-2-Co^{III} could efficiently catalyze PO hydration with a 97% yield of propylene glycol under similar conditions. Furthermore, the reaction rate of POF-1-Co^{III} shows a second-order dependence on Co content in POF-1-Co^{III} framework (Co content in the network varies in the range of 0.2 to 0.9 mmol/g, the initial reaction rate increases dramatically from 0.009 to 0.33 mmol/min) (Figure 3), confirming that POF-1-Co^{III} catalyzes the PO hydration reaction via a cooperation activation pathway. CMP-Co^{III[16]} with Co^{III}(salen) integrated in conjugated microporous polymer only gave 6% conversion under similar conditions. The low activity of CMP-Co^{III} is possibly due to its rigidity and the longer distance between two Co^{III}(salen) active sites, which impedes the cooperation of Co^{III}(salen) during the catalytic process.

Under similar reaction conditions, The TOF value of POF-1-Coll is much higher than that of its homogeneous counterpart (3300 versus 2670 h⁻¹), indicating that the cooperation of crosslinked Co(salen) is enhanced in comparison with Co^{III}(salen) in the homogeneous system. PO conversion on POF-2-Co^{III} is more than five folds that of Coll(salen)-2. The poor activity of Coll(salen)-2 is possibly due to its low solubility in the reaction system and different molecular structure and electronic properties comparing to Co^{III}(salen)-1. In addition, the activity of POF-1-Co^{III} in PO hydration reaction is higher than most of heterogeneous catalytic systems, such as Sn zeolites, Co^{III}(salen) based porous polymers and ordered mesoporous silica materials (Table S2). More interestingly, the TOF of POF-Co^{III} increases enormously as the S/C increases from 500 to 2000 (Figure S7). On the contrary, the TOF of Co^{III}(salen)-1 decreases from 2670 to 2040 h⁻¹. This further confirms the enhanced cooperative activation effect for POFs based catalysts.

POF-1-Co^{III} and POF-2-Co^{III} could efficiently convert epoxides with aliphatic substitute to corresponding diols with high conversion and selectivity (Table 2, Entry 1-6). Even for sterically hindered cyclohexene oxide, POF-2-Co^{III} could afford 64% conversion (Table 2, Entry 8). A sterically more demanding substrate, ethylene oxide substituted with triphenyl glycidyl ether, which size is much larger than the pore diameter of POFs catalysts, was also used for hydration reaction. Encouragingly, POF-1-Co^{III} gives 67% conversion (Table 2, Entry 9). The fact that POFs could catalyse the over-sized substrates confirms that the pore size expansion occurs during catalytic process. This is possibly due to the flexible network of POFs which may change their structure and expand their pore size for accommodating substrates. For cyclohexene oxide and ethylene oxide substituted with triphenyl glycidyl ether, POF-1-Co^{III} and POF-2-Co^{III} show different substrate specificity.

To evaluate the stability of the solid catalyst, the filtration test and recycling experiment were performed. POF-1-Co^{III} was filtrated out of the reaction system after glycol yield approaching 46 % (Figure S8). No further increase in glycol yield was observed for another 180 min. This result clearly shows that the reaction is catalyzed by solid catalyst and no leaching of Co(salen) occurs during the catalytic process. Furthermore, POF-1-Co^{III}could be recycled for at least 7 times for PO hydration with only slightly decrease in activity (Figure S9). ICP analysis shows that reused POF-1-Co^{III} has Co content of 5 wt% (0.85 mmol/g), comparable to fresh sample. This suggests that POF-1-Co^{III} has enough stability to be used as solid catalyst for epoxide hydration.

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In summary, this work successfully endowed the solid catalyst with flexibility demonstrated by porous organic frameworks integrated with Co(salen). Multi adsorption steps for benzene and solid ³¹P NMR suggest that POFs may have changeable porous structure and flexibility. Consequently, the POFs could efficiently catalyze the hydration of epoxide via a cooperative activation way as evidenced by the second-order rate dependence on Co(salen) concentration in the network. The high activity of POFs is mainly attributed to the flexible network for facilitating the cooperation of nearby Co(salen) during the catalytic process. Furthermore, the POFs enhance the cooperative activation, affording much higher activity than homogeneous counterpart.

Table 2. Catalytic performance of POF-Co^{III} for catalyzing hydration of epoxides with different molecular size^[a].

Entry	Substrate	Cat.	Time (h)	Conv. (%)	Sel. (%)
1	Ņ	POF-1- Co ^{III}	3	99	>99
2		POF-2- Co ^{III}	4	89	95
3		POF-1- Co ^{III}	6	97	>99
4	M_3	POF-2- Co ^{III}	6	88	97
5		POF-1- Co ^{III}	8	92	99
6	M_{12}	POF-2- Co ^{III}	8	66	99
7	$\int \int 0$	POF-1- Co ^{III}	48	21	85
8	\checkmark	POF-2- Co ^{III}	48	64	93
9 ^[b]	$\overset{\circ}{\frown}$	POF-1- Co ^{III}	72	67	99
10 ^[b]	[∽] CPh ₃	POF-2- Co ^{III}	72	26	99

[a] T = 40 °C, S/C = 500, H₂O/PO molar ratio of 2. [b] S/C = 50, 1 mL CH₂Cl₂ was added and conversion was tested by ¹H NMR.

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Keywords: Co(salen) • porous organic frameworks • flexibility • epoxide hydration • cooperation

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Layout 1:

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Porous organic frameworks with high concentration of Co(salen) in the network and flexible nature efficiently promote the cooperation between the nearby Co(salen) in the epoxide hydration reaction.



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Page No. – Page No.

Cooperative activation promoted on flexible porous organic frameworks