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Ionic Liquid/H₂O-Mediated Synthesis of Mesoporous Organic Polymers and Their Application in Methylation of Amines

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Mesoporous Tröger's base-functionalized polymers (Meso-TBPs) were prepared using sulfonic acid group functionalized ionic liquid/H₂O, with surface areas up to 431 $m^2 g^{-1}$ and pore sizes of 3~15 nm. Ir(II) coordinated Meso-TBP exhibited extraordinary catalytic performance in the Nmethylation of amines using methanol.

In recent years, porous organic polymers (POPs) have attracted considerable attention because of their permanent porosity, diversified chemical synthetic routes, and extensive applications in the fields of gas adsorption, photocatalysis, electrical energy storage, heterogeneous catalysis and so on.¹ Especially, POPs with nitrogen- or phosphine-rich functionalities exhibit excellent coordination ability with transition-metal complex, and the resultant metalized materials perform well as heterogeneous catalysts in organic reaction, even with higher catalytic efficiency than the corresponding homogeneous ones owing to the synergistic effect of the POPs backbone and the metal center.² Up to now, most of the reported POPs possess micropores (<2nm), which are quite promising materials for efficient gas adsorption (e.g. CO₂, H₂).³ However, micropores are unfavorable to mass transfer, inferior in contact of substrates with the catalytic center within the pores, and in diffusion of products out of the cavities, thus limiting their applications in catalysis.⁴ Only a few examples have been reported for the preparation of mesoporous organic polymers. For example, mesoporous ohydroxyazobenzene polymers have been developed by our group based on diazo-coupling reaction in aqueous solution.⁵

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Xiao et al. reported an organic solvent mediated approach for the synthesis of mesoporous phosphine-functionalized POPs based on divinylbenzene polymerization.⁶ Dai's group prepared mesoporous phenolic polymers and ionic organic networks using F127 as soft template⁷ and SiO₂ as hard template,⁸ respectively. Though such progress has been made, the synthesis of POPs with mesoporous structures, especially those functionalized by nitrogen-containing ligands, is still challenging.

lonic liquids (ILs) are tunable solvents with extremely low vapor pressure, wide electrochemical window, nonflammability, high thermal stability and wide liquid range.9 Such unique properties offer ILs opportunities to replace conventional solvents in a variety of applications in chemistry and material science.¹⁰ For example, ILs combined with water or CO₂ have been used as solvents and templates for the preparation of porous metal organic frameworks (MOFs) and metal oxides,¹¹ which provides green routes for the synthesis of these functional materials. However, utilization of ILs for the synthesis of mesoporous organic polymers is rarely reported. On the other hand, in most cases, noble-metal catalysts are required for the synthesis of POPs in organic media, which is environmentally unfriendly and has the drawbacks of high economic cost and metal residual. Hence, synthesis of POPs under metal-free conditions, especially in aqueous media is highly desirable.

Tröger's base-functionalized polymers (TBPs) is a kind of POPs with TB ligand, which can be prepared based on the strong acid-catalyzed reaction of amines with dimethoxymethane (DMM), affording micropores in the polymer.¹² Herein we proposed a facile route for the preparation of mesoporous TBPs (**Meso-TBPs**), in which sulfonic acid group functionalized IL, 1-butylsolfonate-3-methylimidazolium chloride (SO₃H-IL), served both as the catalyst and reaction medium together with H₂O without any other template and surfactant. Changing the mass ratio of H₂O to SO₃H-IL, the Brunauer–Emmett–Teller (BET) surface areas of the resultant TBPs could be tuned in the range

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of 85~431 m² g⁻¹, and the highest surface area of 431 m² g⁻¹ was obtained at the H₂O/SO₃H-IL ratio of 1.5. The pore sizes of the resultant TBPs were in the range of 3~15 nm, and the H₂O/SO₃H-IL ratio had little effect. Mesoporous benzimidazole-functionalized POPs was also synthesized in the aqueous SO₃H-IL solutions. The integration of TB ligand gave the **Meso-TBPs** the ability to chelate with Ir(II) complex, and the Ir(II)-chelated TBP exhibited extraordinary catalytic performance in the N-methylation of amines using methanol without any other additives, even better than their corresponding homogeneous analogues, together with high stability and easy recyclability.

Firstly, the metal-free synthesis of Meso-TBP-1 was conducted through the reaction of a planar rigid aromatic amine building block (A-1) with DMM using the mixture of SO₃H-IL and H₂O as reaction medium without any other additives (Figure 1A, for synthetic procedures, see ESI). Changing the mass ratio of H₂O to SO₃H-IL in the range of 0.5~3, a series of polymers (denoted as Meso-TBP-1-r, where r stands for the ratio of H₂O to SO₃H-IL) as brown powders were obtained with BET surface areas of 85~368 m² g⁻¹, and the corresponding total volume of 0.119~0.439 cm³ g⁻¹ (Figure 1B, for BET plots, see Figure S1, ESI). Notably, Meso-TBP-1 with the highest BET surface area and total pore volume was achieved at a H₂O/SO₃H-IL mass ratio of 1.5. That is, Meso-TBP-1-1.5 had the highest surface area and total pore volume. The textual information of the resultant Meso-TBP-1-r was investigated by N2 sorption analysis. As shown in Figure 1D, the N₂ adsorption-desorption isotherm of the **Meso-TBP-1-r** (r = 1.0 to 2.5) exhibited an intermediate mode between type I and type IV, indicating the coexistence of microand mesopores dominated with mesopores. For polymers Meso-TBP-1-0.5 or Meso-TBP-1-3.0, only mesopores were observed accompanied by relatively small BET surface areas. From the pore size distribution (Figure 1E), it is obvious that the pore sizes of Meso-TBP-1-r were in the range of 3~15 nm, and changed a little via changing the mass ratios of H₂O to SO₃H-IL. Considering that the TB-functionalized organic polymer with the same monomers obtained using strong acidic trifluoroacetic acid as the catalyst only showed micropores of 1.69 nm,¹³ the porous structures of the Meso-TBPs obtained in this work should be ascribed to the specific properties of the reaction media. The morphology of Meso-TBP-1-1.5 was observed by scanning electron microscopy (SEM) and transmission electron microscopy (TEM) (Figure 1F and 1G). It is shown that Meso-TBP-1-1.5 was composed of layer structure with obvious mesopores.

The microstructure of the SO₃H-IL/H₂O reaction medium was investigated to reveal the possible formation mechanism of the mesopores in **Meso-TBP-1-r**. Firstly, the conductivities of the H₂O/SO₃H-IL mixtures were determined, which sharply increased along with the increase in the H₂O/SO₃H-IL mass ratios from 0.5 to 1.5, and then slowly decreased with further increasing the ratios, as shown in Figure 1C. Hence, the maximum conductivity was obtained at the H₂O/SO₃H-IL mass ratio of 1.5, in consistent with the variation tendency of BET surface areas and total pore volumes of **Meso-TBP-1-r** obtained with various H₂O/SO₃H-IL of mass ratios (Figure 1B). H₂O/IL mass ratio of 1.5 was also the



Figure 1. (A) Synthetic route. (B) BET surface area and total pore volume of Meso-TBP-1-r. (C) Conductivity of H2O/SO₃H-IL with mass ratio of 0.5 $^{\circ}$ 3. (D) Adsorption (filled) and desorption (empty) isotherms of N₂ at 77 k for Meso-TBP-1-r. Number within the figure stands for the mass ratio of H₂O/SO₃H-IL. (E) Pore size distributions of Meso-TBP-1-r based on the calculation results of the NLDFT method. (F) SEM and (G) TEM images of Meso-TBP-1-1.5. Scale bars for F and G was 250 nm and 50 nm, respectively.

optimal ratio to balance the catalyst concentration as well as the vicosity of the reaction system. For polymers obtained in H₂O/SO₃H-IL with mass ratio of, e.g. 0.5 or 3.0, the BET surface areas/total pore volumes were small, which was due to the high viscosity of SO₃H-IL with a small quantity of H₂O, or low SO₃H-IL (catalyst) concentration with too much H₂O, and hence low degree of polymerization. Cyro-TEM technology was employed to observe the microstructure of H₂O/SO₃H-IL mixture with mass ratio of 1.5. As shown in Figure S2 in ESI, self-assembly micelles were formed between SO₃H-IL and H₂O. Non-uniformity of the micelles was probably induced by the local high temperature during the TEM detection. In addition, for the mixture of H₂O/SO₃H-IL with mass ratio of 1.5, micelles with hydrodynamic diameter around 0.72 nm was detected at room temperature through the dynamic light scattering (DLS) method (Figure S3, ESI). A possible mechanism for the formation of mesoporous organic polymers in H₂O/SO₃H-IL was proposed (Figure 2). Firstly, self-assembly micelles were formed in the mixture of

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 SO_3H -IL and H_2O , due to the coexistence of hydrophilic - SO_3H group and hydrophobic alkyl group in the structure of SO_3H -IL. The amine substrate (**A-1**) was mainly soluble and dispersed around the interface of SO_3H -IL and H_2O due to acidification of the amino group by - SO_3H group. Secondly, the polymerization reaction occured around the interface of the micelles under the catalysis of SO_3H -IL. Finally, after removing the SO_3H -IL and H_2O , the TB-functionalized organic polymers with mesopores derived from the H_2O cavity and micropores formed due to the rigid backbone structure were obtained.



Figure 2. Proposed mechanism for the formation of mesoporous TB-functionalized organic polymers in $\rm H_2O/SO_3H\text{-}IL$ mixture.

The synthesis of other Meso-TBPs was performed utilizing A-2 and A-3 as monomers, respectively, with a H₂O/SO₃H-IL mass ratio of 1.5 (for monomer structures and synthetic procedures, see ESI). They were obtained as brown powers, and insoluble in water and common organic solvents. N₂ sorption analysis together with the pore size distribution curves showed the existence of mesopores (3~15 nm) (Figure S4, ESI), together with BET surface areas of 368, 273 and 431 m² g⁻¹ for Meso-TBP-1, Meso-TBP-2 and Meso-TBP-3, respectively (for BET plot, see Figure S1, ESI). The porous morphology of Meso-TBP-2/3 was also observed by TEM and SEM (Figure S5, ESI). The whole X-ray photoelectron spectroscopy (XPS) spectra showed no signals for S (S2s: ~228 eV, S2p: 164~165 eV), and for CI (Cl2p_{3/2}: ~198.5 eV), demonstrating the complete removal of SO₃H-IL (Figure S6A, ESI). C1s XPS spectra (Figure S6B, ESI) varified the existence of aromatic C (284.5 eV), C-N bond (285.0 eV), a small quantity of C-O bond (286.4 eV) and trace carbon cation (288.9 eV) as the intermediate for TB formation. N1s spectra (Figure S6C, ESI) mainly showed the presence of C-N bond among the backbone (398.9 eV) and the residual unreacted -NH₂ group (399.6 eV). In the FTIR spectra (Figure S7, ESI), the TB structure was confirmed by the characteristic band of methylene group around 2925 cm⁻¹, C-N stretching vibration at 1180 cm⁻¹, aromatic ring in the range of 1450~1600 cm⁻¹. For Meso-TBP-3, respective band for C=C vibration was also observed at 1678 cm⁻¹. The presence of chemical shifts in crosspolarization magic-angle spinning (CP/MAS) ¹³C NMR spectra (Figure S8, ESI) at 125~146 ppm was attributed to the aromatic carbons in the backbone and those at 44 ppm to the methylene carbon, further confirming the formation of the TB-linked aromatic polymer. Powder X-ray diffraction (PXRD) pattern exhibited the amorphous nature of the polymer (Figure S9, ESI). The POPs with benzimidazole groups embedded among the backbone exhibited high CO₂ adsorption capacity.¹⁴ Herein, to

further expand the utilization of H_2O/SO_3H -IL as catalytic systems and reaction media for the preparation of mesoporous POPs, **A-4** and **C-1** was utilized as monomers for the synthesis of **Meso-BIMP** with a H_2O/SO_3H -IL mass ratio of 1.5 (for structures and synthetic procedure, see ESI). N_2 sorption analysis of **Meso-BIMP** showed obvious combination of type I and type IV modes (Figure S4A, ESI), and the pore size distribution curve showed the existence of mesopores in the range of 2~10 nm (Figure S4B, ESI), together with BET surface area of 423 m² g⁻¹ (for BET plot, see Figure S1, ESI). The porous structure of **Meso-BIMP** was also revealed by TEM (Figure S5, ESI). Therefore, the reaction system developed in this work was proved to be very efficient for the preparation of mesoporous POPs functionalized with TB or benzimidazole.

Subsequently, utilization of the resultant mesoporous POPs in heterogeneous catalysis was investigated. Development of novel methods for amine synthesis is of paramount importance in organic chemistry.¹⁵ N-Methylamines are key structure motifs in various biologically active natural compounds and hence are an integral part of tremendous pharmaceutical and agrochemical products.¹⁶ However, industrial production of methylamines still depends on the reductive amination using toxic formaldehyde.¹⁷ Hence, development of efficient methylation protocols using safer methylating agents, especially with stable and recyclable heterogeneous catalytic systems, is highly desirable. Although procedures using dimethyl carbonate, formic acid or CO2 as methylation regents have been investigated, drawbacks including the need of excess reducing agent (e.g. hydrosilane or hydroborane), higher temperature (>150 °C), high pressure of H₂ and operation under inert atmosphere severely hinder their applications in industrial production.¹⁸ An attractive methylation strategy is utilizing the readily available and inexpensive methanol as the methylating agent, with H₂O as the only byproduct. In this method, homogeneous catalytic systems based on Ir¹⁹ and Ru²⁰ complexes together with phosphine ligands and inorganic bases have been reported. However, heterogeneous catalysts are mainly metal oxides, requiring high temperature (>240 °C).²¹ Hence, development of highly efficient heterogeneous catalytic materials under mild reaction conditions is crucial for further industrialization of the methylamines production with methanol.

Herein, in order to integrate the advantages of high efficiency of homogeneous catalysts and easy recyclability of heterogeneous ones, mesoporous POP (e.g. **Meso-TBP-1**) was taken as the support for further metallization based on the significant coordination ability of TB functionality with metal complex.¹³ Ir(II)-metalated **Meso-TBP-1-1.5** (denoted as **Meso-TBP-1@Ir**) was obtained by treating the polymer with [IrCp*Cl₂]₂ in CH₂Cl₂ (for experimental details, see ESI). The content of Ir species immobilized onto **Meso-TBP-1** was 7.78 wt%, as determined by inductively coupled plasma-optical emission spectroscopy (ICP-OES) analysis. FTIR spectra of **Meso-TBP-1 @Ir** showed the existence of Cp* functionality (Figure S10, ESI). Then energy dispersive spectroscopy (EDS) profile obtained during the TEM observation indicated the presence of Ir metal species (Figure S11, ESI). Compositional EDS mapping of

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Meso-TBP-1@Ir exhibited homogeneous distribution of C, N, Ir elements (Figure S12, ESI). No aggregated Ir particles was observed by (HR)TEM image of Meso-TBP-1@Ir, probably due to the strong coordination ability of TB functionality towards Ir complex (Figure S13, ESI). XPS measurement was performed to investigate the coordination site and oxidation state of Ir species (Figure S14, ESI). BE of Ir4f7/2 in Meso-TBP-1@Ir was located at 62.3 eV, together with BE of 65.3 eV for $Ir4f_{5/2}$, which was similar with Ir(II) in the half-sandwich Ir-bipyridine derivatives reported in the literature (Ir4f7/2: 62.1 eV).22 Correspondingly, the BE of Cl2p_{3/2} was observed at 197.8 eV. Subsequently, the resultant Meso-TBP-1@Ir was applied in the methylation of amines using MeOH without any other additives. The methylation reaction of morpholine (1a) with methanol was selected to investigate the influence of various reaction parameters (For reaction parameters screening, see Table S1, ESI). Almost no Nmethylmorpholine (2a) was formed in the absence of any catalyst (entry 1, Table S1, ESI), while 89% yield of 2a was obtained at 110 °C within 24 h catalyzed by Meso-TBP-1@Ir without any other additives, which was even higher than that obtained over the corresponding homogeneous [IrCp*Cl2]2 (2a yield: 81%) under the same other conditions (Scheme 1). The yield of 2a was 83% and 89% respectively taking Meso-TBP-2@Ir and Meso-TBP-3@Ir as catalyst under the same reaction conditions. Notably, 2a yield was decreased to 63% using microporous TB-MOP (pore width: 1.69 nm) with the same skeleton as support using the same catalyst loading of Ir, indicating the advantageous mesoporosity of Meso-TBP-1-1.5 for mass transfer. Methylation products of other aliphatic secondary amines (2b-2e) were also obtained in moderate to high yields (60~94%) (Scheme S1, ESI). N, N-Dimethylbenzylamines with electro-donating (2f) or electro-withdrawing (2g) group on the benzene ring with yield of 78% and 95% were achieved, respectively. For aliphatic primary benzylamines with various functional groups on the benzene ring, dimethylation products with moderate yields in the range of 82~93% were observed for 2h-2k. While n-butylamine gave lower yield of the corresponding dimethylation product (21, 62%). Notably, 48% yield of Nethylmorpholine (2m) and 94% yield of N-isobutylmorpholine (2n) were obtained taking ethanol and isobutanol as the corresponding alkylation reagents. Based on the previous $\operatorname{reports}^{19\text{-}20}$ and results in this work, a possible reaction mechanism was also proposed (Scheme S2, ESI). In the repeating reactions, the catalytic activity of Meso-TBP-1@Ir was well retained after recycling the catalyst for five times (Figure S15, ESI). The XPS analysis on the recycled catalyst showed that the Ir species were still in the Ir(II) form (Figure S16, ESI). For leaching test, the heterogeneous catalyst was separated through centrifugation after the reaction being performed for 6 h (2a yield: 69%), and the liquid supernatant was detected by ICP-OES. It was demonstrated that Ir species were undetectable (<10 ppb) in the liquid phase and the yield of 2a kept constant after the reaction was further conducted for 24 h at 110 °C. All these indicated the excellent stability and activity of Meso-TBP-1@Ir as a heterogeneous catalyst for the methylation of amines with MeOH.



Scheme 1. Methylation of amines with methanol. ^a Reaction conditions: morpholine 1 mmol, **[Ir]** 3 mol%, MeOH 4 mL, 110 °C, 24 h. Yield of **2a** was determined by GC using dodecane as the internal standard.

In summary, mesoporous TB-functionalized POPs have been synthesized using sulfonic acid group functionalized IL both as catalyst and reaction medium together with H₂O. This aqueous acidic IL solution was also feasible in the preparation of mesoporous benzimidazole-functionalized POPs. The integration of TB ligands gave the polymer strong coordination ability with Ir(II) complex. The resultant **Meso-TBP-1@Ir** exhibited extraordinary catalytic performance in the N-methylation of amines using methanol together with high stability and easy recyclability. We thank the National Natural Science Foundation of China (Nos. 21673256, 21402208, 21403252, 21503239).

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Mesoporous Tröger's base-functionalized organic polymers were prepared using sulfonic acid group functionalized ionic liquid/H₂O system without any templates and surfactants.