Design of Porous Organic Polymer based Promising Molecular Nanoarchitecture in Advanced Heterogeneous Catalysis

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Abstract

Porous organic polymers (POPs) having very high surface areas, tunable pore volumes, excellent stabilities and controllable chemical composition have recently emerged a growing research interest as a most promising molecular nanoarchitecture for binding of metal or metal oxide nanoparticles (NPs) in the development of new advanced heterogeneous catalysts. Here in this featured article, we have focused on development of various kinds of POPs and immobilization of NPs in the framework of these POPs with their prospective employment as heterogeneous nanocatalysts to catalyze several catalytic reactions. These POP-based materials exhibit superior catalytic performance in coupling, hydrogenation and various acid catalyzed reactions with tremendous recyclability and combination of best features of both homogeneous and heterogeneous catalysts.

Keywords: Porous Organic Polymers; Heterogeneous Nanocatalysis; Rigid Framework; Heterogeneous Organocatalyst
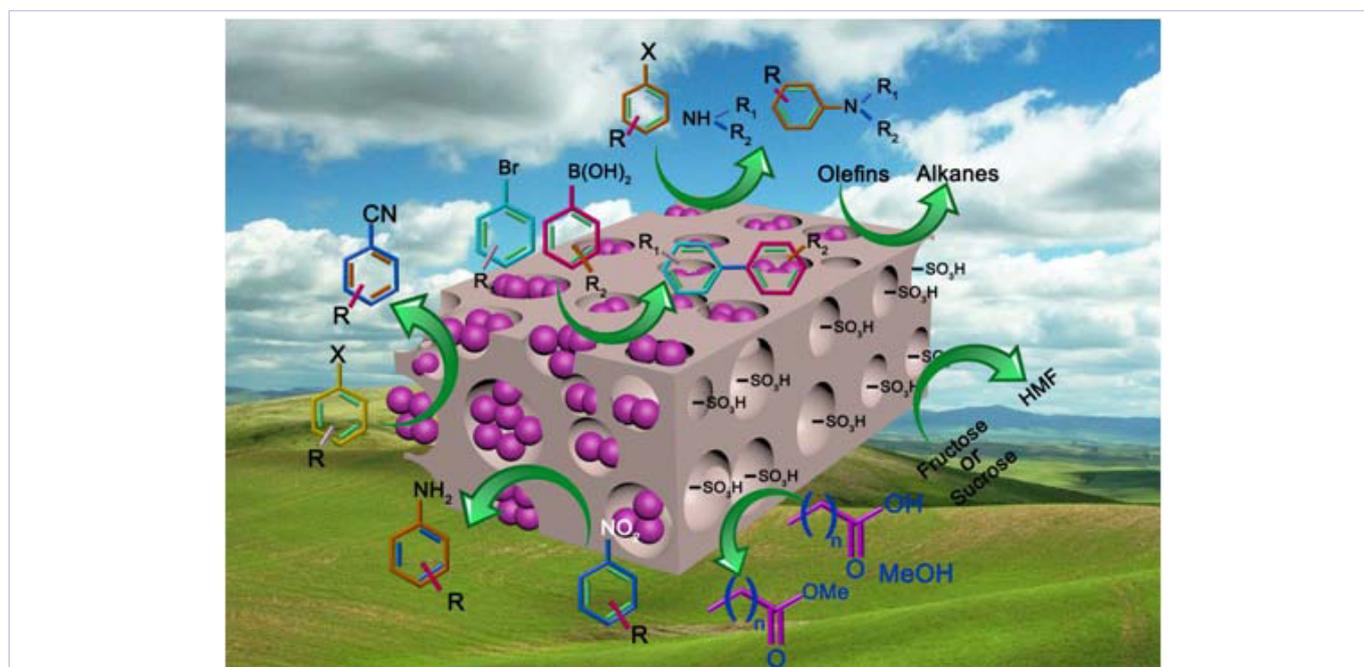
Introduction

The synthesis of Porous Organic Polymer (POP) support metal and metal oxides, it has received an increasing attention over the last decade in interdisciplinary research area for the development of new heterogeneous catalysts owing to their high chemical activity and wide-ranging applicability [1,2]. Generally, ultrafine metal NPs are very efficient in catalytic reactions than their bulk metal particles owing to their high surface to volume ratio so that they can provide a large number of available active sites per unit area for reaction substrates. But owing to their high surface energy, aggregation and thermally induced sintering of nanoparticles during catalytic reactions accompanied by a diminishment in catalytic activity always take place [3]. However, numerous obdurate problems including usual detachment of metal nanoparticles from the support and general tendency to grow or aggregate during the catalytic process are still associated which hamper the progress of this research area. To address these above mentioned problems, various molecular architecture for encapsulation or fabrication of NPs including mesoporous silica, zeolite, metal organic framework (MOFs) have been developed in recent years [4,5]. But among them Porous Organic Polymers (POPs) have been emerged a incredible degree of awareness as a most promising molecular nanoarchitecture for immobilization, fabrication, encapsulation followed by stabilization of ultrafine metal NPs because of their huge surface area with well-defined pore size distribution, high thermally and mechanically stable covalent bonds, low skeleton density, and presence of hydrophobic components in their framework which exhibits high catalytic activities and also high selectivity [6,7]. Spatial control of catalytically active sites in porous organic materials can be tuned and modified through various choice of organic building blocks and synthetic diversity, which provides them potential ability for catalysis [8,9]. Practical application of MOF (Metal Organic Framework) encapsulated NPs in heterogeneous catalysis remains yet unexplored owing to their moisture sensitivity, physiochemical instability in acids or bases and presence of metal and co-ordination bonds [10,11]. An additional stability and progressive catalytic activity of metal NPs in Porous Organic Polymer compared with the other catalytic supports is obtained owing to the interaction between organic linkers in POPs and metal NPs that can be readily enhanced by introducing coordination groups into metal precursors. In addition, easy recovery of these heterogeneous catalysts by simple filtration technique after the reaction and tremendous recyclability with negligible lose of catalytic activity have attracted a broad research interest in catalysis community for production of fine chemicals, value-added products in petroleum refining and chemical industry. The past decade has witnessed performance of various metal-catalyzed couplings, including Yamamoto, Suzuki-Miyaura, Sonogashira-Hagihara, Cu-catalyzed click reaction and Friedel-Crafts reactions, radical polymerization with AIBN for the preparation of such POPs [12-17]. To date, extensive efforts have been focused in the development of such POPs supported materials with facile fabrication or encapsulation of metal or metal oxide NPs on the external surface as well as in the interior cavity of those respective functionalized POPs. Recent reports in the literature demonstrate that heterogeneous catalysts for morphology-tuned aerobic oxidation [18] hydrogenation [19] coupling [20] cycloaddition reactions, [21] and

so forth can be developed through the fabrication and immobilization of metal NPs inside the nanoporous channel of POPs. Recently a number of research groups have reported many palladium NPs immobilized in imidazolium-based POPs used in advanced heterogeneous catalysis with high catalytic efficiency and recyclability. Wang, *et al.* discovered an interesting synthetic strategy for in situ formation of well-dispersed palladium NPs immobilized in imidazolium-based organic ionic polymers (OIP-1 and OIP-2) through Suzuki-Miyaura cross-coupling reaction of 1,3-bis(4-bromophenyl) imidazolium bromide with 1,3,5-tri(4-pinacholatoborolanephenyl) benzene in 1,4-dioxane and DMF for hydrogenation of nitroarenes to produce aniline derivatives at 25 °C [19]. They have found that Pd@OIP-2 offers higher catalytic activity and recyclability at least 6th competitive run without significant loss of catalytic activity compared with Pd@OIP-1 with their exposed Pd(111) crystalline facets making a better contact between substrate and catalytic active Pd-sites. They have also developed various palladium N-heterocyclic carbene polymers. They have synthesized poly-imidazolium particles with wire-shaped, spherical and ribbon-shaped morphologies by adjusting solvent polarity, solubility, concentration and temperatures. These morphologies remains unaltered after complexation of the particles with $Pd(OAc)_2$ to produce the corresponding ionic polymers containing palladium N-heterocyclic carbene units (NHCs) which showed morphology dependant catalytic activity in heterogeneous Suzuki-Miyaura cross-coupling reaction [22]. Wire-shaped palladium-NHC polymer exhibits excellent catalytic activity, probably due to larger contact areas with the reactants of wire-shaped particles. Due to superparamagnetic nature of Fe_3O_4, the newly developed material has recently emerged a growing attention as a novel magnetically separable catalyst. Recently, Wang *et. al.* also synthesized two spherical magnetic core-shell palladium-NHC particles (Fe_3O_4@PNP1 and Fe_3O_4@PNP2) by a one-pot reaction of tripodal imidazolium salts and $Pd(OAc)_2$ in the presence of magnetite nanoparticles [23]. These magnetically separable heterogeneous catalysts display outstanding catalytic activity and recyclability in Suzuki-Miyaura cross-coupling reaction at 25 °C in H_2O-EtOH mixture. Karimi's group recently developed a new protocol to synthesize another N-heterocyclic carbene-Pd polymers, which was found an efficient precatalyst for cyanation of various aryl halides by using non-toxic cyanic source [$K_4Fe(CN)_6$] under mild condition to produce corresponding benzonitrile derivatives [24]. Palladium NPs encapsulated 1,2,3-triazolyl containing porous organic polymers have been described by Wang's group [25]. These well designed CPP-C and CPP-Y polymers have been synthesized through click reaction and Yamamoto coupling reaction, respectively with the corresponding BET surface areas are 156 and 408 $m^2 g^{-1}$, respectively, and their total pore volumes are 0.19 and 0.25 $cm^3 g^{-1}$, respectively. Ultrafine palladium NPs are successfully incorporated in the interior cavities of these polymers to synthesize corresponding Pd@CPP-C and Pd@CPP-Y materials and its application as heterogeneous catalysts in hydrogenation of olefins to alkanes at 25 °C and 1 atm of H_2 was investigated and high efficiencies and selectivities were achieved. Furthermore, Pd@CPP-Y was reused for at least 7 catalytic runs without obvious activity loss. More re-

cently, another two click-based porous organic polymers (CPP-1 and CPP-2) are designed by click reaction of tetrakis-(4-ethynyl-phenyl) methane with 4,4'-diazidobiphenyl and tetrakis(4-azidophenyl)-methane with 4,4'-diethynylbiphenyl, respectively in the presence of copper sulfate and sodium ascorbate with very high BET surface areas 826 and 894 $m^2 g^{-1}$, respectively [26]. Ultrafine Pd-NPs with dual distributions and no apparent aggregation are successfully confined in the organic skeleton to develop Pd-based nanocatalyst in hydrogenation of nitroarenes at 25 °C and 1 atm of H_2 in ethanol medium with superior stability, recyclability without loss of catalytic activity and enhancement in activity compared with the palladium on charcoal. Mondal, et al. have reported the synthesis of a three-dimension benzene-benzylamine based porous organic polymer (BBA-1) network with a high BET surface area by adopting a novel strategy through Friedel-Crafts alkylation of benzylamine and benzene by using formaldehyde dimethyl acetal as a cross-linker and anhydrous $FeCl_3$ as a promoter [27]. Ruthenium Nanoparticles (Ru-NPs) anchored BBA-1 POP based materials (Ru-**A**, Ru-**B** and Ru-**C**) have been successfully synthesized through $NaBH_4$, ethylene glycol, and hydrothermal reduction routes. These newly developed materials exhibited excellent catalytic performance in room temperature catalytic transfer hydrogenation of nitroarene with very high conversion, selectivity, improved catalytic activity compared with the conventional catalysts such as Ru@C, $Ru@SiO_2$, and $Ru@TiO_2$ owing to the synergetic effect of nanoconfinement and electron donation offered by the 3D POP network. For the selective aerobic oxidation of hydrocarbons and alcohols to produce more functional compounds including aldehydes, ketones and imines with atmospheric air or molecular oxygen, several heterogeneous catalysts have been developed. New salen-(Mn)-based porous organic polymer (Mn-POP) with BET surface area 836 $m^2 g^{-1}$, and 3.4 nm pore dimension has been synthesized by Cao *et al.* employing palladium catalyzed Sonogashira-Hagihara coupling reaction of 1, 3, 5-triethynylbenzene and Mn-salen complex [28]. This newly designed Mn-POP was utilized as an efficient heterogeneous catalyst for olefin epoxidation with high activity and excellent selectivity to epoxide using O_2 as an oxidiser. The catalyst is easily separable and could be recycled at least five consecutive catalytic runs and no considerable drop in conversion and epoxide selectivity is observed during recycling. Morphology-tuned catalytic activity of POPs supported metal oxides has attracted considerable interest in recent time. Zhao *et. al.* have performed an extensive investigation on shape-controlled catalytic activity for Mn_3O_4 nanoparticles with sphere, nanowire and octahedron morphologies fabricated on divinyl benzene-triallyl amine (DVTA) POP in aerobic sp^3 C-H bond oxidation of aromatic hydrocarbons and alcohols [29]. The synthesis of a new class of divinyl benzene based Porous Organic framework with huge surface area 903 $m^2 g^{-1}$ and pore volume is 0.472 ccg^{-1} was achieved by Non-aqueous polymerization of a mixture of divinylbenzene and triallyl amine under hydrothermal conditions in presence of Azobisisobutyronitrile (AIBN) as the radical initiator. Moreover, Mn@DVTA-3 nanocatalyst having octahedron morphology exhibited exceptionally higher catalytic activity compared with Mn@DVTA-1 and Mn@DVTA-2 owing to more exposed (101)

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Figure 1: Schematic presentation Porous Organic Polymer (POP) based molecular Nanoarchitecture used in Advanced Heterogeneous Catalysis.

Table 1: Catalytic activity of various porous organic polymer supported metal NPs and acid fuctionalized microporous polymers.

Materials	Reaction conditions	Application in catalysis	Reference
Pd@OIP-1 and Pd@OIP-2	NaBH4, H2O/THF (9:1), 25 oC, 1.5 h	hydrogenation of nitroarenes to aniline derivatives	19
PIS-Pd	K2CO3, H2O/DMF, 100 oC, 3 h	Suzuki-Miyaura cross-coupling reactions	22
Fe3O4@PNP1 and Fe3O4@PNP2	25 oC in H2O-EtOH mixture	Suzuki-Miyaura cross-coupling reactions	23
NHC–Pd polymer	[K4Fe(CN)6], DMF, K2CO3, 95 oC, 12 h	cyanation of various aryl halides	24
Pd@CPP-C and Pd@CPP-Y	25 oC, 2 h, 1 atm of H2	hydrogenation of olefins to alkanes	25
CPP-1 and CPP-2	25 oC, 1 h, 1 atm of H2 in ethanol medium	hydrogenation of nitroarenes	26
Ru-A, Ru-B and Ru-C	NaBH4, THF/H2O (1:3), 25 oC	catalytic transfer hydrogenation of nitroarene	27
Mn-POP	Isobutyraldehyde, O2	olefin epoxidation	28
Mn@DVTA-1, Mn@DVTA-2 and Mn@DVTA-3	TBHP, 1 atm O2, 80 oC	aerobic sp3 C-H bond oxidation of aromatic hydrocarbons and alcohols	29
PDVB-SB-Cu	Cs2CO3, 115 oC, 16 h	Ullmann biaryl ether coupling of iodobenzene and phenol	30
Cu-A, Cu-B	Cs2CO3, 110 oC	Ullmann coupling of aryl halides with primary and secondary amines	31
SPPPOP-1, SPPPOP-2, SPPPOP-3	25 oC	Esteriifcation and transesterification reactions	32
HMP-1-SO3H	25 oC	Esteriifcation and transesterification reactions	33
SPPTPA-1	Microwave (140 oC), 20 min	conversion of carbohydrates to HMF	34

crystallographic facets in imine production using 1 atm pressure of molecular oxygen. This DVTA-POP based catalyst possess extraordinary thermal and chemical stability providing accessible coordinating sites for uptake of metal ions or metal nano-particles thereby making them an attractive candidate as new ideal platform in heterogeneous catalysis community. The synthesis of an efficient and stable Cu-incorporated porous Schiff base-modified polydivinylbenzene (PDVB-SB-Cu) has been reported which

was obtained by performing multiple steps reactions include nitration of benzene ring, reduction of nitro group, Schiff-base condensation, and followed by coordination with Cu(OAc)$_2$ preserving its porous framework [30]. The heterogeneous PDVB-SB-Cu catalyst exhibited high catalytic activity in the Ullmann biaryl ether coupling of iodobenzene and phenol similar to that of the homogeneous Schiff base-stabilized copper catalyst with remarkable recyclability. Zhao, *et al*. have newly developed heteroge-

neous Cu^o NPs supported on porous organic polymer (Cu-B) catalyst for carrying out Ullmann coupling of aryl halides with primary and secondary amines in aqueous solution [31]. Cu-B showed higher catalytic performance compared with Cu-A catalyst which was synthesized by chelate-assisted co-assembly strategy using Cu(acac)$_2$, soluble phenol formaldehyde resin and poly (ethylene oxide)-block-poly (propylene oxide)-block-poly-(ethylene oxide) triblock copolymer Pluronic F127 due to higher pore volume which is advantageous for access of reactants, and the hierarchical porosity is also favourable for easy mass transfer of reactants and products. The versatile substrate scope of this catalytic system with high product yield, high selectivity, and high mechanical stability of the polymer under the harsh reaction condition with no leaching of catalytic active centers proved this system very attractive in nature. Porous Organic Polymer was employed as a potential heterogeneous solid metal free organocatalyst which was developed with the introduction of active functional groups in the polymer framework by post-synthetic modification or precisely adjusting polymer matrix by selection of monomer. Recently, Bhaumik's group have rationally designed a series of new microporous organic polymers through Friedel-Crafts alkylation reactions and one-step oxidative polymerization of triphenylamine in presence of anhydrous FeCl$_3$. Friedel-Crafts alkylation reaction between pyrene and bis(1,4-dibromo-methyl)benzene at their different molar ratios in presence of FeCl$_3$ has been adopted to synthesize a series of POPs namely PPOP-1, PPOP-2, and PPOP-3 [32]. All these pyrene based POP materials provide BET surface areas 615, 570 and 508 m^{2}g^{-1}, respectively. Same strategy was again followed for synthesis of a new hypercrosslinked supermicroporous polymer (HMP-1) with a BET surface area of 913 m^{2}g^{-1} via FeCl$_3$ assisted Friedel-Crafts alkylation of carbazole and α,α′-dibromo-p-xylene [33]. Nanoporous polytriphenylamine (PPTPA-1) polymer with exceptionally high surface area about 1437 m^{2}g^{-1} has been synthesized by one-step oxidative polymerization of triphenyl amine in presence of anhydrous FeCl$_3$ [34]. All these microporous organic polymers were further functionalized with –SO$_3$H groups to produce highly efficient acidic heterogeneous organocatalysts by reaction with a strong oxidizing reagent, ClSO$_3$H. Temperature programmed desorption of NH$_3$ (NH$_3$-TPD) measurement signifies high loading of –SO$_3$H group in the porous framework. The organocatalysts exhibited high catalytic performance in production of biofuel and HMF. The performances of these solid sulfonated porous polymers PPOP acid catalysts were evaluated in the context of biodiesel synthesis via esterification/transesterification of long chain fatty acids/esters at room temperature. Similarly transesterification reaction of long chain fatty acids with methanol for biodiesel production with the corresponding product yield 93-99% was performed employing HMP-1-SO$_3$H carbocatalyst. On the other hand, sulfonic acid functionalized PPTPA-1 (SPPTPA-1) catalyst was employed in catalytic conversion of carbohydrates to HMF which is a key intermediate of many industrially important chemicals and liquid fuels. All these aforementioned solid POP based organocatalysts exhibit outstanding recyclability and stability in the reaction conditions thereby offering useful alternatives suppressing the use of toxic homogeneous Lewis acid catalysts.

In this article, in a nutshell we have discussed the employment of porous organic polymers and various metal or metal oxide nanoparticles fabricated in porous organic polymers as heterogeneous and also metal free organocatalyst in performing various catalytic reactions.

An increasing interest has been paid for the development of POPs with high density of functionalities for choosing an efficient platform for loading active metal or metal oxide NPs in their framework due to their high surface area, suitable pore volume, chemical and thermal stability, strong binding ability and high catalyst loading. All the heterogeneous catalysts become a promising candidate in coupling reactions, hydrogenation reactions and various acid catalyzed reactions for production of fine chemicals, value-added products in petroleum refining and chemical industry. The versatile nature of Porous Organic Polymers (POPs) will make them attractive candidate in heterogeneous catalysts community addressing all the profound challenges in near future. Therefore researchers are keener in finding some sustainable, easy and low-cost methodologies for large scale preparation of such Porous Organic Polymer based materials that can be easily channelized in the design and development of novel heterogeneous catalysts.

Acknowledgement

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