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Catalytic Zirconium/Hafnium-Based Metal–Organic Frameworks

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ABSTRACT: Metal–organic frameworks (MOFs) are highly versatile materials that find applications in several fields. Highly stable zirconium/hafnium-based MOFs were recently introduced and nowadays represent a rapidly growing family. Their unique and intriguing properties make them privileged materials and outstanding candidates in heterogeneous catalysis, finding use either as catalysts or catalyst supports. Various techniques have been developed to incorporate active species into Zr-MOFs, giving rise to catalysts that often demonstrate higher performances or unusual activity when compared with their homogeneous analogues. Catalytic functions are commonly incorporated at the zirconium-oxide node, at the linker, or encapsulated in the pores. Representative examples are discussed, and advantages in adopting Zr- and Hf-MOFs in catalytic applications are highlighted.

KEYWORDS: *metal–organic framework, zirconium, hafnium, catalysis, node, linker, encapsulation*

INTRODUCTION

Heterogeneous catalysis has played a prominent role in the development of abiotic catalytic systems of practical relevance. Currently, the most successful heterogeneous catalysts span from oxide materials such as aluminas, aluminum silicates, or zeolites to various forms of supported metals such as metal nanoparticles, metal oxides, or organometallic complexes.[1−4] In the field of heterogeneous catalysis, there is a need for well-defined, highly tunable catalysts that are also stable and easily attainable. Other relevant properties of heterogeneous catalysts include high surface area, crystallinity, and uniformity. In fact, the majority of heterogeneous catalysts lack structural uniformity and contain multiple kinds of active sites, potentially differing in both reactivity and selectivity. As a result, significant effort must be devoted to their characterization and, in particular, to the identification of the most catalytically relevant species. For these reasons, unambiguous conclusions regarding catalyst-structure/activity relationships in heterogeneous catalytic systems can, at times, be difficult to reach. In the past two decades, highly porous metal–organic frameworks (MOFs)[5,6]—crystalline and self-organizing reticular structures composed of metal-based building units sharing polytopic organic linkers—have emerged as promising materials for applications in heterogeneous catalysis. MOFs have great potential both as catalyst supports and as catalysts themselves, because they can display many of the properties of an ideal heterogeneous catalyst, the most notable being crystallinity, active-site uniformity, high surface area, and porosity.

Undeniable virtues of MOFs—and ones that are lacking in typical porous carbons and various other solid supports—are their meso- and/or molecular-scale structural periodicity and their *atomically* precise crystallinity. These features are enormously useful for X-ray-based structural characterization, including probing the structures of active sites and therefore making MOFs amenable to predictive computational modeling.[7] In addition, mesoporous MOFs, when compared to purely microporous materials such as various zeolites for instance, facilitate faster transport (diffusion) of molecular reactants and products, making these materials attractive alternatives to conventional heterogeneous catalysts and catalyst supports for liquid-phase reactions.[8,9]

Metal–organic frameworks (MOFs) have been explored for a broad range of catalytic transformations (albeit, chiefly in the condensed phase) as evidenced by an impressive and growing number of reports.[7,10−34] Interestingly, solid MOF-based catalysts often display activity that is higher than the corresponding homogeneous catalysts; indeed, examples have been reported for a broad range of reactions including condensation, ring opening, N-methylation, isomerization, hydrogenation, and oxidation reactions.[18] It is also noteworthy that MOF-based materials have been found to be catalytically competent for the production of certain fine chemicals.[35]

In the past few years, zirconium-based MOFs (Zr-MOFs) have generated disproportionately high interest and, as a consequence, have found proof-of-concept application in several areas, including sensing,[36−79] chemical separations,[80−102] gas storage,[103−118] in vivo drug delivery and therapeutic treatment,[119−132] toxic analyte[133−161] and

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gas[162−190] capture, and chemical and biochemical catalysis.[191−463] Most Zr-MOFs feature Zr_6O_8 clusters as nodes, coordinated to carboxylate-terminated organic linkers. (The combination of core cluster, carboxylates, charge-balancing protons (on node oxygen ions), and, if needed, aqua ligands, define secondary-building units (SBUs) for the materials.) While less common and less well-known, several examples of hafnium(IV) analogues of Zr(IV)-containing MOFs exist. Measurable properties, such as node aqua and hydroxo ligand pK_a values are similar for hafnium versus zirconium MOFs.[464] For simplicity, we will normally refer to compounds as Zr-MOFs even if hafnium analogues are known. The prototypical Zr-MOFs are those in the UiO-family (UiO is University of Oslo), a class of 12-connected MOFs featuring **fcu** topology.[465] By varying the geometry and symmetry of the organic linker, a large library of Zr-MOFs with varying node connectivity (Figure 1)[466] and framework topology (Figure 2)

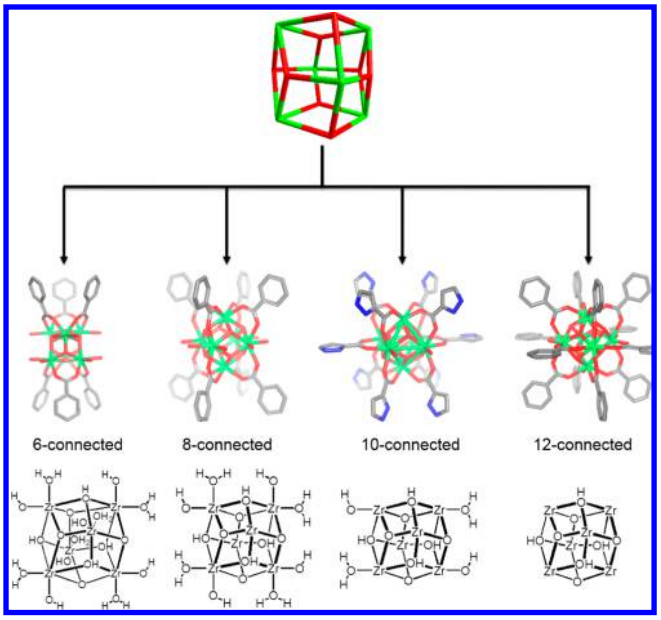

Figure 1. Node connectivity and proton topology in zirconium-based MOFs. Proton topologies are known for 8-connected and 12-connected Zr-nodes, whereas proton topologies of 6- and 10-connected nodes as shown in Figure 1 represent only putative structures.

can be obtained. (The "12" in 12-connected refers to the number of linker-terminating functional groups coordinated to a single node, not the number of bonds formed between the node and surrounding linkers or nonstructural ligands.)

Zr-MOFs comprising 12-, 10-, 8-, or 6- connected Zr_6O_8 clusters (Figure 1) have been reported with di-, tri-, and tetratopic organic linkers. However, it is common for nominally 12-connected frameworks to contain defects[467,468] (missing nodes and/or missing linkers that decrease the actual connectivity), which introduce inhomogeneity in the crystalline structure (Figure 3) as well as terminal −OH and $-OH_2$ groups on the node (typically one aqua, hydroxo pair for each absent carboxylate).[468,469] (Even without defects, 12-connected hexa-zirconium(IV) nodes present four μ_3−OH groups per node. Note that the four bridging hydroxyls contribute four of the oxygen atoms present in the Zr_6O_8 node core. As such, they are highly resistant to displacement or substitution, although deprotonation is possible. A typical μ_3−OH pK_a value for a Zr-MOF is 3.4—weakly acidic.)

Terminal (i.e., nonbridging, nonstructural), hydrogen-bonding-competent −OH and $-OH_2$ groups can also be introduced by design—specifically, by intentionally targeting structures based on 10-, 8-, or 6-connected nodes. As discussed further below, the terminal hydroxo, and especially aqua, sites are somewhat labile, making them susceptible to displacement by intentionally added, nonstructural ligands, including chromophoric, hydrophobic, luminescent, redox-active, acidic, and basic ligands.[166,202,399] As discussed below, the sites are also reminiscent of Lewis and/or Brønsted acidic or basic sites prevalent on the surfaces of conventional metal-oxides, but with the distinction in MOF materials of being periodically sited, and being structurally and stoichiometrically well-defined.

There are a few examples of Zr-MOFs containing nodes other than Zr_6O_8. Among them are PCN-221[191] (PCN is porous coordination network), which is based on Zr_8O_8 units, and the Zr-MIL-140 series, which features monometallic ZrO_7 units.[470,471] In addition, Zr-MOFs have been prepared with linkers other than carboxylates such as phosphonate[180,472,473] and phenolate[474,475] with monometallic ZrO_6 and ZrO_8 units, respectively.

Zr-MOFs are characterized by exceptional thermal,[465,470,476,477] chemical,[475,476,478,479] and mechanical[470,480−482] stability properties that are important for their practical application,[483,484] and particularly in heterogeneous catalysis. The catalytic benefits of high thermal stability were first shown in an investigation with vanadium-metalated (defect-site metalated) UiO-66 (V-UiO-66).[192] This catalyst was found to withstand temperatures as high as 350 °C, even under oxidizing conditions (O_2-based oxidative dehydrogen-

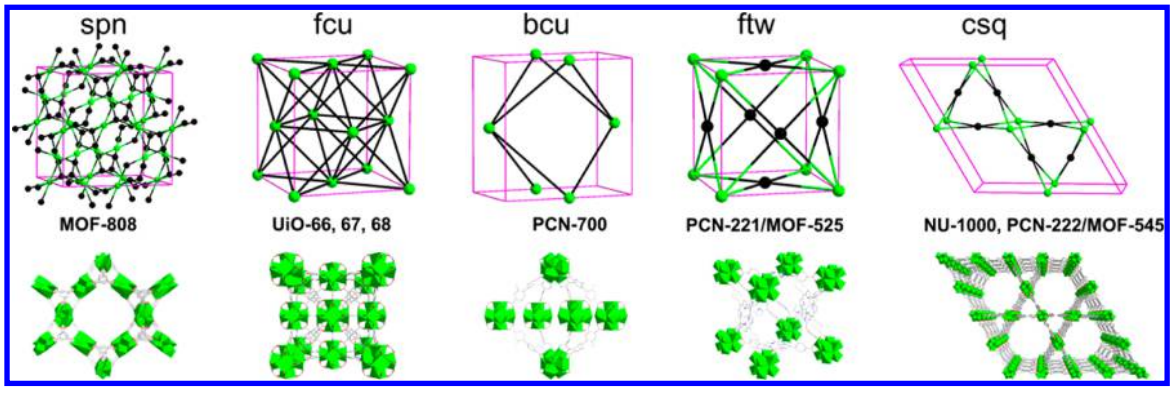

Figure 2. Schematic representation of selected Zr-MOF topologies.

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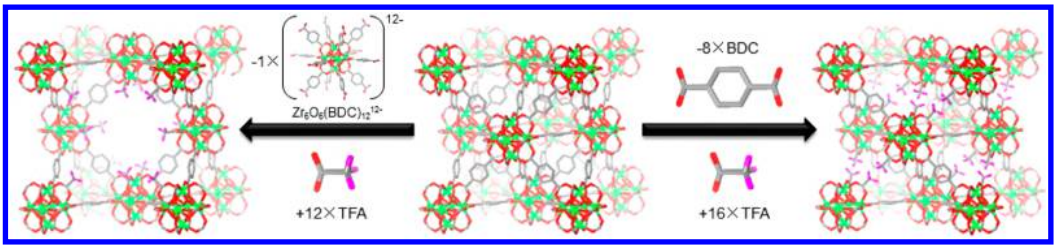

Figure 3. Nature of defects on a 12-connected Zr-MOF (UiO-66): missing node (left) and missing linker (right).

ation of cyclohexene). The significance of this study is not in the particulars of the reaction examined but in the demonstration that MOF-based catalyst supports can withstand fairly demanding heterogeneous catalysis conditions. While it is tempting to extrapolate and assume that MOF-supported heterogeneous catalysts, capable of withstanding 350 °C in air, can make the leap from lab to commercial utilization, long-term (i.e., industrially relevant) stability has yet to be proven. The real significance of the lab-scale demonstrations of high thermal and structural stability, including postcatalysis characterization, is in demonstrating that the catalysts and structures studied ex situ are the same as those present under operando conditions. In turn, these circumstances set the stage for hypothesis-driven research on catalyst structure/activity/mechanism relationships and for predictive and explanative computational studies that probe the actual catalysts under study, rather than idealized precatalyst structures. To the extent that these conditions are satisfied (i.e., catalysts present as uniform, well-defined, and temporally stable arrays of clusters or related moieties), we can expect mechanistic and transferrable catalyst design rules to emerge from studies of MOF-supported heterogeneous catalysts for gas-phase transformations.

Zr-MOFs—owing to their chemical stability—have also been shown to be stable in challenging acidic and basic (condensed phase) environments; such environments may be necessary for catalytic tests or to obtain catalytically active MOFs or MOF-supported species. Recent examples of chemical stability and robustness for Zr-MOFs include the use of (i) H_2SO_4 to install active sites within frameworks,[193] (ii) H_2O_2 to perform selective oxidation of a linker,[194] and (iii) basic conditions to facilitate catalytic electro-oxidation of water (pH 10), as well as the activation of MOF-based precatalysts using aggresive reagents such as MeLi, $NaBHEt_3$, or KO^{t}Bu.[196−198]

Zr-MOFs are typically synthesized de novo by solvothermal methods. However, in some cases, incorporation of catalytic units or other moieties cannot be accomplished by direct synthesis. Fortunately, alternative techniques have been developed for postsynthetic incorporation[231] of catalysts including solvent-assisted ligand incorporation (SALI),[166] solvent-assisted ligand exchange (SALE) and also known as postsynthetic ligand exchange (PSE),[485,486] atomic layer deposition in MOFs (ALD in MOFs - AIM), solvothermal deposition in MOFs (SIM),[487] and catalyst encapsulation (incorporation of noncovalently bound species into framework pores).[23]

In this Perspective, we have chosen to focus mainly on the use of Zr-MOFs as catalysts or catalyst supports. The active site may be at the zirconium *node*, at the *linker*, or a separate chemical entity located in the *void* space of the channels and/or pores (Figure 4). Selected examples are organized according to the location of the active sites, and the significance of each example is outlined. The detailed synthesis and characterization

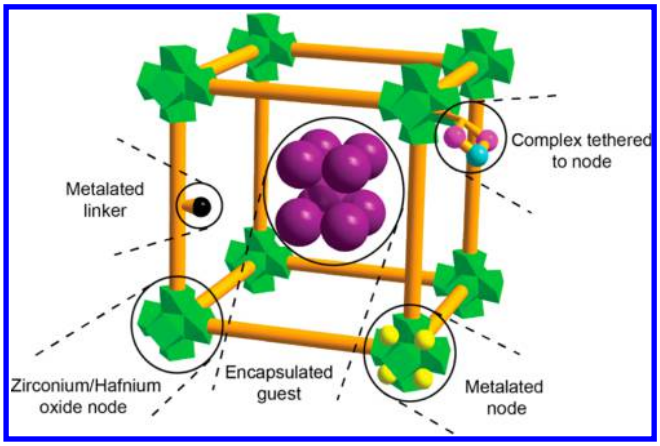

Figure 4. Location of catalytically active sites on zirconium/hafnium oxide-based MOFs.

of the relevant Zr-MOFs is outside the scope of this Perspective. Instead, we have emphasized the approaches and techniques, for catalyst preparation, as well as the activity and selectivity of the resulting catalytic material.

CATALYSIS AT THE ZIRCONIUM OXIDE NODE

When considering the use of MOFs as catalysts, it is common to make use of the metal oxide node component of the framework as the active site.[488] The nature of the oxide cluster node dictates catalytic activity, and its variable coordination environment allows for a high degree of catalytic tunability. MOFs composed of "Zr_6-oxide" cluster nodes with varying node connectivity (Figure 1), electronic environment, and framework topology (Figure 2) have been broadly studied for catalytic applications and primarily employed as Lewis acid catalysts.[199−204] For simplicity, we will describe nodes as metal oxides even if they include hydroxy and aqua ligands. Both $-OH_2$ and terminal $-OH$ groups, present on nodes either as a consequence of structural defects[205,376,382,489] or by intentional design, are generally both substitutionally and thermally labile (although temperatures as high as 230 °C may be required).[204,465] Terminal hydroxo ligands thermally exit as water molecules by recruiting protons from bridging hydroxo ligands, giving easy access to Lewis acidic Zr(IV) metal centers.

In the ideal case, the Zr-based MOF, UiO-66 (Figure 3), is composed of 12-connected Zr_6-oxide nodes,[465] meaning that the most desirable Lewis acidic catalytic sites are occupied by structural linkers. It is well-known, however, that the structure of UiO-66 is rarely ideal and that the identity and concentration of the modulator used in the synthesis of this framework and its derivatives dictates the quantity and identity of defects present in the final structure.[206,489,490] (A modulator is a linker-competitive, nonstructural ligand that serves to slow the growth of a MOF and facilitate the reversal of coordination "mistakes"

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so that crystalline compounds can be obtained. Under certain circumstances, modulators can also take the place of some fraction of linkers—rendering, for example, materials that are nominally 12-connected (e.g., UiO-66) only 10, 9, or even 8 connected. Modulators are particularly effective for controlling the growth of MOFs featuring Zr(IV) and oxy-anion terminated linkers. Zr−O bonds are among the strongest known ionic bonds; nevertheless, the barrier to ligand substitution (linker for modulator; modulator for linker, etc.) is unusually low. These are precisely the requirements for effective control of formation and growth of crystalline MOFs.)

It has been demonstrated that by controlling the concentration and ratios of two modulators, hydrochloric acid (HCl) and trifluoroacetic acid (TFA), the number of defects and hence the catalytic activity of UiO-66 toward the Lewis acid-catalyzed transformation of citronellal to isopulegol can be enhanced.[206] The presence of defects in UiO-66 has also been shown to give rise to promising activity for the catalytic hydrolysis of phosphonate ester bonds[207−209]—a reaction which is important for the degradation and detoxification of nerve agents and their simulants.[210] Given that accessibility of Zr(IV) sites contributes to Lewis acidic catalytic activity of the MOF, 8-connected (NU-1000, NU is Northwestern University)[195] and 6-connected (MOF-808)[211] Zr-based MOFs were studied for hydrolysis of the nerve agents (GD, VX) and their simulant dimethyl 4-nitrophenyl phosphate (Figure 5).

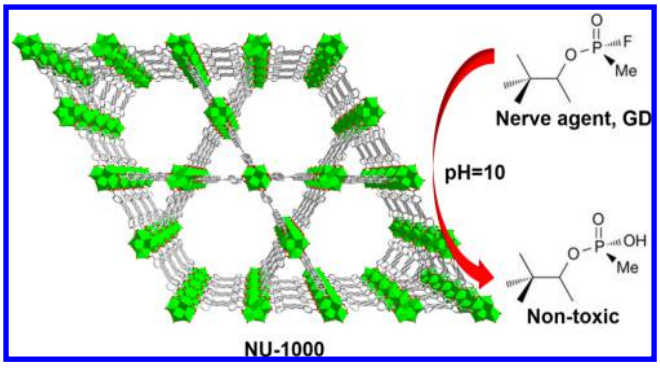

Figure 5. Hydrolysis of GD is catalyzed by NU-1000, an 8-connected Zr-MOF.

Significantly enhanced hydrolysis rates were observed for the 8-connected and 6-connected MOFs with half-lives for DMNP hydrolysis of 15 min and <0.5 min respectively (compared to 50 min for "close to defect-free" UiO-66 and 35 min for defective UiO-66). In addition to lowering node connectivity, removal of the terminal −OH and $-OH_2$ ligands from the 8-connected node of NU-1000 via thermal treatment can also be used to increase catalytic activity and the half-life for DMNP hydrolysis can be reduced to 1.5 min.[195] It is important to note that upon dehydration, the zirconium oxide node undergoes structural rearrangements involving changes in the Zr−Zr distances but preserving the larger framework structure, as demonstrated by EXAFS (extended X-ray absorption fine structure spectroscopy) analysis on UiO-66[465] and by PDF (pair distribution function) analysis on UiO-66 and NU-1000.[491] The structural change, in turn, greatly diminishes the affinity of the node for water ligands. With the hydrated version of NU-1000, the rate-determining step in the hydrolysis reaction is the substitutional displacement of an aqua ligand by the phosphoryl oxygen atom of the nerve-agent simulant. For the dehydrated and structurally rearranged version of the catalyst, water is barely ligated and, therefore, is much more readily/rapidly displaced by the agent or simulant. Interestingly, related rearrangements are known for hydrated zirconia, but they occur at much higher temperature than in UiO-66 and NU-1000. The temperature difference is illustrative of the kinds of enhancement in chemical reactivity that can be anticipated when bulk materials are reduced to minimalist clusters.

In addition to Zr(IV) site accessibility, electronic effects imposed on the Zr_6-oxide node have also been shown to affect Lewis acidic catalytic activity.[212] For example, the presence of electron-withdrawing groups on the structural organic linkers of UiO-66 (Figure 6) was shown to enhance the MOFs activity for the conversion of citronellal to isopulegol.[212]

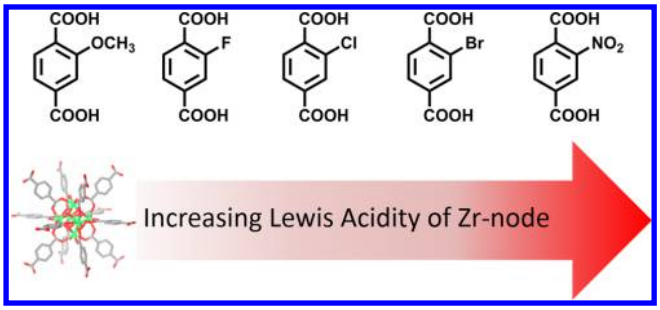

Figure 6. Electron-withdrawing groups on the linker influence the Lewis acidity of the Zr-node.

This increase in catalytic activity was attributed to an increase in Lewis acidity of the Zr(IV) sites as a result of the electron-withdrawing groups on the linker. As well as electronic effects, structural organic linkers can also affect the local chemical environment around the Zr_6-oxide node. A compelling example is the activity of UiO-66-NH_2 versus UiO-66 for the catalytic hydrolysis of the nerve agent simulant DMNP.[213] The addition of a basic ($-NH_2$) moiety proximal to the node shortens the reaction half-life from 35 min to just 1 min. Inspiration for the catalyst modification came from the mechanism employed by enzyme phosphotriesterase to catalytically hydrolyze phosphonate esters. Key to its high activity is the presence of histidine and aspartate residues (proton acceptors) near substrate-activating zinc ions. Sustained hydrolysis within the enzyme appears to be facilitated by proton relay steps involving the two bases.[214] Whether a similar effect is responsible for the remarkable enhancement of activity by pendant NH_2 groups is unclear. What is clear is that second-sphere effects in MOF-catalyzed reactions can be large and favorable. Their systematic exploration no doubt will be an important element of investigations going forward.

Although less commonly reported, Zr-oxide (and to a slightly greater extent, Hf-oxide) MOF nodes can also act as Brønsted acid catalysts.[215] Similar to the Lewis acidic sites, the Brønsted acidic sites also arise primarily from low node connectivity or structural defects resulting in Zr/Hf−OH and Zr/Hf−OH_2 active sites where the hydroxide or water ligands can act as proton donors. The Brønsted acidity as well as the quantity of these node-based protons can be measured by potentiometric acid−base titrations. As one might anticipate, distinctly different pK_a values are obtained for chemically distinct protons (i.e., μ_3−OH, − OH_2, and − OH).[464] Despite the high density of protons (four on each node of defect-free UiO-6X; 16 on each node of NU-1000; etc.) no evidence has been found

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experimentally for polyelectrolyte-type behavior. Thus, the pK_a values of the four μ_3−OH ligands of UiO-6X are essentially identical as are the pK_a values of the four aqua ligands of NU-1000. One possible explanation is that the preponderance of carboxylate anchoring groups at the node serves to electrostatically screen acid or conjugate-base sites from each other.

Returning to chemical catalysis, a study of an extended series of Zr- and Hf-based MOFs (UiO-66, UiO-67, PCN-57, NU-1000, and MOF-808) featuring varying numbers of node-based Brønsted acid sites—either by design or as a consequence of structural defects—showed that their efficacy as Brønsted acid catalysts for styrene oxide ring-opening (to yield a secondary alcohol) varied with the number of highly acidic sites presented.[216] Hf-NU-1000 was also shown to be an effective catalyst for the Brønsted acid catalyzed CO_2 insertion into epoxides to give cyclic carbonates under very mild conditions.[217]

Superacidity has been engendered in a Zr-MOF, MOF-808, simply by soaking the porous material in aqueous sulfuric acid. Sulfate was found to replace nonstructural formate ligands remaining from the MOF synthesis. By varying the concentration of sulfuric acid in the soaking solution, the 6-connected node of MOF-808 could be loaded with 0.65, 1.3, 2.3, or 2.5 sulfates (where the theoretical maximum loading should be 3). MOF-808-xSO$_4$ was tested as a catalyst for the cyclization of citronellal to isopulegol. The selectivity of this reaction is typically highly sensitive to the Lewis/Brønsted acidity of the catalyst and, indeed, with MOF-808-xSO$_4$, the selectivity for isopulegol systematically decreases with increasing sulfate loading, suggesting that higher sulfate loadings lead to a more strongly Brønsted acidic MOF. Furthermore, conversion of α-pinene to camphene and limonene was nearly quantitative using MOF-808−2.5SO$_4$ as a catalyst, illustrating the superacidic nature of the MOF.[193] While these observations are both exciting and intriguing, the greatest value of these catalysts may be in providing uniquely well-defined microcosms of sulfated zirconia, a widely used but rather poorly atomically defined support material. Well-defined analogues, such as MOF-808-xSO$_4$, NU-1000−2SO$_4$, and UiO-66−4SO$_4$, should provide a basis for high-quality, predictive computational modeling and understanding of not only the catalytic activity of sulfated zirconia but also its exemplary behavior as an activity-enhancing support material for a variety of transition-metal catalysts. Finally, while the ability of hexa-zirconium(IV) nodes to bind other oxyanions has recently received attention in the context of purification of effluent water, little, if anything, has been done yet to explore the utility of these alternative ensembles as catalysts or catalyst supports.

CATALYSIS AT THE METALATED NODE

Apart from being used as Brønsted acid functions, the −OH and −OH$_2$ groups on the Zr-cluster node also provide sites for anchoring metal ions to give structurally well-defined catalysts. In addition, because nodes are separated/isolated by linkers, aggregation of the node-supported metal ions is prevented or limited, giving rise to uniform active sites.

Only a few examples of metals grafted on zirconium nodes have been reported, the most explored synthetic route being metalation in the solution phase (solvothermal deposition in MOFs (SIM)).[192,205,218−224] The reported examples include the use of common and readily available halide metal precursors or organometallic complexes bearing ligands that can be easily protonated (including Grignard reagents[492]). UiO-66 was metalated with the vanadium complex VO(acac)$_2$ (acac is acetylacetonate) to give a catalyst that is stable at high temperature and active in oxidative cyclohexene dehydrogenation,[192] and similarly, exposure to $CrCl_3(THF)_3$ (THF is tetrahydrofuran) has been shown to lead to node metalation of UiO-66 and UiO-67.[205] Interestingly, it has been proposed that the Cr concentration in UiO-67—a nominally 12-connected MOF—is related to the defect density in the framework. Similarly, the Zr-node of PCN-700 was functionalized with metal halides to obtain PCN-800(M) (M=Ni, Co, In).[223] In this example, the metal incorporation on the nodes of PCN-700 was elucidated using single-crystal X-ray diffraction (SCXRD) because metalation occurs via a single crystal to single crystal transformation. Both Cr-UiO-67[205] and PCN-800(In)[223] were employed in acetaldehyde trimerization, a transformation of industrial relevance, the former showing activity and selectivity comparable to the homogeneous catalyst $CrCl_3(THF)_3$, and the latter demonstrating conversion of 76% and selectivity up to 99% within 2 h.

Lin et al. reported the postsynthetic metalation of Zr-UiO-68 and Zr/Hf-MTBC (MTBC is methane-tetrakis(p-biphenylcarboxylate)) via deprotonation with n-buthyl lithium of the bridging hydroxyls followed by reaction with cobalt and iron halides to obtain species able to catalyze broad scope hydrogenation reactions[218] and C−H functionalizations.[219] Interestingly, cobalt hydride species installed on UiO-type MOFs showed chemoselectivity for the functionalization of the less-hindered C−H bonds, as a result of the steric confinement imposed by the MOF environment to the metal species.[219]

Chemistry and reactivity developed on metal (and semimetal) oxide supports such as MgO, ZrO_2, and SiO_2 has been transferred successfully to Zr-MOFs as supports. For instance, [Ir(acac) $(CO)_2$] and [Ir(acac)$(C_2H_4)_2$] were reacted with the Zr-nodes of UiO-66 and NU-1000 to give isolated iridium species.[222] The grafted iridium species proved to be catalytically competent for ethylene hydrogenation, giving TOFs (turnover frequencies) of 0.010 s^{-1} and 0.017 s^{-1} and selectivity of 99.5% and 98.5% with NU-1000 and UiO-66, respectively (Figure 7).

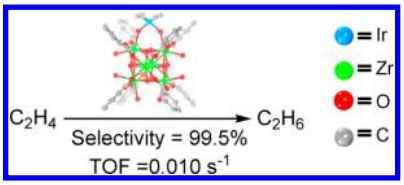

Figure 7. [Ir$(C_2H_4)_2$] fragment installed on NU-1000 catalyzes ethene hydrogenation.

Similarly, a single-site catalyst has been prepared using Hf-NU-1000 as a scaffold—a MOF featuring hafnium oxide nodes isostructural to the Zr_6O_8 units of NU-1000—and tetrabenzylzirconium as an organometallic precursor for node metalation. Upon release of toluene, the zirconium precursor reacts with the hafnium node to give highly electrophilic Zr(IV) centers shown to be active in alkene polymerization.[220] The catalyst was found active in ethene polymerization to give linear polyethylene, whereas, when exposed to 1-hexene, it was found to produce >95% isotactic-poly(1-hexene) with a bimodal mass distribution (M_n = 274 000 g/mol and M_n = 570 g/mol) as result of different activity between the interior and the exterior Zr(IV) sites on the MOF. This example is the first report of stereoregular polymerization catalyzed by a MOF.

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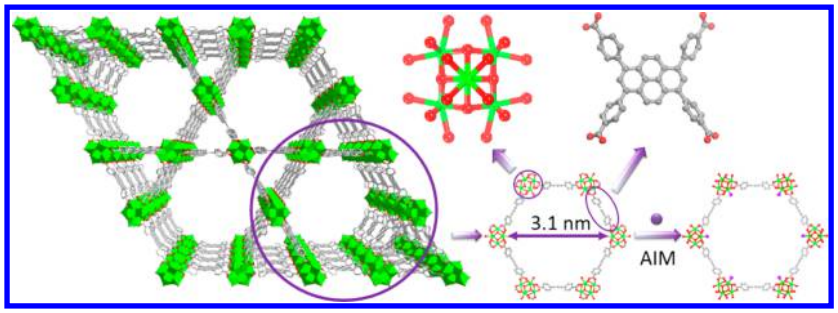

Figure 8. Atomic layer deposition in a metal–organic framework (AIM).

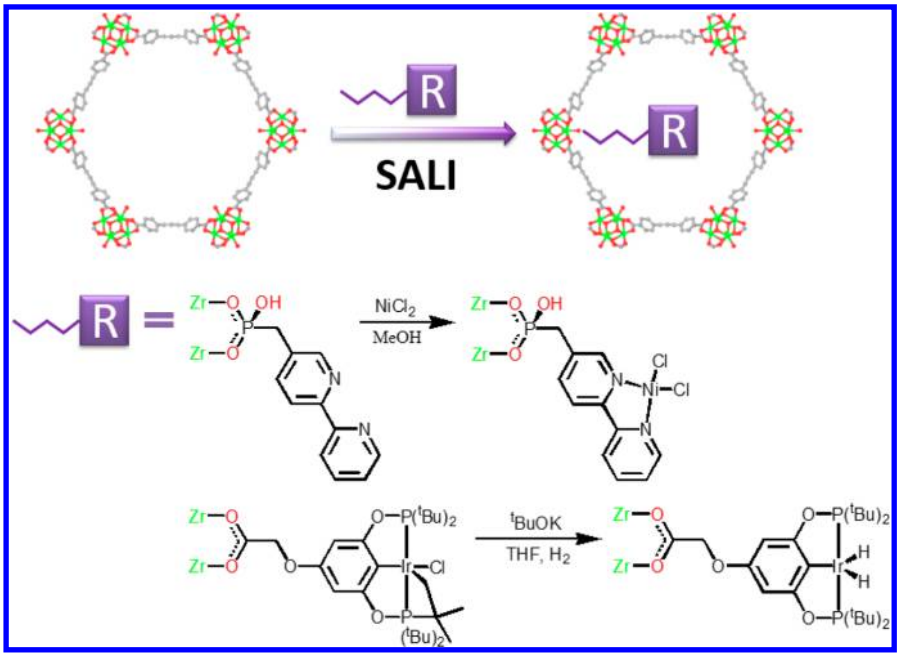

Figure 9. Solvent-assisted ligand incorporation onto Zr-nodes of NU-1000 by means of carboxylic and phosphonic acids groups.

Atomically precise control over metal deposition in solid supports is vital when considering heterogeneous catalyst design. Recently, atomic layer deposition in MOFs (AIM) has successfully been adopted as an alternative way to efficiently metalate the node of Zr-MOFs postsynthetically.[224,226−228] AIM brings the advantages of a vapor-phase technique, avoiding thus the use of solvents and eliminating the need for drying and activation processes. The goal of this technique is to achieve single-site and efficient metal deposition for applications in catalysis. AIM requires a MOF platform with high chemical and thermal stability as well as large pores in order to achieve optimal deposition performances. One example of a catalyst prepared using AIM involves the use of a cobalt amidinate metal precursor and H_2S as coreactant to deposit cobalt sulfide (CoS) in NU-1000 (Figure 8).[227] The as-prepared CoS-AIM material performed better than cobalt sulfide and oxide reference materials for the hydrogenation of *m*-nitrophenol to *m*-aminophenol. Extension of the AIM concept to NiS yields node-sited clusters that, in combination with visible-light absorbing linkers (NU-1000), are *photo*catalytic for the reduction of neutral water to H_2.[462] AIM-based installation of cobalt(II)oxo,hydroxy clusters (four cobalt ions per node) yields a MOF that in thin-film form is *electro*catalytic, but now for the other half of the water-splitting reaction, O_2 formation.[404]

Sulfur-free nickel species have also been deposited in NU-1000 by AIM and are observed, after thermal and chemical activation (presumably loss of an aqua ligand), to possess high activity and long-term stability for the gas-phase hydrogenation of ethene as a result of the antisintering properties of spatially isolated metallic centers.[226] The crystalline nature of the support allowed for the use of computational modeling to elucidate mechanistic details of the catalytic ethene hydrogenation and oligomerization. Other metals such as Al,[228] Zn,[228,493] and Ti[224] have also been deposited this way.

Solvent-assisted ligand-incorporation (SALI) is another technique that takes advantage of hydroxyl and water ligands on Zr-nodes to anchor carboxylic or phosphonic-acid-functionalized molecules postsynthetically. SALI has been used to anchor metal-based catalysts to the node of Zr-MOFs. As shown in recent reports, coordination complexes can be tethered to the node of NU-1000 while the nature of the active site is preserved and catalytic activity retained (Figure 9).

More precisely, an iridium pincer complex bearing a −COOH functionality was installed in NU-1000 to give a catalytic species that showed enhanced performance for the liquid-phase hydrogenation of 1-decene and styrene compared to the homogeneous analogue. The catalyst was also active for gas-flow ethene hydrogenation.[198] A similar strategy was reported using a two-step postsynthetic functionalization technique.[225] 5-Methylphosphonate-2,2′-bipyridine—a biden-

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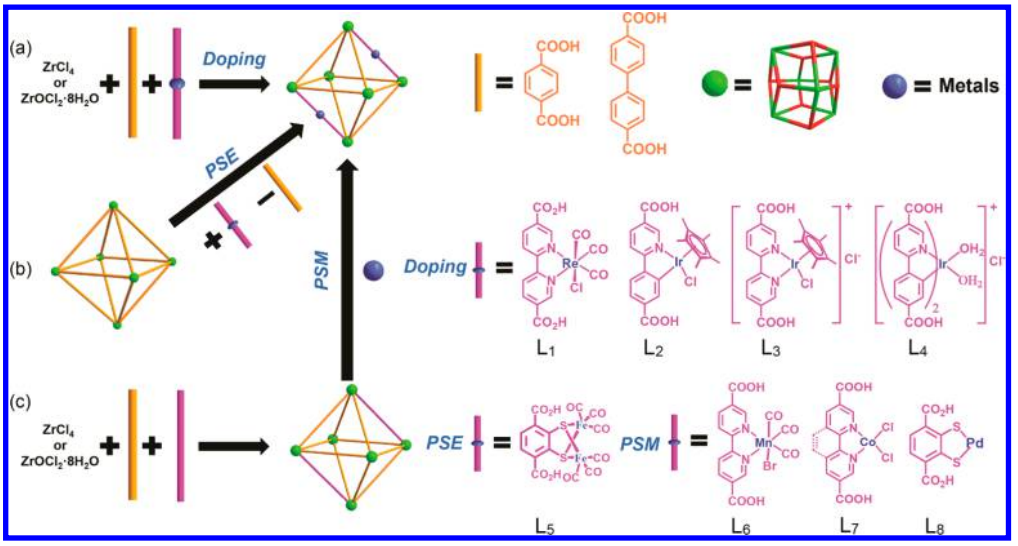

Figure 10. Synthesis of metalated Zr-MOFs via (a) doping, (b) postsynthetic exchange (PSE), and (c) postsynthetic metalation (PSM).

tate ligand—was first affixed on the node of NU-1000 and subsequently metalated with $NiCl_2$ to obtain NU-1000-bpy-$NiCl_2$ (bpy is 2,2′-bipyridine). Compared to the corresponding homogeneous analogues, the single-site catalyst not only showed activity an order of magnitude higher for the liquid-phase dimerization of ethylene but also showed interesting activity at ambient temperature for gas-phase ethylene dimerization. We speculate that immobilization on a heterogeneous support induces stabilization of the catalysts and/or alters the stability of intermediate species generated during the catalytic cycles, thus influencing the overall reaction rate or selectivity. For example, the isolated nature of a supported molecular catalyst might prevent intermolecular catalyst interactions that can be responsible for a loss in activity.

CATALYSIS AT THE LINKER

Installation of catalytic functions at the linker may represent the most widely explored catalyst incorporation approach in Zr-MOFs. As opposed to traditional homogeneous catalysts that require bulky ligands to protect the metal center from deactivation pathways, site-isolation of metals periodically installed at the linker of MOFs can inhibit undesired side-reactions. Unlike more common heterogeneous catalyst supports, MOF linkers can be strategically designed to provide an environment that resembles that of the active homogeneous counterpart.

The synthesis of MOFs using linkers that already include metallic species in the form of organometallic complexes is rarely successful. A linker designed on the skeleton of a palladium pincer complex, however, was used for the direct synthesis of a Zr-MOF, producing a catalyst for transfer hydrogenation of aldehydes.[229] The success in the synthetic approach can be attributed mainly to the intrinsic stability of the metal complex induced by the strongly chelating ligand. Typically, de novo attempts at synthesizing MOFs using metalated linkers, as opposed to nonmetalated analogues, give materials that lack crystallinity. To overcome this difficulty, alternative direct methods and postsynthetic modification approaches have been developed to obtain MOFs with metalated linkers.

Incorporation of metalated linkers in a MOF can be achieved by the so-called "doping" approach. This synthetic protocol has been documented with a series of coordination complexes containing carboxylic-acid-functionalized bipyridyl-type ligands in UiO-67 (Figure 10a, L_1 - L_4).[230] Under conditions typically used to synthesize UiO-67 but with the addition of a small amount (4%, as reported) of the desired metalated linker, the MOF can be obtained. Incorporation of only a small amount of the sterically demanding metalated linker is compatible with the framework structure and does not cause loss of crystallinity or porosity. It is important to emphasize that this procedure requires the metalated and nonmetalated linkers to match in length in order to obtain the desired MOF. The applicability of this approach was demonstrated with the incorporation of iridium, rhenium, and ruthenium complexes bearing different ligands with varying steric bulk. For instance, the complex $Re^{I}(CO)_3$(dcbpy)Cl (dcbpy is 5,5-dicarboxylate-2,2-bipyridine) could be incorporated into UiO-67 (Figure 10a, L_1) and was reported to be active for photocatalytic CO_2 reduction, the first example of this reaction using a MOF-based catalyst.[230] The photocatalytic reaction—performed in the presence of trimethylamine as a sacrificial agent—gave a turnover number (TON) for CO production of 11 after 20 h. Iridium-containing molecular catalysts were similarly immobilized in UiO-67 (Figure 10a, L_2 - L_4) and were found to be catalytic for water oxidation in the presence of a Ce(IV) oxidant (ceric ammonium nitrate).[230] However, poisoning experiments revealed that the catalytic reaction occurs only at the surface of the MOF particles, rendering the homogeneous catalysts more active than the heterogeneous ones.

In some cases, direct methods are not applicable to linkers containing metallic species that cannot withstand solvothermal conditions. Therefore, *solvent-assisted linker exchange* (SALE) or *postsynthetic exchange* (PSE)—a technique that involves replacing some (or all) of the structural linkers in a framework with new linkers—has been developed as a modification method (Figure 10b).[231] For instance, the proton reduction catalyst [FeFe](bdt)$(CO)_6$ (bdt is benzenedithiolate)—similar in structure to the active site of *hydrogenase* and showing a decomposition temperature of 50 °C—has been incorporated in UiO-66 using PSE (Figure 10b, L_5).[233] After successful incorporation into the Zr-MOF, the catalyst UiO-66[FeFe]-(dcbdt)$(CO)_6$ (dcbdt is 1,4-dicarboxylbenzene-2,3-dithiolate) was found to be much more active in the photocatalytic

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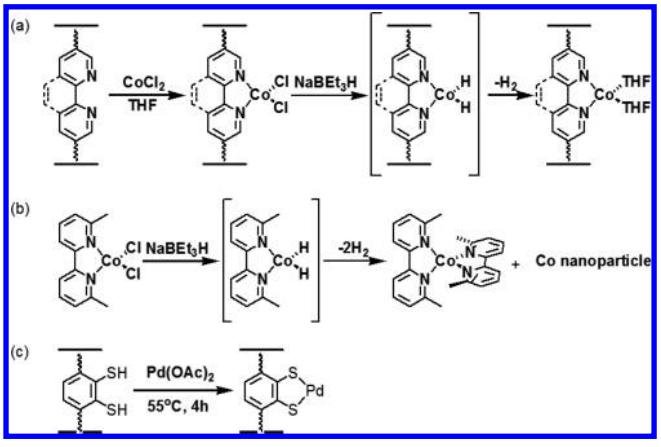
production of hydrogen compared to the homogeneous catalyst—supporting the idea of catalyst stabilization upon heterogenization.

Another example which takes advantage of the stabilization of thermally unstable species in a MOF involves the incorporation of the photocatalyst *fac*-Mn(bpy)$(CO)_3$Br (Figure 10c, L_6) in UiO-67 by *postsynthetic linker metalation*. UiO-67-Mn(dcbpy)$(CO)_3$Br was shown to photocatalytically reduce CO_2 in the presence of 1-benzyl-1,4- dihydronicotinamide as a sacrificial reducing agent and $[Ru(dmb)_3]^{2+}$ (dmb is 4,4′-dimethyl-2,2′-bipyridine) as a photosensitizer in DMF/triethanolamine. A respectable TON of 110 ± 13 in the production of formate (HCOO−) was found over the course of 18 h. The higher catalytic efficiency of the heterogenized catalyst compared to the homogeneous system was attributed to both the stabilization of the $Mn(CO)_3$ center within the robust Zr-UiO framework and the inhibition of Mn−Mn dimer formation, known to be a product of the one-electron reduction of the parent species [Mn(bpy)$(CO)_3$Br].

Postsynthetic metalation of a MOF organic linker has been extensively used to develop a wide array of single-site solid catalysts,[194,196,197,229,234−283] and in particular, it has been demonstrated that this technique can be used to prepare undercoordinated species otherwise unattainable de novo. If prepared in solution, coordinatively unsaturated species usually undergo rearrangement, intermolecular reactions, and aggregation. For these same reasons, confinement in a rigid structure such as a MOF is essential for their stabilization. Also, installation of a metal on a linker unambiguously generates single-site species.

The use of this technique has been extensively demonstrated, for instance, in a series of UiO-type MOFs directly synthesized using 2,2′-bipyridine- or phenanthroline-based linkers (Figure 10c, L_7).[196] Postsynthetic metalation of the UiO-67 derivative with cobalt(II) chloride followed by treatment with $NaBEt_3H$ gives (bpy)Co$(THF)_x$ units throughout the framework (Scheme 1a,b). Control reactions show that the homogeneous catalyst (bpy)Co$(THF)_2$ is highly unstable in solution due to intermolecular ligand-disproportionation leading to dimerization products and cobalt nanoparticle formation (Scheme 1b), thus highlighting the requirement of a MOF support.

Scheme 1. (a) Metalation with $CoCl_2$ by Post-Synthetic Linker Metalation to Achieve Undercoordinated Species; (b) Attempted Solution-Phase Synthesis of Homogeneous Cobalt Analogues; (c) Metalation of a Thiocatechol Linker with $Pd(OAc)_2$



The cobalt-based MOF was found to be catalytically competent for alkene hydrogenation and hydroboration, aldehyde and ketone hydroboration, and arene C−H borylation. In particular, it showed excellent activity for the hydrogenation of a wide range of mono-, di-, tri-, and tetra-substituted alkenes with an extraordinary TON of 2.5×10^6 in 1-octene hydrogenation. Furthermore, the MOF catalyst was found to be tolerant of a variety of functional groups and could be reused several times without loss of catalytic activity and with negligible metal leaching.

Using a similar approach, a thiocatechol-functionalized UiO-66 framework (UiO-66-TCAT) was postsynthetically metalated with palladium acetate under mild conditions (Figure 10c, L_8). The thiocatechol linker, acting as dianionic chelating ligand, stabilized monometallic Pd(II) species with open coordination sites (Scheme 1c).[244] Undercoordinated metal centers are usually highly active catalysts, and therefore, stabilization of these centers is desirable. It is important to point out that such undercoordinated species would not be obtainable as homogeneous catalysts. UiO-66-PdTCAT (with 5% Pd loading), tested for regioselective aromatic C−H oxidation in the presence of iodobenzene diacetate as an oxidant, almost quantitatively converted benzoquinoline to methoxy functionalized quinoline, whereas in the presence of N-halosuccinimides, it successfully halogenated benzoquinoline and bipyridine derivatives in excellent yield.

The majority of linker-based Zr-MOF catalysts feature metal ions or atoms on the linker; however, a significant number of *organocatalysts* have been reported. The covalent attachment of organocatalysts onto the organic struts of MOFs has been successfully exploited for a variety of bond-forming transformations.[259,284−297,299−302,308] Although unrelated, immobilization of organocatalysts gives rise to similar advantages to those highlighted with respect to metalated linkers. In fact, the most remarkable examples describe heterogenization of organic catalytic species either to prevent deactivation or to tune activity and selectivity.

Squaramide—a hydrogen-bond-donating organocatalyst—has been affixed onto the biphenyl-4,4′-dicarboxylate linker of UiO-67 (UiO-67-Squar) for the Friedel−Crafts reaction between indole and *β*-nitrostyrene.[294] The homogeneous squaramide catalyst is remarkably *in*effective (0% Friedel−Crafts yield), likely due to strong self-association via the same hydrogen-bond-forming functionalities that would otherwise catalyze the reaction. Under the same conditions, and with the same reactants, the heterogeneous UiO-67-Squar catalyst elicited 80% conversion. Thus, the framework serves to prevent squaramides from self-associating, thus freeing them to bind and activate susbtrate molecules. While the UiO-67-Squar catalyst is perhaps the most striking example to date of framework-facilitated catalyst "activation via isolation," the theme is a widely recurring one in MOF catalytic chemistry.

Kaskel et al. reported the immobilization of L-proline in UiO-68 to achieve reverse diastereoselectivity for the aldol condensation between 4-nitrobenzaldehyde and cyclohexanone.[292] In situ deprotection of the L-proline groups resulted in a nearly enantiopure heterogeneous catalyst that gave 97% yield under optimized conditions with a *syn*:*anti* diastereomeric ratio of 88:12. The homogeneous species proceeded with 60% yield and d.r. value of 23:77 (*syn*:*anti*) under the same conditions (Scheme 2).

The diastereoselectivity of UiO-68-NHPro for the *syn* product is relatively unheard of in the literature. Previously

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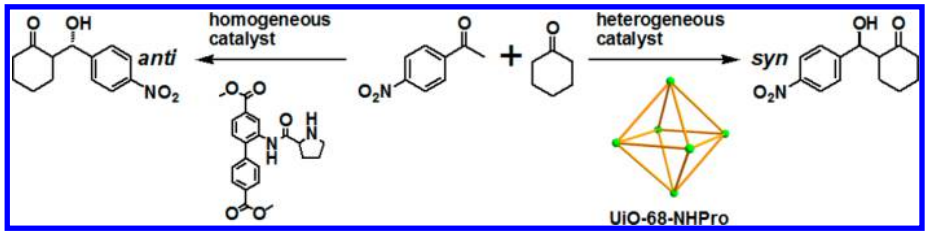
Scheme 2. Diastereoselectivity of UiO-68-NHPro in Aldol Condensation Reaction



reported proline-functionalized MOFs all exhibit a preference for the *anti* product in stereoselective aldol reactions. Further, utilization of L-proline in both the homogeneous and heterogeneous form for the aldol addition of 4-nitrobenzaldehyde and cyclohexanone results in the *anti*-adduct being favored over the *syn*. The preference for the *syn* product, using UiO-68-NHPro as a catalyst, can be attributed to the high concentration of proline side groups around the active site, potentially altering the transition states involved in the catalytic transformation. Further, it has been documented that Lewis acids strongly influence the stereoselectivity of L-proline-based aldol additions.

■ ENCAPSULATED CATALYSTS

The strategy of encapsulating various materials within Zr-based MOFs for catalytic applications has recently been of great interest, and metal nanoparticles are the most widely used guests.[303−332] These hierarchical core−shell structures provide numerous advantages with respect to traditional catalyst supports, including increased durability by inhibiting sintering, imparting substrate size and shape selectivity, as well as controlling product selectivity through steric or chemical control.

Encapsulated nanoparticles have been extensively reported for catalysis and used for a broad range of reactions including hydrogenation,[303−313,333,334] C−H activation,[329] C−C coupling,[313−316] dehydrogenation,[310] isomerization,[324] oxidation,[318−321,335] photocatalysis,[327,336−339] and reduction.[322] Nanoparticle encapsulation procedures fall mostly into two general categories that are commonly designated as "ship-in-a-bottle" and "bottle-around-ship" approaches.[494,495] The former entails impregnating a desired MOF with an ionic or molecular precursor followed by reduction or decomposition to generate the encapsulated nanoparticles.[496] The Bottle-around-Ship method involves the controlled growth of a MOF around presynthesized nanoparticles.[497,498] Both procedures have been used to prepare nanoparticle@MOF structures.

An alternative encapsulation method involves the incorporation of the metal precursor as a metalated linker. Subsequent exposure to reducing conditions allows for the formation of nanoparticles evenly distributed throughout the framework. This approach, for instance, has been demonstrated for the preparation of palladium nanoparticles by utilizing 5-dicarboxylate-2,2-bipyridine metalated with [$PdCl_2$] as a linker.[304,308,419,463]

The ability of MOFs to act as a "molecular sieve" and thereby enable size or shape selective catalysis has been reported numerous times. The "sieving effect" was first demonstrated in a Zr-MOF in the chemoselective liquid-phase hydrogenation of cinnamaldehyde (Figure 11).[333] The "ship-in-a-bottle" approach was employed to incorporate platinum nanoclusters (Pt NCs) in amino-functionalized UiO-66 (UiO-66-NH_2), resulting in the growth of extremely small and narrowly

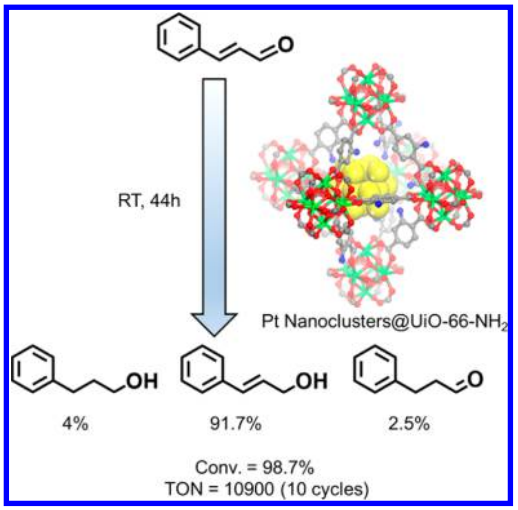


Figure 11. Encapsulation of platinum clusters within UiO-66-NH_2 leads to enhanced selectivity in the hydrogenation of cinnamaldehyde to cinnamyl alcohol.

dispersed clusters (diameter = 1.16 ± 0.16 nm). This composite material (Pt@UiO-66-NH_2) catalyzes cinnamaldehyde hydrogenation with 98.7% conversion and 91.7% selectivity for cinnamyl alcohol after 44 h at room temperature. In contrast, the nonencapsulated Pt/UiO-66-NH_2, where clusters reside on the external surface of the MOF, shows 52% conversion and 72% selectivity. The higher selectivity of the Pt@UiO-66-NH_2 composite—as compared with Pt/UiO-66-NH_2—is a direct result of the Pt clusters being confined in octahedral cages 12 Å in diameter accessible via 6 Å wide triangular windows. The steric restrictions prevent the internal C═C bond of cinnamaldehyde from interacting with the Pt clusters, and as a result, the terminal C═O bond more easily adsorbs to the cluster surface. These circumstances lead to high selectivity for the unsaturated alcohol, which is the thermodynamically less favorable hydrogenation product. In contrast, in Pt/UiO-66-NH_2, clusters deposited on the surface of the MOF crystallites are less sterically hindered and are therefore capable of preferentially adsorbing cinnamaldehyde via the internal C═C bond. This leads to a decrease in selectivity for cinnamyl alcohol and an increase in selectivity for hydrocinnamaldehyde and hydrocinnamyl alcohol, products of C═C bond hydrogenation.

Upon encapsulation, the surfaces of metal nanoparticles are in close proximity with the components of the MOF, either the node or the linker (or both). Because the catalytically relevant sites on nanoparticles are at their surface, catalytic transformations can be considered to occur at the interface between the metal particles and the framework itself. Selective incorporation of different functional groups in the MOF therefore changes the chemical environment of the nanoparticle

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catalysts. This concept was demonstrated for the first time with Pt nanoparticles encapsulated via the "bottle-around-ship" strategy in UiO-66, whose ligands were modified by introducing sulfonic acid (UiO-66-SO_3H) or ammonium (UiO-66-NH_3^+) functionalities.[324] These composites were used to identify the effect of different acidic functionalities on the gas-phase conversion of methylcyclopentane (MCP). Compared to Pt NPs embedded in unmodified UiO-66 (Pt@UiO-66), Pt@UiO-66-SO_3H was found to have markedly different product selectivity, yielding no acyclic isomers and displaying a dramatic increase in selectivity for cyclohexane selectivity (62% versus 22%)—one of the highest selectivities reported at that time—and a 2-fold enhancement in catalytic activity. The Pt@UiO-66-NH_3^+ derivative also showed changes in product selectivity (i.e., an increase in acyclic isomers and a decrease in benzene and cyclohexane) and activity similar to Pt@UiO-66. Control experiments elucidated the importance of functional group acidity in altering the catalytic behavior. Neutralized functional groups engendered little change of product distribution as compared to the unmodified Pt@UiO-66. The combined results point to a synergy between the strongly acidic functional group and Pt NP, which results in a decreased activation energy for the formation of cyclohexane. Most intriguingly, when colocated in the same MOF, the two acidic functional groups ($-SO_3H$ and $-NH_3^+$) change the reaction pathway. The hybrid catalyst containing both $-SO_3H$ and $-NH_3^+$ displays a strikingly different pattern of product selectivity, showing an increase in benzene and olefin production and a decrease in cyclohexane formation, but still exhibiting the high activity that characterizes the sulfonate-acid only analogue.

CONCLUSION AND OUTLOOK

MOFs are porous structures that can be used to incorporate various active functionalities for application in catalysis. Other recent work, not discussed, includes the incorporation of guests such as polyoxometalates,[340−342] quantum dots,[327,340,343] and enzymes,[332,344,345] clearly indicating some important future directions for the use of MOFs in catalysis.

Because all MOF components (linkers, nodes, and pores) can be modified to accommodate various functional catalysts, multifunctional catalysis,[346] tandem or cascade reactions can be realized.[269,271,347] This approach has been recently explored, for instance, with the preparation of bifunctional catalysts that combine the use of a metalated linker and node,[348] a metalated linker and encapsulated polyoxometalate,[341] and encapsulated nanoparticles and metal nodes.[349] MOFs incorporating several functionalities can potentially be prepared to target complex transformations that require the simultaneous presence of different catalytic sites.

Computational modeling clearly will play an increasingly important role in the screening of multicomponent catalysts and predicting the most efficient and effective combinations. Crystallographic uniformity—an intriguing aspect that differentiates MOFs from many other solid supports or solid catalysts—will help to enhance the efficacy of modeling in predicting reactivity and simulating structures. In recent computational work, for example, a CO_2 reduction catalyst in a Zr-MOF has been designed. Here modeling revealed that a series of frustrated Lewis pairs (such as 1-(difluoroboranyl)-4-methyl-1H-pyrazole), when incorporated on the linker of Zr-UiO-66, could facilitate the heterolytic splitting of dihydrogen followed by reaction of the proton and hydride with carbon dioxide.[301,302] The importance of computational modeling was also recently demonstrated in a study involving the hydrolysis of chemical warfare agents. Experimental work demonstrated that the Zr-nodes of NU-1000 are efficient catalysts for the hydrolysis of DMNP via cleavage of the P−O bond (Figure 4). In the same study, it was predicted via computational modeling that the selective hydrolysis of VX by the Zr-node would occur at the preferred position for detoxification (the P−S bond) as opposed to occurring at the P−O bond (which would produce a highly toxic byproduct).[195] A follow-up experimental study showed that selective catalytic hydrolysis of the P−S bond of VX can be achieved with a half-life of 1.8 min, confirming the validity of the computational predictions.[350]

Finally, the robust and highly tunable nature of Zr-MOFs has led to many elegant studies involving the use of these materials as catalysts. A number of different approaches for catalyst incorporation and/or stabilization using Zr-MOFs have been developed, and impressive strides have been made since the first introduction of Zr-MOFs just 8 years ago. With so many tunable components and the continued development of new Zr-MOFs, the potential of these materials as heterogeneous catalysis is only really beginning to be realized.

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Notes

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