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Porous organic polymer supported rhodium as a heterogeneous catalyst for hydroformylation of alkynes to α,β-unsaturated aldehydes†

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A new porous organic polymer supported rhodium catalyst (Rh/POL-BINAPa&PPh$_3$) has been developed for the hydroformylation of various alkynes to afford the corresponding α,β-unsaturated aldehydes with high chem- and stereoselectivity, excellent catalytic activity and good reusability (10 cycles). The heterogeneous catalyst exhibited more catalytic activity than the comparable homogeneous Rh/BINAPa/PPh$_3$ system.

As one of the largest homogeneous industrial processes, hydroformylation is an efficient method for the synthesis of aldehydes. Compared with well-studied hydroformylation of olefins,[1] the hydroformylation of alkynes has attracted relatively less attention in the past few decades.[2] Hydroformylation of alkynes provides a 100% atom-efficient route for producing α,β-unsaturated aldehydes, which are versatile building blocks for organic synthesis.[3] The transformation remains a challenge, not only owing to the hardly suppressed hydrogenated product, but also to the poor regioselectivity and reactivity. Over the past several decades, a variety of effective catalysts have been successfully developed for hydroformylation of various alkynes. Rh-Biphephos and heterobimetallic catalyst [$PdCl_2(PCy_3)_2$]/[$Co_2(CO)_8$] introduced by Buchwald[4] and Hidai[5] respectively were mainly efficient for internal alkyl alkyne substrates. Subsequently, Alper and Van den Hoven applied a zwitterionic rhodium complex into the selective hydroformylation of enynes and thiophenyl substituted alkynes.[6] A Rh/self-assembling bisphosphine system and a Pd/*N*-phenylpyrrole-based bisphosphine system were developed by Breit[7] and Beller[8] respectively for the chemo- and stereoselective hydroformylation of dialkyl- as well as diaryl-substituted alkynes. In 2016, Zhang and coworkers reported a Rh/tetraphosphoramidite catalyzed hydroformylation of various symmetrical and unsymmetrical alkynes to afford the corresponding α,β-unsaturated aldehyde in high yields with excellent efficiency.[9] Recently, You demonstrated a syngas-free rhodium-catalyzed transfer hydroformylation of alkynes with high chemo-, regio-, and *E*/*Z*-selectivities.[10] Although these homogeneous catalytic systems are efficient for hydroformylation of alkynes, catalyst separation and recycling problems severely impede their industrial application.

In recent years, porous organic polymers (POPs) have provided tremendous opportunities for potential applications in heterogeneous catalysis owing to their high surface areas, tunable pore size distribution, excellent stabilities, and synthetic diversification.[11] As a new kind of porous organic polymer, porous organic ligands (POLs) have a high ligand concentration, which would improve the loading capability of the active metals, and thus reduce the loss of metals in the catalytic process. A series of rhodium-loaded porous organic ligands,[12] such as Rh/POL-PPh$_3$,[13] Rh-dppe-POP,[14] Rh/CPOL-bp&PPh$_3$[15] and Rh/POL-BPa&PPh$_3$,[16] have been successfully developed as recyclable catalysts for hydroformylation of olefins. So far, heterogeneous catalysts have been rarely explored in the hydroformylation of alkynes. In the present work, we report a new Rh-loaded porous organic ligand (Rh/POL-BINAPa&PPh$_3$) for hydroformylation of symmetrical and unsymmetrical alkynes to afford various α,β-unsaturated aldehydes with high chem- and stereoselectivity, excellent catalytic activity and good recyclability.

The new porous organic polymer **5** (POL-BINAPa&PPh$_3$) was synthesized *via* a free-radical polymerization reaction using the vinyl-functionalized bisphosphoramidite **3** and tris(4-vinylphenyl)-phosphine (Scheme 1). Subsequently, treatment of the porous organic polymer with Rh(acac)(CO)$_2$ in THF for 24 h afforded the supported rhodium catalyst Rh/POL-BINAPa&PPh$_3$. Meanwhile, BINAPa **4**, Rh/POL-BPa&PPh$_3$ **6** and Rh/POL-PPh$_3$ **7** were also prepared for hydroformylation of alkynes (for details, see the ESI†).

^{13}C MAS NMR spectra were used to characterize the Rh/POL-BINAPa&PPh$_3$ catalyst. The signals at 130.2 ppm and 39.2 ppm

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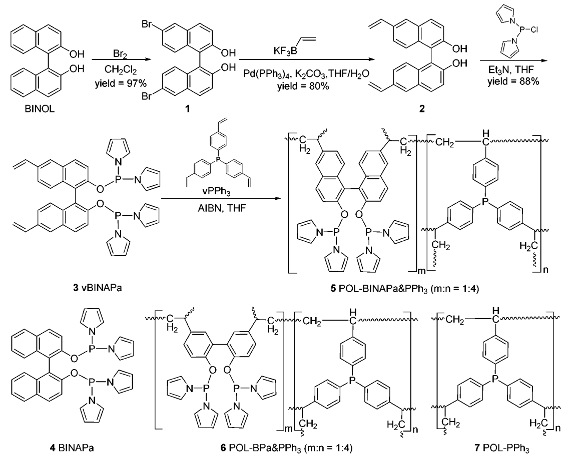


Scheme 1 Synthesis of ligand **4** and POLs (**5**, **6**, **7**).

are attributed to the aromatic carbons and polymerized vinyl groups, respectively (Fig. S1, ESI†). In the solid ^{31}P MAS NMR spectrum, the peaks at around 108.0 ppm are ascribed to the phosphorus signals of polymerized vBINAPa moieties, and the peak at −9.1 ppm is attributed to the phosphorus signals of polymerized $vPPh_3$ moieties. Remarkably, two peaks at 126.9 ppm and 24.8 ppm are assigned to the phosphorus signals of polymerized vBINAPa and $vPPh_3$ moieties coordinated to Rh, respectively (Fig. S2, ESI†). The XPS spectra of Rh/POL-BINAPa&PPh_3 (Fig. S8, ESI†) show the binding energies of P2p at 132.5 eV (PPh_3 units) and 130.6 eV (BINAPa units), which are lower than those of POL-BINAPa&PPh_3 (132.6 eV and 130.9 eV, Fig. S7, ESI†). Meanwhile, compared with the XPS spectra of Rh3$d_{3/2}$ (314.0 eV) and Rh3$d_{5/2}$ (309.2 eV) in $Rh(CO)_2(acac)$,[15a] Rh/POL-BINAPa&PPh_3 also gives lower binding energies (313.0 eV and 308.5 eV, Fig. S10, ESI†). The results indicate the successful coordination of Rh with two types of P species in the polymer framework. TG analysis demonstrates that significant decomposition starts at around 456 °C, which indicates that the Rh/POL-BINAPa&PPh_3 catalyst is thermally stable (Fig. 1a). The nitrogen sorption isotherm exhibits a combined curve of type-I and type-IV, indicating that the catalyst possesses micro- and mesopores in the catalyst (Fig. 1b). The pore sizes of two types are distributed at 1.1–2.0 and 2.0–20 nm respectively according to the calculations of the nonlocal density functional theory (NLDFT). The Brunauer–Emmett–Teller (BET) surface area of Rh/POL-BINAPa&PPh_3 is as high as 492.2 $m^2\ g^{-1}$ with a pore volume of 0.41 $cm^3\ g^{-1}$ (Fig. S4, ESI†). Scanning electron microscopy (SEM, Fig. 1c) and transmission electron microscopy (TEM, Fig. 1d) images of Rh/POL-BINAPa&PPh_3 further confirm the existence of hierarchical porosities. In addition, the copolymer (vBINAPa : $vPPh_3$ = 1 : 1.5) was prepared and exhibited a low BET surface area and pore volume, which demonstrated that $vPPh_3$ moieties played an important role in the construction of a porous framework (Fig. S6, ESI†). Inductively coupled plasma mass spectrometry (ICP-MS) and FT-IR (Fig. S13, ESI†) were also used to characterize the Rh/POL-BINAPa&PPh_3 catalyst.

It is well known that catalysts have a crucial effect on the hydroformylation reactions. Thus, we began our initial investigation by evaluating a series of catalysts in the hydroformylation of diphenylacetylene as the model substrate. As shown in Table 1, Rh/POL-PPh_3 exhibited low activity in formation of the desired product (Table 1, entry 1). When Rh/POL-BPa&PPh_3 was used, 79% yield of **9a** and 17% yield of hydrogenation product **10a** was observed (Table 1, entry 2). The application of Rh/POL-BINAPa&PPh_3 provided the desired product **9a** in high yield with good stereoselectivity and a little hydrogenation product (Table 1, entry 3). It could be concluded that the BIANPa and BPa ligand moieties were more active than the PPh_3 ligand moiety owing to high π-acidity of the *N*-pyrrolylphosphorus unit, which facilitated the CO dissociation from the metal centers.[9] Meanwhile, steric hindrance of the BIANPa ligand moiety may suppress the hydrogenation step. The efficiency of the corresponding homogeneous catalysts in the hydroformylation of diphenylacetylene was also investigated. When a BINAPa ligand was applied, a similar result was obtained (Table 1, entries 4 *vs.* 3). Adding the PPh_3 ligand to the Rh/BINAPa catalyst led to a declined yield (Table 1, entries 5 *vs.* 4), and further adding excess PPh_3 ligand resulted in a significant degradation in catalytic activity (Table 1, entries 6 *vs.* 4).

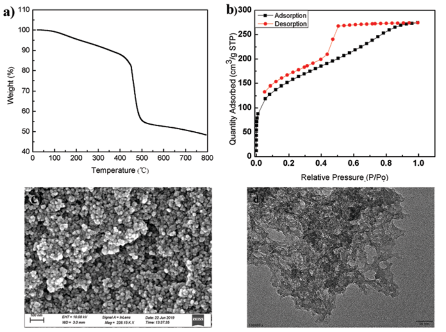
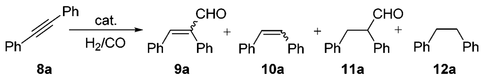


Fig. 1 (a) TG curve, (b) N_2 sorption isotherm, (c) SEM image, and (d) TEM image of Rh/POL-BINAPa&PPh_3.

Table 1 Screening conditions for hydroformylation of diphenylacetylene[a]



Entry	Catalyst	Conv. (%)	**9a** (*E*/*Z*) (%)	**10a** (%)
1[b]	Rh/POL-PPh_3	37	27 (112)	10
2[c]	Rh/POL-BPa&PPh_3	97	79 (160)	17
3	Rh/POL-BINAPa&PPh_3	100	90 (60)	10
4[d]	Rh/BINAPa	100	90 (91)	10
5[e]	Rh/BINAPa/PPh_3	75	65 (113)	10
6[f]	Rh/BINAPa/PPh_3	12	11(110)	1
7	Rh/Al_2O_3	0	0	0
8[g]	Rh/POL-BINAPa&PPh_3	94	80 (64)	14
9[h]	Rh/POL-BINAPa&PPh_3	100	82 (91)	18

[a] **8a** (96.0 mg), 2.7 mg of Rh/POL-BINAPa&PPh_3 (Rh loading at 1.32 wt%), S/C = 1000, toluene (1.0 mL), 20 h, *p*-xylene as the internal standard; yields and ratios of *E* to *Z* isomers were determined on the basis of GC analysis; only a trace amount of **11a** and **12a** (<1%) was detected in each case. [b] 2.0 mg of Rh/POL-PPh_3 (Rh loading at 2.1 wt%). [c] 3.0 mg of Rh/POL-BPa&PPh_3 (Rh loading at 1.2 wt%). [d] $Rh(acac)(CO)_2$: BINAPa = 1 : 3. [e] $Rh(acac)(CO)_2$: BINAPa : PPh_3 = 1 : 3 : 12. [f] $Rh(acac)(CO)_2$: BINAPa :PPh_3 = 1 : 3 : 18. [g] S/C = 5000. [h] S/C = 50 000, 80 °C, 40 h.

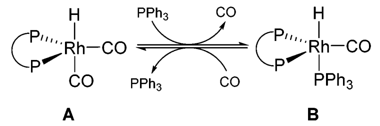
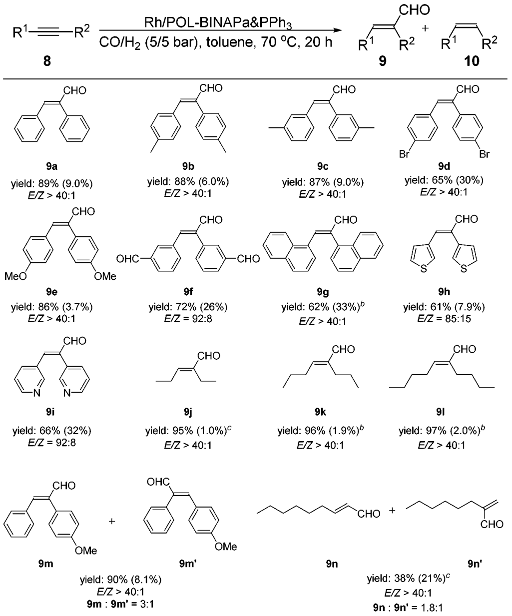


Fig. 2 Catalytically active species **A** and **B**.

According to our previous work,[17] the catalytically active species **A**, which adopted a trigonal bipyramidal structure wherein the bidentate ligand coordinates to Rh in a bis-equatorial (eq–eq) fashion, was observed in homogeneous Rh/BINAPa catalyzed hydroformylation. When the PPh_3 ligand was added, dissociation of CO and subsequent coordination of the PPh_3 ligand to Rh resulted in the formation of active species **B** (Fig. 2). Compared with active species **A**, active species **B** had more steric hindrance at the rhodium metal, which probably reduced the rate of addition of the alkyne to the hydrido rhodium complex. So we concluded that species **A** was more active than species **B**, and the results also corresponded to the hypothesis (Table 1, entries 4–6). When the ligands were fixed on the long polymeric chain in the Rh/POL-BINAPa&PPh_3 catalyst, the polymer framework and less mobility of the ligands may reduce the synergetic effect of BINAPa and PPh_3 ligands with Rh species. Therefore, the heterogeneous catalyst afforded more catalytic activity compared with its homogeneous counterpart (Table 1, entries 3 *vs.* 5). In addition, the commercial Rh/Al_2O_3 didn't show any catalytic activity under the optimized reaction conditions (Table 1, entry 7).

Subsequently, the influence of reaction parameters such as temperature, pressure of syngas, and reaction solvents were investigated in the presence of Rh/POL-BINAPa&PPh_3 as the catalyst (Table S1, ESI†). Decreasing the reaction temperature from 70 to 60 °C provided a low yield, and increasing the reaction temperature to 80 °C resulted in an enhancement of the hydrogenation product (Table S1, entries 1–3, ESI†). Furthermore, either increasing the H_2/CO pressure from 5/5 to 10/10 bar, or decreasing the H_2/CO pressure to 3/3 bar, led to decreased yield of **9a** (Table S1, entries 4 and 5, ESI†). The reactions proceeded well in non-polar solvent and polar solvents (Table S1, entries 6–11, ESI†). We reasoned that the porous structure of the catalyst probably contributed to the good solvent compatibility.[18] In addition, it may be due to the encapsulation of proton solvent in the metal center, which resulted in a decrease of selectivity in the step of alkyne insertion into the rhodium–hydrogen bond, so a low *E*/*Z* ratio of hydroformylation product **9a** was obtained in EtOH. In order to evaluate the activity of the catalyst, the substrate/catalyst (S/C) ratios were surveyed in Rh/POL-BINAPa&PPh_3 catalyzed hydroformylation of diphenylacetylene. Decreasing the catalyst loading to 0.02 mol% (S/C = 5000), the reaction provided the hydroformylation product **9a** in 80% yield (Table 1, entry 8). Further decreasing the catalyst loading to 0.002 mol% (S/C = 50000), **9a** was still obtained in 82% yield with a TON value of up to 5.0×10^4, albeit with a slightly increased hydrogenation product **10a** (Table 1, entry 9). It is noteworthy that such a highly active heterogeneous catalyst might be a valuable attribute for industrial applications.



Scheme 2 Study scope for hydroformylation of various alkynes catalyzed by Rh/POL-BINAPa&PPh_3[a]. [a] Rh/POL-BINAPa&PPh_3 (1.5 mg), S/C = 1000, 20 h, yield of isolated product after column chromatography. The yields of **10** are in parentheses. Yields of **10** and ratios of *E* to *Z* isomers were determined on the basis of GC analysis and/or ^1H NMR. [b] Reaction temperature was 90 °C. [c] The yield was determined by GC analysis.

Under the optimized reaction conditions, various alkynes were investigated as substrates for Rh/POL-BINAPa&PPh_3 catalyzed hydroformylation. As shown in the Scheme 2, a variety of diaryl alkynes were readily amenable to the hydroformylation procedure, affording the corresponding α,β-unsaturated aldehydes (**9a–9i**) in good to excellent yields (61–89%) with high *E* stereoselectivity. Various functional groups on the phenyl ring of the diarylacetylenes, such as methyl, methoxy, bromo and formyl, were well tolerated. In the case of electron poor aryl substituents (**8d** and **8f**), some amount of hydrogenation products was found. It is notable that the aryl bromide and aldehyde substrates could be easily further modifiable. Moreover, the use of the more bulky substrate **8g** gave product **9g** in a good yield. Based on the results, we assumed that the steric effect and electron-deficient properties of the substrates could decrease the hydroformylation rate in the competing process of hydroformylation reaction and hydrogenation reaction. The heteroaromatic dithiophene and dipyridine alkynes (**8h** and **8i**) proceeded smoothly in this catalytic system with good yields. In addition, different dialkyl substituted alkynes performed well and produced the corresponding α,β-unsaturated aldehydes (**9j–9l**) in excellent yields. The challenging unsymmetric alkynes and terminal alkynes were also investigated. 1-Methoxy-4-(phenylethynyl)benzene (**8m**) and 1-octyne (**8n**) gave the corresponding desired aldehydes (**9m** and **9n**) with moderate regioselectivities.

The reusability of the Rh/POL-BINAPa&PPh_3 catalyst in the hydroformylation of diphenylacetylene was evaluated. As shown in Fig. 3, the Rh/POL-BINAPa&PPh_3 catalyst can be reused for at least 10 cycles (for details, see the ESI†). The slight decrease in yield of **9a** and increase in yield of the hydrogenation product may be due to the decomposition of partial BINAPa ligand moieties. After separating the solid

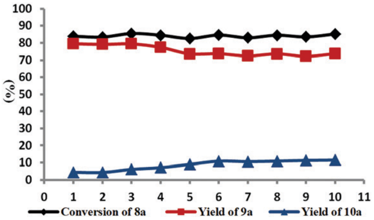


Fig. 3 Recycling studies of the Rh/POL-BINAPa&PPh_3 in diphenylacetylene hydroformylation. Reaction conditions: 28.0 mg Rh/POL-BINAPa&PPh_3, diphenylacetylene (0.95 g), S/C = 1000, CO/H_2 = 5/5 bar, toluene (7.0 mL), 70 °C for 8 h.

catalyst, the residual liquid showed no activity in the hydroformylation of diphenylacetylene. The Rh species in the filtrate after each run were undetectable (<0.1 ppm) by ICP-MS analysis, indicating the strong coordination ability of the polymer with Rh species. The recovered catalyst was characterized by XPS and FT-IR (Fig. S9, S11 and S14, ESI†), which exhibited similar results to the original catalyst. Additionally, the N_2 sorption isotherm showed decreased surface area and pore volume (Fig. S5, ESI†).

In summary, a new porous organic polymer (POL-BINAPa&PPh_3) was synthesized through copolymerization of the vinyl-functionalized phosphoramidite and triphenylphosphane ligands. After supporting with Rh species, the obtained heterogeneous catalyst (Rh/POL-BINAPa&PPh_3) showed good performance in the hydroformylation of various symmetrical and unsymmetrical alkynes to afford the corresponding α,β-unsaturated aldehyde products with high selectivity, activity and recyclability. Remarkably, the Rh/POL-BINAPa&PPh_3 heterogeneous catalyst exhibited more catalytic activity than the comparable homogeneous Rh/BINAPa/PPh_3 system.

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Conflicts of interest

The authors declare no conflict of interest.

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