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# Heterogeneous Enantioselective Hydrogenation of Aromatic Ketones Catalyzed by Rh Nanoparticles Immobilized in Ionic Liquid

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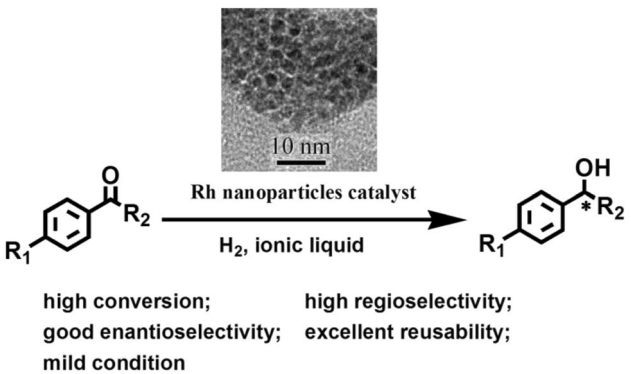
## Abstract

Rhodium nanoparticles (Rh NPs) stabilized by natural cinchona alkaloids were synthesized in imidazolium-based ionic liquids using $H_2$ as the reductant. Characterization showed well-dispersed Rh NPs of about 1.96 nm (TEM and HRTEM) and confirmed the ionic liquid and cinchona alkaloid stabilization to the Rh(0) NPs (XPS). When modified by chiral diamine, including (1*R*,2*R*)-diphenylethylenediamine ((1*R*,2*R*)-DPEN) or cinchona alkaloid derivatives, the Rh NPs catalysts exhibited good activity, chemoselectivity and enantioselectivity in the heterogeneous enantioselective hydrogenation of aromatic ketones. Synergistic effect between (*1R,2R*)-DPEN and cinchonidine was also observed, which significantly accelerated the reaction rate and enhanced the enantioselectivity. 63.0% enantioselectivity and 98.9% chemoselectivity could be achieved in the acetophenone enantioselective hydrogenation; up to 70.2% enantioselectivity and 100% chemoselectivity was obtained in the isobutyrylbenzene catalytic enantioselective hydrogenation. Catalytic system could be reused several times without significant loss in activity, chemoselectivity as well as enantioselectivity. This catalytic protocol opens the door to heterogeneous enantioselective hydrogenation of aromatic ketones with metal Rh NPs immobilized in ionic liquid.

### Graphical Abstract




Cinchona alkaloid and ionic liquid stabilized Rh NPs catalyst, when modified by chiral diamine like (1R,2R)-DPEN, exhibited high activity, high chemoselectivity and good enantioselectivity in the challenging heterogeneous enantioselective hydrogenation of aromatic ketones to corresponding aromatic alcohols under mild conditions. Synergistic effect between (1R,2R)-DPEN and cinchonidine was also observed, which significantly accelerated the reaction rate and enhanced the enantioselectivity. 63.0% enantioselectivity and 98.9% chemoselectivity could be achieved in the acetophenone enantioselective hydrogenation; up to 70.2% enantioselectivity and 100% chemoselectivity was obtained in the isobutyrylbenzene catalytic enantioselective hydrogenation. Catalytic system could be reused several times without significant loss in activity, chemoselectivity as well as enantioselectivity. This catalytic protocol opens the door to heterogeneous enantioselective hydrogenation of aromatic ketones with metal Rh NPs immobilized in ionic liquid.

**Keywords** Aromatic ketone · Cinchona alkaloid · Heterogeneous enantioselective hydrogenation · Ionic liquid · Nanoparticle

Extended author information available on the last page of the article

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## 1 Introduction

Over the past decades, metal nanoparticles synthesized in ionic liquids have been receiving more and more attention in catalytic hydrogenation and other applications [1–3]. With many advantages, such as negligible volatility, excellent thermal stability, remarkable solubility and a variety of available structures, ionic liquids can act as a support and adhere to the metal surface through either electrostatic, van der Waals as well as covalent interactions, which provide stability to the nanoparticles through electrostatic or steric repulsion between neighbouring particles [4–6]. Dupont and co-workers [7] prepared well-dispersed transition metal nanoparticles in various imidazole-based ionic liquids with narrow particle size distribution and showed interesting activity and selectivity in the ketones hydrogenation. Leitner and co-workers [8] successfully employed Ru(0) nanoparticles immobilized in ionic liquids for chemoselective hydrogenation of biomass-derived substrates. We [9, 10] previously reported the use of phosphine functionalized ionic liquid-stabilized ruthenium nanoparticles to catalyze the tunable chemoselective hydrogenation of aromatic ketones, aromatic aldehydes as well as quinolines. Good to excellent catalytic performance was achieved.

Asymmetric hydrogenation of simple aromatic ketones is an important organic conversion because the resulting chiral alcohols are a versatile precursor to many natural products and drug molecules [11, 12]. However, in the field of heterogeneous enantioselective hydrogenation of simple aromatic ketone and its derivatives, successful catalytic example is rather limited [13–16]. Such as, Reyes and co-workers [17] reported enantioselective hydrogenation of acetophenone on cinchonidine modified $Ir/SiO_2$ catalysts and up to 62% enantiomeric excess was obtained. Li et al. reported diphenylethylenediamine modified $1\%Ru/\gamma\text{-}Al_2O_3$/2tpp (tpp:triphenylphosphine) for the asymmetric hydrogenation of aromatic ketones with the best enantioselectivity of 78% [18]. Chen previously reported heterogeneous enantioselective hydrogenation of aromatic ketones employing cinchona- and phosphine-modified iridium as catalysts [19–22], with enantioselectivity up to 96%. We herein originally report the heterogeneous enantioselective hydrogenation of aromatic ketones, catalyzed by cinchona alkaloids stabilized pretty small rhodium nanoparticles (Rh NPs) modified by chiral diamine, such as (1*R*,2*R*)-diphenylethylenediamine ((1*R*,2*R*)-DPEN)) or cinchona alkaloid derivatives, in imidazolium-based ionic liquids, with high activity, chemoselectivity and up to 70.2% enantioselectivity to corresponding aromatic alcohols.

## 2 Results and Discussion

### 2.1 Synthesis and Characterization of Rh NPs

The synthesis of Rh NPs was achieved through the $H_2$ reduction of $RhCl_3.3H_2O$ in $BMIMBF_4$ (BMIM = 1-butyl-2,3-dimethylimidazolium) in the presence of 2.0 equivalent of cinchona alkaloids, which afforded a dark suspension. For comparison, we also synthesized Rh NPs without any additional stabilizer. A black powder could be separated from the black suspension by the addition of acetone followed by centrifugation (5000 rpm for 10 min). Washed three times with acetone and dried under reduced pressure, the isolated powder was analyzed by transmission electron microscopy (TEM) and X-ray photoelectron spectroscopy (XPS).

TEM analysis was used to characterize the obtained Rh NPs and determine the average diameter (Fig. 1, image A and A#). The TEM and HRTEM image of cinchonidine stabilized Rh NPs exhibited a regular spherical shape and a narrow size distribution with an average diameter of 1.96 nm.

The XPS analysis of the cinchonidine stabilized Rh NPs was employed to elucidate the nature of the stabilizing layer of the nanoparticles (Fig. 2). XPS analysis of Rh NPs stabilized by cinchonidine showed the presence of rhodium, boron, nitrogen and oxygen, which signified the presence of the $BMIMBF_4$ and cinchonidine in the ligand sphere of the Rh NPs. The binding energies for Rh, 307.1 eV and 311.7 eV (Fig. 2), which indicated the Rh NPs were composed of Rh(0) [23]. In short, the TEM and XPS results indicated that the rhodium (III) species was completely reduced to Rh NPs, and these Rh NPs were protected by the $BMIMBF_4$ and cinchonidine without any change of valence.

### 2.2 Catalytic Hydrogenation

Enantioselective hydrogenation was performed in a 20 mL stainless autoclave with a magnetic stirrer bar, by using cinchonidine stabilized Rh NPs as a catalyst in the presence of chiral diamine like (1*R*,2*R*)-diphenylethylenediamine ((1*R*,2*R*)-DPEN) or cinchona alkaloid derivatives as chiral modifiers. Acetophenone was chosen as a model substrate to explore the enantioselective catalytic hydrogenation performance of Rh NPs. In heterogeneous enantioselective catalysis, reaction activity, chemoselectivity as well as enantioselectivity are always rather sensitive to the solvent used [24]. In $BMIMBF_4$ solvent, 20.0% conversion, up to 96.2% chemoselectivity and 51.6% ee were obtained for the acetophenone asymmetric hydrogenation

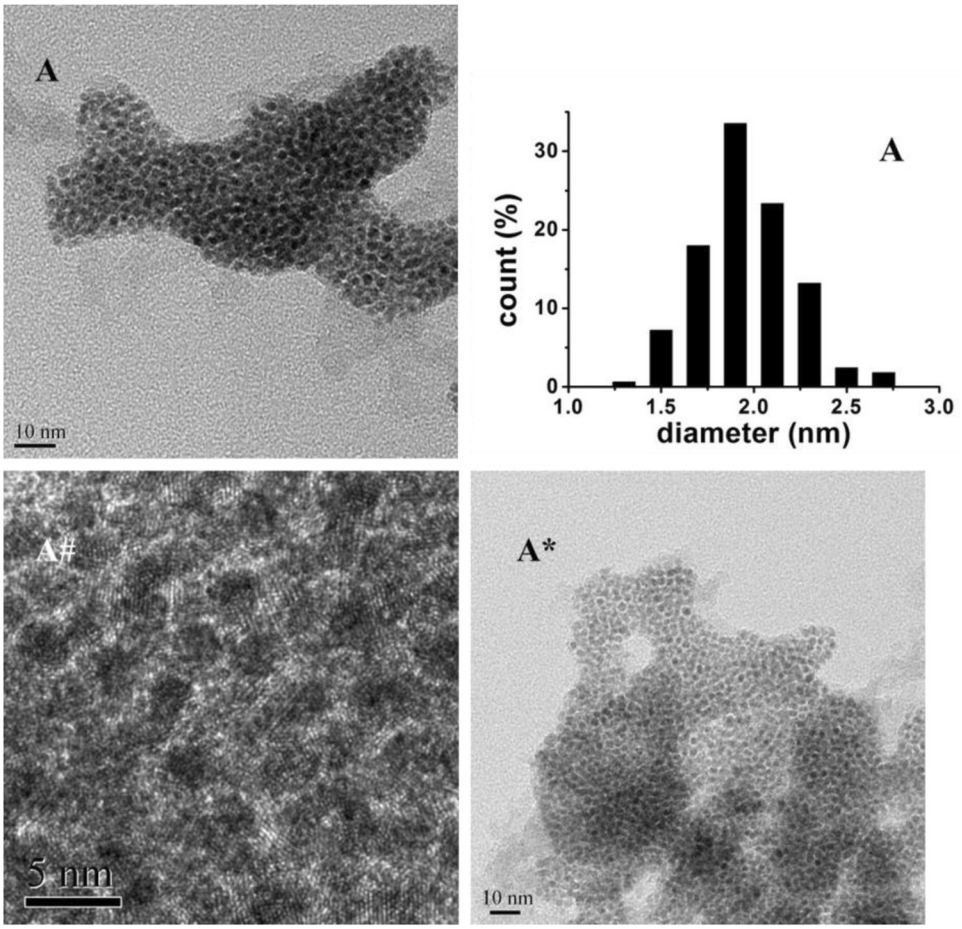



**Fig. 1** TEM image of cinchonidine stabilized Rh NPs **A**, HRTEM image of cinchonidine stabilized Rh NPs **A#** and TEM image of the spent Rh NPs after 3 recycles of acetophenone hydrogenation **A***

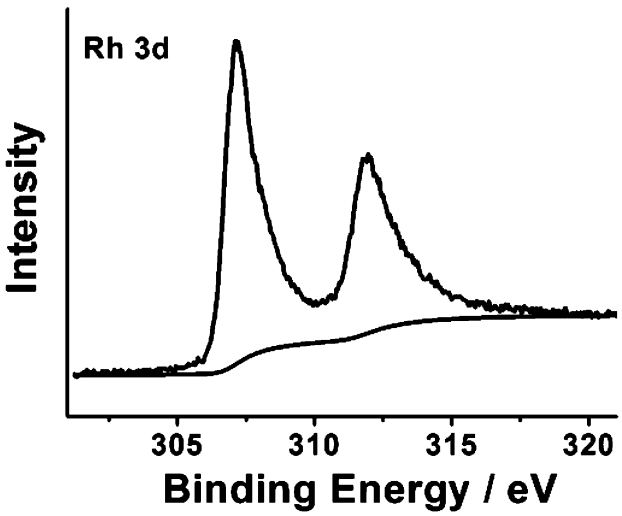
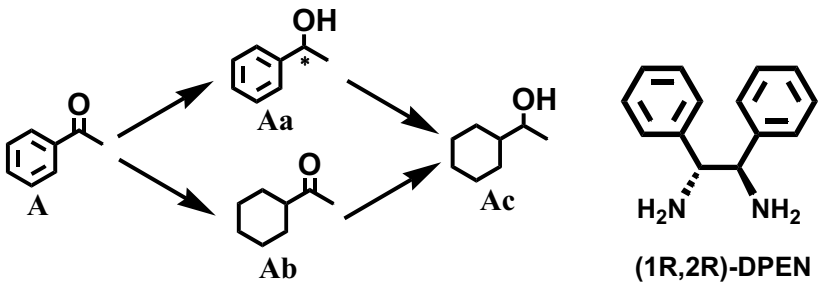



**Fig. 2** XPS spectrum of Rh 3d in cinchonidine stabilized Rh NPs

(Table 1, entry 1). However, when $BMIMPF_6$, $BMIMNTf_2$ and BMIMOTf were employed as the solvent, the conversion were rather low, accompany with sharply decreased ee (Table 1, entries 2–4 vs. 1). We believe the ionic liquid viscosity, the possible coordination effect between anion and Rh NPs as well as the difference in stabilizer/modifier conformation in different ionic liquids sharply influenced the catalytic performance. In order to further optimize the catalytic activity, chemoselectivity and enantioselectivity, we attempted to introduce co-solvent into the catalytic system. Catalytic activity increased while co-solvents like alcohols and water were introduced (Table 1, entries 5–8); chemoselectivity of acetophenone to α-phenethyl alcohol were slightly increased accompany with all co-solvents introduction tested (Table 1, entries 5–10); however, improvement of the hydrogenation enantioselectivity were only observed while EtOH or $H_2O$ co-solvents were introduced. The catalytic performance, 92.1% conversion, 98.9% chemoselectivity and 63.0% ee in mixture solvent $BMIMBF_4$-EtOH, is the best in acetophenone heterogeneous enantioselective hydrogenation herein. Upon the optimization of solvents, we further investigated the effects of modifiers, base additives as well as transition metal species on catalytic performance. Similar to homogeneous reactions and some heterogeneous reactions [25–27], base additive is important to accelerate the reaction. In the absence of base additive BMIMOH, the activity,

**Table 1** Optimization of reaction conditions for the enantioselective hydrogenation of acetophenone




| Entry | Ionic liquid | Co-solvent | Conversion (%) | Selectivity (%) | | | ee (%) | Config[a] |
|---|---|---|---|---|---|---|---|---|
| | | | | Aa | Ab | Ac | | |
| 1 | $BMIMBF_4$ | – | 20.0 | 96.2 | 2.7 | 1.1 | 51.6 | *S* |
| 2 | $BMIMPF_6$ | – | 2.7 | 98.3 | 1.7 | 0.0 | 2.6 | *S* |
| 3 | $BMIMNTf_2$ | – | 0.3 | 99.0 | 1.0 | 0.0 | 8.6 | *S* |
| 4 | BMIMOTf | – | 3.0 | 95.1 | 4.9 | 0.0 | 35.0 | *S* |
| 5 | $BMIMBF_4$ | MeOH | 74.3 | 99.6 | 0.2 | 0.2 | 36.0 | *S* |
| 6 | $BMIMBF_4$ | EtOH | 92.1 | 98.9 | 0.6 | 0.5 | 63.0 | *S* |
| 7 | $BMIMBF_4$ | *i*PrOH | 62.0 | 99.5 | 0.5 | 0.0 | 22.0 | *S* |
| 8 | $BMIMBF_4$ | $H_2O$ | 92.3 | 96.9 | 0.0 | 3.1 | 54.0 | *S* |
| 9 | $BMIMBF_4$ | THF | 12.0 | 99.2 | 0.0 | 0.8 | 28.0 | *S* |
| 10 | $BMIMBF_4$ | Toluene | 23.0 | 98.7 | 0.0 | 0.3 | 24.0 | *S* |
| 11[b] | $BMIMBF_4$ | EtOH | 34.0 | 70.5 | 17.0 | 12.5 | 0.7 | *S* |
| 12[c] | $BMIMBF_4$ | EtOH | 96.3 | 62.1 | 7.4 | 30.5 | 0.9 | *S* |
| 13[d] | $BMIMBF_4$ | EtOH | 93.5 | 94.7 | 2.0 | 3.3 | 35.0 | *S* |
| 14[e] | $BMIMBF_4$ | EtOH | 2.5 | 100.0 | 0.0 | 0.0 | 3.3 | *S* |

Reaction was carried out at 40 °C for 10 h, $PH_2$:5.0 MPa, substrate:0.86 mmol, cinchonidine act as the stabilizer, substrate/Rh/modifier (1*R*,2*R*)-DPEN = 200:1:2, Vionic liquid:1 mL, Vionic liquid:Vco-solvent = 1:1, BMIMOH = 0.20 mol $L^{-1}$ was added. Products were analyzed by a GC instrument with an FID detector and β-DEX120 capillary column

[a]Determined by compare the optical rotation of hydrogenation products and known compounds

[b]No BMIMOH introduced

[c]No modifier (1*R*,2*R*)-DPEN and no BMIMOH introduced

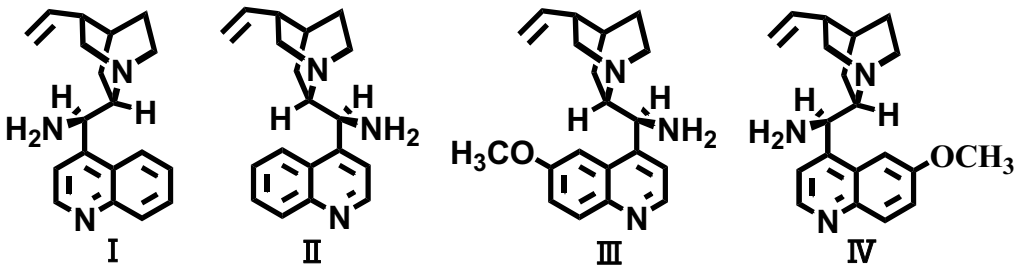
[d]Ru NPs as catalyst

[e]Pt NPs as catalyst

chemoselectivity as well as enantioselectivity were sharply decreased (Table 1, entries 11 vs. 6). In order to clarify the importance of (1*R*,2*R*)-DPEN modification effect to Rh NPs, both (1*R*,2*R*)-DPEN and BMIMOH were removed (Table 1, entry 12), accompany with the catalytic reaction activity recover, the chemoselectivity toward α-phenethyl alcohol was further decreased. This should be explained that the removal of the modifier made it easier for both the carbonyl and benzene ring in acetophenone to get close to the active site of the Rh NPs. Ru and Pt nanoparticles were also tested under similiar conditions (Table 1, entries 13–14), however, moderate to poor catalytic performance were obtained.

The introduction of ligands as metal particle stabilizers is one of the most effective methods to improve the catalytic performance of supported metal catalysts [28, 29]. Without the stabilizer, the hydrogenation resulted in conversion as low as 16.3%, chemoselectivity of 73.0% and ee value of 26.0% (Table 2, entry 1). Different cinchona alkaloid stabilizers have significant different effects on catalytic performance especially in activity and enantioselectivity (Table 2, entries 2–5). When cinchonidine act as the stabilizer, 92.1% conversion, 98.9% chemoselectivity and 63.0% ee were achieved. However, only 38.9% conversion, 99.5% chemoselectivity and 22.0% ee were obtained in the presence of stabilizer cinchonine (Table 2, entry 3). On the other hand,

**Table 2** Effect of different stabilizers and modifiers on enantioselective hydrogenation of acetophenone



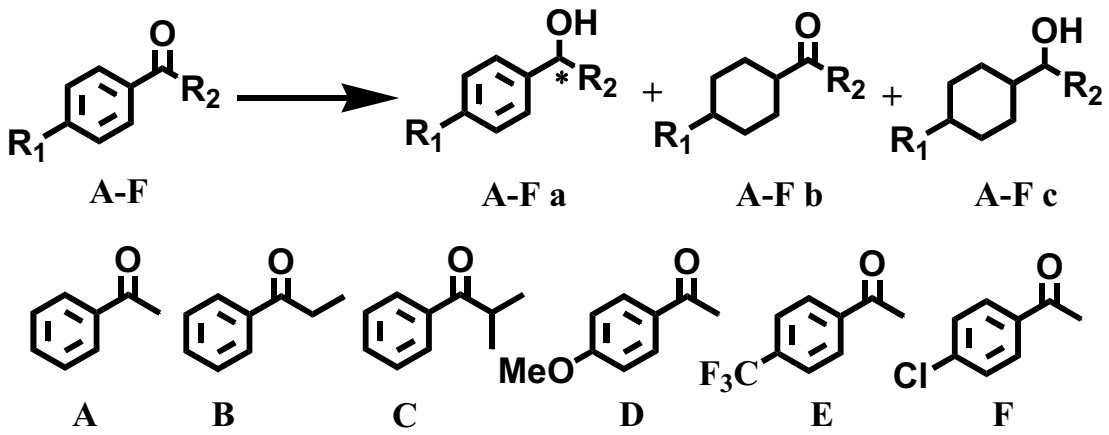

| Entry | Stabilizer | Modifier | Conversion (%) | Selectivity (%) | | | ee (%) | Config. |
|---|---|---|---|---|---|---|---|---|
| | | | | a | b | c | | |
| 1 | – | (1*R*,2*R*)-DPEN | 16.3 | 73.0 | 20.8 | 6.2 | 26.0 | *S* |
| 2 | Cinchonidine | (1*R*,2*R*)-DPEN | 92.1 | 98.9 | 0.6 | 0.5 | 63.0 | *S* |
| 3 | Cinchonine | (1*R*,2*R*)-DPEN | 38.9 | 99.5 | 0.5 | 0.0 | 22.0 | *S* |
| 4 | Quinidine | (1*R*,2*R*)-DPEN | 62.0 | 98.7 | 0.7 | 0.6 | 37.1 | *S* |
| 5 | Quinine | (1*R*,2*R*)-DPEN | 62.5 | 99.2 | 0.3 | 0.5 | 39.2 | *S* |
| 6 | Cinchonidine | I | 64.0 | 99.2 | 0.4 | 0.4 | 26.0 | *S* |
| 7 | Cinchonidine | II | 98.7 | 99.1 | 0.0 | 0.9 | 9.4 | *R* |
| 8 | Cinchonidine | III | 77.4 | 98.8 | 0.5 | 0.7 | 10.0 | *S* |
| 9 | Cinchonidine | IV | 99.5 | 98.7 | 0.5 | 0.8 | 30.0 | *S* |

The reaction conditions are the same as in Table 1 (V $BMIMBF_4$:V EtOH = 1:1)

modifiers have critical important impact on the activity, chemoselectivity as well as enantioselectivity in most existed heterogeneous catalysis systems too [30, 31]. Considering cinchona alkaloid derivative modifiers exhibited excellent catalytic performance in some previous research [15, 32]. We further tested the effect of different cinchona alkaloid derivative modifiers on the catalytic performance (Table 2, entries 6–9). However, the enantioselectivity in the acetophenone heterogeneous enantioselective hydrogenation were obviously decreased. The decrease in catalytic performance should reasonably be ascribed to the steric hindrance caused by cinchona alkaloids derivatived modifiers and which indicated the high specific correlation between the modifier and substrate in heterogeneous enantioselective catalysis. Above results indicate that a proper solvent, stabilizer, and combination of chiral diamine modifier and base additive contribute to the formation of the catalytic species and the chiral induction. Compared with the catalyst using phosphine as the stabilizer [19–22], cinchona alkaloids stabilized Rh catalysts exhibited unique feature. Obviously, synergistic effect between (*1R,2R*)-DPEN and cinchonidine significantly accelerated the reaction rate and enhanced the enantioselectivity.

Some representative examples of the asymmetric hydrogenation of the aromatic ketones catalyzed by cinchonidine-stabilized Rh NPs modified by (1*R*,2*R*)-DPEN are listed in Table 3. The extent of catalytic activity, chemoselectivity, and enantioselectivity appeared to be delicately affected by substituent in the substrate. The catalytic activity decreased and the chemoselectivity and enantioselectivity increased by increasing the bulkiness of the alkyl group from methyl or primary alkyl to isopropyl (Table 3, entries 1–3). It is worth mentioning that the chemoselectivity of isobutyrylbenzene hydrogenation was 100% and enantioselectivity could reach as high as 70.2%. When the substituent was on the aromatic ring, the catalytic hydrogenation enantioselectivity was generally decreased (Table 3, entries 4-6). The catalytic activity decreased with the increase of the electron-donating effect in the *para*-position of aromatic ring. While the chemoselectivity decreased with the increase of the electron-drawing effect in the *para*-position of aromatic ring. It is worth mentioning that a certain percentage of dehalogenation products were detected in the 4'-chloroacetophenone hydrogenation (Table 3, entry 6). Additionally, enantioselective hydrogenation of ketoesters including ethyl pyruvate and ethyl acetoacetate was also tested. Ethyl pyruvate could be hydrogenated to corresponding *R*-ethyl lactate with 100% conversion and 9.9% ee value with the same reaction conditions as in Table 3; however, no enantiodifferentiating ability was detected during the ethyl acetoacetate hydrogenation. Above all, the effect of the steric bulk and the

**Table 3** Enantioselective hydrogenation of aromatic ketones catalyzed by (1*R*,2*R*)-DPEN modified cinchonidine stabilized Rh nanoparticles




| Entry | Substrate | Conversion (%) | Selectivity (%) | | | ee (%) | Config. |
|---|---|---|---|---|---|---|---|
| | | | A–F a | A–F b | A–F c | | |
| 1 | A | 92.1 | 98.9 | 0.6 | 0.5 | 63.0 | *S* |
| 2 | B | 78.0 | 99.0 | 0.0 | 1.0 | 65.0 | *S* |
| 3 | C | 66.0 | 100.0 | 0.0 | 0.0 | 70.2 | *S* |
| 4 | D | 47.0 | 100.0 | 0.0 | 0.0 | 29.0 | *S* |
| 5 | E | 99.5 | 87.1 | 2.5 | 10.4 | 38.0 | *S* |
| 6[a] | F | 62.0 | 90.0 | 0.0 | 0.0 | 35.0 | *S* |

The reaction conditions are the same as in Table 1 (V $BMIMBF_4$:V EtOH = 1:1)

[b]10% dehalogenation product was detected

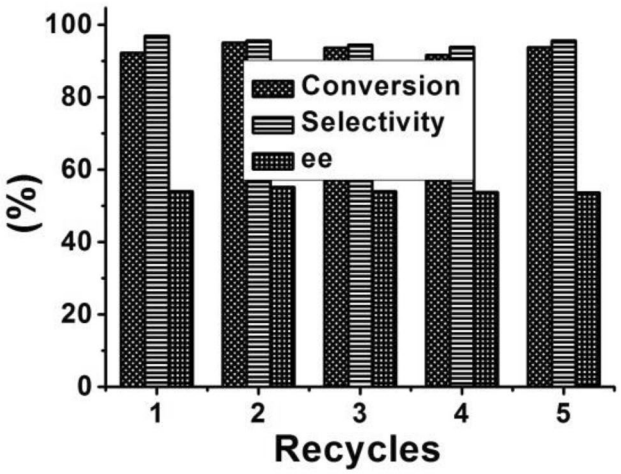



**Fig. 3** Recyclability of enantioselective hydrogenation of acetophenone in a mixture solvent of $BMIMBF_4$ and $H_2O$. Reaction conditions are the same as in Table 1

electronic nature of the substrate influence the activity, chemoselectivity as well as the enantioselectivity in the catalytic reaction.

Figure 3 indicated the recyclability of enantioselective hydrogenation of acetophenone in a mixture solvent of $BMIMBF_4$ and $H_2O$, and it was found that the leaching amount of the Rh NPs catalyst was negligible by ICP-AES analysis in continuous catalytic cycle. The catalyst could be reused by simple liquid–liquid extraction after the reaction. The process was repeated 5 times and the results showed that Rh NPs could be recycled without significant loss of catalytic activity, chemoselectivity and enantioselectivity.

## 3 Conclusion

In conclusion, cinchona alkaloids stabilized Rh NPs catalyst, when modified by chiral diamine like (1*R*,2*R*)-DPEN, exhibited high activity, chemoselectivity and up to 70.2% enantioselectivity in challenging heterogeneous enantioselective hydrogenation of aromatic ketones to corresponding aromatic alcohols in imidazolium-based ionic liquids under mild conditions. In comparison with the catalyst using phosphine as the stabilizer, cinchona alkaloids stabilized Rh catalysts exhibited unique feature. Apparently, synergistic effect between (*1R,2R*)-DPEN and cinchonidine significantly

accelerated the reaction rate and enhanced the enantioselectivity. Catalytic system could be reused 5 times without significant loss in activity, chemoselectivity and enantioselectivity. The work reported herein provides new direction for heterogeneous enantioselective nanometal catalysis in ionic liquids. Additional work is currently in progress in this and related areas.

## 4 Experimental Section

### 4.1 Materials

All manipulations involving air-sensitive materials were carried out using standard Schlenk line techniques under an atmosphere of nitrogen. Various substrates and other reagents were analytical grade. The purity of hydrogen was over 99.99%. Products were analyzed by GC instrument with an FID detector and Chrompack Chirasil-DEX column (25 m × 0.25 mm). Products were confirmed by GC–MS and NMR. The TEM analyses were performed in a JEOL JEM 2010 transmission electron microscope operating at 200 kV with nominal resolution of 0.25 nm. The X-ray photoelectron spectroscopy (XPS) measurements were performed on a Thermo ESCALAB 250 spectrometer.

### 4.2 Synthesis of Rh NPs

In a typical experiment, $RhCl_3 \cdot 3H_2O$ (0.014 mmol) and cinchonidine (0.028 mmol) was well dispersed in $BMIMBF_4$ (1 mL) (BMIM = 1-butyl-2,3-dimethylimidazolium) and the reaction mixture was placed in a 20 mL stainless-steel high pressure reactor. After stirring the mixture at room temperature under an atmosphere of argon for 30 min, a constant pressure of $H_2$(g) (4 MPa) was admitted to the system and the content was stirred for 1 h at 60 °C. The reactor was cooled to ambient temperature and carefully vented. A dark solution was obtained. The Rh NPs embedded in $BMIMBF_4$ were employed for hydrogenation studies (see below). Isolation of the Rh NPs for TEM and XPS analysis was achieved by dissolving the mixture in acetone (5 mL), centrifuging (5000 rpm for 10 min), washing with acetone (3 × 5 mL) and drying under vacuum.

### 4.3 General Procedure for the Heterogeneous Enantioselective Hydrogenation

In stainless steel autoclave, previously prepared Rh(0) catalyst was charged with the appropriate modifier, co-solvent and substrate, and then the autoclave was sealed and purged with pure hydrogen several times. After the reactants were heated to predetermined temperature, the reaction timing began. After completion of the reaction and cooling to ambient temperature, the products were isolated by high speed centrifugation or liquid–liquid extraction and analyzed by gas chromatography. Isolation of the Rh NPs for TEM analysis after catalytic cycles was achieved by dissolving the reaction mixture in acetone (5 mL), centrifuging (5000 rpm for 10 min), washing with acetone (3 × 5 mL) and drying under vacuum.

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