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FULL PAPER

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Selective hydrogenation of aromatic compounds using modified iridium nanoparticles

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Till now, Ionic liquid-stabilized metal nanoparticles were investigated as catalytic materials, mostly in the hydrogenation of simple substrates like olefins or arenes. The adjustable hydrogenation products of aromatic compounds, including quinoline and relevant compounds, aromatic nitro compounds, aromatic ketones as well as aromatic aldehydes, are always of special interest, since they provide more choices for additional derivatization. Iridium nanoparticles (Ir NPs) were synthesized by the H_2 reduction in imidazolium ionic liquid. TEM indicated that the Ir NPs is worm-like shape with the diameter around 12.2 nm and IR confirmed the modification of phosphine-functionalized ionic liquids (PFILs) to the Ir NPs. With the variation of the modifier, solvent and reaction temperature, substrate like quinoline and relevant compounds, aromatic nitro compounds, aromatic ketones as well as aromatic aldehydes could be hydrogenated by Ir NPs with interesting adjustable catalytic activity and chemoselectivity. Ir NPs modified by PFILs are simple and efficient catalysts in challenging chemoselective hydrogenation of quinoline and relevant compounds, aromatic nitro compounds, aromatic ketones as well as aromatic aldehydes. The activity and chemoselectivity of the Ir NPs could be obviously impacted or adjusted by altering the modifier, solvent and reaction temperature.

KEYWORDS
aromatic ketone, chemoselective hydrogenation, ionic liquids, iridium nanoparticles, quinoline

1 | INTRODUCTION

The metal nanoparticles with characteristics of small diameter and narrow size distribution are receiving more and more attention in both academic and industrial research because of their inherent large surface-to-volume ratio and quantum size effect.[1–5] Additional stabilize or modify effects, generally provided by amines, phosphines and thiols, to nanoparticles influence the catalytic activity, selectivity and recyclability.[6–12] With many advantages, such as negligible volatility, excellent thermal stability, remarkable solubility and a variety of available structures, ionic liquids can act as both the solvent and the stabilizer in the preparation of metal nanoparticles. Up to now, ionic liquid stabilized nanometal particles were tested as catalysts generally in the hydrogenation of olefins or arenes.[3,4,13–16]

The selective hydrogenation of aromatic compounds (quinoline and its analogues, aromatic nitro compounds, aromatic ketones, aromatic aldehydes etc.) by means of heterogeneous catalysis is among the most important

subjects in industrial organic chemistry.[17–26] The controllable hydrogenation products of aromatic compounds are always of interest, as they offer various options for further derivatization. Dupont and co-workers[27] prepared well-dispersed transition metal nanoparticles with narrow size distribution in a variety of imidazolium-based ionic liquids, which showed very interesting activity and selectivity in the hydrogenation of ketones. Leitner and co-workers[18] successfully used Ru(0) nanoparticles prepared in ionic liquids in the chemoselective hydrogenation of biomass-derived substrates. Herein, we report originally that iridium nanoparticles (Ir NPs), with the modification of phosphine-functionalized ionic liquids (PFILs), exhibit interesting chemoselectivity in the hydrogenation of quinoline and its analogues, aromatic nitro compounds, aromatic ketones and aromatic aldehydes.

2 | RESULTS AND DISCUSSION

2.1 | Synthesis and characterization of Ir NPs

The synthesis of Ir NPs was achieved through the reduction of $[Ir(cod)Cl]_2$ in $[BMIM]PF_6$ (BMIM=1-butyl-2,3-dimethylimidazolium), which afforded a dark suspension. A black powder could be isolated from the black suspension by adding acetone and then centrifuging (8000 rpm for 10 min). Washed three times with acetone and dried under reduced pressure, the isolated powder was analyzed by transmission electron microscopy (TEM), X-ray photoelectron spectroscopy (XPS), X-ray diffraction (XRD), and infrared spectroscopy (IR).

The Ir NPs isolated before or after catalytic hydrogenation were placed as a thin film on a carbon-

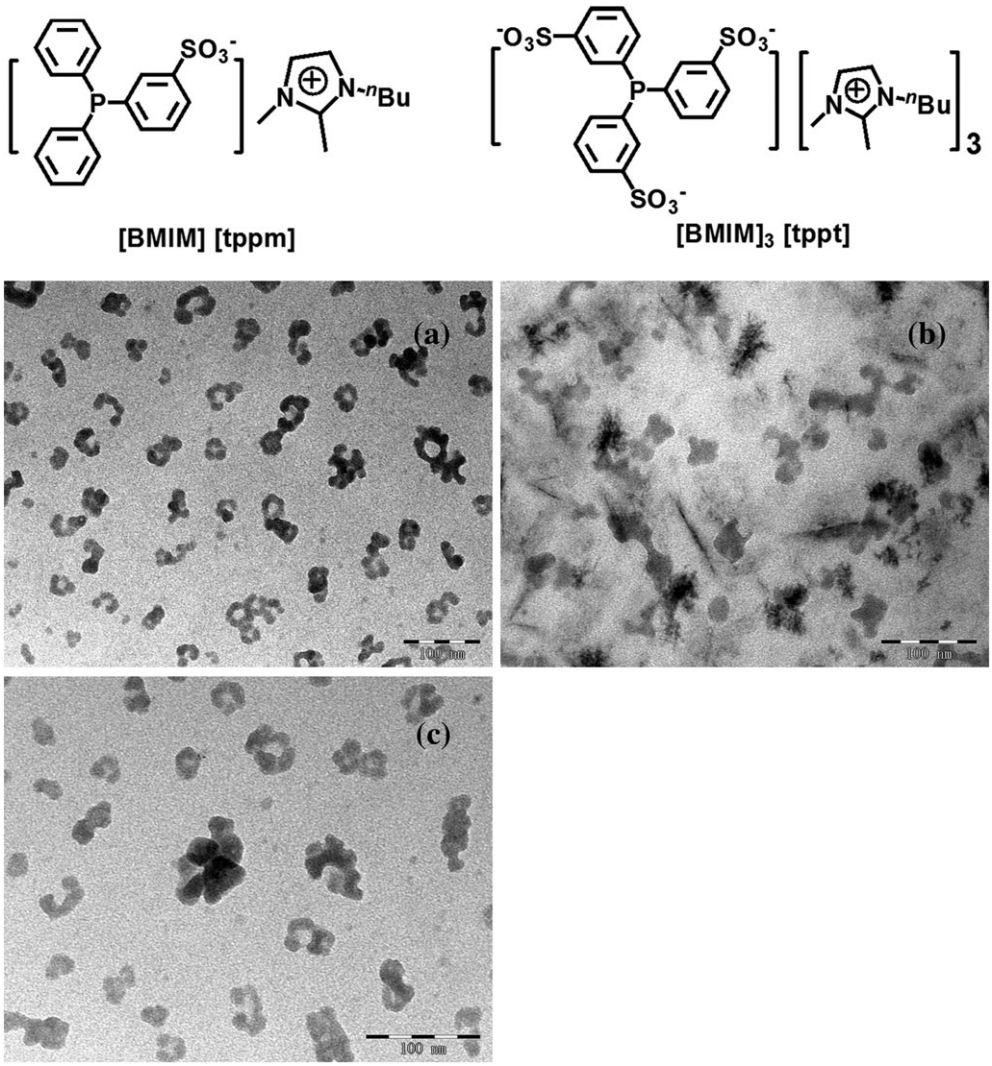


FIGURE 1 TEM images of (a) Ir NPs as prepared, (b) Ir NPs after three consecutive recycles of styrene hydrogenation, (c) Ir NPs after three consecutive recycles of styrene hydrogenation in the presence of phosphine-functionalized ionic liquid modifier [BMIM][tppm]

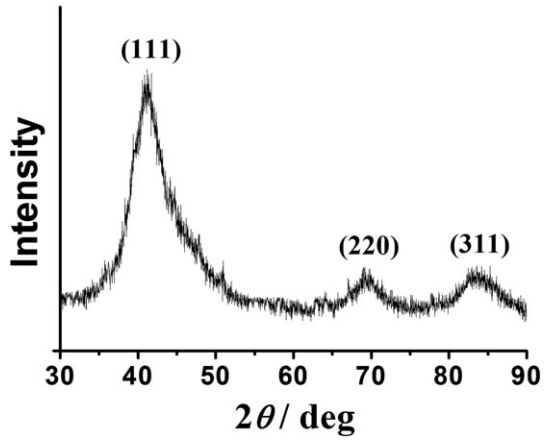


FIGURE 2 X-Ray diffraction pattern of isolated Ir NPs as prepared

coated copper grid and characterized by TEM analysis, which was employed to characterize the obtained Ir NPs and determine their mean diameters (Figure 1). The TEM image of Ir NPs as prepared exhibited a worm-like shape, with an average diameter of 12.2 nm (Figure 1, image A). After three consecutive recycles of styrene hydrogenation, the TEM image showed a trend toward agglomeration of the particles (Figure 1, image B). However, Ir NPs (Figure 1, image C) remained almost the same shape in the presence of phosphine-functionalized ionic liquid modifier [BMIM][tppm] after three consecutive hydrogenation recycles.

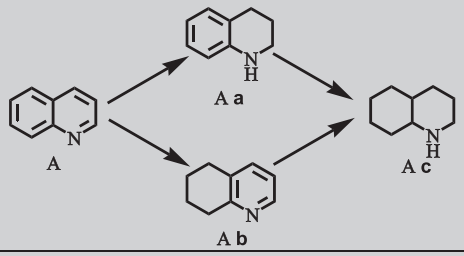
The surface characteristics of Ir NPs were investigated by XPS. XPS analysis of Ir NPs prepared in $[BMIM]PF_6$ showed the presence of iridium, nitrogen and carbon, which signified the presence of the $[BMIM]PF_6$ in the Ir NPs. The BEs for Ir, 60.7 eV and 63.6 eV, indicated Ir NPs were composed of Ir(0) (Figure S1 in supporting information). XRD analysis (Figure 2) showed the presence of broad reflection lines indicating the diffraction by very small crystals. The three diffraction peaks were assigned as the Ir(0) (111), (220) and (311) reflection planes.

IR is effective for the investigation of the mutual ligand-metal interactions of nanometal catalysts. Information on the interaction between the Ir NPs and the phosphine-functionalized ionic liquid modifier [BMIM][tppm] was confirmed by FTIR methods in which the red shift of the $P\text{-}C_6H_5$ deformation around 1469 cm^{-1} was an indication of the coordination of [BMIM][tppm] to the metal Ir(0) (Figure S2 in supporting information).

2.2 | Catalytic hydrogenation

Hydrogenation of quinoline and its analogues is of considerable industrial interests for the production of petrochemicals, fine chemicals, and pharmaceuticals.[20,21] The effect of different reaction factors on the chemoselective hydrogenation of quinoline is listed in Table 1. The phosphine-functionalized ionic liquid modifiers were fairly important in the catalytic system; without the modifier, the hydrogenation resulted in a

TABLE 1 Optimization of reaction conditions for the chemoselective hydrogenation of quinoline[a]



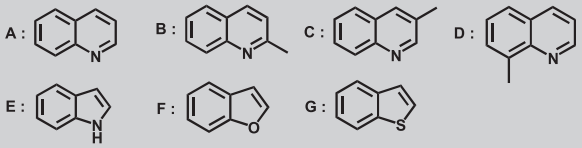
Entry	Catalyst	Yield (%)	Selectivity (%)		
			Aa	Ab	Ac
1	**1**	94.4	100.0	0.0	0.0
2	**2**	92.0	100.0	0.0	0.0
3	**3**	32.8	100.0	0.0	0.0
4	Ir/C	18.8	100.0	0.0	0.0
5	**1**[b]	93.0	100.0	0.0	0.0
6	**1**[c]	90.9	100.0	0.0	0.0

[a]Reaction was carried out at 50 °C. Substrate: in a 1 ml $[BMIM]PF_6$ at [1.8 M], PH_2:3.0 MPa. Substrate/Ir=100:1, Ir/C (5 wt%, 68 mg). Reaction time: 10 h. **1**=Ir NPs modified by [BMIM][tppm], **2**=Ir NPs modified by [BMIM][tppt], **3**=Ir NPs as prepared. Products were analyzed by a GC instrument with an FID detector and HP-5 column.

[b]Third run in the quinoline hydrogenation.

[c]Fifth run in the quinoline hydrogenation.

TABLE 2 Hydrogenation of quinoline and its analogues catalyzed by Ir NPs modified by [BMIM][tppm][a]



Entry	Substrate	Yield (%)	Selectivity (%)		
			A-Ga	A-Gb	A-Gc
1	A	94.4	100.0	0.0	0.0
2	B	100.0	100.0	0.0	0.0
3	C	90.0	100.0	0.0	0.0
4	D	81.0	100.0	0.0	0.0
5	E [b]	33.4	46.1	0.0	53.9
6	F	22.5	100.0	0.0	0.0
7	G	0.0	-	-	-

[a]The reaction conditions are the same as in Table 1.

[b]Reaction time: 15 h.

conversion of 32.8% to 1,2,3,4-tetrahydroquinoline (Table 1, entry 3). Ir NPs modified by [BMIM][tppm] showed higher activity to 1,2,3,4-tetrahydroquinoline than Ir NPs modified by [BMIM][tppt] (Table 1, entries 1-2). The dramatically activity increase of Ir(0) particles in the chemoselective hydrogenation of quinoline (Table 1, entries 1-2 vs 3) should be ascribed to the modification effect of PFILs to Ir(0) particles and the modification effect may be good for the rate-determining dissociative adsorption of H_2 on the Ir(0) site.[26] In comparison, the commercially available Ir/C was utilized as a catalyst for the selective hydrogenation of quinoline, it showed poor activity to 1,2,3,4-tetrahydroquinoline (Table 1, entry 4). The catalyst **1** could be reused 5 times without obvious loss in catalytic activity and chemoselectivity during the quinoline hydrogenation (Table 1, entries 5-6).

We investigated the catalytic performance of [BMIM][tppm] modified Ir NPs for the hydrogenation of quinoline and its analogues (Table 2). Quinoline could be easily hydrogenated to 1,2,3,4-tetrahydroquinoline with absolute chemoselectivity in [BMIM]PF_6 (Table 2, entry 1). The hydrogenation activity of 2-, 3- or 8-methylquinoline was similar to quinoline. Interestingly, the presence of substituent in quinoline had no influence on the hydrogenation chemoselectivity (Table 2, entries 2-4). Other substrates, including indole, 2,3-benzofuran and 1-benzothiophene, were also tested. In comparison to

TABLE 3 Hydrogenation of aromatic nitro compounds by Ir NPs[a]

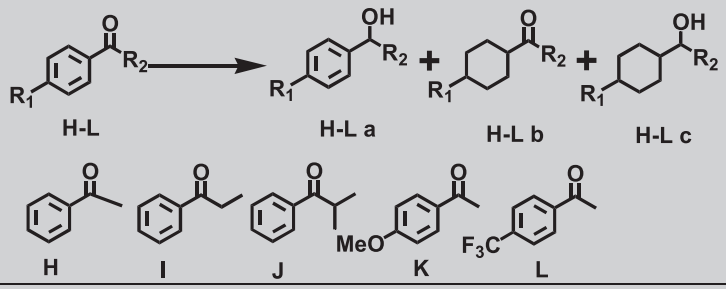
Entry	Substrate	Catalyst	Product	Yield (%)
1	nitrobenzene	**1**	aniline	100.0
2	nitrobenzene	**2**	aniline	100.0
3	nitrobenzene	**3**	aniline	98.2
4	nitrobenzene	Ir/C	aniline	85.1
5	*m*-methylnitrobenzene	**1**	*m*-methylaniline	96.0
6	*p*-methylnitrobenzene	**1**	*p*-methylaniline	100.0
7	*o*-methoxylnitrobenzene	**1**	*o*-methoxyaniline	72.0
8	*p*-methoxylnitrobenzene	**1**	*p*-methoxyaniline	6.0
9	*p*-fluoronitrobenzene	**1**	*p*-fluoroaniline	100.0
10	*o*-chloronitrobenzene	**1**	*o*-chloroaniline	100.0
11	*p*-chloronitrobenzene	**1**	*p*-chloroaniline	100.0

[a]Reaction was carried out at 50 °C. Substrate: in a 1 ml [BMIM]PF_6 at [1.8 M], PH_2:3.0 MPa. Substrate/Ir=100:1, Ir/C (5 wt%, 68 mg). Reaction time: 5 h. **1**=Ir NPs modified by [BMIM][tppm], **2**=Ir NPs modified by [BMIM][tppt], **3**=Ir NPs as prepared. Products were analyzed by a GC instrument with an FID detector and HP-5 column. Only corresponding amino aromatic products were detected during all the catalytic reactions.

quinoline, indole, 2,3-benzofuran and 1-benzothiophene exhibited lower catalytic activity. Indole could be hydrogenated with a high chemoselectivity to complete hydrogenation product (Table 2, entry 5). 2,3-benzofuran could be hydrogenated with the complete chemoselectivity to 2,3-dihydrobenzofuran. 1-Benzothiophene was not reduced (Table 2, entry 7), and the absence of activity may be attributed to the affinity of sulfur atoms for its classical η^1-S adsorption onto the surface of Ir NPs **1** catalyst.

Selective hydrogenation of aromatic nitro compounds by metal nanoparticles have seen remarkable growth in recent years [28,29], we also investigated the hydrogenation of aromatic nitro compounds over PFILs modified Ir NPs. As shown in Table 3, with the superior [BMIM][tppm] modified Ir NPs **1** as the catalyst, almost all the substrates could be efficiently converted into the corresponding substituted amino aromatic compounds under mild conditions except substrates with a methoxy substituent (Table 3, entries 7-8). The methyl- or chloro-substituted nitrobenzene afforded the corresponding amino aromatic compounds and showed comparable reactivity as that of unsubstituted nitrobenzene. [BMIM][tppm] modified Ir NPs **1** catalyst showed high activity for chemoselective hydrogenation of nitrobenzene and its derivatives, which was not apparently affected by the steric effect, but possibly by the electronic effects. In comparison, commercially available Ir/C catalyst was also tested under similar reaction conditions in the hydrogenation of nitrobenzene (Table 3, entry 4). However, Ir/C catalyst showed poor activity towards aniline.

TABLE 4 Chemoselective hydrogenation of aromatic ketones by Ir NPs [a]



Entry	Substrate	Catalyst	Yield (%)	Selectivity (%)		
				H-L a	H-L b	H-L c
1 [b]	H	**3**	0.0	-	-	-
2	H	**3**	89.0	12.6	0.0	87.4
3	H	**1**	39.3	88.0	1.8	10.2
4	H	**2**	19.8	85.4	7.6	7.0
5 [c]	H	**3**	96.6	89.5	0.0	10.5
6 [c]	H	**1**	90.2	94.8	0.9	1.7
7	H	Ir/C	0.0	-	-	-
8 [c]	H	Ir/C	60.0	83.5	0.0	16.5
9	I	**3**	60.8	71.5	13.7	14.8
10 [c]	I	**1**	20.0	93.5	4.0	2.5
11	J	**3**	17.7	54.8	24.9	20.3
12 [c]	J	**1**	8.0	89.1	5.9	5.0
13	K	**3**	0.0	-	-	-
14 [c]	K	**1**	0.0	-	-	-
15	L	**3**	86.8	89.0	9.2	1.8
16 [c]	L	**1**	37.0	96.2	3.0	0.0

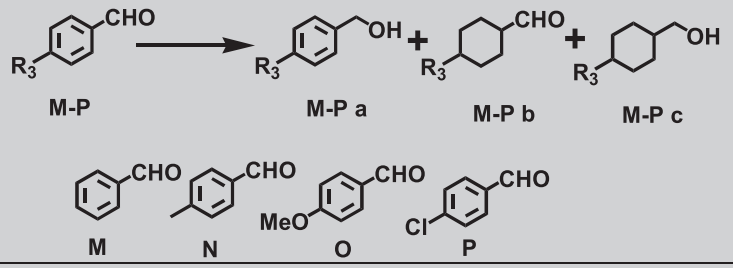
[a]Reaction was carried out at 75 °C. Substrate: in a 1 ml [BMIM]PF_6 solution at [1.8 M], PH_2:3.0 MPa. Substrate/Ir=100:1. Ir/C (5 wt%, 68 mg). Reaction time: 10 h. **1**=Ir NPs modified by [BMIM][tppm], **2**=Ir NPs modified by [BMIM][tppt], **3**=Ir NPs as prepared. Products were analyzed by a GC instrument with an FID detector and β-DEX120 capillary column.

[b]Reaction was carried out at 30 °C.

[c]1 ml water was introduced to catalytic reaction.

The chemoselective reduction of aromatic ketones by means of heterogeneous catalysis is of considerable importance in the synthesis of fine chemicals, particularly intermediates in the fragrance and pharmaceutical industries [17–19]. Additionally, tuning the hydrogenation chemoselectivity of aromatic ketones compounds on heterogeneous catalysis remains a formidable scientific challenge. The effect of different reaction factors on the chemoselective hydrogenation of acetophenone is listed in Table 4 (entries 1-8). The Ir NPs had no catalytic activity under ambient temperature in the hydrogenation of acetophenone (Table 4, entry 1). The phosphine-functionalized ionic liquid modifiers had important influence on the catalytic system. Without the modifier, the hydrogenation resulted in a conversion of 89.0% and a chemoselectivity as high as 87.4% to cyclohexylethanol (Table 4, entry 2). Interestingly, with the PFILs modifiers, the acetophenone hydrogenation showed low activity with high chemoselectivity switched to 1-phenylethanol (Table 4, entries 3-4). Ir NPs modified by [BMIM][tppm] **1** showed higher activity and chemoselectivity to 1-phenylethanol than Ir NPs modified by [BMIM][tppt] **2**. Different from activity change in quinoline hydrogenation with catalysts **1, 2** and **3** (Table 1, entries 1-3), chemoselective reduction of acetophenone with catalysts **1** and **2** showed lower catalytic activity than catalyst **3** (Table 4, entries 2-4), above comparison indicated that all interactions between PFILs modifiers, substrates, as well as the corresponding hydrogenation products and Ir(0) particles have important influence on the rate-determining dissociative adsorption of H_2 on the Ir(0) site. Additionally, the chemoselectivity adjustment in acetophenone hydrogenation with **1, 2** and **3** (Table 4, entries 2-4) suggested that the PFILs modifiers had influence on preferential adsorption of acetophenone to the Ir(0) catalysts followed by reduction with H atoms on the Ir(0) site [26]. Water is an attractive alternative to traditional organic solvents because it is cheap, readily available, nontoxic, non-flammable and safe to environment.[30] Water was introduced to catalytic system to improve the catalytic performance (Table 4, entries 5-6). To our surprise, in the presence of water, the catalytic activity of Ir NPs catalysts **1** and **3** sharply increased with improved chemoselectivity to 1-phenylethanol. We speculate that C=O in acetophenone not only interacts with Ir(0) particles, but it also forms the hydrogen bond with water. As a result, the hydrogen bond between C=O and water improves the activity and chemoselectivity of Ir(0) particles for the hydrogenation of acetophenone to 1-phenylethanol. Commercially available Ir/C catalyst was also tested under similar

TABLE 5 Chemoselective hydrogenation of aromatic aldehydes by Ir NPs[a]



Entry	Substrate	Catalyst	T (°C)	Yield (%)	Selectivity (%)		
					M-P a	M-P b	M-P c
1	M	**1**	30	100.0	100.0	0.0	0.0
2 [b]	M	**1**	75	100.0	0.0	0.0	100.0
3	M	Ir/C	30	83.0	97.5	0.0	2.5
4 [b]	M	Ir/C	75	81.9	97.0	0.0	3.0
5	N	**1**	30	100.0	100.0	0.0	0.0
6 [b]	N	**1**	75	99.0	99.8	0.0	0.2
7	O	**1**	30	42.0	100.0	0.0	0.0
8 [b]	O	**1**	75	82.0	100.0	0.0	0.0
9	P	**1**	30	100.0	100.0	0.0	0.0
10 [b]	P	**1**	75	90.2	0.0	0.0	100.0

[a]Substrate: in a 1 ml [BMIM]PF_6 solution at [1.8 M], PH_2:3.0 MPa. Substrate/Ir=100:1. Ir/C (5 wt%, 68 mg). Reaction time: 10 h. **1**=Ir NPs modified by [BMIM][tppm]. Products were analyzed by a GC instrument with an FID detector and β-DEX120 capillary column.

[b]1 ml water was introduced to catalytic reaction.

reaction conditions in the hydrogenation of acetophenone (Table 4, entries 7-8). Ir/C catalyst showed poor activity and chemoselectivity during the test.

Some representative examples are also listed in Table 4 (entries 2, 6, 9-16) for the chemoselective hydrogenation of aromatic ketones catalyzed by Ir NPs **3** in $[BMIM]PF_6$ or by [BMIM][tppm] modified Ir NPs **1** in a mixture of $[BMIM]PF_6$ and water. In comparison to acetophenone, other substrates generally exhibited lower catalytic activity with major chemoselectivity to aromatic ethanols during the test. The activity and chemoselectivity decreased by increasing the bulkiness of the alkyl group from methyl or primary alkyl to isopropyl (Table 4, entries 2, 6, 9-12). When the substituent was in the *para* position, substrate with electron-donating group showed no reaction activity (Table 4, entries 13-16).

The chemoselective hydrogenation of aromatic aldehydes were also tested (Table 5). In comparison to the chemoselective hydrogenation of aromatic ketones, aromatic aldehydes could be hydrogenated to corresponding C=O hydrogenation products easily. Benzaldehyde could be hydrogenated to corresponding benzyl alcohol in $[BMIM]PF_6$ at 30 °C (Table 5, entry 1). Complete hydrogenation product of benzaldehyde could also be obtained in the mixture solvent of $[BMIM]PF_6$ and water (Table 5, entry 2). Commercially available Ir/C catalyst was also tested under similar reaction conditions in the hydrogenation of benzaldehyde (Table 5, entries 3-4). Ir/C catalyst showed poor activity during the test. When the substituent was in the *para* position of benzaldehyde, C=O and full hydrogenation could be achieved in electron-drawing group substituted substrate (Table 3, entries 9-10). With electron-donating group in the *para* position, *p*-anisaldehyde could be hydrogenated with absolute chemoselectivity to C=O hydrogenation product (Table 3, entries 7-8).

3 | CONCLUSION

In summary, we have demonstrated that Ir NPs modified by PFILs are simple, efficient and recyclable catalysts for the challenging selective hydrogenation of quinoline and its analogues, aromatic nitro compounds, aromatic ketones and aromatic aldehydes. The activity and chemoselectivity of the Ir NPs can be obviously influenced or controlled by variation of the modifier, solvent and reaction temperature. The catalytic performance is complementary to both classical homogeneous and heterogeneous catalysts. 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. $[Ir(cod)Cl]_2$ and Ir/C were purchased from Acros. Various substrates and other reagents were analytical grade. The purity of hydrogen was over 99.99%. Phosphine-functionalized ionic liquids were synthesized according to literature.[31,32] Products were analyzed by GC instrument with an FID detector and HP-5 column (30 m × 0.25 mm) or 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. The XRD analysis was performed in a D/MAX 2550 VB/PC using a graphite crystal as monochromator.

4.2 | Synthesis of Ir NPs

In a typical experiment, $[Ir(cod)Cl]_2$ (0.009 mmol) was well dispersed in $[BMIM]PF_6$ (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) (1 MPa) was admitted to the system and the content was stirred for 10 min at 75 °C. The reactor was cooled to ambient temperature and carefully vented. A dark solution was obtained. The Ir NPs embedded in $[BMIM]PF_6$ were employed for hydrogenation studies (see below). Isolation of the Ir NPs for TEM, XPS and XRD analysis was achieved by dissolving the mixture in acetone (5 ml), centrifuging (8000 rpm for 10 min), washing with acetone (3 × 5 ml) and drying under vacuum.

4.3 | General procedure for the heterogeneous chemoselective hydrogenation

In stainless steel autoclave, PFIL (0.018 mmol) was dispersed in previously prepared Ir(0) catalyst. After stirring at room temperature under an atmosphere of argon for 60 min, the mixture was charged with the appropriate 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 liquid-liquid extraction with diethyl ether and analyzed by gas chromatography. Isolation of the Ir NPs for TEM analysis after catalytic cycles was achieved by dissolving the reaction mixture in acetone (5 ml), centrifuging (8000 rpm for 10 min), washing with acetone (3 × 5 ml) and drying under vacuum.

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