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Asymmetric hydrogenation of α,β-unsaturated ketones catalyzed by Ru–TPPTS–(*S*,*S*)-DPENDS complex in ionic liquids

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Abstract—The asymmetric hydrogenation of α,β-unsaturated ketones catalyzed by the achiral ruthenium monophosphine complex $RuCl_2(TPPTS)_2$ [TPPTS: $P(m\text{-}C_6H_4SO_3Na)_3$] modified by (*S*,*S*)-DPENDS [disodium salt of sulfonated (*S*,*S*)-1,2-diphenyl-1,2-ethylene-diamine] was investigated in ionic liquid [RMIM]Ts (1-alkyl-3-methylimidazolium *p*-methylphenylsulfonates, R = ethyl, butyl, octyl, dodecyl). Chemoselectivity of 100% and 75.9% ee were obtained for benzalacetone under the optimized conditions. The resulting products can be easily separated from the catalyst immobilized in ionic liquid [EMIM]Ts by extraction with *n*-hexane, while the catalyst can be reused seven times without the loss of catalytic activity and enantioselectivity. Especially, the addition of water can improve the performance of the catalyst.

1. Introduction

Catalytic asymmetric hydrogenation is a powerful tool for the preparation of enantiomerically pure compounds. Homogeneous catalysts show distinguished catalytic properties in the asymmetric hydrogenation of simple ketones,[1] but these catalysts are very expensive and difficult to recycle and reuse.[2] Immobilization of the homogeneous catalysts offers an interesting approach to solve these problems.[3] Ionic liquids (ILs) have attracted a great deal of interest because of their capability to immobilize homogeneous asymmetric hydrogenation catalysts and to facilitate the catalysts recycling.[2,4] In recent years, the asymmetric hydrogenations of enamides, arylacrylic acids, aromatic ketones, imines, and β-ketoesters in ionic liquids have been reported,[5] and the results indicate that the organic products can be easily separated by extraction with a less polar organic solvent and the catalysts could be reused several times without a loss in catalytic activity or enantioselectivity.

A highly selective hydrogenation of α,β-unsaturated ketones to chiral allylic alcohols has remained difficult since most existing homogeneous or heterogeneous catalysts tend to catalyze the hydrogenation of the C=C bond preferentially over a coexisting C=O bond.[6] This longstanding problem can be solved by applying *trans*-$RuCl_2$(binap)(1,2-diamine) as a precatalyst.[1a] Recently, asymmetric hydrogenation of α,β-unsaturated ketones has been investigated by diversified chiral homogeneous catalysts.[6,7,1a] We are interested in the asymmetric hydrogenation of α,β-unsaturated ketones in ionic liquids for catalyst recycling. Previously, we reported the asymmetric hydrogenation of aromatic ketones catalyzed by $RuCl_2(TPPTS)_2$–(*S*,*S*)-DPENDS (see Fig. 1) complex in ionic liquids,[8] and the catalyst immobilized in ionic liquid can be reused several times without a significant decrease in activity and enantioselectivity. These promising results encouraged us

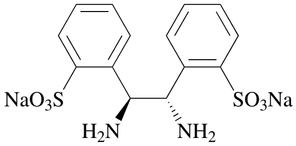

Figure 1. (*S*,*S*)-DPENDS.

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to continue examining the asymmetric hydrogenation of α,β-unsaturated ketones catalyzed by $RuCl_2(TPPTS)_2$–(*S*,*S*)-DPENDS complex in ionic liquids [RMIM]Ts (see Fig. 2). The results show that the catalyst not only exhibits excellent chemoselectivity and good enantioselectivity, but also can be recycled and reused.

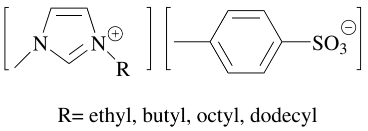


Figure 2. Ionic liquid [RMIM]Ts.

2. Results and discussion

2.1. Asymmetric hydrogenation of benzalacetone in different ionic liquids medium

To examine the catalytic performance of $RuCl_2(TPPTS)_2$–(*S*,*S*)-DPENDS complex in ionic liquids for asymmetric hydrogenation of α,β-unsaturated ketones, we chose benzalacetone as the standard substrate. Three different possibilities of hydrogenation are expected to form saturated ketone (SK), unsaturated alcohol (UA), and saturated alcohol (SA) in the asymmetric hydrogenation of benzalacetone (see Scheme 1). The results in Table 1 indicate that the R-groups in [RMIM]Ts markedly influence the enantioselectivity of the catalyst. When the chain length of the R-group in [RMIM]Ts was increased from ethyl to dodecyl, the catalytic activity and selectivity of allylic alcohol decreased only slightly, but the enantioselectivity obviously decreased from 71.8% to 59.9% ee. The increase of alkyl chain length in [RMIM]Ts is not favorable to the solubility of water-soluble chiral ligand (*S*,*S*)-DPENDS in ionic liquids and the chiral modification of (*S*,*S*)-DPENDS to Ru catalyst is reduced, and then the enantioselectivity decreases. A similar phenomenon was observed in asymmetric hydrogenation of aromatic ketones in our previous work.[8]

2.2. Influence of base on asymmetric hydrogenation of benzalacetone

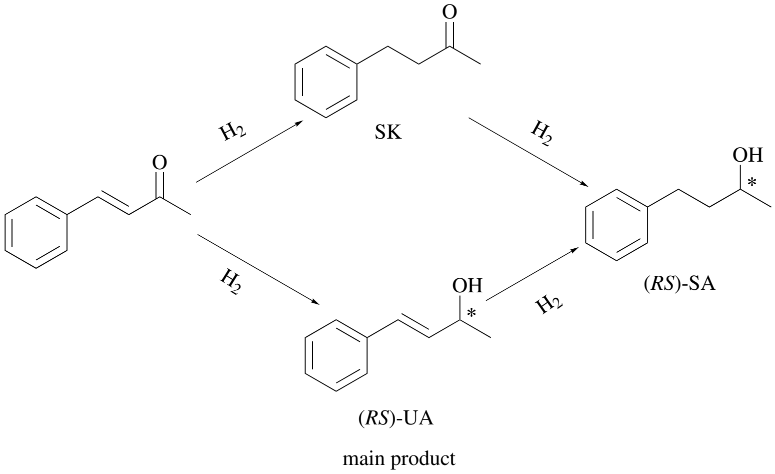
Asymmetric hydrogenation of benzalacetone catalyzed by the $RuCl_2(TPPTS)_2$–(*S*,*S*)-DPENDS complex under different base conditions in the mixture of ionic liquid [EMIM]Ts and water was investigated, and the results are summarized in Table 2. As co-catalyst, the base obviously influences the catalytic activity, chemoselectivity, and enantioselectivity of the catalyst. When K_2CO_3, a weak base, was used, very low conversion (only 4.6%) was observed. However, in the presence of these strong bases, such as LiOH, NaOH, and KOH, the catalyst displays excellent activity and enantioselectivity, high conversions and chemoselectivities as well as good ee values as shown in Table 2. Among the strong bases, KOH represents the highest catalytic performance. The conversion and chemoselectivity as well as ee value can reach up to 100%, 100%, and 71.8%, respectively.

2.3. Effect of water on the asymmetric hydrogenation of benzalacetone

According to the literature, water affects the reactions in ionic liquids.[9] To improve the performance of $RuCl_2$-$(TPPTS)_2$–(*S*,*S*)-DPENDS–KOH catalyst, we evaluate the effect of water on the asymmetric hydrogenation of benzalacetone with the results summarized in Table 3. The results suggest that the addition of appropriate amount of water is not only beneficial to exerting the catalytic activity and chemoselectivity of the catalyst, but also helps improve the enantioselectivity, because water enhances the solubility of catalyst in ionic liquids. However, lower activity and chemoselectivity are observed when only water is used as solvent.

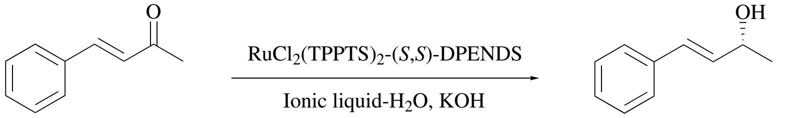
2.4. Effect of different ruthenium precatalysts on asymmetric hydrogenation of benzalacetone

The asymmetric hydrogenation of benzalacetone catalyzed by different ruthenium precatalysts was investigated in ionic liquid [EMIM]Ts with the results summarized in



Scheme 1. Asymmetric hydrogenation of benzalacetone.

Table 1. Asymmetric hydrogenation of benzalacetone in different ionic liquids[a]



R	[RMIM]Ts	Conversion[b] (%)	Selectivity[b] (%)			ee[c] (%)
			SK	SA	UA	
Ethyl	[EMIM]Ts	100	—	—	100	71.8
Butyl	[BMIM]Ts	100	0.5	—	99.5	66.3
Octyl	[OMIM]Ts	98.4	1.1	—	98.9	61.2
Dodecyl	[DoMIM]Ts	99.5	1.3	—	98.7	59.9

[a] Reaction conditions: benzalacetone: 0.85 mmol; Ru/benzalacetone/(S,S)-DPENDS = 1:112:3; ionic liquid: 0.5 mL, ionic liquid/H_2O (v/v) = 1:1; KOH: 0.36 mol/L; P_{H_2}: 5.0 MPa; T: 40 °C; t: 2 h.

[b] The conversions and selectivities were determined by GC with a Chrompack Chirasil-DEX column (25 m × 0.25 mm).

[c] The ee values were determined by HPLC with a Chiralcel OD-H column (25 cm × 4.6 mm). All products were in the (R)-configuration.

Table 2. Influence of the base on the asymmetric hydrogenation of benzalacetone[a]

Base	Conversion (%)	Selectivity (%)			ee (%)
		SK	SA	UA	
K_2CO_3	4.6	6.0	—	94.0	58.7
LiOH	98.8	0.6	—	99.4	70.5
NaOH	100	0.2	—	99.8	70.0
KOH	100	—	—	100	71.8

[a] Reaction conditions: ionic liquid/[EMIM]Ts; the others are the same as in Table 1.

Table 3. Influence of water on asymmetric hydrogenation of benzalacetone[a]

V_{ILs}/V_{H_2O}	Conversion (%)	Selectivity (%)			ee (%)
		SK	SA	UA	
No water	34.2	9.8	—	90.2	46.6
19:1	71.7	8.6	—	91.4	64.0
3:1	98.4	0.9	—	99.1	60.5
1:1	100	—	—	100	71.8
1:3	100	—	—	100	75.9
No IL	74.9	7.9	1.0	91.1	66.0

[a] Reaction conditions: ionic liquid/[EMIM]Ts, $V_{\text{ILs}} + V_{H_2O} = 1$ mL, the others are the same as in Table 1.

Table 4. Effect of different ruthenium precatalysts on asymmetric hydrogenation of benzalacetone[a]

Ruthenium precatalyst	Conversion (%)	Selectivity (%)			ee (%)
		SK	SA	UA	
$[RuCl_2(TPPTS)_2]_2$	100	—	—	100	75.9
$RuCl_2$(COD)-2TPPTS	52.9	1.3	—	98.7	75.0
$RuCl_3$-2TPPTS	14.4	13.5	—	86.5	76.4
$RuCl_3$-4TPPTS	43.1	0.9	—	99.1	76.2

[a] Reaction conditions: ionic liquid/H_2O (v/v) = 1:3, the others are the same as in Table 3.

Table 4. The results indicate that different ruthenium catalyst precursors can significantly influence the activity and selectivity. The data show that $[RuCl_2(TPPTS)_2]_2$ is the most efficient catalyst precursor: the conversion, ee value and chemoselectivity of the products can reach up to 100%, 75.9%, and 100%, respectively. With respect to $RuCl_2$(COD), the low activity was observed because COD is exchanged with TPPTS to form an active catalyst. In the case of $RuCl_3$ as a catalyst precursor, the three-valent ruthenium was reduced to a low oxidation state, and then coordinated with TPPTS, while a part of TPPTS was oxidized to OTPPTS.[10] Accordingly, there was a longer induction period for $RuCl_3$, which resulted in very lower activity. Furthermore, the lower chemoselectivity was obtained due to the oxidation of TPPTS. The activity and chemoselectivity increased with an increase in molar ratio of TPPTS to $RuCl_3$, which demonstrate that catalyst precursor $RuCl_3$ is propitious to hydrogenate C=C, before forming catalytic active species.

2.5. Asymmetric hydrogenation of different α,β-unsaturated ketones

To further explore the scope of substrates, we also investigated the effectiveness of catalyst $RuCl_2(TPPTS)_2$–(S,S)-DPENDS–KOH for the asymmetric hydrogenation of other three α,β-unsaturated ketones in ionic liquid [EMIM]Ts with the results summarized in Table 5. The data demonstrate that the catalyst exhibits high activity and selectivity for 4-phenyl-3-buten-2-ol with good enantioselectivity (75.9% ee) in the asymmetric hydrogenation of benzalacetone. For 4-methyl-3-penten-2-one and 3-methyl-2-cyclohexen-1-one, as well as 2-cyclohexen-1-one, the catalyst exhibited good to excellent chemoselectivity for allylic alcohol. However, low conversions (29.1% for 4-methyl-3-penten-2-one and 50.2% for 3-methyl-2-cyclohexen-1-one) were obtained although the reaction time was prolonged to 6 and 12 h, respectively. The highest enantioselectivity was observed (84.7% ee) for 4-methyl-3-penten-2-one, but for 3-methyl-2-cyclohexen-1-one, only 13.5% ee was achieved. For 2-cyclohexen-1-one, the catalyst displayed good activity and reasonable enantioselectivity (48.1% ee).

2.6. Catalyst recycling

For catalyst recycling, the separation of the hydrogenation products from the catalyst was investigated, using

Table 5. Asymmetric hydrogenation of different α,β-unsaturated ketones

Substrate	Time (h)	Conversion (%)	Selectivity (%)			ee (%)
			SK	SA	UA	
Benzalacetone[a]	2	100	—	—	100	75.9
4-Methyl-3-penten-2-one[b]	6	29.1	—	15.1	84.9	84.7
3-Methyl-2-cyclohexen-1-one[b]	12	50.2	—	—	100	13.5
2-Cyclohexen-1-one[b]	6	97.4	2.6	—	94.1	48.1

[a] Reaction conditions are the same as in Table 2.

[b] Except for reaction time, the other conditions are the same as in Table 2. The conversions, selectivities and ee values were determined by GC with a Chrompack Chirasil-DEX CB column (25 m × 0.25 mm). 4-Methyl-3-penten-2-one was of the (*R*)-configuration, the other two products were of the (*S*)-configuration.

Table 6. Recycling of catalyst RuCl$_2$(TPPTS)$_2$-(1*S*,2*S*)–DPENDS–KOH in ionic liquid[a]

Run	Conversion (%)	Selectivity (%)			ee (%)
		SK	SA	UA	
1	100	—	—	100	75.9
2	99.3	0.7	—	99.3	75.1
3	98.9	0.6	—	99.4	73.5
4	92.1	0.8	—	99.2	72.7
5	91.6	0.7	—	99.3	71.9
6	91.0	0.8	—	99.2	72.3
7	87.9	0.9	—	99.1	72.5
8[b]	96.0	0.6	—	99.4	74.9

[a] Reaction conditions are the same as in Table 4.

[b] 0.18 mmol of KOH was added.

benzalacetone as a model substrate. The experiments demonstrate that the products can be easily separated by extraction with *n*-hexane, while the catalyst and modifiers immobilized in ionic liquid [EMIM]Ts can be recycled and reused several times. As shown in Table 6, the RuCl$_2$(TPPTS)$_2$–(*S*,*S*)-DPENDS catalyst shows extremely high efficiency upon seven successive cycles without significant loss of enantioselectivity and catalytic activity. Although the activity slightly decreased in the seventh run, the conversion rose from 87.9% (run 7) to 96.0% (run 8) when 0.18 mmol KOH was added into the reaction system in the eighth run, which further proves the notable influence of the base on catalytic activity. Similar phenomena were observed in asymmetric hydrogenation of aromatic ketones in ionic liquids.[8]

3. Conclusions

In conclusion, we have successfully applied the RuCl$_2$(TPPTS)$_2$–(*S*,*S*)-DPENDS–KOH catalyst to the asymmetric hydrogenation of α,β-unsaturated ketones in ionic liquids, which give high activity and chemoselectivity for the α,β-unsaturated alcohol with good enantioselectivity. The resulting products can be easily separated from the catalyst system by extraction with *n*-hexane. The catalyst immobilized in ionic liquid not only exhibits excellent chemoselectivity and enantioselectivity in the asymmetric hydrogenation of benzalacetone, but can also be reused several times without significant loss of the ee value, selectivity for conversion.

4. Experimental

4.1. Materials and methods

Benzalacetone (Acros, >98%), 3-methyl-2-cyclohexen-1-one (Acros, >98%), 4-methyl-3-penten-2-one (TCI, >97%), cyclohexen-1-one (Acros, >97%), RuCl$_2$ (COD) [dichloro(1,5-cyclooctadiene)ruthenium(II), 99%] (Strem Chemical Co.) and hydrogen (99.99%) were used as received. RuCl$_3$·3H$_2$O and other reagents are analytical grades. TPPTS, ionic liquids [RMIM]Ts, RuCl$_2$(TPPTS)$_2$, and chiral modifier (*S*,*S*)-DPENDS were synthesized according to known methods in our laboratory.[11]

4.2. A typical procedure for asymmetric hydrogenation of benzalacetone

To a 60 mL stainless autoclave with a glass linear and magnetic stirrer were added the ionic liquid [EMIM]Ts, RuCl$_2$(TPPTS)$_2$, (1*R*,2*R*)-DPENDS, KOH, H$_2$O, and benzalacetone. Hydrogen was introduced to the desired pressure after the reaction mixture had been purged five times with H$_2$. The mixture was stirred at 40 °C for a predetermined period of time. After hydrogen was vented, *n*-hexane (5 mL) was added to extract the organic product. The resultant two layers were mixed thoroughly by vigorous stirring for 5 min. The upper solution layer was separated by decantation. This manipulation was repeated one more time. The combined extracts containing the product was purified by silica gel (yield 98%). The products were analyzed by GC and HPLC. The conversions and selectivities were determined by GC with a Chrompack Chirasil-DEX column (25 m × 0.25 mm). The ee values were determined by HPLC with a Chiralcel OD-H column (25 cm × 4.6 mm). The ee value was calculated from the equation: ee (%) = $100 \times (R - S)/(R + S)$. The recovered catalyst in ionic liquids was reused in the next batch of the catalytic reaction under identical conditions.

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