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Asymmetric Epoxidation of Chromenes Using Manganese(III) Complexes with Novel Chiral Salen-like Schiff Base Ligands

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Abstract Three novel chiral salen-like schiff base ligands and their Mn(III) complexes containing different amino acid unit have been synthesized and characterized. Asymmetric epoxidation reactions show these complexes are effective catalysts for the chromenes with buffer NaOCl as terminal oxidant and pyridine *N*-oxide as co-catalyst in the presence of ionic liquid. Good-to -excellent enantioselectivity and acceptable yields can be obtained under optimum reaction conditions. Catalyst **4c** gives the highest ee (95%) for 6-chloro-2,2-dimethylchromene among these catalytic performances. Furthermore, compared the enantioselectivity of catalyst **4c** with the other two catalysts **4a** and **4b**, the positive experimental results suggest that the steric effect of the ligands plays an important role in the asymmetric catalysis.

Keywords Manganese(III) complex · Schiff base · Asymmetric catalysis · Epoxidation · Ionic liquid · Amino acid

1 Introduction

Chiral epoxides are extremely useful building blocks in the synthesis of chiral compounds for the pharmaceuticals as well as for fine chemicals [1–4]. The use of metal complexes of chiral salen ligands in asymmetric synthesis has been widespread in recent years [5, 6]. Numerous metal complexes containing salen derivatives have been synthesized and extensively used as catalysts for a range of asymmetric reactions, such as aziridination [7], cyclopropanation [8], Diels–Alder cycloaddition [9], lactide polymerization [10], Michael addition [11], CO_2 fixation [12], Sulfide oxidation [13], hydrolytic kinetic resolution [14] and so on. Chiral Mn(III) salen complexes have received much interest due to their scope of applications as homogeneous catalysts in asymmetric epoxidation of un-functionalised alkenes, which have been studied extensively by Jacobsen, Katsuki and so on [15, 16]. Although these metallosalen complexes are excellent catalysts for asymmetric reactions, the separation and recycling of homogeneous catalysts is problematic, making the entire catalytic process economically nonviable for industrial processes. Therefore, many attempts have been made to discover the recoverable metallosalen complexes, such as immobilization of catalyst onto inorganic materials [17, 18] or polymer [19], ionic liquid-functionalized of catalyst [20, 21], oligomer [22, 23] and highly active salen catalysts [24, 25]. Among these methods, ionic liquids have generated much excitement in the field of organic synthesis, particularly for metal complex catalysis, because ionic liquids are green reaction media and can improve the separation, activity and recycling of the catalysts [26–29].

The structural and electronic properties of salen ligands play the important roles in the catalytic activities. A great deal of chiral schiff base ligands have been synthesized for enantioselective catalytic reactions over the past decade, but the vast majority of such studies have been focused on the Jacobson–Katsuki type chiral catalysts, the study of novel salen-like chiral schiff base ligands has attracted less attention [30, 31]. Amino acids are one of the most important classes of the building blocks of life: they are the structural subunits of proteins, peptides, and many secondary metabolites. They are also the interesting ligands

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with cheap chiral center and some of their metal complexes have been approved as excellent catalysts for many important asymmetric organic transformations [32]. Amino acids have received considerable attention to get cheaper and highly active chiral salen catalyst. To the best of our knowledge, few people plough into creating amide-based salen-like schiff ligands. Herein, we first report the synthesis and characterization of three novel chiral salen-like schiff base ligands and their Mn(III) complexes derived from different amino acids. Their activities in asymmetric epoxidation of chromenes using NaClO as the oxidant, PyNO as the axial base, and ionic liquid as the reaction solvent have been studied and discussed systematically.

2 Experimental Section

2.1 Materials

All starting materials were purchased from the Tianjin Chemical Reagent Factory or Aldrich Chemical Company. All solvents and raw materials were of analytical grade and used without further purification unless otherwise stated. 2,2-Dimethylchromene and its derivatives [33], α-amino amide and its derivatives (**2a–2c)** [34], and ionic liquid L-1-ethyl-3-(1′-hydroxy-2′-propanyl)imidazolium bromide [35] were synthesized according to the literature procedures.

2.2 Physical Methods and Analysis

^{1}H NMR and ^{13}C NMR spectra were recorded on a Mercury Plus 400 spectrometer with TMS as internal standard. IR spectra were obtained on a Nicolet 170SX FT-IR spectrophotometer as KBr discs. LC–MS spectra were performed on a Bruker Daltonics Esquire 6000 mass spectrometer. Elemental analyses were taken using a Perkin-Elmer 240C analytical instrument. All reactions were monitored by TLC. TLC was performed on glass plates coated with silica gel 60F254. The crude products were purified by flash chromatography. The enantiomeric excesses of the chiral epoxides were determined by chiral high-performance liquid chromatograph analysis (Daicel Chiralcel OD-H chiral column, *n*-hexane: *i*-PrOH = 99:1 (v/v), 1.0 mL/min, 254 nm) using a water 600 controller with 2996 photodiode array detector.

2.3 Preparation

*2.3.1 General Procedure for the Preparation of the Novel Chiral Schiff Base Ligands (**3a–3c**)*

The ethanol solution (40 mL) of 3,5-di-*tert*-butylsalicylaldehyde (0.24 g, 1.0 mmol) was added dropwise to the ethanol solution (40 mL) of compound (**2a–2c**, 1.0 mmol). The reaction mixture was stirred under refluxing for 8 h before stirring was discontinued. After cooling down to room temperature, the solvent was evaporated under reduced pressure. The residue was purified by flash chromatography on silica gel, eluted with petroleum ether-EtOAc (5:1, v/v) to afford the desired product as a yellow solid.

*2.3.1.1 Compound **3a*** Yield: 0.33 g (83%). ^{1}H NMR (400 MHz, $CDCl_3$): δ 12.55 (1H, s), 8.71 (1H, s), 8.49 (1H, s), 8.21 (1H, s), 7.49 (1H, d, $J = 2.4$ Hz), 7.17 (1H, d, $J = 2$ Hz), 7.13 (1H, t, $J = 7.6$ Hz), 7.02 (2H, d, $J = 8$ Hz), 6.87 (1H, t, $J = 7.6$ Hz), 4.21 (1H, d, $J = 6.8$ Hz) 1.68 (2H, d, $J = 6.8$ Hz), 1.48 (9H, s), 1.32 (9H, s). ^{13}C NMR (100 MHz, $CDCl_3$): δ 172.3, 169.1, 157.6, 148.6, 141.3, 137.1, 128.6, 127.4, 126.9, 124.9, 122.2, 120.5, 119.7, 117.5, 68.6, 35.1, 34.2, 31.4, 29.4, 21.3. LC–MS: *m/z* 397.4 $[M+H]^+$. Anal. Calcd for $C_{24}H_{32}N_2O_3$: C, 72.70; H, 8.13; N, 7.06. Found: C, 72.80; H, 8.18; N, 7.14. FT-IR (KBr): 3385, 2959, 2870, 1658, 1622, 1595, 1539, 1455, 1386, 1248, 1174, 1103, 1038, 750 cm^{-1}.

*2.3.1.2 Compound **3b*** Yield: 0.38 g (89%). ^{1}H NMR (400 MHz, $CDCl_3$): δ 12.63 (1H, s), 8.74 (1H, s), 8.41 (1H, s), 8.13 (1H, s), 7.50 (1H, d, $J = 2.4$ Hz), 7.19 (1H, d, $J = 2.4$ Hz), 7.11 (1H, t, $J = 7.2$ Hz), 6.99 (2H, dd, $J = 7.2$, 1.2 Hz), 6.87 (1H, t, $J = 7.6$ Hz), 3.89 (1H, d, $J = 4$ Hz), 2.61–2.57 (1H, m), 1.48 (9H, s), 1.33 (9H, s), 1.08 (3H, d, $J = 6.8$ Hz), 1.09 (3H, d, $J = 6.8$ Hz). ^{13}C NMR (100 MHz, $CDCl_3$): δ 171.1, 169.7, 157.6, 148.7, 141.1, 137.0, 136.3, 129.7, 128.6, 127.4, 127.0, 126.9, 124.8, 122.3, 120.5, 119.7, 117.4, 41.3, 35.1, 34.2, 31.4, 29.4. LC–MS: *m/z* 425.4 $[M+H]^+$. Anal. Calcd for $C_{26}H_{36}N_2O_3$: C, 73.55; H, 8.55; N, 6.60. Found: C, 73.67; H, 8.62; N, 6.69. FT-IR (KBr): 3380, 2960, 2871, 1657, 1626, 1532, 1457, 1390, 1273, 1248, 1173, 1103, 826, 750 cm^{-1}.

*2.3.1.3 Compound **3c*** Yield: 0.38 g (81%). ^{1}H NMR (400 MHz, $CDCl_3$): δ 12.45 (1H, s), 8.63 (1H, s), 8.12 (1H, s), 8.05 (1H, s), 7.46 (1H, d, $J = 2.4$ Hz), 7.26–7.14 (5H, m), 7.13 (1H, t, $J = 6.4$ Hz), 7.09–6.93 (3H, m), 6.85 (1H, t, $J = 6.4$ Hz), 4.23 (1H, dd, $J = 4.8$, 3.6 Hz), 3.46 (1H, dd, $J = 10$, 3.6 Hz), 3.23 (1H, dd, $J = 8.4$, 5.2 Hz), 1.47 (9H, s), 1.28 (9H, s). ^{13}C NMR (100 MHz, $CDCl_3$): δ 171.6, 169.9, 157.8, 148.9, 141.3, 137.2, 128.7, 127.5, 126.9, 124.9, 122.3, 120.5, 119.9, 117.5, 79.1, 35.2, 34.2, 32.6, 31.4, 29.4, 19.7, 17.2. LC–MS: *m/z* 473.4 $[M+H]^+$. Anal. Calcd for $C_{30}H_{36}N_2O_3$: C, 76.24; H, 7.68; N, 5.93. Found: C, 76.33; H, 7.76; N, 6.01. FT-IR (KBr): 3357, 2957, 2864, 1656, 1627, 1534, 1454, 1388, 1238, 1174, 1087, 1033, 847, 751 cm^{-1}.

2.3.2 General Procedure for the Preparation of the Mn(III) Complexes (*4a–4c*)

A mixture of $Mn(OAc)_2{\cdot}4H_2O$ (3.0 mmol) and new ligand (**3a–3c**) (1.0 mmol) in ethanol (40 mL) was heated to 80 °C for 6 h under nitrogen atmosphere. After cooling down to room temperature, lithium chloride (6.0 mmol) was added and the resulting mixture was refluxed for additional 2 h while exposed to air. The solvent was removed under reduced pressure and the residue was extracted with dichloromethane (3 × 10 mL). The extract was washed with water (2 × 10 mL), brine and dried over anhydrous Na_2SO_4, and then concentrated to give the crude product. The crude product was recrystallized from petroleum ether yielding the desired complex as a dark brown powder.

2.3.2.1 Complex ***4a*** Yield: 0.36 g (74%). LC–MS: *m/z* 449.2 $[M-Cl]^+$. Anal. Calcd for $C_{24}H_{30}ClMnN_2O_3$: C, 59.45; H, 6.24; N, 5.78. Found: C, 59.36; H, 6.17; N, 5.71. FT-IR (KBr): 3379, 2957, 2866, 1615, 1568, 1530, 1476, 1385, 1242, 1171, 1103, 1027, 749 cm^{-1}.

2.3.2.2 Complex ***4b*** Yield: 0.38 g (74%). LC–MS: *m/z* 477.3 $[M-Cl]^+$. Anal. Calcd for $C_{26}H_{34}ClMnN_2O_3$: C, 60.88; H, 6.68; N, 5.46. Found: C, 60.81; H, 6.59; N, 5.39. FT-IR (KBr): 3382, 2959, 2870, 1609, 1556, 1474, 1385, 1271, 1242, 1172, 1104, 840,748 cm^{-1}.

2.3.2.3 Complex ***4c*** Yield: 0.45 g (80%). LC–MS: *m/z* 525.2 $[M-Cl]^+$. Anal. Calcd for $C_{30}H_{34}ClMnN_2O_3$: C, 64.23; H, 6.11; N, 4.99. Found: C, 64.16; H, 6.03; N, 4.90. FT-IR (KBr): 3420, 2957, 2868, 1609, 1558, 1473, 1385, 1243, 1171, 1083, 1027, 844, 749 cm^{-1}.

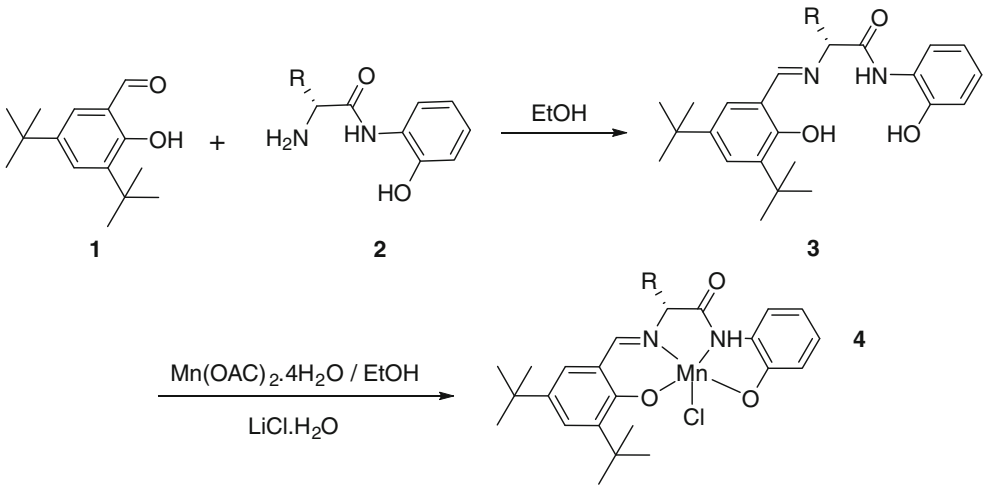
2.3.3 General Procedure for the Asymmetric Epoxidation of Chromenes

The substrate (2.0 mmol) and PyNO (0.3 mmol) were added to the CH_2Cl_2 (4 mL) solution of Mn(III) complex (0.10 mmol) in the presence of ionic liquid L-1-ethyl-3-(1′-hydroxy-2′-propanyl)imidazolium bromide. After the addition of buffered NaOCl (4.0 mmol) (pH 11.3) at 0 °C, the resulting mixture was stirred vigorously and monitored by TLC. After the reaction was totally completed, the mixture was diluted with CH_2Cl_2 (2 × 10 mL). The organic layer was separated, washed with water and brine, and then dried with $MgSO_4$. The solvent was removed under reduced pressure to afford the crude product, which was purified by flash chromatography on silica gel (petroleum ether/CH_2Cl_2 = 2:1) to afford the corresponding epoxide. The enantiomeric excesses of the chiral epoxide were determined by chiral high-performance liquid chromatograph analysis.

3 Result and Discussion

3.1 Synthesis and Characterizations

The outline of the synthesis of three novel chiral salen-like schiff base ligands **3a–3c** and their Mn(III) complexes



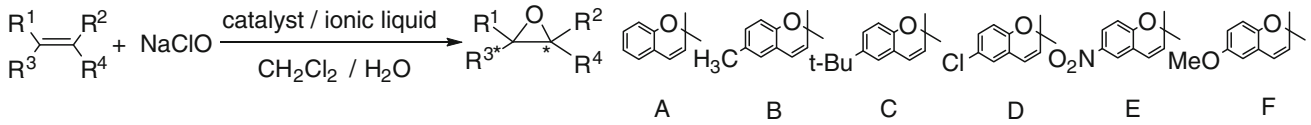
Scheme 1 Synthesis of ligands **3a–3c** and corresponding Mn(III) complexes **4a–4c**

4a–4c is presented in Scheme 1. These ligands have been prepared by two steps, the α-amino amide and its derivative (**2a–2c)** bearing a cheap chiral center have been first prepared according to the reported procedure [34], then compound (**2a–2c)** reacted with 3,5-di-*tert*-butylsalicylaldehyde, respectively, in a 1:1 molar ratio affording salen-like schiff base ligand (**3a–3c**). To elucidate electronic and steric effects of chiral center in the catalytic reaction systematically, L-alanine, L-valine, and L-phenylalanine were used as the starting materials, which bear a methyl, isopropyl or benzyl group as the donor substituent on the amino acid moiety, respectively. The compounds **3a–3c** in EtOH solution were heated with excess $Mn(OAc)_2{\cdot}4H_2O$ for insertion of the Mn(II) center. Then, LiCl was added and the mixture was heated to reflux for an additional 2 h to obtain the Mn(III) complexes. These compounds were well characterized by NMR, LC–MS, FT-IR, and Elemental analyses.

3.2 Catalytic Activity Studies

It is known that some 2,2-disubstituted-3,4-epoxychromene derivatives show unique physiological activity and have wide range of applications [36]. The asymmetric catalytic activity of the Mn(III) complexes **4a–4c** are systematically investigated with the substrates of 2,2-dimethylchromene (A), 2,2,6-trimethylchromene (B), 6-*tert*-butyl-2,2-dimethylchromene (C), 6-chloro-2,2-dimethylchromene (D), 6-nitro-2,2-dimethylchromene (E), and 6-methoxy-2,2-dimethylchromene (F). Ionic liquid L-1-ethyl-3-(1′-hydroxy-2′-propanyl)imidazolium bromide is used as the reaction solvent to improve the separation of chiral Mn(III)

Table 1 Asymmetric epoxidation of chromenes using complexes **4a**, **4b** and **4c** as catalysts[a] in the presence of ionic liquid[b]



A B C D E F

Entry	Alkene	Catalyst	Time (h)[c]	Yield (%)[d]	ee (%)[e]	Configuration[f]
1	A	**4a**	3	86	81	3*R*,4*R*-(+)
2	B	**4a**	3	77	83	3*R*,4*R*-(+)
3	C	**4a**	3	79	83	3*R*,4*R*-(+)
4	D	**4a**	3	89	88	3*R*,4*R*-(+)
5	E	**4a**	3	83	81	3*R*,4*R*-(+)
6	F	**4a**	3	81	85	3*R*,4*R*-(+)
7	A	**4b**	3	89	84	3*R*,4*R*-(+)
8	B	**4b**	3	86	91	3*R*,4*R*-(+)
9	C	**4b**	3	83	89	3*R*,4*R*-(+)
10	D	**4b**	3	92	91	3*R*,4*R*-(+)
11	E	**4b**	3	88	87	3*R*,4*R*-(+)
12	F	**4b**	3	83	90	3*R*,4*R*-(+)
13	A	**4c**	3	90	91	3*R*,4*R*-(+)
14	B	**4c**	3	87	94	3*R*,4*R*-(+)
15	C	**4c**	3	88	92	3*R*,4*R*-(+)
16	D	**4c**	3	98	95	3*R*,4*R*-(+)
17	E	**4c**	3	92	93	3*R*,4*R*-(+)
18	F	**4c**	3	88	94	3*R*,4*R*-(+)

A 2,2-dimethylchromene, *B* 2,2,6-trimethylchromene, *C* 6-*tert*-butyl-2,2-dimethylchromene, *D* 6-chloro-2,2-dimethylchromene, *E* 6-nitro-2,2-dimethylchromene, *F* 6-methoxy-2,2-dimethylchromene

[a] Reaction conditions: substrate (2 mmol), catalyst (5 mol%), NaOCl (4 mmol), PyNO (15 mol%)

[b] Ionic liquid: L-1-ethyl-3-(1′-hydroxy-2′-propanyl)imidazolium bromide

[c] Monitored by TLC every other 20 min

[d] Isolated yield

[e] Determined by HPLC on chiral OD-H column

[f] Absolute configuration is determined by comparison of the sign of [α] D with the literature value

salen complexes [35]. Organic co-catalyst plays an important role in the Jacobsen epoxidation, therefore, pyridine *N*-oxide is used as co-catalyst due to its remarkable effects on both the activity and enantioselectivity of the enantioselcetive epoxidation [37, 38]. Asymmetric epoxidation reactions are performed with 5 mol% of the complexes **4a**–**4c** with substrates in CH_2Cl_2 at 0 °C using buffer NaOCl (pH 11.3) as terminal oxidant and pyridine *N*-oxide as co-catalyst in the presence of ionic liquid. Table 1 summarizes the catalytic performance of complexes **4a**–**4c** in the enantioselective epoxidation of chromenes. As expected, reaction solvent plays a crucial role in the asymmetric epoxidation of chromene. The reaction time of ionic liquid system is very short, and the product can be easily separated due to the solvent power of the ionic liquid for many organic and inorganic substances compared with the well-documented reaction time of Jacobson type chiral catalysts [39, 40]. The experimental results show these complexes are effective catalysts for the asymmetric epoxidation of chromenes in the presence of ionic liquid. It is noteworthy that the catalyst **4c** shows high enantioselectivity in excess of 91% and excellent chemical yield (yield 87–98%, ee 91–95%) (Table 1, entries 13–18), especially epoxidation of 6-chloro-2,2-dimethylchromene (yield 98%, ee 95%). The use of catalyst **4a** insteading of **4c** leads to remarkable reduced entantioselectivity and chemical yield (yield 77–89%, ee 81–88%) (Table 1, entries 1–6). This feature can be attributed to the steric factors of the chiral center of the catalyst, the steric hindrance of benzyl group is lager than the methyl group. To elucidate the steric effect of catalyst in the catalytic reaction systematically, the catalyst **4b** with isopropyl group has been prepared. The asymmetric epoxidation of chromenes with catalyst **4b** is examined under the same condition. As expected, the catalyst **4b** gives better chemical yield and entantioselectivity than the catalyst **4a**, but poorer than that of the catalyst **4c** (Table 1, entries 7–12). Furthermore, compared the epoxidation activities of catalyst **4c** with the other two catalysts **4a** and **4b**, we can conclude that different substituent groups of chiral schiff base complex have crucial effects on the catalytic performances. This result supports the assumption that steric factors and electronic effects play the important roles in the asymmetric catalysis.

4 Conclusion

In conclusion, this work emphasizes a combinational strategy to encourage more attempts to explore new highly effective catalysts for the enantioselective epoxidation of nonfunctional olefins, three novel chiral salen-like schiff base ligands and the corresponding Mn(III) complexes functionalized by different amino acids have been prepared. These Mn(III) complexes are efficient catalysts for the asymmetric epoxidation of chromenes with good-to-excellent chemical yields and enantioselectivity using NaClO as the oxidant in the presence of PyNO as the axial base, especially epoxidation of 6-chloro-2,2-dimethylchromene with catalyst **4c** in the presence of ionic liquid. The steric effect of chiral center in the catalyst has crucial effect on the catalytic performance of the Mn(III) complex. The complex **4c** bearing a benzyl group exhibits the best catalytic performance in the epoxidation reaction. These observations suggest that the cooperation of steric factor and chiral center of the complex plays an important role in the catalytic performances.

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