Lewis-Acid-Assisted Highly Selective Oxidative Cross-Coupling Polymerization with Copper Catalysts

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An asymmetric oxidative cross-coupling polymerization with a binary catalyst system of an achiral or chiral copper complex and a Lewis acid was investigated. Both the copper and Lewis acid catalysts significantly affected the catalyst activity, cross-coupling and stereoselectivities during the polymerization. The polymerization of methyl 3,7-dihydroxynaphthalene-2-carboxylate using a copper(I)-bisoxazoline catalyst at room temperature under an O_2 atmosphere in the presence of $Yb(OTf)_3$ proceeded with a cross-coupling selectivity of 97%.

KEY WORDS: Asymmetric Oxidative Coupling / Cross-Coupling / Lewis Acid / Binaphthol /

The oxidative coupling reaction of a mixture of two differently substituted 2-naphthol derivatives generally affords a mixture of three coupling products, a cross-coupling and two homo-coupling products, but there have been few reports on a catalytic and selective cross-coupling process, which is attractive because of directly affording a 1,1'-bi-2-naphthol (BINOL) with a C_1 symmetrical structure.[1–6] Recently, we found that a catalytic asymmetric oxidative coupling between 2-naphthol and 3-hydroxy-2-naphthoate derivatives using CuCl-(S)-2,2'-isopropylidenebis-(4-phenyl-2-oxazoline) [CuCl-(S)Phbox] (Scheme 1) proceeds in a highly cross-coupling selective manner.[7–10] This was further used for the polymerization of the unsymmetric 6,6'-bi-2-naphthol derivatives, such as **1** (Scheme 2), to give poly(BINOL),[11–14] which is interesting as an optically active and electroluminescent material, etc.[15–17] However, during these reactions, the cross-coupling and stereo-selectivities were significantly affected by the substrate or monomer structure and still have not been sufficiently controlled.

The oxidative coupling reaction includes a radical coupling step,[1,18–20] in which the radical species generated by the one-electron oxidation participate in the coupling process. On the other hand, a Lewis acid catalyst, such as the trifluoromethanesulfonate salts of ytterbium and scandium [$Yb(OTf)_3$ and $Sc(OTf)_3$], is often used as an effective tool for selectivity control during the radical reaction, especially, the radical addition process.[21–27] Therefore, in our initial study, the oxidative coupling reaction of the 2-naphthol and 3-hydroxy-2-naphthoate derivatives with a copper catalyst in the presence of $Yb(OTf)_3$ was examined, and a significant effect on controlling both the cross-coupling and stereoselectivity was observed.[28]

This novel binary catalyst system for the oxidative cross-coupling was then used for the polymerization. This study focused on the polymerization of 3,7-dihydroxynaphthalene-2-carboxylate **2**, which is facilely prepared from the commer-

Scheme 1.

R=Me : **1a**
nHex : **1b**
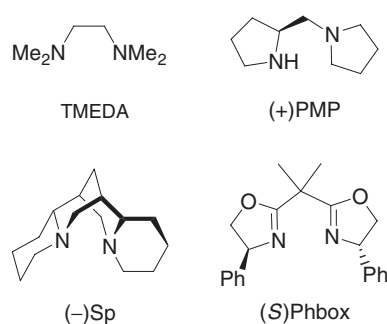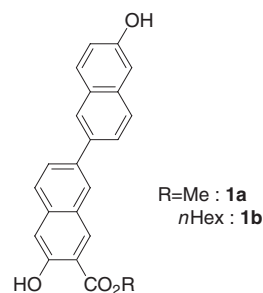
Scheme 2.

cially available acid by esterification and leads to a polymer bearing a mixture of three BINOL-unit structures, a cross-coupling (Y) and two homo-coupling ones (X and Z).[13] The polymerization with various copper catalysts in the presence of the Lewis acid (LA) was investigated (Scheme 3).

EXPERIMENTAL

Materials

Dry THF (Kanto) was used for the oxidative coupling reactions. The optically active ligands, (S)-(+)-1-(2-pyrrolidi-

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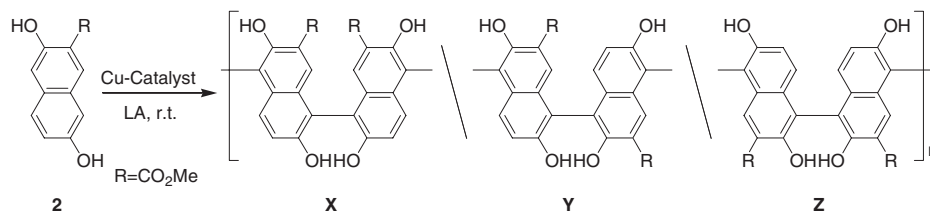
Scheme 3.

nylmethyl)pyrrolidine [(+)PMP] (TCI), (−)-sparteine [(−)Sp] (Sigma), and Phbox (Aldrich), were used as received. The achiral copper complex, di-μ-hydroxo-bis[(N,N,N′,N′-tetramethylethylenediamine) copper(II)] chloride [CuCl(OH)-TMEDA], was purchased from TCI (Scheme 1).

Monomer **2** was synthesized by esterification of 3,7-dihydroxy-2-naphthoic acid (TCI) with methanol and sulfuric acid in dichloromethane.

Model compound **4**: M.p.: >250 °C (decompose). ^{1}H NMR (DMSO-d_6): δ 3.69 (s, 3H, OCH$_3$), 4.00 (s, 3H, OCH$_3$), 6.85–7.49 (m, 6H, aromatic), 7.76 (s, 1H, aromatic), 8.47 (s, 1H, aromatic), 9.06 (s, 1H, –OH), 9.54 (s, 1H, –OH), 9.87 (s, 1H, –OH), 10.00 (s, 1H, –OH). ^{13}C NMR (DMSO-d_6): δ 52.66, 52.67, 110.04, 111.74, 114.47, 115.40, 115.95, 117.32, 122.20, 122.48, 125.79, 127.09, 127.39, 127.54, 128.05, 130.19, 131.48, 132.12, 151.02, 151.25, 152.46, 153.59, 168.76, 169.86. IR (KBr, cm^{-1}): 3421, 2923, 1681, 1623, 1519, 1446, 1392, 1342, 1234, 1103, 1045. Anal. calcd for C$_{24}$H$_{18}$O$_8$: C, 66.36; H, 4.18. Found: C, 66.36; H, 4.33.

Model compound **5**: M.p.: >250 °C (decompose). ^{1}H NMR (DMSO-d_6): δ 3.98 (s, 6H, OCH$_3$), 6.81–7.29 (m, 6H, aromatic), 8.47 (s, 2H, aromatic), 9.68 (s, 2H, –OH), 10.08 (s, 2H, –OH). ^{13}C NMR (DMSO-d_6): δ 52.67, 110.00, 114.51, 117.13, 122.48, 125.66, 128.04, 129.72, 131.18, 150.77, 152.52, 169.80. IR (KBr, cm^{-1}): 3401, 2925, 1677, 1629, 1515, 1446, 1398, 1340, 1224, 1081, 1024. Anal. calcd for C$_{24}$H$_{18}$O$_8$: C, 66.36; H, 4.18. Found: C, 66.35; H, 4.22.

Cross-coupling compound **9**: M.p.: >250 °C (decompose). ^{1}H NMR (CDCl$_3$): δ 3.73 (s, 3H, OCH$_3$), 3.90 (s, 3H, OCH$_3$), 3.97 (s, 3H, OCH$_3$), 4.06 (s, 3H, OCH$_3$), 4.89 (s, 1H, –OH), 7.06–7.47 (m, 6H, aromatic), 7.83 (s, 1H, aromatic), 8.62 (s, 1H, aromatic), 10.67 (s, 1H, –OH). IR (KBr, cm^{-1}): 3444, 2952, 1733, 1718, 1677, 1600, 1508, 1232, 1137, 1029. Anal. calcd for C$_{26}$H$_{22}$O$_8$: C, 67.53; H, 4.80. Found: C, 67.52; H, 4.76.

Polymerization (Typical Procedure)

A mixture of CuCl and diamine in THF ([CuCl]/[diamine] = 0.1/0.12) was stirred for 30 min under an O$_2$ atmosphere. To this mixture, a monomer and a Lewis acid were added ([monomer]/[Lewis acid] = 1/0.2, [monomer] = 0.35 M). After stirring at room temperature for a specified time, a polymer was isolated as the methanol-1N HCl (9/1 v/v)-insoluble fraction by centrifugation and drying *in vacuo*.

Measurements

The ^{1}H and ^{13}C NMR spectra were measured by a Varian Unity Inova (500 MHz for ^{1}H) or Mercury (200 MHz for ^{1}H) spectrometer. The infrared (IR) spectra were recorded by a Horiba FT-720 spectrometer. The optical rotations were measured by a Jasco P-1010 polarimeter. The circular dichroism (CD) spectra were obtained with a Jasco J-720WI apparatus. The size exclusion chromatography (SEC) analyses were performed by a Jasco PU-2080 plus equipped with a Jasco UV-2075-plus UV detector with KF-806F and KF-803F columns connected in series (eluent = THF, flow rate = 1.0 mL/min). Calibration was carried out with standard polystyrenes. The high-performance-liquid-chromatography (HPLC) analyses were performed by a Jasco 986-PU chromatograph equipped with a UV (Jasco 970-UV) detector at 25 °C (eluent, acetonitrile; flow rate, 0.5 mL/min).

RESULTS AND DISCUSSION

In a previous study, the oxidative coupling polymerization of **2** with various copper catalysts in the absence of the Lewis acid was carried out, and the CuCl-(S)Phbox catalyst successfully produced a polymer in quantitative yield, whose cross-coupling selectivity (Y) was estimated to be about 80% from the model coupling reaction. However, the other copper complexes, such as the TMEDA and (+)PMP catalysts, only afforded an oligomer in a low yield.[13]

Table I shows the results of the oxidative coupling polymerization of **2** with various copper catalysts in the presence of the Lewis acid, together with those of the polymerization without the Lewis acid. Every binary catalyst system of the copper complex and Yb(OTf)$_3$ resulted in a good yield to give a polymer including a THF-insoluble part within a much shorter reaction time than that for the polymerization in the absence of the Lewis acid. Especially, during the polymerization with TMEDA and (+)PMP catalysts, a significant effect of the Lewis acid on the catalyst activity was observed (entries 5 and 6). The polymerization with the CuCl-(−)Sp catalyst in the absence of the Lewis acid also resulted in a very low molecular weight oligomer (entry 3), whereas, during the polymerization in the presence of Yb(OTf)$_3$, a methanol-1N HCl (9/1, v/v)-insoluble polymer was obtained in an 89% yield, whose THF-soluble fraction showed a number average molecular weight (M_n) of 8.7×10^3 (entry 7). Y(OTf)$_3$ was also effective and gave a polymer in quantitative yield (entry 11), whereas the polymerization in the presence of Sc(OTf)$_3$ resulted in no yield. These results quite resemble to those for the coupling reaction of 2-naphthol and 3-hydroxynaphthoate previously

Table I. Oxidative coupling polymerization of **2** in the presence of lewis acid[a]

| Entry	Catalyst (eq.)	Lewis acid (eq.)	Time (h)	Yield (%)[b]	M_n (×10³) (M_w/M_n)[c]	M_n (×10³)[d]	$[\alpha]_D^{25}$[e]	Unit ratio X:Y:Z[d]
1[f]	CuCl(OH)-TMEDA (0.2)	None	24	60	1.5 (—)	1.3	—	16:80:4
2[f]	CuCl-(+) PMP (0.2)	None	24	3	1.0 (—)	—	−14	—
3	CuCl-(−) Sp (0.1)	None	6	99	0.6 (—)	0.9	−5	14:74:12
4[f]	CuCl-(S)Phbox (0.2)	None	24	99 (25)[g]	12.7 (2.1)	11.9	0	9:90:1
5	CuCl(OH)-TMEDA (0.1)	Yb(OTf)₃ (0.2)	1	94 (56)[g]	7.4 (2.2)	5.9	—	4:92:4
6	CuCl-(+) PMP (0.1)	Yb(OTf)₃ (0.2)	3	84 (13)[g]	4.3 (2.3)	6.2	+8	4:94:2
7	CuCl-(−) Sp (0.1)	Yb(OTf)₃ (0.2)	2	89 (31)[g]	8.7 (1.8)	5.5	0	4:93:3
8	CuCl-(S)Phbox (0.1)	Yb(OTf)₃ (0.2)	3	87 (17)[g]	9.1 (2.2)	6.7	−43	1:97:2
9	CuCl-(R)Phbox (0.1)	Yb(OTf)₃ (0.1)	3	67 (51)[g]	4.2 (1.6)	4.2	+38	2:96:2
10	CuCl-(S)Phbox (0.2)	Yb(OTf)₃ (0.1)	5	99 (86)[g]	8.9 (2.0)	7.6	−55	<1:95:4
11	CuCl-(S)Phbox (0.1)	Y(OTf)₃ (0.2)	3	99 (65)[g]	10.4 (2.1)	10.6	−20	<1:93:6
12	CuCl-(S)Phbox (0.1)	Sc(OTf)₃ (0.2)	48	0	—	—	—	—

[a]Conditions: [**2**] = 0.35 M, solvent = THF, temperature = r. t., O₂ atmosphere. [b]Methanol-1N HCl (9/1 v/v)-insoluble part. [c]Determined by SEC in THF (polystyrene standard). [d]Estimate by ¹³C NMR analysis. [e]In THF. [f]Reference 13. [g]Methanol-insoluble and THF-soluble part.

reported.[28] The Lewis acid catalyst significantly affected the catalyst activity for the polymerization of **2**.

The coupling selectivity of poly(**1**) was determined by the ¹H NMR analysis of the hydroxyl protons.[11–13] However, as previously reported, the ¹H NMR spectrum of the obtained poly(**2**) hardly showed a clear peak splitting based on the coupling selectivity, and then it was estimated from the model reaction.[13] Therefore, further investigation of the unit ratio, that is, two homo-coupling units (X and Z) and a cross-coupling unit (Y), was performed.

Figure 1 depicts the ¹³C NMR spectra of carbons attached to the hydroxyl group of the model homo-polymer, poly(**3**), the model compounds, **4** and **5**, which correspond to the coupling units Y and Z in poly(**2**), respectively, and poly(**2**) obtained with the (+)PMP catalyst in the presence of Yb(OTf)₃ (entry 6) in dimethyl sulfoxide-d_6 (DMSO-d_6) (Scheme 4). Poly(**3**), consisting of the homo-coupling units, X and Z, with 2-naphthol terminals, was synthesized by the polymerization of dimethyl 2,2′,6,6′-tetrahydroxy-1,1′-binaphthalene-3,3′-dicarboxylate with CuCl(OH)-TMEDA at room temperature for 48 h under an O₂ atmosphere (yield 60%, $M_n = 4.6 \times 10^3$, $M_w/M_n = 1.6$). Both the THF-insoluble and -soluble parts of poly(**2**) were soluble in DMSO and were then analyzed. The internal (**a–d**) and terminal (**e** and **f**) carbons of the model compounds with different chemical shifts appeared as shown in the figure. Each observed peak for poly(**2**) was then assigned based on the chemical shifts observed for the models, poly(**3**), **4** and **5**. The peak integral ratio of carbon **b** is equal to that of **c**, accordingly, the unit ratio (X:Y:Z) for the polymer obtained with CuCl-(+)PMP and Yb(OTf)₃ was determined to be 4:94:2.

The evaluated coupling ratio and the M_n value for the obtained polymers by the ¹³C NMR analysis are also listed in Table I. The SEC analysis was performed for the THF-soluble fraction of the obtained polymer, whereas both parts of poly(**2**) being soluble and insoluble in THF were used for the NMR analysis, as aforementioned. Most of the M_n values observed by the latter method are almost equal to or somewhat smaller than that evaluated by the former. These facts indicate that the
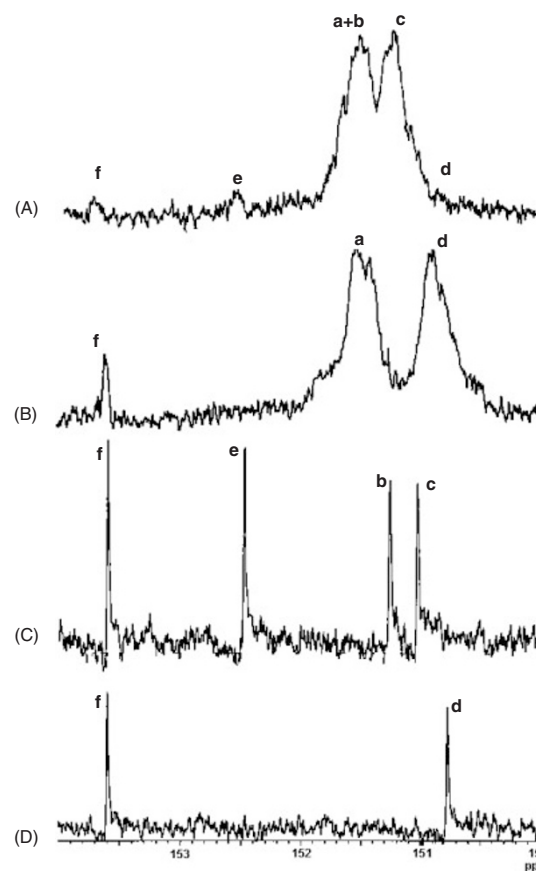
Figure 1. ¹³C NMR spectra of carbons attached with hydroxyl group of (A) poly(**2**) (Table I, entry 6), (B) poly(**3**), (C) **4** and (D) **5** (in DMSO-d_6, 50 °C).

polymer has a rigid main chain. The cross-coupling reactions in the absence of the Lewis acid predominantly proceeded with a cross-coupling selectivity Y of 74–90% (entries 1–4). The observed cross-coupling selectivity for the polymerization using the (S)Phbox ligand, 90%, was slightly higher than that estimated from the model coupling reaction (80%),[13] probably due to the effect of the protecting group for the hydroxyl group,
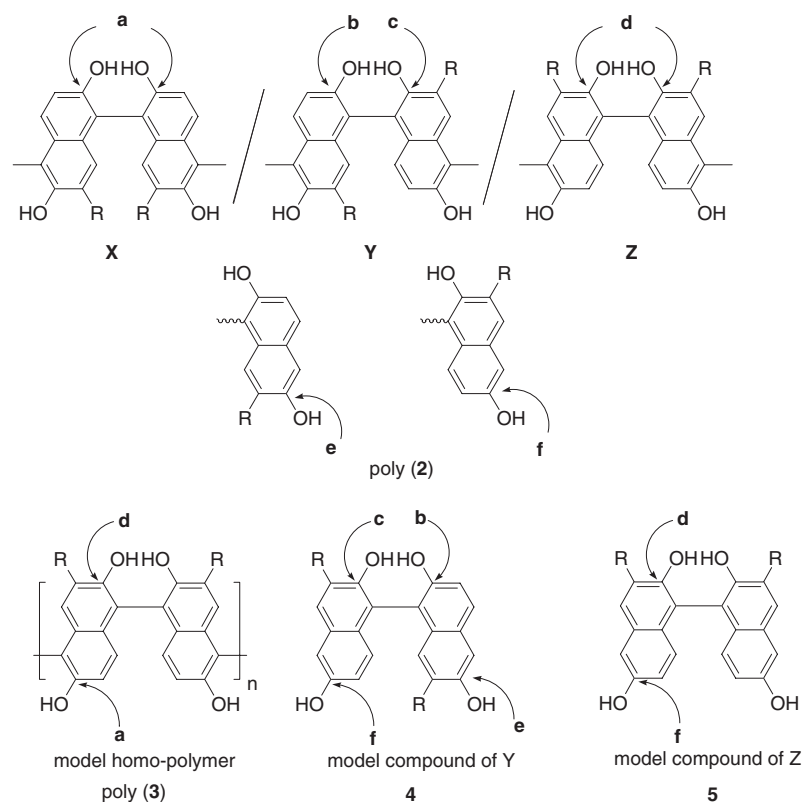
Scheme 4.

a methoxymethyl group, of the model monomers. Regardless of the copper catalyst, a significant increase in the cross-coupling selectivity was observed for the polymerization when the Lewis acid was used as the co-catalyst, and it reached 97% during the polymerization with the (S)Phbox ligand (entry 8). The cross-coupling selectivity observed for the polymerization with Y(OTf)$_3$ was slightly smaller than that for the polymerization using Yb(OTf)$_3$ (entry 11). The Lewis acid catalyst is quite effective for cross-coupling control during the polymerization of **2**.

The CD spectra of the obtained poly(**2**) are shown in Figure 2. The spectral patterns of the polymers with positive and negative specific rotations are almost mirror images of each other. The absorption intensities of the polymers around 250 and 300 nm showed a good relation to the absolute $[\alpha]_D$ values and the spectral patterns indicate that the polymer obtained with the CuCl-(S)Phbox catalyst is rich in the S-configuration,[4] whereas the R-structure is preferentially constructed during the polymerization with CuCl-(R)Phbox. The polymerization in the absence of the Lewis acid hardly showed the specific rotation. Therefore, the Lewis acid also significantly affects the stereochemistry during the polymerization of **2**, although the details were not currently clear.

The stereoselectivity was then estimated from the model reaction between two naphthol derivatives, **6** and **7**, in which one of two hydroxyl groups is protected by the methyl group, and the coupling products, **8–10**, corresponding to the coupling units, X–Z in poly(**2**), respectively, were isolated (Scheme 5,
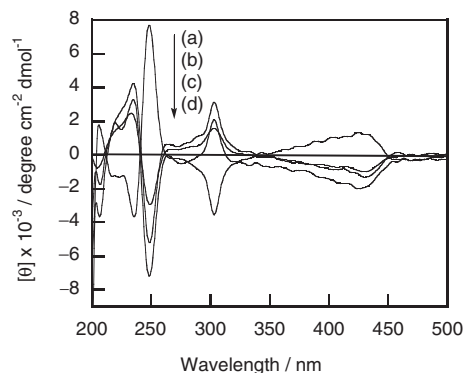
Figure 2. CD spectra of poly(**2**) obtained with (a) (R)Phbox (Table I, entry 9), (b) (S)Phbox in the presence of Y(OTf)$_3$ (entry 11), (c) (S)Phbox in the presence of Yb(OTf)$_3$ (entry 8), and (d) (S)Phbox in the presence of Yb(OTf)$_3$ (entry 10) (monomer unit, in THF).

Table II). The reaction with the CuCl-(S)Phbox catalyst in the presence of Yb(OTf)$_3$ proceeded in a cross-coupling specific manner with a 94% yield, which showed an enantioselectivity of 19% ee (S), while the reaction with the TMEDA ligand resulted in a 97% cross-coupling selectivity. These cross-coupling selectivities are consistent with those observed for the polymerization, and the stereostructure of the cross coupling compound, **9**, obtained with (S)Phbox is identical with that preferentially constructed during the polymerization. Therefore, the polymerization with the CuCl-(S)Phbox catalyst in the
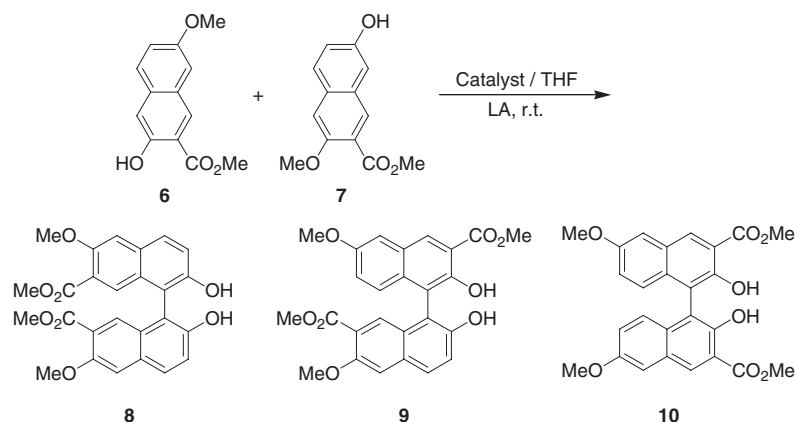
Scheme 5.

Table II. Asymmetric oxidative coupling reaction between **6** and **7**[a] for 48 h

Entry	Catalyst	Yb(OTf)$_3$ (eq.)	Coupling ratio 8:9:10[b]	Cross-coupling product 9	
				Yield (%)[c]	ee (%)[d]
1	CuCl-(S)Phbox	0.2	0:>99:0	94	19 (S)
2	CuCl(OH)-TMEDA	0.2	0:97:3	54	—

[a]Conditions: [copper]/[diamine]/[**6**]/[**7**] = 0.20/0.25/1/1, [**6** + **7**] = 0.35 M, solvent = THF, O$_2$ atmosphere. [b]Ratio of isolated yields. [c]Isolated yield. [d]Determined by HPLC analysis (Chiralpak AD).

Table III. Oxidative coupling polymerization of **1** in the presence of Yb(OTf)$_3$[a]

Entry	1	CuCl-(S)Phbox (eq.)	Lewis acid (eq.)	Time (h)	Yield (%)[b]	M_n (×10^3) (M_w/M_n)[c]	$[\alpha]_D^{25}$[d]	Unit ratio X:Y:Z[e]
1[f]	1a	0.2	None	24	71(47)[g]	4.5 (2.8)	+20	3:96:1
2	1a	0.1	Yb(OTf)$_3$(0.2)	72	68	2.9 (1.3)	+45	<1:99:0
3[h]	1b	0.2	None	24	95	9.1 (2.3)	+55	0:97:3
4	1b	0.1	Yb(OTf)$_3$(0.2)	60	98	4.4 (1.4)	+58	<1:99:0

[a]Conditions: [**1**] = 0.13 M, solvent = THF, temperature = r. t., O$_2$ atmosphere. [b]Methanol-ethyl acetate-1N HCl (2/1/0.3 v/v/v)-insoluble part. [c]Determined by SEC in THF (polystyrene standard). [d]In THF. [e]Estimate by ^{1}H NMR analysis. [f]Reference 11. [g]THF-soluble part. [h]Reference 13.
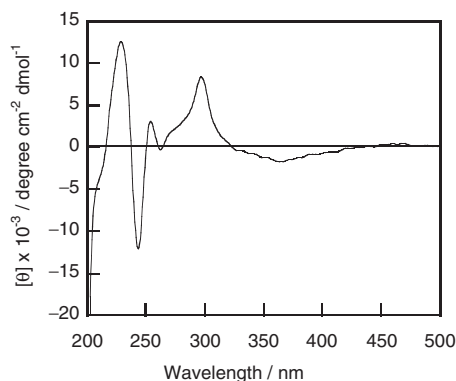

Figure 3. CD spectra of the cross-coupling compounds **9** obtained with CuCl-(S)Phbox in the presence of Yb(OTf)$_3$ (Table II, entry 2) (binaphthalene unit, in THF).

presence of Yb(OTf)$_3$ should proceed with about the observed selectivity for this model reaction. The CD spectrum of the obtained **9** is shown in Figure 3.

The oxidative cross-coupling polymerization of **1** with the CuCl-(S)Phbox in the presence of Yb(OTf)$_3$ (0.2 eq.) at room temperature was also carried out. The results are summarized in Table III (Scheme 6). When Yb(OTf)$_3$ was used for the polymerization, a much longer reaction time was needed to attain a good polymer yield, and the M_n value was also reduced, in comparison with that of the polymers obtained without Yb(OTf)$_3$. Those results are different from those for the polymerization of **2**. The cross-coupling selectivity, however, was again increased, and a cross-coupling specific polymerization was achieved (entries 2 and 4).

The CD spectra of the obtained poly(**1**) using the CuCl-(S)Phbox catalyst are shown in Figure 4. The spectral patterns indicate that the polymers are rich in the S-configuration, and the absorption intensities around 270 and 290 nm again showed a good relation to the absolute values of the specific rotation.[11–13,29] The Lewis acid catalyst also improved the stereoselectivity as well as the cross-coupling selectivity, and the monomer structure significantly affected the catalyst activity.

A plausible mechanism for the catalytic oxidative cross-coupling reaction has been suggested in previous reports.[7–10,28] The mechanism for the polymerization should be similar to that for the cross-coupling reaction. That is, during the polymerization, the naphthol unit with an electron-withdrawing group functions as an acceptor part for the radical species generated by the one-electron oxidation of the other naphthol unit, and the Lewis acid effectively activates the former acceptor unit by coordinating to the electron-withdrawing group. As aforementioned, regardless of the structure of the copper complex, the Lewis acid assistance for the cross-coupling selectivity was observed. Thus, the coordination by the Lewis acid plays an important role during the cross-coupling process. During the polymerization of **2**, the Lewis acid coordination to the ester group more greatly influenced the reactivity of the 2-naphthol part than that in the monomer **1**, and this should cause the observed difference in the polymerizability between these two monomers. Both the Lewis acid and the copper complex affected the cross-coupling and stereoselectivies. Therefore,
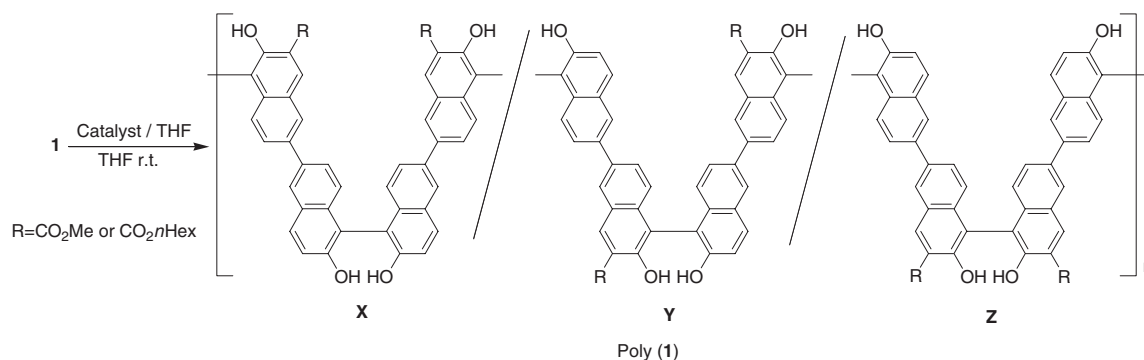
Scheme 6.
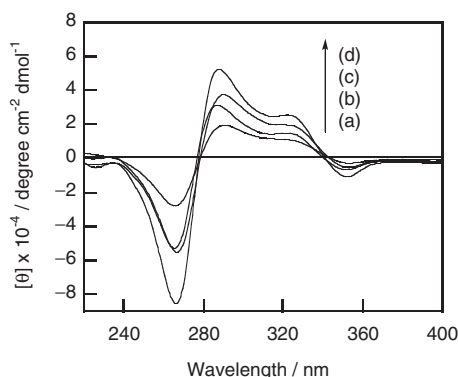
Figure 4. CD spectra of poly(**1**)s obtained with CuCl-(*S*)Phbox. (a) poly(**1a**) in the absence of Yb(OTf)$_3$ (Table III, entry 1) and (b) in the presence of Yb(OTf)$_3$ (entry 2), (c) poly(**1b**) in the absence of Yb(OTf)$_3$ (entry 3) and (d) in the presence of Yb(OTf)$_3$ (entry 4) (monomer unit, in THF).

both catalysts cooperate in the cross-coupling intermediate. These facts support the proposed mechanism well.

In conclusion, a novel binary catalyst system for asymmetric oxidative cross-coupling was successfully used for the polymerization, that is, the polymerization using the copper catalyst in the presence of Yb(OTf)$_3$ showed a much higher cross-coupling selectivity than that observed during the polymerization in the absence of the Lewis acid. The Lewis acid co-catalyst also significantly affected the catalyst activity and the coupling stereoselectivity.

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