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Homo- and copolymerization of 5-ethylidene-2-norbornene with ethylene by [2-C_5Me_4-4,6-$^tBu_2C_6H_2O$]$TiCl_2$/Al^iBu_3/$Ph_3CB(C_6F_5)_4$ catalyst system and epoxidation of the resulting copolymer

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ABSTRACT

5-Ethylidene-2-norbornene (ENB) polymerization and copolymerization with ethylene (E) catalyzed by constrained geometry catalyst 2-tetramethylcyclopentadienyl-4,6-di-*tert*-butylphenoxytitanium dichloride (**1**) were studied in the presence of Al^iBu_3 and $Ph_3CB(C_6F_5)_4$ (TIBA/B). The catalyst system shows moderate activity on the homopolymerization of ENB, and higher activity on the copolymerization of E/ENB under atmospheric pressure of ethylene. ^{1}H NMR analysis indicates that ENB was regioselectively incorporated into the copolymer backbone through the endocyclic double bond, leaving the ethylidene double bond unreacted. The ethylidene group contained in the poly(E-*co*-ENB) was quantitatively converted to the epoxy group with *m*-chloroperbenzoic acid, producing functionalized poly(E-*co*-ENB)s. The parent and functionalized copolymers have been characterized using IR, NMR, DSC, and GPC techniques.

1. Introduction

Copolymerization of ethylene with cyclic olefins like cyclopentene [1–3], cyclohexene [4], dicyclopentadiene [5] and norbornene [6–12] catalyzed with various metallocene catalysts has been widely investigated in the past decade because of the unique properties of the resultant copolymers such as high transparency, high glass transition temperature (T_g), high chemical resistance and so on. On the other hand, one of the most attractive subjects in this field is the introduction of polar functional groups in the polyolefin molecules [13,14]. This is primarily because polar functionality in polyolefins substantially improves the polymer properties such as permeability, dye ability, adhesion, and compatibility with other materials [15–18]. However, the remarkable drawback of group 4 metallocene catalysts is their limited ability to polymerize polar monomers [15]. Thus, much work has recently been focused on the chemical modification of polymers containing reactive groups, like vinyl or vinylidene group, etc. [17,19–21]. Copolymerization of ethylene with non-conjugated dienes, such as 4-vinyl-1-cyclohexene [20], 5-vinyl-2-norbornene [16,17,21], and dicyclopentadiene [22], has been reported to produce copolymers with unsaturated carbon–carbon double bond that can be functionalized via various standard chemical reactions. 5-Ethylidene-2-norbornene (ENB) is a relatively cheap norbornene derivative with an additional carbon–carbon double bond (ethylidene) that does not interfere in the polymerization reaction [23–27]. It should thus be a good comonomer for introducing functional groups into the copolymers. We have previously found that 2-tetramethylcyclopentadienyl-4,6-di-*tert*-butylphenoxytitanium dichloride (**1**) (Chart 1) exhibits high catalytic activity for the homo- and copolymerization of norbornene with ethylene upon activation and obtained high norbornene incorporation copolymers with high molecular weight [28]. In this paper, we wish to report the results of the ENB homopolymerization and the E/ENB copolymerization catalyzed by the **1**/TIBA/B catalyst system, as well as the epoxidation of the resulting poly(E-*co*-ENB) (Scheme 1).

2. Experimental

2.1. Materials

All experiments involving air-sensitive compounds were performed using standard Schlenk techniques in an atmosphere of high purity nitrogen or glove-box techniques. Toluene was distilled from sodium/benzophenone ketyl under nitrogen prior to use. ENB (Aldrich) was dried over calcium hydride for several days and filtered under nitrogen prior to use. Al^iBu_3 was purchased from Aldrich. Complex **1** [29] and $Ph_3CB(C_6F_5)_4$ [30] were synthesized according to literature procedures. Polymerization grade ethylene was further purified by passage through columns of 5 Å molecular sieves and MnO.

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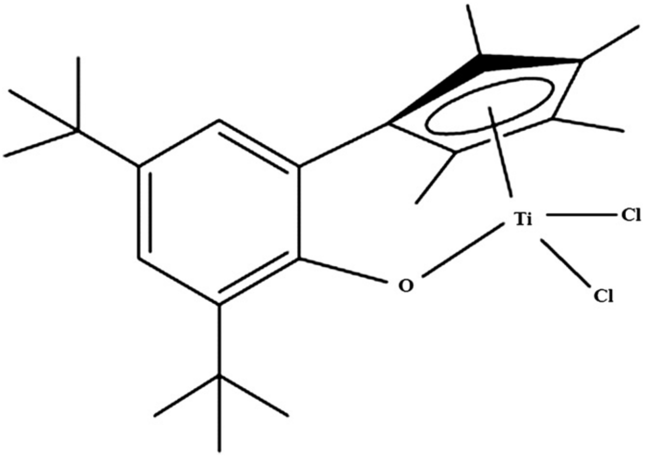
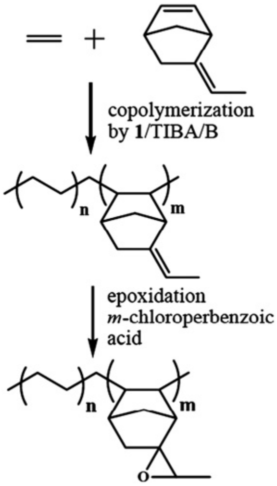


Chart 1. Structure of complex **1**.



Scheme 1. E/ENB copolymerization and epoxidation of poly(E-*co*-ENB).

2.2. Polymerization

ENB polymerization experiments were performed as follows: a prescribed amount of ENB and $Al^{i}Bu_3$ (2/3 of total amount) in toluene (40 mL) were added into a 100 mL glass flask under N_2 with stirring, and the solution was heated to a desired temperature in an oil bath. The polymerization reaction was started by injection of a mixture of the catalyst and $Al^{i}Bu_3$ (1/3 of total amount) in toluene (5 mL) and a solution of $Ph_3CB(C_6F_5)_4$ in toluene (5 mL) at the same time. After a certain period of time, the polymerization was terminated by injecting acidified methanol [HCl (3 M)/methanol = 1:1]. The polymer was collected by filtration, washed with water and methanol, and dried at 60 °C in vacuo to a constant weight.

E/ENB copolymerization experiments were performed in a similar way: a dry 100 mL glass reactor with a magnetic stirrer was charged with a solution of appropriate amount of ENB and $Al^{i}Bu_3$ (2/3 of total amount) in toluene (40 mL), thermostated at the desired temperature, and saturated with ethylene (1 atm). The polymerization reaction was started by injection of a mixture of the catalyst and $Al^{i}Bu_3$ (1/3 of total amount) in toluene (5 mL) and a solution of $Ph_3CB(C_6F_5)_4$ in toluene (5 mL) at the same time. After a certain period of time, the polymerization was quenched by injecting acidified methanol. The polymer was collected by filtration, washed with water and methanol, and dried at 60 °C in vacuo to a constant weight.

2.3. Epoxidation

A poly(E-*co*-ENB) sample (0.20 g) containing 50.5 mol% of ENB and 40 mL of dry toluene were placed in a 100 mL three-necked round-bottom flask, and the mixture was heated at 55 °C until the copolymer was completely dissolved. A total of 0.35 g of *m*-chloroperbenzoic acid dissolved in 10 mL of toluene was added dropwise and stirred for 4 h. The reaction was then stopped by the addition of methanol. The epoxidized copolymer was filtered, washed with an excess amount of methanol and dried in vacuum at 60 °C for 24 h.

2.4. Characterization

^{1}H NMR spectra of the copolymers were recorded on a Bruker Avance-500 NMR spectrometer. ^{13}C NMR spectra of the copolymers were recorded on a Varian Unity-400 NMR spectrometer. Gel permeation chromatographic (GPC) measurements were carried out on a Waters instrument (515 HPLC pump) equipped with a Wyatt interferometric refractometer, eluted with THF at 25 °C at 1 mL/min. The molecular weights were calibrated against polystyrene standards. Differential scanning calorimetric (DSC) measurements were performed on a NETZSCH DSC 204 differential scanning calorimeter. Each sample was heated from 0 to 300 °C at a heating rate of 10 °C/min and reheated at the same rate. IR spectra were recorded on a Perkin–Elmer 2000 FT-IR spectrometer.

3. Results and discussion

3.1. ENB homopolymerization

The homopolymerization of ENB catalyzed by the **1**/TIBA/B catalyst system was studied in detail and the results are summarized in Table 1. The effects of the polymerization temperature and initial ENB concentration on the homopolymerization reaction were examined. The catalytic activity, polymer yield, and ENB conversion increase with the increase in polymerization temperature until reaching the maximum value at about 70 °C. Further increase in the polymerization temperature results in a decrease in the catalytic activity, polymer yield, and ENB conversion. The intrinsic viscosity of the obtained polymer decreases with the increase in polymerization temperature. The catalytic activity increases with the increase in initial ENB concentration. In addition, the increase in the initial ENB concentration results in an increase in the yield and intrinsic viscosity of the polymer. These results are similar to those observed in the homopolymerization of norbornene by the **1**/TIBA/B catalyst system [28]. The catalytic activity of the **1**/TIBA/B catalyst system is obviously higher than that of a similar constrained geometry (t-BuNSiMe$_2$Flu)TiMe$_2$/MAO catalyst system even at lower polymerization temperature with longer polymerization time [23].

Table 1
ENB polymerization results by the **1**/TIBA/B catalyst system[a]

Run	T[b] (°C)	Concentration[c] (mol/L)	Yield (g)	Activity (kg/mol h)	Conversion (%)	η[d] (dL/g)
1	30	1.0	0.46	77	7.7	0.31
2	50	1.0	0.52	87	8.7	0.29
3	70	1.0	0.70	116	11.6	0.27
4	90	1.0	0.65	108	10.8	0.25
5	70	2.0	1.28	213	10.6	0.39
6	70	3.0	1.76	293	9.8	0.58
7	70	4.0	2.15	358	8.9	0.79
8	70	5.0	2.64	440	8.8	1.01

[a] Reaction conditions: catalyst **1** 2 μmol, molar ratio of Al/Ti 120, molar ratio of B/Ti 1.5, toluene + ENB total 50 mL, polymerization time 3 h.
[b] Polymerization temperature.
[c] Initial ENB concentration.
[d] Intrinsic viscosity measured at 135 °C in decahydronaphthalene.

3.2. E/ENB copolymerization

The results of E/ENB copolymerization catalyzed by the **1**/TIBA/B catalyst system under different conditions are summarized in Table 2. The influence of Al/Ti molar ratio on the catalytic activity was examined in the range of 60–150. Maximum catalytic activity was obtained at the Al/Ti molar ratio of 120. The copolymerization was also carried out at different temperatures (30–90 °C) with fixed initial ENB concentration and Al/Ti molar ratio. It was observed that the catalytic activity increases first with the increase in polymerization temperature and reaches the maximum value at about 70 °C. Similar results have been reported previously [28,31]. The content of ENB in the poly(E-*co*-ENB) was found to increase with the increase in polymerization temperature due to the decrease of the ethylene concentration. This is accompanied by an increase in the T_g of the poly(E-*co*-ENB). The molecular weight of the obtained poly(E-*co*-ENB) decreases with the increase in polymerization temperature. Besides the Al/Ti molar ratio and polymerization temperature, the initial ENB concentration is another important issue for the copolymerization reaction, and the influence of the initial ENB concentration on the E/ENB copolymerization was also examined. It was observed that the catalytic activity increases as the initial ENB concentration increases from 0.4 to 1.2 mol/L (Runs 3 and 8–11 in Table 2), and decreases then with the further increase in the initial ENB concentration (Run 12 in Table 2). A similar "comonomer effect" on the catalytic activity was also observed previously in the ethylene/5-vinyl-2-norbornene copolymerization [15]. The content of ENB in the poly(E-*co*-ENB) increases as the initial ENB concentration is raised and reaches as high as 50.5 mol% at an initial ENB concentration of 1.4 mol/L, which, in turn, leads to an increase in the T_g of the poly(E-*co*-ENB) from 92.2 to 171.9 °C (Runs 3 and 8–12 in Table 2). The molecular weight of the poly(E-*co*-ENB) is also sensitive to the initial ENB concentration and decreases with the increase in the ENB concentration (Runs 3 and 8–12 in Table 2). In comparison to the reported results from the (t-BuNSiMe$_2$Flu)TiMe$_2$/MAO catalyst system, the catalytic activity of the **1**/TIBA/B catalyst system and the molecular weight of the produced poly(E-*co*-ENB) are relatively high, while the ENB content in the poly(E-*co*-ENB) is relatively low under similar conditions [23].

^{1}H NMR spectra of ENB and a poly(E-*co*-ENB) sample are illustrated in Fig. 1. The absence of the endocyclic double bond signal at 5.8–6.0 ppm and the presence of ethylidene proton between 4.8 and 5.3 ppm in the spectrum of the poly(E-*co*-ENB) indicate that the ENB comonomer is copolymerized regioselectively through the endocyclic double bond with the ethylidene group unreacted (Scheme 1) because of the ring strain of the endocyclic double bond [16,32]. The content of ENB in the poly(E-*co*-ENB) was calculated based on the ^{1}H NMR spectra according to the following equation [23]:

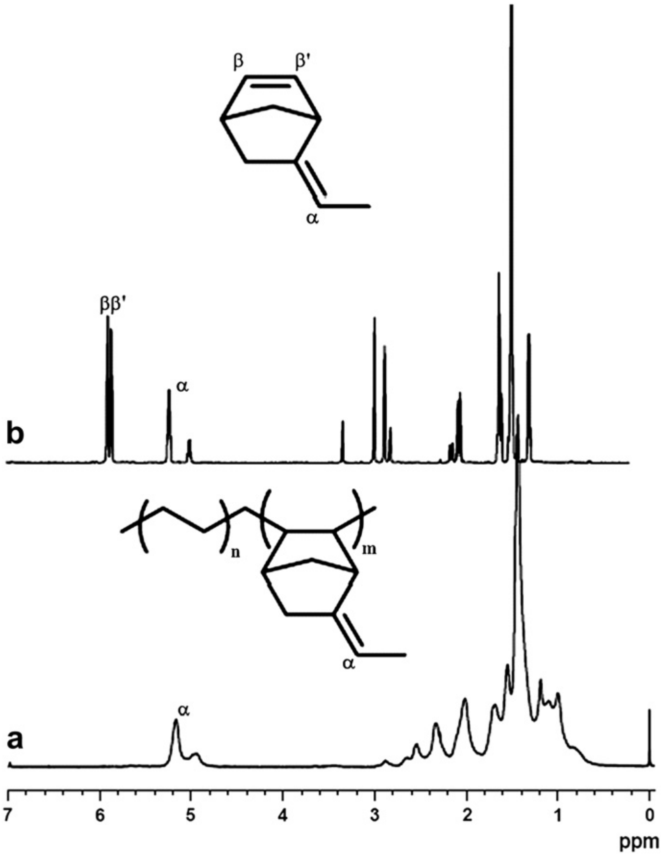


Fig. 1. ^{1}H NMR spectra of (a) a poly(E-*co*-ENB) sample (Run 12 in Table 2) and (b) ENB in CDCl$_3$ at room temperature.

$$\text{ENB (mol\%)} = \frac{A}{(1/4)(B - 11A) + A} \times 100 \qquad (1)$$

where A is the integral of ethylidene hydrogen signals in the range from 4.8 to 5.3 ppm and B is the integral over all other hydrogen signals from 0.5 to 3.0 ppm.

A normal ^{13}C NMR spectrum and a corresponding DEPT 135 spectrum (where the signal intensity of methylene (–CH$_2$–) carbons appears in negative intensity and the signal intensity of quaternary carbons disappears) of a poly(E-*co*-ENB) sample are shown in Fig. 2. The ^{13}C NMR spectrum presents the region of resonances from 13.5 to 148 ppm, and the assignments on these resonances can be made by comparing to a similar spectrum of the ethylene/5-vinyl-2-norbornene copolymer reported in literature [15] with the help of the DEPT spectrum. The resonances in the regions of 145–148 and 110–113 ppm can be assigned to the double bond C5 and C8, respectively. The resonances in the region of 49.5–52.0 ppm should be from C4, while the signals of C1 overlap with the resonances of C3 in the region between 44.0 and 49.5 ppm with the signals of C3 located at the higher field. The secondary carbon resonances of C6 and C7 can be easily recognized in the DEPT spectrum in the regions of 35.0–38.0 and 33.0–35.0 ppm, respectively. The resonances in the region of 28.2–33.0 ppm are for the ethylene units, and the resonances in the region of 13.5–15.5 ppm can be assigned to C9.

Table 2
E/ENB copolymerization results by the **1**/TIBA/B catalyst system[a]

Run	Al/Ti[b]	T[c] (°C)	ENB[d] (mol/L)	Activity[e]	ENB content[f] (mol%)	M_w[g] (10^3)	PDI[g]	T_g[h] (°C)
1	60	70	1.0	160	n.d.[i]	n.d.	n.d.	n.d.
2	90	70	1.0	400	n.d.	113	2.06	n.d.
3	120	70	1.0	1380	45.8	117	1.92	152.5
4	150	70	1.0	860	n.d.	110	1.95	n.d.
5	120	30	1.0	840	25.4	256	2.20	69.2
6	120	50	1.0	1040	38.2	195	2.04	115.3
7	120	90	1.0	1260	48.6	48	2.13	167.5
8	120	70	0.4	680	31.0	197	2.12	92.2
9	120	70	0.6	820	36.3	161	2.05	111.4
10	120	70	0.8	1060	41.3	135	1.96	134.6
11	120	70	1.2	1580	48.7	109	2.01	168.2
12	120	70	1.4	1320	50.5	105	2.05	171.9

[a] Reaction conditions: ethylene pressure 1 atm, catalyst **1** 1 μmol, toluene + ENB total 50 mL, molar ratio of B/Ti 1.5, polymerization time 0.5 h.
[b] Molar ratio of Al/Ti.
[c] Polymerization temperature.
[d] Initial ENB concentration.
[e] Activity in kg polymer/mol Ti·h.
[f] Determined by ^{1}H NMR spectroscopy.
[g] Determined by GPC.
[h] Determined by DSC.
[i] n.d. not determined.

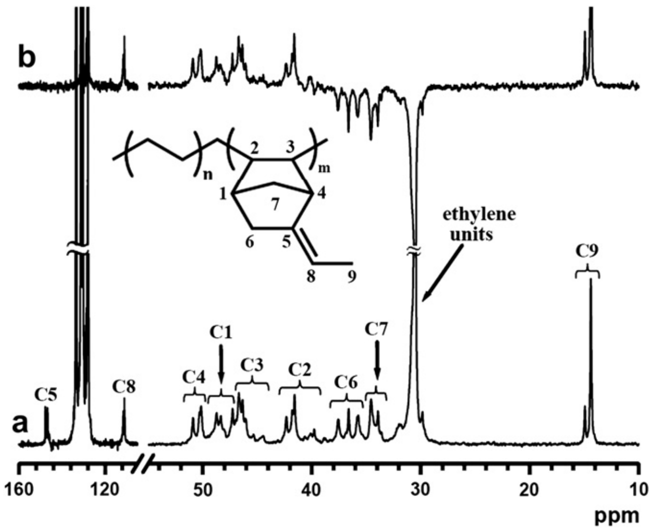


Fig. 2. ^{13}C and DEPT NMR spectra of a poly(E-*co*-ENB) sample with 25.4 mol% ENB content (Run 5 in Table 2) in 1,2,4-trichlorobenzene/benzene-d_6 (90/10 wt%) at 110 °C. (a) the normal ^{13}C NMR spectrum and (b) the corresponding DEPT spectrum.

^{13}C NMR spectra of poly(E-*co*-ENB) samples with different ENB incorporation are shown in Fig. 3. It can be seen that the signal groups in these spectra are becoming broader with the increase in the ENB content in the poly(E-*co*-ENB), due probably to the increase of different ENB repeat units of varying lengths linked in both meso and racemic positions. Similar results have been reported in literature for ethylene–norbornene copolymers [11,33].

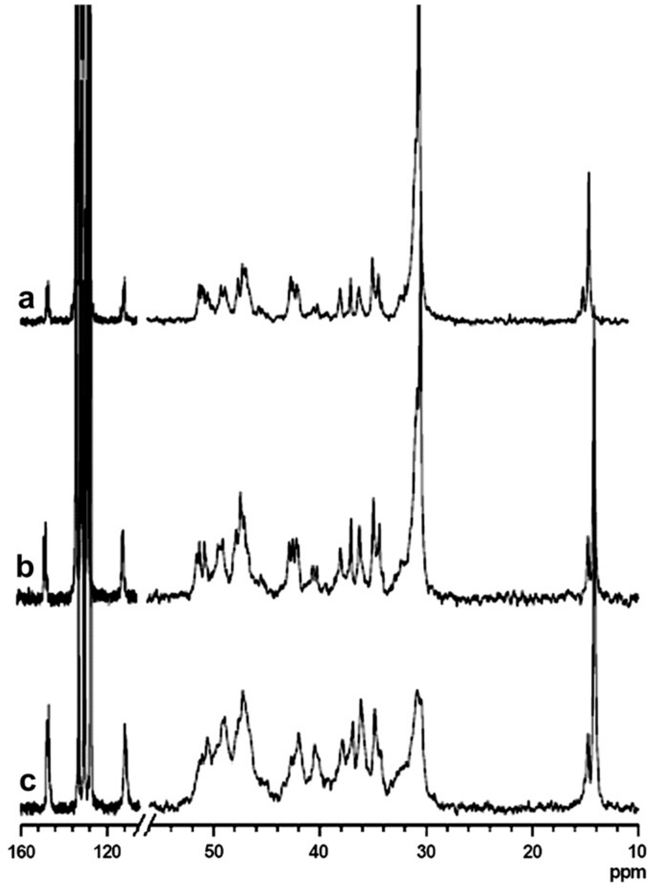


Fig. 3. ^{13}C NMR spectra of poly(E-*co*-ENB) samples with different ENB contents in *o*-$C_6D_4Cl_2$ at 125 °C. (a) 31.0 mol% (Run 8 in Table 2), (b) 41.3 mol% (Run 10 in Table 2), and (c) 50.5 mol% (Run 12 in Table 2).

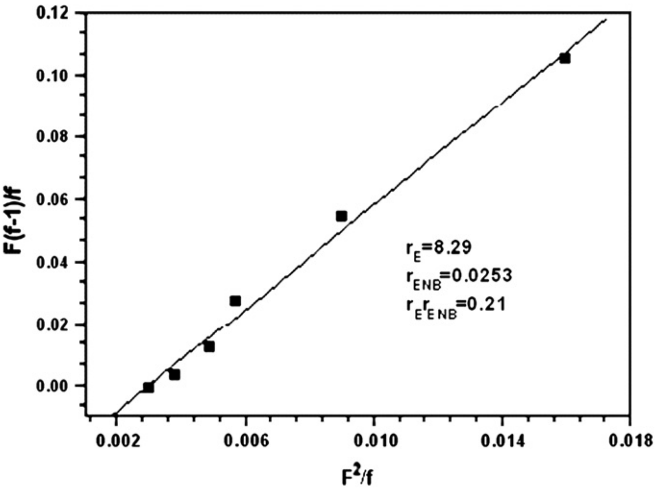


Fig. 4. Fineman–Ross plot for E/ENB copolymerization with the **1**/TIBA/B catalyst system.

The ethylene and ENB reactivity ratios (r_E and r_{ENB}) were calculated according to the Fineman–Ross method [34]. Copolymerization was performed with different initial ENB concentrations. In all cases, the conversion of ENB was restricted to less than 10%. The Fineman–Ross plot for the **1**/TIBA/B catalyst system is shown in Fig. 4. The obtained monomer reactivity ratios of r_E and r_{ENB} are 8.29 and 0.0253, respectively, with the product of reactivity ratio, $r_E r_{ENB} = 0.21$.

The GPC analysis reveals that the obtained poly(E-*co*-ENB) samples possess moderate molecular weights with narrow molecular weight distributions ($M_w/M_n = 1.92$–2.20). The polydispersity is close to 2, which suggests that the polymerization takes place at a single active site [35]. The T_g values of the poly(E-*co*-ENB) samples determined by DSC increase with the increase in the comonomer content, which is similar to the reported results observed for poly(ethylene-*co*-norbornene)s [11,28] and poly(ethylene-*co*-ENB)s [23,25,27,36]. It should be noted that the T_g values of the poly(E-*co*-ENB) samples with high ENB contents obtained by **1**/TIBA/B catalyst system are higher ($T_g = 115.3$ and 171.9 °C for the samples with 38.2 and 50.5 mol% ENB contents, respectively) than those of similar samples produced by the (*t*-BuNSiMe_2Flu)TiMe_2/MAO catalyst system ($T_g = 130$ °C for a sample with 49 mol% ENB content) [23] and the $Me_2Si[Ind]_2ZrCl_2$/MAO catalyst system ($T_g = 72.6$ °C for a sample with 37.0 mol% ENB content) [25]. The T_g of the poly(E-*co*-ENB) depends on the several factors, such as the molecular weight, the ENB content, and the comonomer distribution in the polymer chain. The poly(E-*co*-ENB)s obtained with **1**/TIBA/B catalyst system may contain more ENB repeat units, which leads to their higher T_g values [37].

3.3. *Epoxidation of poly(E-*co*-ENB)*

The poly(E-*co*-ENB)s prepared above possess C=C double bonds, and therefore can be functionalized by epoxidation. As an example, a poly(E-*co*-ENB) sample containing 50.5 mol% of ENB (Run 12 in Table 2) was epoxidized with *m*-chloroperbenzoic acid in toluene at 55 °C for 4 h as shown in Scheme 1. The ^{1}H NMR spectrum of the epoxidized poly(E-*co*-ENB) is shown in Fig. 5. In comparison with Fig. 1a, complete epoxidation is confirmed by the disappearance of the peaks of =CH in ethylidene group at 4.8–5.3 ppm and the appearance of the peaks of –CH in epoxy group at

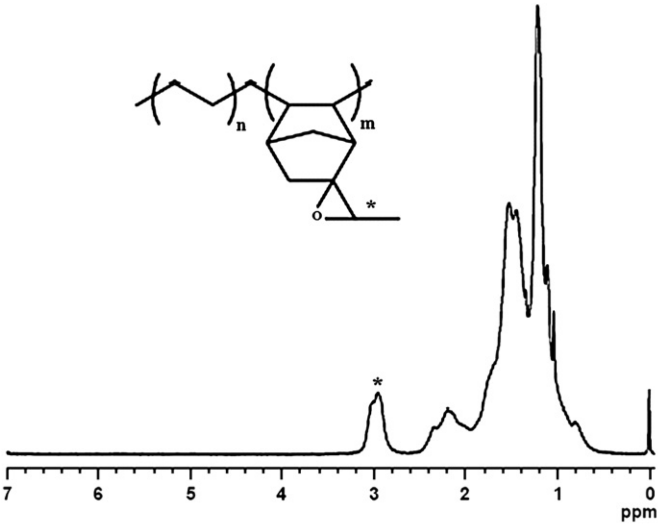


Fig. 5. ^{1}H NMR spectrum of an epoxidized poly(E-*co*-ENB) sample (Run 12 in Table 2) in $CDCl_3$ at room temperature.

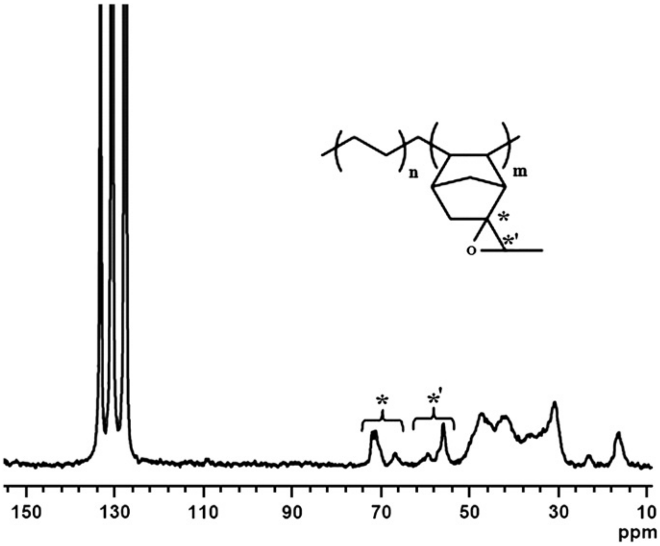


Fig. 6. ^{13}C NMR spectrum of an epoxidized poly(E-*co*-ENB) sample (Run 12 in Table 2) in *o*-$C_6D_4Cl_2$ at 125 °C.

3.0 ppm. The ^{13}C NMR spectrum of the epoxidized poly(E-*co*-ENB) is shown in Fig. 6. The peaks appearing in the regions of 53.1–62.2 and 65.1–74.3 ppm in Fig. 6 can be assigned to the two C atoms in the epoxy group. The peaks for the C═C double bonds in the regions of 108.1–114.9 and 144.8–149.7 ppm in Fig. 3c have completely disappeared in Fig. 6, which further confirms the complete epoxidation of the C═C double bonds. The IR spectra of the poly(E-*co*-ENB) sample and its epoxidation product are shown in Fig. 7, in which the disappearance of the absorption for the C═C stretching vibration (1688 cm^{-1}) and the appearance of the absorption for the epoxy ring stretching vibration (1335 cm^{-1}, symmetric, and 870 cm^{-1}, asymmetric) are also indicative of complete epoxidation. Because of the ring strain, the absorption of the C–O symmetric stretching vibration shifts to 1335 cm^{-1} from about 1260 cm^{-1} [17]. The epoxidized poly(E-*co*-ENB) sample possesses a molecular weight of $M_w = 112 \times 10^3$ with a narrow molecular weight distribution ($M_w/M_n = 2.08$) and a melting temperature of 111.3 °C.

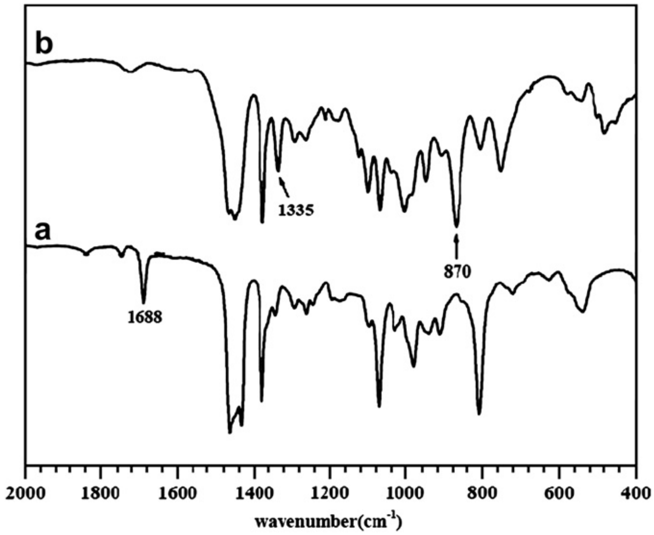


Fig. 7. IR spectra of (a) a poly(E-*co*-ENB) sample (Run 12 in Table 2) and (b) its epoxidation product.

4. Conclusion

In summary, the **1**/TIBA/B catalyst system can effectively catalyze the ENB homopolymerization and E/ENB copolymerization with good catalytic activities and produce poly(E-*co*-ENB)s with moderate molecular weights. ^{1}H NMR analysis of the poly(E-*co*-ENB)s indicates that the ENB comonomer is copolymerized regioselectively through the endocyclic double bond with the ethylidene group unreacted. For the copolymerization, obvious effects of temperature and comonomer can be observed. The ENB content in the poly(E-*co*-ENB) increases with the increase in polymerization temperature and the initial ENB concentration. The ethylidene group remained in the poly(E-*co*-ENB) can be converted into the epoxy group quantitatively with *m*-chloroperbenzoic acid.

Acknowledgments

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