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### Polymerization of Cyclohexene Oxide By Organoaluminum Compounds

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## POLYMERIZATION OF CYCLOHEXENE OXIDE BY ORGANOALUMINUM COMPOUNDS

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### ABSTRACT

The polymerization of cyclohexene oxide (CHO) by various organoaluminum compounds such as  $R_3Al$  ( $R = Me, Et, i-Bu$ ),  $Et_2AlCl$ , and  $EtAlCl_2$  is reported. Ethyl-substituted aluminum compounds were found to be very effective for this polymerization. As more chlorine atom is substituted on the aluminum atom, the polymer yield was increased though the molecular weight was decreased. The polymer yields at varying monomer-to-catalyst mole ratios (M/C) were similar (80–93%). The temperature and solvent effect for the present polymerization were also studied. The present poly(CHO) was a less stereoregular (atactic) isomer, regardless of catalysts and polymerization conditions. The resulting

poly(CHO) was a white powder and was soluble in aromatic and halogenated hydrocarbon solvents such as benzene, chlorobenzene,  $\text{CCl}_4$ , chloroform, etc.

## INTRODUCTION

Various 1,2-epoxides have been polymerized with a number of catalysts [26–28]: Lewis acids, such as  $\text{AlCl}_3$ ,  $\text{SnCl}_4$ ,  $\text{BF}_3 \cdot \text{OEt}_2$ , trialkylaluminum [1–3]; metal alcoholates, such as  $\text{Ca}(\text{OR})_2$  and  $\text{Ba}(\text{OR})_2$  [4]; oxonium ions or carbenium ions, such as  $(\text{OEt}_3)\text{BF}_4$ ,  $(\text{OPh}_3)\text{AsF}_6$ , and  $(\text{OPh}_3)\text{PF}_6$  [5]; Si–O–Al linkages, such as  $[(\text{CH}_3)_3\text{SiO}]_3/\text{Al}$  [6] and  $\text{Ph}_3\text{SiOH}/\text{Al}$  complexes [7–9].

The organoaluminum-based catalysts for the polymerization of propylene oxide are as follows:  $\text{Et}_3\text{Al}/\text{nickel dimethylglyoxime}$  [10],  $\text{Et}_3\text{Al}/N,N\text{-disalicyclidene}(1R,2R)\text{-1,2-cyclohexane-diyldiaminato cobalt(II)}$  [11],  $(i\text{-C}_4\text{H}_9)_3\text{Al}/\text{succinimide}$  [12],  $\text{Al}(\text{O}-i\text{-C}_3\text{H}_7)_3$  [13, 14],  $\text{Al}(\text{O}-i\text{-C}_3\text{H}_7)_3/\text{Zn}(\text{C}_2\text{H}_5)_2$  or  $\text{ZnCl}_2$  [15, 16],  $\text{Et}_3\text{Al}/\text{FeCl}_3$  [17],  $\text{Et}_3\text{Al}/\text{alumina}$  [18],  $\text{Et}_3\text{Al}/\text{primary amine}$  [19], etc.

To date, the polymerization of cyclohexene oxide (CHO) has been carried out by  $\text{Ti}(i\text{-OPr})_4/\text{alcohol}$  (protected aryl alcohol) [20], Al complex/silanol [21,22], diaryliodonium salt/benzoin redox couple [23], etc. Al-based catalyst systems have been especially widely used. However, there have been no systematic studies on the catalytic activity of simple organoaluminum compounds such as  $\text{Et}_3\text{Al}$ ,  $\text{Et}_2\text{AlCl}$ ,  $\text{EtAlCl}_2$ , etc.

This article deals with the polymerization behavior of CHO with various organoaluminum compounds such as  $\text{Et}_2\text{AlCl}$ ,  $\text{EtAlCl}_2$ , and  $\text{R}_3\text{Al}$  ( $\text{R} = \text{Me}$ ,  $\text{Et}$ ,  $i\text{-Bu}$ ), and the characterization of the resulting poly(CHO).

## EXPERIMENTAL

### Materials

CHO (Aldrich Chemicals, 98%) was dried with calcium hydride and fractionally distilled.  $\text{Me}_3\text{Al}$  (Aldrich Chemicals, 2.0 M in toluene),  $\text{Et}_3\text{Al}$  [Aldrich Chemicals, 25 wt% (1.9 M) in toluene],  $(i\text{-Bu})_3\text{Al}$  [Aldrich Chemicals, 25 wt% (0.1 M) in toluene],  $\text{Et}_2\text{AlCl}$  [Aldrich Chemicals, 25 wt% (1.8 M) in toluene], and  $\text{EtAlCl}_2$  [Aldrich Chemicals, 25 wt% (1.8 M) in toluene] were used as received. All polymerization solvents were analytical-grade materials. They were dried with an appropriate drying agent and fractionally distilled.

### Polymerizations

The preparations of the catalyst system and the polymerizations were carried out under a dry nitrogen atmosphere. Organoaluminum compounds were dissolved in chlorobenzene as 0.2 or 0.05 M solutions before use. An example of a typical polymerization is as follows.

A catalyst solution is prepared by mixing  $\text{EtAlCl}_2$  (0.024 mL 0.2 M chlorobenzene solution, 0.040 mmol) and chlorobenzene (3.8 mL,  $[\text{M}]_0 = 2$ ). To this solution is added CHO (1 g, 10.2 mmol). Polymerization was carried at  $-20^\circ\text{C}$  for 24 hours and terminated with a small amount of methanol. The resulting polymers were

dissolved in chloroform followed by precipitation into excess methanol. The precipitated polymers were filtered from the solution and dried under vacuum at 40°C for 24 hours. The polymer yield was calculated by gravimetry. The samples for characterization were reprecipitated into methanol, filtered, and dried under vacuum.

### Instruments

<sup>1</sup>H- and <sup>13</sup>C-NMR spectra were recorded on a Bruker AM-200 spectrometer. Infrared spectra were taken on a Bio-Rad Digilab FTS-60 spectrometer using KBr pellet. The average molecular weights of polymers were measured by means of a Waters GPC-150C using calibration curves for polystyrene in tetrahydrofuran solution. Elemental analyses (C, H) were carried out with a 240C Elemental Analyzer. Thermogravimetric analysis (TGA) was performed under a nitrogen atmosphere at a heating rate of 10°C/min up to 600°C.

## RESULTS AND DISCUSSION

The catalytic activity of various organoaluminum compounds was investigated.

Table 1 shows the results for the polymerization of CHO by five organoaluminum compounds: Me<sub>3</sub>Al, Et<sub>3</sub>Al, (*i*-Bu)<sub>3</sub>Al, Et<sub>2</sub>AlCl, and EtAlCl<sub>2</sub>. Me<sub>3</sub>Al and (*i*-Bu)<sub>3</sub>Al were found to be less effective than ethyl-substituted aluminum compounds. Et<sub>3</sub>Al, Et<sub>2</sub>AlCl, and EtAlCl<sub>2</sub> were found to be very active catalysts for the polymerization of CHO. The polymer yield according to ethyl-substituted organoaluminum compounds increased in the order EtAlCl<sub>2</sub> > Et<sub>2</sub>AlCl > Et<sub>3</sub>Al. However, the molecular weight decreased as the chloride content of the organoaluminum compounds increased.

TABLE 1. Polymerization of CHO by Organoaluminum Compounds<sup>a</sup>

No.	Organoaluminum	Polymer yield <sup>b</sup>	$\overline{M}_n^c$	$\overline{M}_w^c$
1	Me <sub>3</sub> Al	Trace	—	—
2	( <i>i</i> -Bu) <sub>3</sub> Al	20	9,200	26,500
3	Et <sub>3</sub> Al	87	36,200	177,800
4	Et <sub>2</sub> AlCl	90	34,600	111,700
5	EtAlCl <sub>2</sub>	93	10,200	34,100

<sup>a</sup>Polymerization was carried out at -20°C for 24 hours in chlorobenzene. The monomer-to-catalyst mole ratio (M/C) and initial monomer concentration ([M]<sub>0</sub>) were 250 and 2 M, respectively.

<sup>b</sup>Methanol-insoluble polymer.

<sup>c</sup>Measured by means of a Waters GPC-150C using the calibration curves for polystyrene.

TABLE 2. The Effect of Initial Monomer Concentration ( $[M]_0$ ) for the Polymerization of CHO by  $\text{EtAlCl}_2$

No.	$[M]_0^b$	Polymer yield <sup>c</sup>	$\overline{M}_n^d$	$\overline{M}_w^d$
1	0.5	10	—	—
2	1	90	15,800	57,900
3	2	95	15,200	51,200
4	3	85	10,700	35,600
5	4	79	8,000	28,900

<sup>a</sup>Polymerization was carried out at  $-20^\circ\text{C}$  for 24 hours in toluene. The monomer-to-catalyst mole ratio (M/C) was 250.

<sup>b</sup>Mol/L, M.

<sup>c</sup>Methanol-insoluble polymer.

<sup>d</sup>Measured by means of a Waters GPC-150C using the calibration curves for polystyrene.

Table 2 shows the effect of the initial monomer concentration ( $[M]_0$ ) on the polymerization of CHO by  $\text{EtAlCl}_2$ . The highest polymer yield was obtained with increased  $[M]_0$ .

Table 3 shows the effects of CHO to catalyst mole ratio (M/C) for the polymerization of CHO by  $\text{EtAlCl}_2$ . The polymer yields according to M/C were similar (81–93%). The highest polymer yield was obtained when the M/C was 250. On the other hand, the molecular weight increased as the M/C increased.

Table 4 shows the temperature effect on the polymerization of CHO by  $\text{EtAlCl}_2$ . Polymerization did not proceed at  $-78^\circ\text{C}$ . This behavior was also observed for the polymerization of CHO by  $\text{Et}_3\text{Al}$  and  $\text{Et}_2\text{AlCl}$ . The best results were obtained at  $-20$  and  $5^\circ\text{C}$  from the standpoint of polymer yield and average molecular weight. The polymerization of CHO at relatively high temperatures gives a low yield of polymers having relatively low molecular weights.

TABLE 3. The Effect of CHO to Catalyst Mole Ratio for the Polymerization of CHO by  $\text{EtAlCl}_2^a$

No.	CHO to $\text{EtAlCl}_2$ (mole ratio)	Polymer yield, <sup>b</sup> %	$\overline{M}_n^c$	$\overline{M}_w^c$
1	50	81	10,600	40,400
2	100	87	11,100	40,300
3	250	93	15,200	54,200
4	500	85	19,100	69,000
5	1000	82	22,900	84,300

<sup>a</sup>Polymerization was carried out at  $-20^\circ\text{C}$  for 24 hours in chlorobenzene. The initial monomer concentration ( $[M]_0$ ) was 2 M.

<sup>b</sup>Methanol-insoluble polymer.

<sup>c</sup>Measured by means of a Waters GPC-150C using the calibration curves for polystyrene.

TABLE 4. Temperature Effect for the Polymerization of CHO by  $\text{EtAlCl}_2^a$ 

No.	Temperature	Polymer yield <sup>b</sup>	$\overline{M}_n^c$	$\overline{M}_w^c$
1	-78	Trace	—	—
2	-20	95	15,200	51,200
3	5	93	14,700	50,800
4	30	76	10,200	38,800
5	60	71	7,800	32,600
6	90	60	7,200	30,100

<sup>a</sup>Polymerization was carried out at  $-20^\circ\text{C}$  for 24 hours in toluene. The monomer-to-catalyst mole ratio (M/C) and initial monomer concentration ( $[\text{M}]_0$ ) were 250 and 2 M, respectively.

<sup>b</sup>Methanol-insoluble polymer.

<sup>c</sup>Measured by means of a Waters GPC-150C using the calibration curves for polystyrene.

Figure 1 shows the time-dependence curve of polymer yield for the polymerization of CHO by  $\text{EtAlCl}_2$ . Polymerization proceeded rapidly initially to give 50% polymer yield after 5 hours, and then polymerization proceeded more slowly to give a final polymer yield after 24 hours.

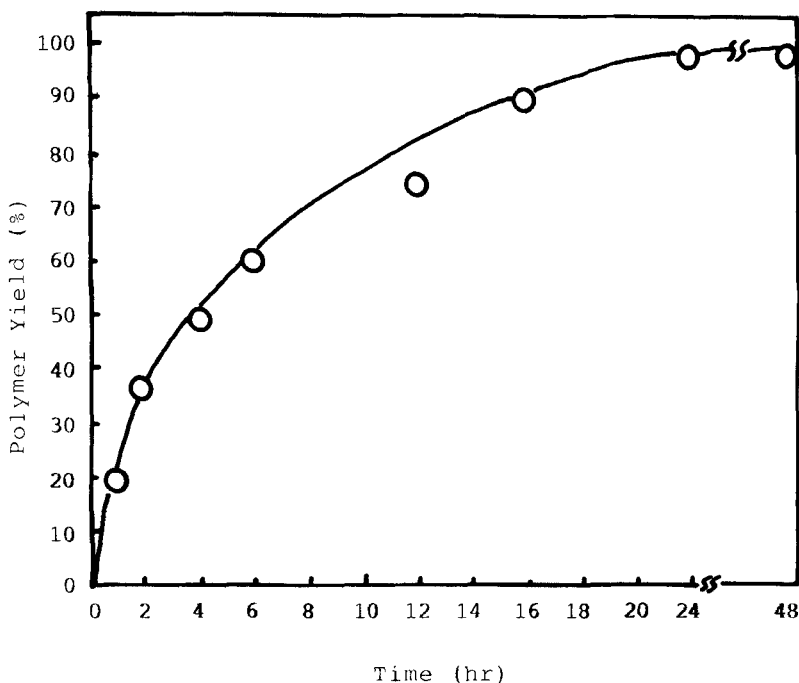


FIG. 1. Time-dependence curve of polymer yield for the polymerization of CHO by  $\text{EtAlCl}_2$ . Solvent, toluene; M/C = 250, temperature  $-20^\circ\text{C}$ ,  $[\text{M}]_0 = 2$ .

Table 5 shows the solvent effect on the polymerization of CHO by EtAlCl<sub>2</sub> and Et<sub>3</sub>Al. Aromatic and halogenated hydrocarbons were used as solvents. Polymerization was good in aromatic solvents such as chlorobenzene, toluene, and benzene. However, polymerization did not occur in nitrobenzene and MEK, probably because of complex formation of the catalyst with nitrobenzene and MEK. Chlorine-containing hydrocarbon solvents such as methylene chloride and 1,1-dichloroethylene were found to be good solvents whereas carbon tetrachloride and chloroform give only a low yield of polymer. EtAlCl<sub>2</sub> and Et<sub>3</sub>Al show similar polymerization behaviors.

The elemental analysis data of poly(CHO) prepared by EtAlCl<sub>2</sub> agreed well with the theoretical value. Calculated for (C<sub>6</sub>H<sub>10</sub>O)<sub>n</sub>: C, 73.47; H, 10.20; O, 16.33%. Found: C, 72.46; H, 10.58; O, 16.35%.

The polymer structure was identified by NMR and IR spectroscopies. Figure 2 shows the IR spectrum of poly(CHO) prepared by EtAlCl<sub>2</sub>. This shows the aliphatic C—H stretching frequency at 2932 cm<sup>-1</sup>, and it shows an absorption peak at 1027 cm<sup>-1</sup> for the C—O—C stretching frequency.

Figure 3 shows the <sup>1</sup>H-NMR spectrum of poly(CHO) prepared by EtAlCl<sub>2</sub>. The peaks between 3.5 and 3.8 ppm are due to the hydrogens of the carbon adjacent to the ether linkage. The proton peaks of methylene carbon were observed at 1.2–1.9 ppm.

TABLE 5. Solvent Effect for the Polymerization of CHO by EtAlCl and Et<sub>3</sub>Al<sup>a</sup>

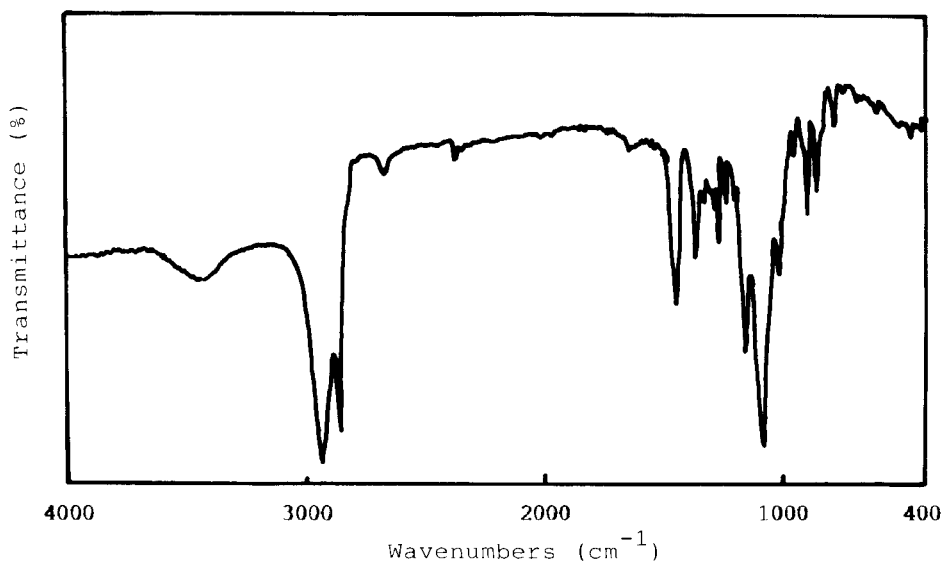
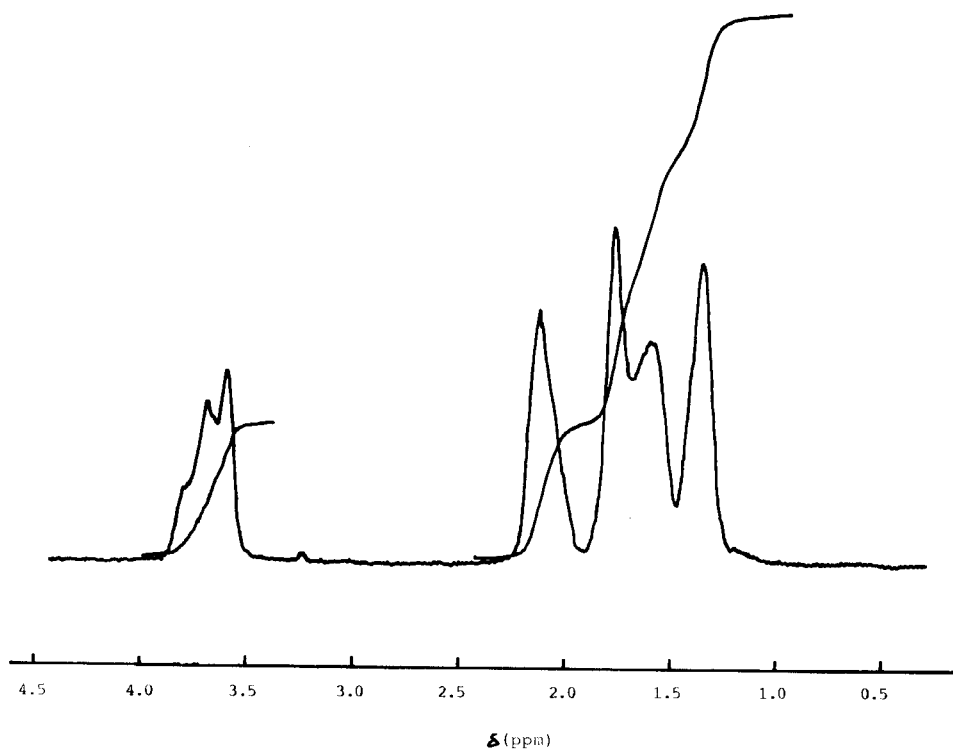
No.	Solvent	Catalyst	Polymer yield, <sup>b</sup> %	$\bar{M}_n^c$	$\bar{M}_w^c$
1	Chlorobenzene	Et <sub>3</sub> Al	87	36,200	177,800
2	"	EtAlCl <sub>2</sub>	93	15,200	54,800
3	Toluene	Et <sub>3</sub> Al	90	35,700	182,000
4	"	EtAlCl <sub>2</sub>	95	16,300	62,000
5	Benzene <sup>d</sup>	Et <sub>3</sub> Al	52	24,800	67,000
6	"	EtAlCl <sub>2</sub>	67	—	—
7	CCl <sub>4</sub>	Et <sub>3</sub> Al	10	—	—
8	"	EtAlCl <sub>2</sub>	17	—	—
9	CH <sub>2</sub> Cl <sub>2</sub>	Et <sub>3</sub> Al	60	29,400	61,800
10	"	EtAlCl <sub>2</sub>	65	9,800	37,600
11	CH <sub>2</sub> =CCl <sub>2</sub>	Et <sub>3</sub> Al	93	18,800	66,500
12	"	EtAlCl <sub>2</sub>	72	10,400	47,800
13	MEK	Et <sub>3</sub> Al	0	—	—
14	"	EtAlCl <sub>2</sub>	0	—	—
15	Nitrobenzene	Et <sub>3</sub> Al	0	—	—
16	"	EtAlCl <sub>2</sub>	0	—	—

<sup>a</sup>Polymerization was carried out at -20°C for 24 hours. The monomer-to-catalyst mole ratio (M/C) and initial monomer concentration ([M]<sub>0</sub>) were 250 and 2 M, respectively.

<sup>b</sup>Methanol-insoluble polymer.

<sup>c</sup>Measured by means of a Waters GPC-150C using the calibration curves for polystyrene.

<sup>d</sup>Polymerization temperature was 10°C.

FIG. 2. IR spectrum of poly(CHO) prepared by EtAlCl<sub>2</sub>.FIG. 3. <sup>1</sup>H-NMR spectrum of poly(CHO) prepared by EtAlCl<sub>2</sub>.



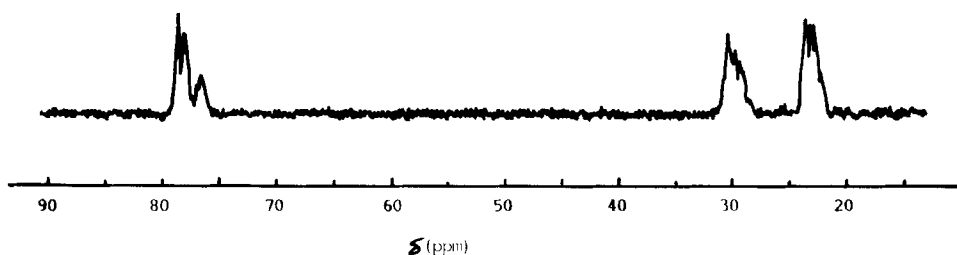


FIG. 4.  $^{13}\text{C}$ -NMR spectrum of poly(CHO) prepared by  $\text{EtAlCl}_2$ .

The shape of  $^1\text{H}$ -NMR spectra of poly(CHO)s prepared by  $\text{ZnEt}_2/(S,S)$ -1,2-diphenyl ethandiol, etc., were found to be very sensitive to the nature of the solvent used and to the recording temperature, indicating that the conformational effects of the cyclohexane ring may superimpose on the tacticity effects [24]. It was reported that the methine proton peaks in the  $^1\text{H}$ -NMR spectrum of poly(CHO) were assigned to isotactic (3.49 ppm), syndiotactic (3.40 ppm), and heterotactic (3.32 ppm) triads [25, 29]. From this viewpoint, the present poly(CHO)s prepared by  $\text{EtAlCl}_2$  are more atactic. Similar stereochemical results were also observed in the poly(CHO)s prepared by  $\text{Et}_3\text{Al}$  and  $\text{Et}_2\text{AlCl}$ , regardless of the catalysts and polymerization solvents used.

Figure 4 shows the  $^{13}\text{C}$ -NMR spectrum of poly(CHO) prepared by  $\text{EtAlCl}_2$ . It is clearly seen in the methine carbon region that the intensity of peaks at 78.57 ppm

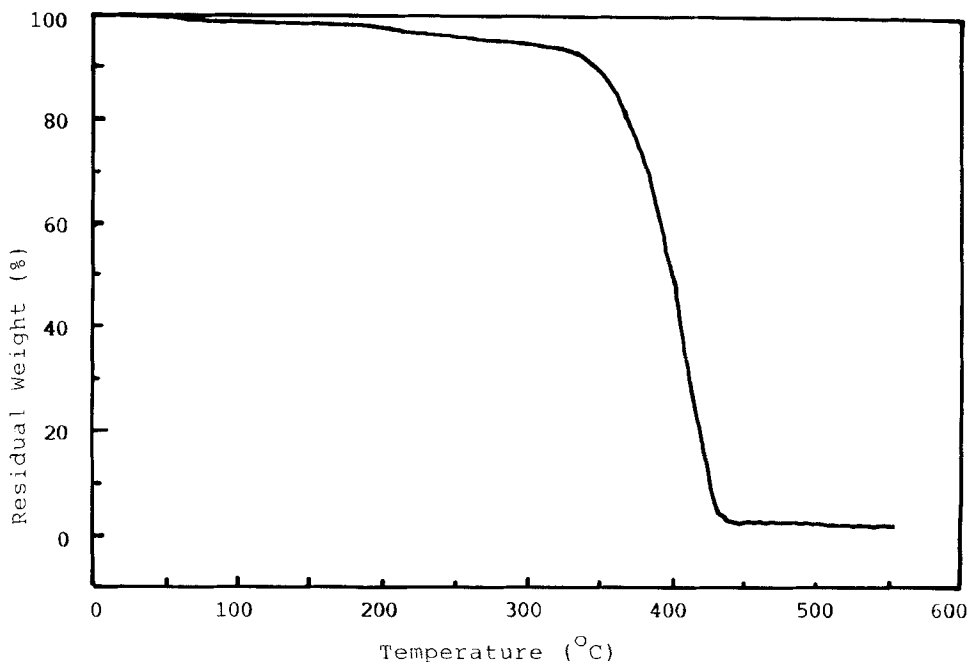


FIG. 5. TGA thermogram of poly(CHO) prepared by  $\text{EtAlCl}_2$ .

become much more intense in the case of optically active samples [24]. These peaks can be assigned to isotactic, e.g.,  $-RR-RR-$ , enchainments. From comparative studies, the peaks at 76.64 and 78.07 ppm correspond to the syndiotactic  $-RR-SS-$  enchainments and atactic enchainments, respectively. The peaks at about 30 and 23 ppm are due to the methylene carbons furthest from the oxygen atom. The  $^{13}\text{C}$ -NMR spectrum of poly(CHO) prepared at a relatively high temperature ( $90^\circ\text{C}$ ) is similar with that of poly(CHO) prepared at  $-20^\circ\text{C}$ .

The resulting poly(CHO) was a white powder. This polymer is soluble in aromatic and halogenated hydrocarbon solvents such as benzene, chlorobenzene,  $\text{CCl}_4$ , chloroform, etc., but insoluble in methanol, ethyl ether, hexane, DMF, etc.

Figure 5 shows the TGA thermogram of poly(CHO) prepared by  $\text{EtAlCl}_2$ . It shows that this poly(CHO) retained 98% of its original weight at  $200^\circ\text{C}$ , 95% at  $300^\circ\text{C}$ , 89% at  $351^\circ\text{C}$ , and 3% at  $450^\circ\text{C}$ .

## CONCLUSIONS

CHO was easily polymerized by ethyl-substituted aluminum compounds. As more chlorine atoms are substituted on the aluminum atom, the polymer yield increases and the molecular weight decreases. The polymerization behavior of CHO according to the initial monomer concentration ( $[\text{M}]_0$ ), the CHO to catalyst mole ratio (M/C), the temperature, and the solvent were studied. The best results were obtained when  $[\text{M}]_0$  and the temperature were 2 and  $-20^\circ\text{C}$ , respectively. The polymer yields according to M/C were similar although the molecular weight increased as M/C increased. Polymerization proceeded well with aromatic solvents such as chlorobenzene, benzene, and toluene, and with some chlorine-containing aliphatic solvents such as  $\text{CH}_2\text{Cl}_2$  and 1,1-dichloroethylene.

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