Polymerization and Characterization of Bis(trichlorophenolato)di(pyridine) Nickel(II) and Bis(tribromophenolato)di(pyridine) Nickel(II) Dihydrate Complexes in Solution in the Presence of Iodine

L. (Karabey) Molu, D. Kısakürek

*Department of Chemistry, Middle East Technical University, 06531 Ankara, Turkey*

ABSTRACT: The syntheses of bis(trihalophenolato)-di(pyridine) nickel(II) complexes were achieved in aqueous solution, and their characterizations were performed by Fourier transform infrared (FTIR) spectroscopy, ultraviolet–visible analysis, differential scanning calorimetry, and elemental analysis. The thermal polymerization of these complexes was studied in toluene solution in the presence of iodine. The effect of time, temperature, and amount of iodine added on the percentage conversion, structure of poly-mers, and intrinsic viscosity ($[\eta]$) were investigated. Poly-mers were characterized by FTIR, ^{1}H-NMR, and ^{13}C-NMR spectroscopic analyses, glass-transition temperatures deter-mined by differential thermal analysis, and $[\eta]$ values deter-mined by the viscometric method. © 2002 Wiley Periodicals, Inc. J Appl Polym Sci 86: 2232–2239, 2002

Key words: solution polymerization; FTIR; UV-vis; DSC

INTRODUCTION

Halogenated poly(phenylene oxide)s have the poten-tial for outstanding mechanical, chemical, electrical, and thermal properties, provided they can be pre-pared as linear polymers with high molecular weights.[1] Such polymers have also been found to be extremely fire resistant.[2] Poly(dihalophenylene ox-ide)s constitute a series of polymers that can be mod-ified easily by placement of the halogen substituents in the 2, 4, and 6 positions of phenol. The polymeriza-tion of trihalophenols by the thermal decomposition of silver salts in solution;[3–6] copper complexes in solu-tion,[6–18] in solid state,[17–21] and by electroinitia-tion;[22–27] cobalt complex in solid state,[21,28,29] nickel complexes in solid state,[30] in solution,[31] and by elec-troinitiation;[32] and cerium(IV) complexes in solid state[33] have been reported. The suggested reaction mechanism is

$$n\ \mathrm{M(OC_6H_2X_3)_yL_m} \rightarrow n\ \mathrm{MX_yL_m} + (-\mathrm{C_6H_2X_2O}-)_{yn}$$
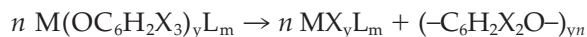
where M is a transition or inner transition metal, L is a neutral ligand, X is a halogen, and y and m are the numbers of trihalophenols and ligands, respectively.

It is well known that the nature of the ligands and transition metals, the coordination of the complex, and the method of synthesis profoundly affect the ease with which thermal decomposition occurs with such complexes. A study that investigated the action of iodine on polymerization[34] showed that the iodine removes the metal atom, forming metal iodide and an unsaturated residue, trihalophenolate, which loses an atom of halogen. This halogen expels the iodine from the metal iodide, forming metal halide. The new, un-saturated residue polymerizes, forming poly(dihalo-phenylene oxide).

The trihalophenolate ions form crystallographically characterized complexes involving chelation through oxygen and chlorine donor atoms to cobalt(II),[29] cop-per(II),[21,35] nickel(II), and silver(I) ions. The five-coor-dinated bis(trichlorophenolato)tri(pyridine) nickel(II) complex lies on a crystallographic twofold symmetry axis, and the geometry about the nickel is described as a distorted square pyramid with one of the pyridine ligands occupying an axial position.[36] However, bis(trihalophenolato)di(pyridine) nickel(II) complexes are in the amorphous form.

In this study, thermal polymerizations of four-coor-dinated bis(trihalophenolato)di(pyridine) nickel(II) complexes were carried out in toluene solution in the presence of iodine. In the previous studies, thermal polymerization of these complexes in solid state and in melt[30] and by electroinitiation[32] were reported. This work was a part of a research project aimed at finding new ways to synthesize poly(dihalophenylene oxide)s through the thermal decomposition of phenolate tran-sition metal complexes in the absence of a chelating

---

Correspondence to: D. Kısakürek (duygu@metu.edu.tr).
Contract grant sponsor: METU.

TABLE I
Elemental Analyses of Complexes

	C (%)	H (%)	N (%)	Ni (%)
Complex 1				
Calculated	43.33	2.31	4.59	9.62
Found	43.26	2.53	4.94	9.39
Complex 2				
Calculated	28.95	1.97	3.07	6.43
Found	29.66	1.94	3.23	5.72

Experimental error = ±0.3.

ligand, yielding greater insight into the polymerization process, the influence of nickel(II) complexes, and the effect of iodine added to the toluene solution on the ease of polymerization.

EXPERIMENTAL

Materials

Analytical-grade 2,4,6-trichlorophenol (TCP; Merck) and 2,4,6-tribromophenol (TBrP; Aldrich Chemical Co.) were used without further purification. The reagent-grade pyridine (Riedel de Hagen AG) was used as a neutral nonchelating ligand. Commercially available reagent-grade nickel sulfate hexahydrate ($NiSO_4 \cdot 6H_2O$; Aldrich) was used in the preparation of trihalophenolate complexes. Toluene (Merck) was used as a solvent in polymerization. Solid I_2 was commercially available and technical grade and was used as an initiator for polymerization. Fractionally distilled ethanol was used as a polymer-precipitating reagent.

Apparatus and procedure

Synthesis of complexes

Nickel complexes of trihalogenated phenols were prepared from aqueous solutions of reagents. A solution of 0.5 mol of NaOH, 0.5 mol of trihalogenated phenol, and 0.25 mol of pyridine in 1 L of distilled water was slowly added to a solution containing 0.25 mol of $NiSO_4 \cdot 6H_2O$ in 250 cm^3 of distilled water, with vigorous stirring for 2 h. The resulting greenish blue complexes were recovered by filtration, washed with distilled water, and dried in a vacuum oven to a constant weight. The greenish blue color of the complexes turned into yellow after drying, most likely due to the change of the water of crystallization.

Synthesis of polymers

The thermal polymerizations of bis(trichlorophenolato)di(pyridine) nickel(II) (complex **1**) and bis(tribromophenolato)di(pyridine) nickel(II) dihydrate (complex **2**) were performed at three different conditions. First, time (3 h) and the amount of iodine (10 wt %)

were kept constant, whereas the temperature was varied from 40 to 110°C. Then, time intervals (3–24 h) were varied at a constant temperature and amount of iodine. Finally, the amount of iodine was varied at a constant temperature and time.

The decomposition of 4-g batches of complex, dissolved in 60 cm^3 of toluene with a known amount of iodine, was performed in a round-bottomed flask inserted in a glycerine oil bath with a temperature fluctuation of ±1°C for a given temperature and period of time. The decomposed complex was poured into a large amount of ethanol containing a few drops of concentrated HCl. The precipitated polymer was recovered by filtration, washed with ethanol, and dried to a constant weight under vacuum.

Characterization of complex

Complexes **1** and **2** were characterized by elemental analysis with a Leco 932 CHN analyzer. The metal content of the complexes were determined with an ATI Unicam solar 929 model atomic absorption spectrometer with an AAS Unicam VP-90 vapor system. The elemental analyses of the complexes are listed in Table I.

The structural analyses of nickel complexes were carried out by Fourier transform infrared (FTIR) spectroscopy in KBr discs and by ultraviolet–visible spectroscopy in *N,N*-dimethylformamide with a Nicolet 510 model and a diode array Hewlett Packard 8452 A, respectively. The molar absorbitivities of the complexes are listed in Table II.

The purity and thermal behavior of the complexes were analyzed by differential scanning calorimetry (DSC) with a scanning rate of 10°C/min for 10-mg samples, as shown in Figure 1.

Characterization of polymers

FTIR analysis. FTIR spectra were recorded on a Nicolet model 510 spectrometer with the polymers dispersed in KBr discs.
NMR analysis. ¹H-NMR and decoupled ¹³C-NMR spectra were recorded on a Brucker GmbH DPX-400 400-MHz high-performance digital FT NMR, with CDCl$_3$ as a solvent and TMS as an internal reference.

TABLE II
Molar Absorbitivities of the Complexes

|	ε (L mol^{-1} cm^{-1})		Absorbance maxima (nm)	
	ε_1	ε_2	λ_1	λ_2
Complex **1**	5144	11104	342	268
Complex **2**	7392	13437	332	268
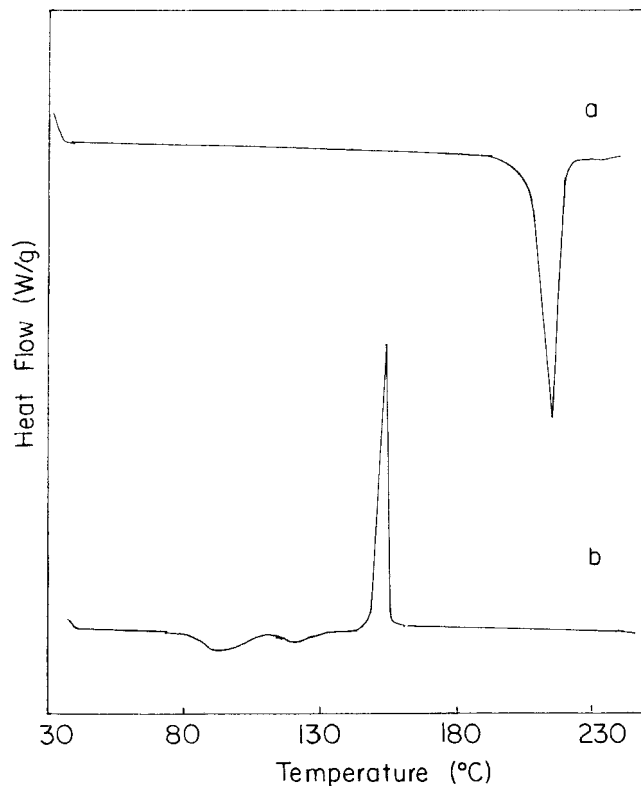
Figure 1 DSC thermograms of complexes (a) **1** and (b) **2**.

DSC. The glass-transition temperatures (T_g's) were determined with the use of a Dupont thermal analysis differential scanning calorimeter (model 2000 DSC 910 S) with a scanning rate of 10°C/min for 10-mg polymer samples.

Intrinsic viscosity ([η]) measurements. [η] values of the polymers were determined at 30°C in toluene with a Schott Geräte AVS 400 automatic viscometer equipped with a Schott Geräte CT 1150 thermostat, with an efflux time of 124 s.

RESULTS AND DISCUSSION

FTIR spectra of complexes **1** and **2** were similar to those of copper and nickel complexes given in the
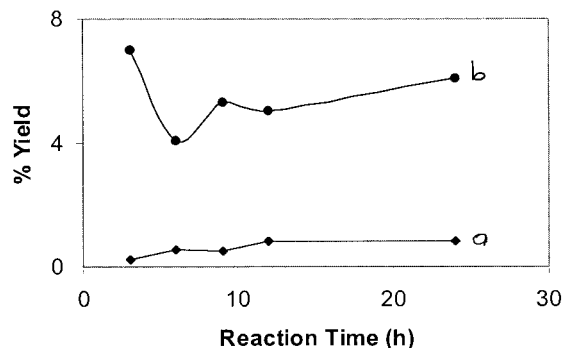
Figure 2 Change in percentage yield with reaction time at a constant amount of I$_2$ (10%) and at a constant temperature of complexes: (a) **P1** at 60°C and (b) **P2** at 90°C.
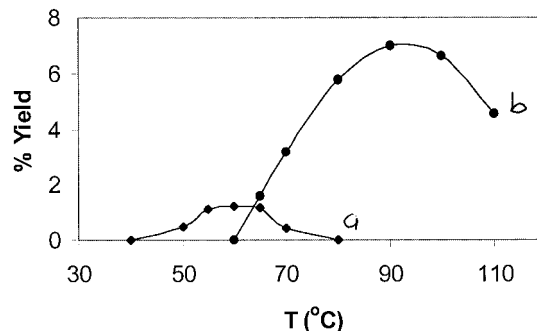
Figure 3 Change in percentage yield of poly(dihalophenylene oxide)s with temperature at the end of 3 h and at a constant amount of I$_2$ (10%): (a) **P1** and (b) **P2**.

literature,[7] exhibiting the characteristic absorption of C—N stretching at about 1064 cm^{-1} and the disappearances of phenolic O—H peaks around 3500 cm^{-1}. In the 3100 cm^{-1} region, C—H stretching of the benzene ring of phenol was also observed. A broad peak at around 3500 cm^{-1} was caused by the presence of water molecules in complex **2**.

The results of elemental analysis for the synthesized amorphous complexes correlated with stoichiometric calculations, 2 mol of nonchelating amine ligand and 2 mol of phenolate ligand in complexes **1** and **2** coordinate per metal ion. Molar absorptivities of the complexes were too high in energy for simple *d–d* transition and were assigned to charge-transfer transitions. The structure of amorphous Ni(THP)$_2$(Py)$_2$ complexes was reported as square planar.[37]

The DSC thermogram of complex **1** had one sharp endothermic peak at 215.33°C due to the melting of the complex [Fig. 1(a)]. In the DSC thermogram of complex **2** [Fig. 1(b)], there was one exothermic peak at 153.25°C, indicating new bond formation taking place, and one broad endothermic peak around 94°C

TABLE III
Percentage Conversion Values of Polymers Obtained at Different Temperatures (*Ts*) at the End of 3 h of Polymerization Time with 10% Iodine

T (°C)	Polymer	Conversion (%)	[η] (dL/g)
40	1	—	—
50	1	—	—
55	1	0.12	—
60	1	0.22	—
	2	—	—
65	1	0.19	—
70	1	—	—
	2	3.17	0.020
80	1	—	—
	2	5.76	0.020
90	1	—	—
	2	6.98	0.020
100	2	6.64	0.020
110	1	—	—
	2	4.54	0.020

TABLE IV
Percentage Conversion and [η] Values of Polymers at Different Times, at Decomposition Temperatures of 60 and 90°C for Complexes 1 and 2, Respectively, and at 10 wt % I$_2$

Time (h)	Complex	I$_2$ (wt %)	Conversion (%)	[η] (dL/g)
3	**1**	10	0.21	—
	2	10	6.98	0.020
6	**1**	10	0.54	—
	2	10	4.09	0.020
9	**1**	10	0.52	—
	2	10	5.31	0.020
12	**1**	10	0.84	—
	2	10	5.04	0.019
24	**1**	10	0.83	—
	2	10	6.08	0.019

corresponding to the melting point of trapped TBrP (87–89°C), as confirmed by elemental analysis results.

The thermal decomposition of nickel trihalophenolate complexes in toluene solution in the presence of iodine was performed at three different conditions. First, time at 3 h and I$_2$ content at 10% were kept constant, and the temperature was varied from the induction temperature of the polymer to toluene's boiling point. Second, at the maximum yield temperature and 10% I$_2$ content, time was varied from 3 to 24 h. Finally, time (3 h) and temperature were kept constant, and the effect of the amount of iodine on polymerization was studied. The yields of polymers were based on the initial weight of the complexes, where the neutral ligands and nickel were not incorporated into the polymer.[7,13]

TABLE V
Percentage Conversion and [η] Values of Polymers at Different Iodine Percentages at the End of 3 h of Polymerization Time and at Decomposition Temperatures of 60 and 90°C for Complexes 1 and 2, Respectively

I$_2$ (wt %)	Polymer	Conversion (%)	[η] (dL/g)	Weight-Average molecular weight ($\times 10^{-3}$)
10	**1**	0.22	—	—
	2	6.98	0.020	—
12.5	**2**	14.19	0.022	—
15	**2**	17.09	0.020	—
20	**1**	13.82	0.026	6
25	**2**	19.42	0.022	—
40	**1**	31.02	0.029	9
50	**2**	30.08	0.024	—
60	**1**	37.67	0.029	9
75	**2**	37.49	0.023	—
80	**1**	47.75	0.029	9
100	**1**	50.54	0.031	11
	2	40.02	0.027	—
200	**1**	54.93	0.031	11
	2	48.82	0.028	—
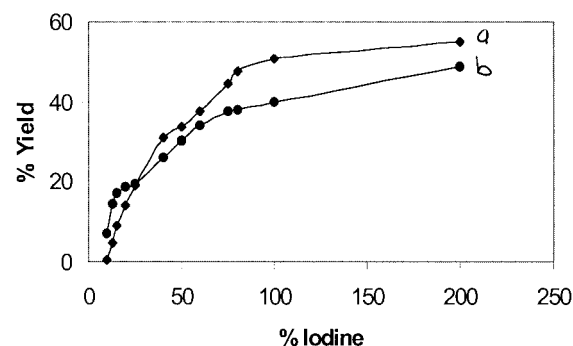
Figure 4 Change in percentage yield of poly(dihalophenylene oxide)s with percentage iodine at the end of 3 h and at a constant temperature: (a) **P1** at 60°C and (b) **P2** at 90°C.

The polymer yields versus time curves for poly(dichlorophenylene oxide) (**P1**) and poly(dibromophenylene oxide) (**P2**) at constant temperatures of 60 and 90°C, respectively, showed that time had a slight effect on the percentage yield of **P1**, as shown in Figure 2(a), with a slight increase up to 12 h followed by a plateau. However, Figure 2(b) illustrates that the maximum percentage yield was observed at 3 h for **P2**, followed by a decrease at the end of 6 h and then a slight increase. **P2** had higher yield values than **P1** at the end of 24 h of polymerization. The changes in the percentage yields of polymers with temperature are listed in Table III and illustrated in Figure 3. Polymerization was started at 40°C with a very small amount for polymer **P1** and then was slightly increased, reaching a plateau around 60°C, followed by a decrease [Fig. 3(a)]. Percentage decomposition of complex **2** showed first a sharp increase up to 6.98% and then a decrease in percentage yield to 4.54% at toluene's boiling point [Fig. 3(b)]. The thermal decomposition of complexes **1** and **2** in solution gave lower percentage yield than both did in solid state[30] and than mixed chelating and nonchelating ligands in bis(trihalophenolato)(ethylenediamine)(pyridine)nickel(II) complexes did in solution.[31] The effect of reaction time and temperature on [η] values of the polymers is listed in Tables III and IV, respectively. The amount of polymeric products
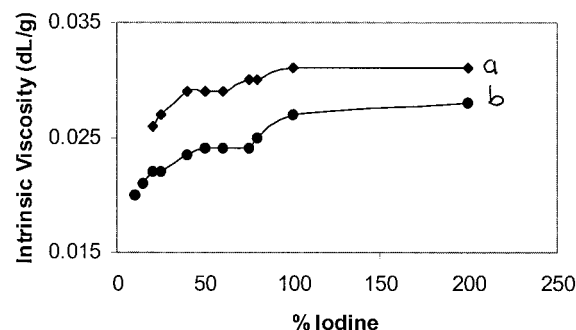
Figure 5 Change in [η] with percentage iodine at the end of 3 h and at a constant temperature: (a) **P1** and (b) **P2**.
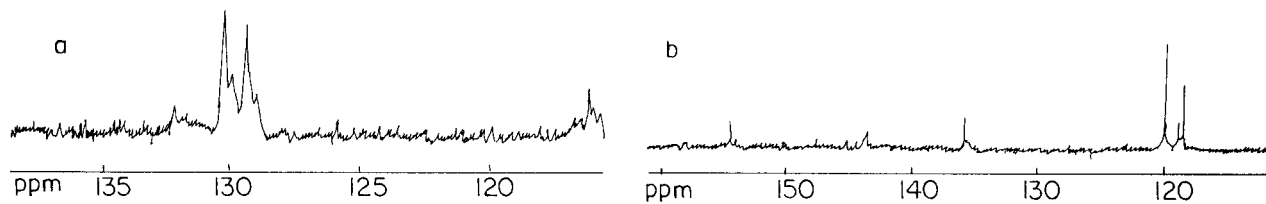
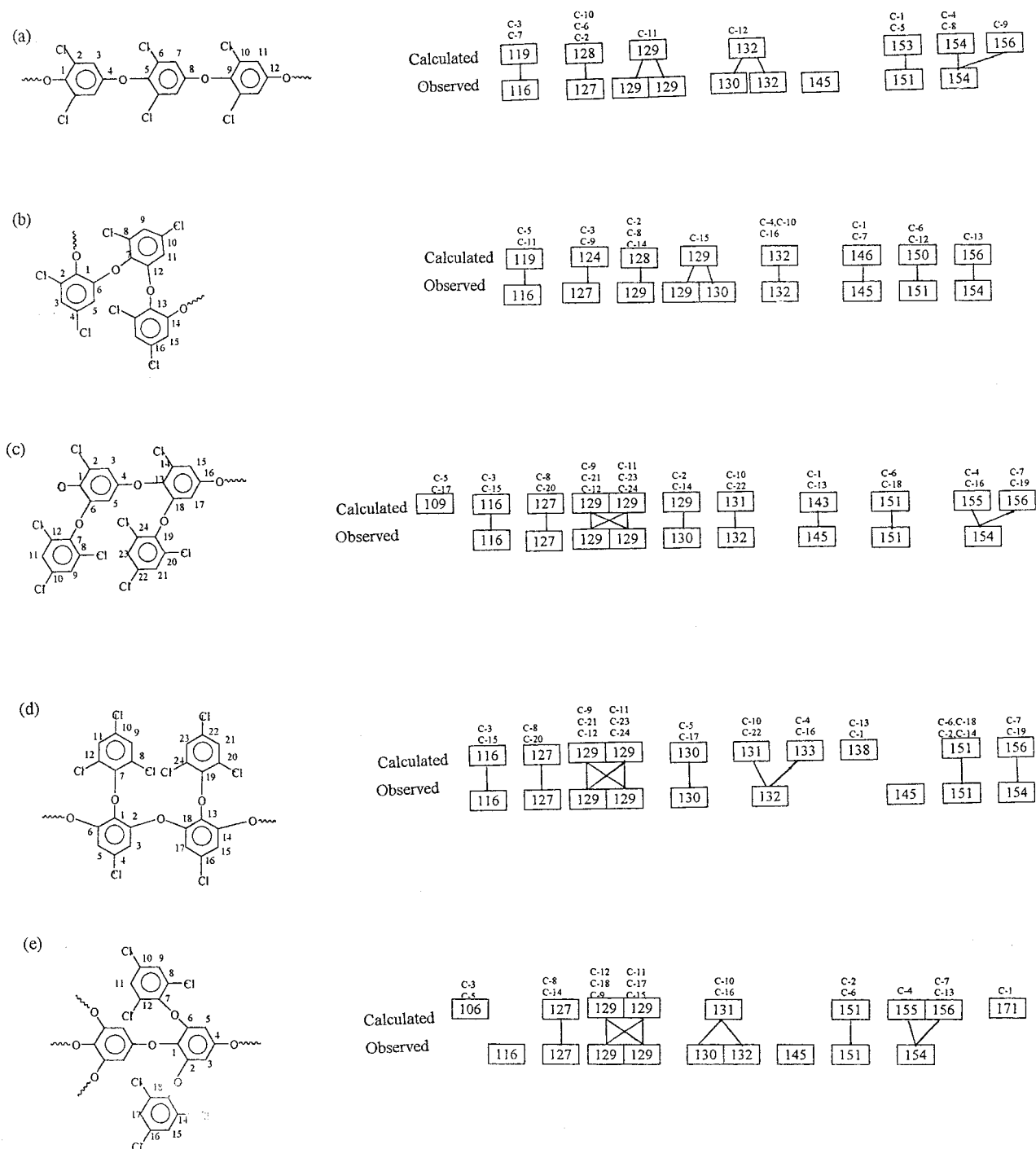**Figure 6** Proton-decoupled ^{13}C-NMR spectra of (a) **P1** and (b) **P2**.

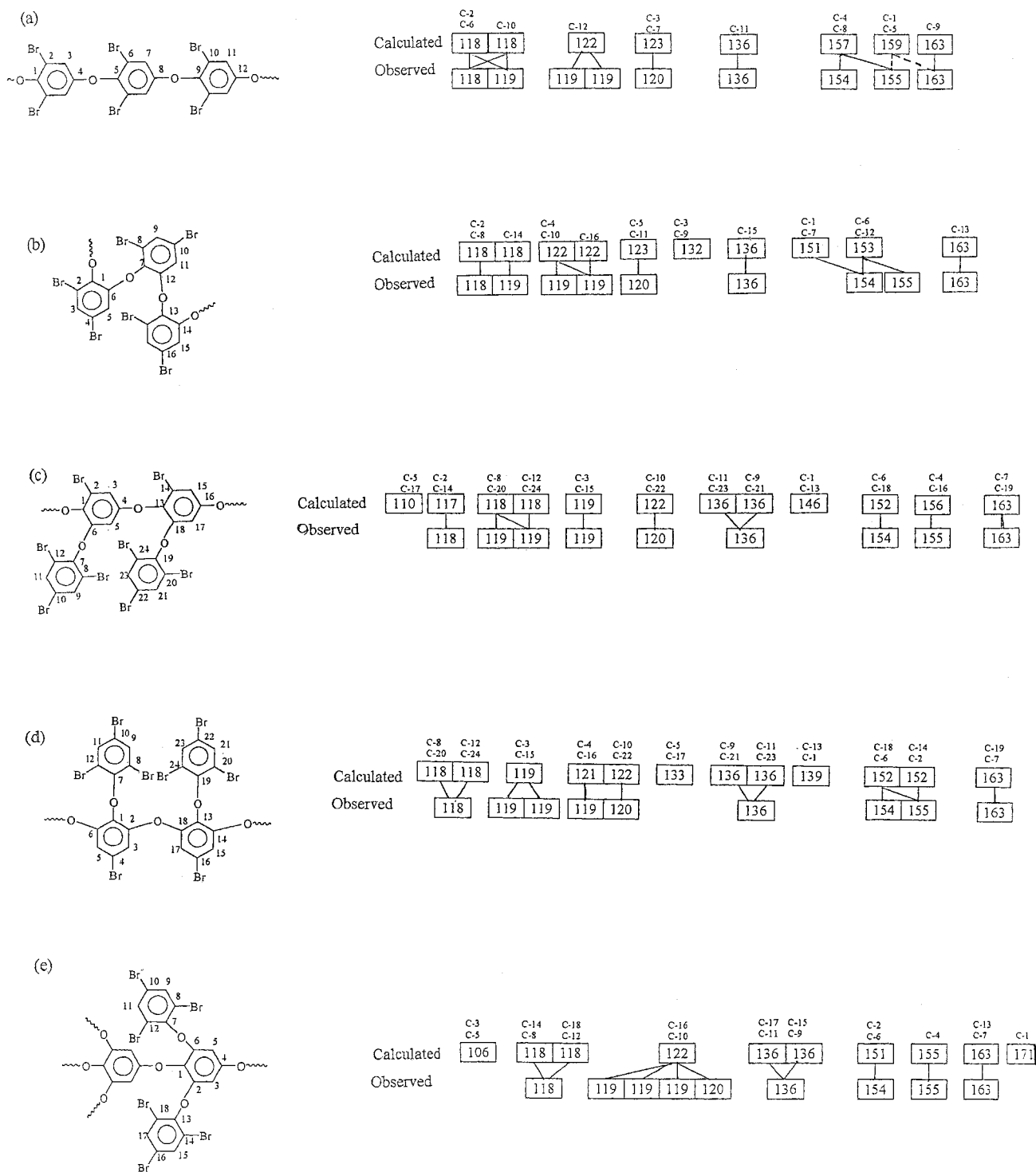**Figure 7** Schematic representation of the observed and calculated ^{13}C-NMR of **P1**.

Figure 8 Schematic representation of the observed and calculated ^{13}C-NMR of **P2**.

obtained for complex **1** was too small to measure [η]. The [η] values were almost constant for **P2**, indicating that polymerization time and temperature had no effect on [η].

The effect of iodine on polymerization was studied at a constant time (3 h) and temperature (60 and 90°C for **P1** and **P2**, respectively) and are listed in Table V.

The decomposition of the both complexes was achieved in solution in the presence of minimum amount of I$_2$ (10 wt %). The yields of polymer showed a rapid increase [Fig. 4(a,b)] as the amount of iodine added to the toluene solution increased. The increase in the percentage yield was observed up to 200% I$_2$. Initially, **P1** had lower percentage yield than did **P2** up
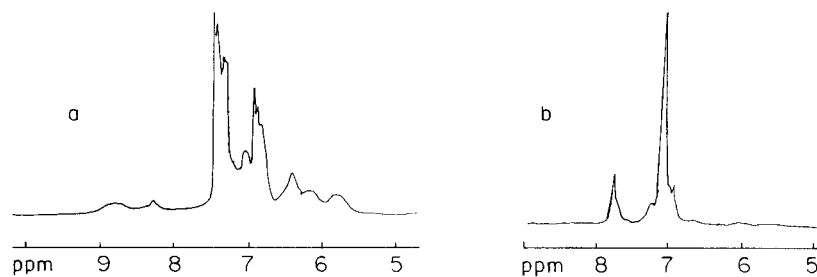
Figure 9 ^{1}H-NMR spectra of (a) **P1** and (b) **P2**.

to 25% I_2. However, it reached a maximum value of 55%, whereas **P2** had a maximum value 49% at 200% I_2. A jump in percentage yield with an increase in I_2 was observed at 40 and 50 wt % I_2 for complexes **1** and **2**, respectively, where the number of moles of THP in the complex had the same molar ratio with the added iodine. The effects of iodine added on $[\eta]$ values of the polymers are listed in Table V. **P1** and **P2** showed a slight increase in $[\eta]$ values up to 40 and 50 wt % I_2 and then reached a plateau having constant chain lengths, whereas the amounts of decomposed complex increased rapidly, as shown in Figure 5(a,b).

The maximum molecular weight calculated with K and α values given in the literature[38] for polymer **P1** was 1.1×10^4, which was lower compared to the same that of the polymer synthesized from the decomposition of Ni(TCP)$_2$(en)(Py) in solution[31] and Ni(TCP)$_2$(Py)$_2$ in solid state.[30]

The absorption bands in the FTIR spectra of polymers were in the ranges 1380–1610 cm^{-1} due to C=C ring stretchings, 1110–1245 cm^{-1} due to C—O absorption, 950–1033 cm^{-1} due to C—O—C stretchings, 820–850 cm^{-1} due to out-of-plane C—H bendings, and 3079 cm^{-1} due to C—H stretchings of the benzene ring as listed in literature.[7,13,15,19,20,28,30]

^{13}C-NMR proton-decoupled spectra of **P1** and **P2** are displayed in Figures 6(a) and 6(b), respectively. Five basic structures could be drawn for each polymer, and the theoretical ^{13}C-NMR chemical-shift data for the main five possible addition products of the polymers were calculated from the related correlation tables given in the literature.[39] The binary relations between the observed and calculated data for polymers **P1** and **P2** are displayed in Figures 7 and 8, respectively. The observed and calculated shift data of **P1** indicated that the structures shown in Figure 7(b,c,d) were possible. The ^{1}H-NMR spectrum [Fig. 9(a)] of **P1** was characterized by a peak at $\delta = 7.2$–7.4 ppm due to the protons of 2,4-dichloro-1,6-phenylene oxide units (1,2 addition) and a peak at $\delta = 6.8$–6.9 ppm due to the protons of 2,6-dichloro-1,4-phenylene oxide units (1,4 addition). The broader peaks at the higher field in Figure 9(a) indicated the presence of both 1,2 and 1,4 addition at the same monomeric unit, indicating that 1,2 and 1,4 addition were taking place

at about equal rates, leading to a branched structure. The observed and calculated shift data of **P2** indicated that both structures were possible, as shown in Figure 8(a,b). The ^{1}H-NMR spectrum, as shown in Figure 9(b), revealed that the peaks at $\delta = 7.8$ and 6.9–7.1 ppm were due to 1,2-(2,4-dibromo-1,6-phenylene oxide units) and 1,4-(2,6-dibromo-1,4-phenylene oxide units), respectively, suggesting that 1,4 addition was predominant over 1,2 addition. The structures of the polymers obtained from the thermal decomposition of the same complexes in solid state[30] were different from those in solution. Both **P1** and **P2**, obtained by solid-state polymerization,[30] led to a branched structure (1,2 addition predominant over 1,4 addition). The T_g's of **P1** and **P2** were 165.48 and 183.35°C (Table VI), respectively, indicating high rigidity.

CONCLUSIONS

Bis(2,4,6-trichlorophenolato)di(pyridine) nickel(II) and bis(2,4,6-tribromophenolato)di(pyridine) nickel(II) dihydrate complexes can be thermally polymerized in the solution in the presence of iodine. The poly(dihalophenylene oxide)s synthesized from the thermal decomposition of these complexes had lower yields and lower $[\eta]$ values compared to the polymers obtained from the decomposition of complexes of tetrahedral cobalt(II) in solid state,[19] octahedral copper(II),[40] and square pyramidal and square planar nickel(II) in solid state or in solution.[30,31] The percentage yield and $[\eta]$ values of polymers slightly increased with polymerization time and temperature at 10% I_2 content. A sharp increase in percentage yield was observed with an increase in the amount of added iodine, which corresponded to the range of the same or higher molar ratio with the phenol in the complexes. The amount of added iodine also increased $[\eta]$ values of the polymers. For complex **1**, the

TABLE VI
T_g **Values of the Two Poly(dihalophenylene oxide)s**

Polymer	T_g (°C)
P1	165
P2	183

polymerization proceeded through 1,2 and 1,4 addition at about equal rates, whereas for complex **2**, 1,4 addition was predominant. All polymers were highly rigid. The optimum conditions to obtain the highest percentage yield from the decomposition of these complexes in solution was at 60°C, 3 h of polymerization time, and 200% I_2 for complex **1** and 90°C, 3 h of polymerization time, and 200% I_2 for complex **2**.

References

1. Steinbuch, I. R. T. Kunstst Plast (Munich) 1965, 3, 3.
2. Blanchard, H. S.; Finkbeiner, H. U.S. Pat. 3,256,243 (1996).
3. Hunter, W. H.; Olson, A. O.; Daniels, E. A. J Am Chem Soc 1916, 38, 1761.
4. Hunter, W. H.; Dahlen, M. A. J Am Chem Soc 1932, 84, 2459.
5. Hunter, W. H.; Joyce, F. E. J Am Chem Soc 1917, 39, 2640.
6. Blanchard, H. S.; Finkbeiner, H.; Russel, G. A. J Polym Sci 1962, 58, 469.
7. Harrod, J. F. Can J Chem 1969, 47(4), 637.
8. Carr, B. G.; Harrod, J. F.; Van Gheluwe, P. Macromolecules 1973, 6, 498.
9. Carr, B. G.; Harrod, J. F. J Am Chem Soc 1973, 95, 5707.
10. Harrod, J. F.; Van Gheluwe, P.; Kısakürek, D.; Shaver, A. Macromolecules 1981, 14, 565.
11. Harrod, J. F.; Shaver, A. Macromolecules 1982, 95, 676.
12. Ünal, H.; Şanlı, O.; Kısakürek, D. Polymer 1989, 30, 344.
13. Kısakürek, D.; Şanlı, O. Makromol Chem 1989, 190, 1843.
14. Kısakürek, D.; Aras, L.; Şanlı, O. METU J Pure Appl Sci 1990, 23(2), 49.
15. Şanlı, O.; Kısakürek, D. Makromol Chem 1992, 193, 619.
16. Şanlı, O.; Pulat, M.; Kısakürek, D. Pol J Chem 1994, 68, 453.
17. Kısakürek, D.; Akbaş, M.; Bilir, N. Makromol Chem Phys 1998, 199, 169.
18. Kısakürek, D. Polymeric Materials Encyclopedia; CRC: New York, 1996; Vol. 8, p 5731.
19. Baştürkmen, M.; Kısakürek, D. Polymer 1993, 34, 625.
20. Şanlı, O.; Pulat, M.; Kısakürek, D. Eur Polym J 1995, 31, 1255.
21. Baştürkmen, M. Ph.D. Thesis, Middle East Technical University, 1997.
22. Türker, L.; Kısakürek, D.; Şen, Ş.; Toppare, L.; Akbulut, U. J Polym Sci Part B: Polym Phys 1988, 26, 2485.
23. Saçak, M.; Akbulut, U.; Kısakürek, D.; Türker, L.; Toppare, L. J Polym Sci Part A: Polym Chem 1989, 27, 1599.
24. Yiğit, S.; Kısakürek, D.; Türker, L.; Toppare, L.; Akbulut, U. Polymer 1989, 30, 348.
25. Kısakürek, D.; Yiğit, S. Eur Polym J 1991, 27, 955.
26. Pulat, M.; Önal, A. M.; Kısakürek, D. New Polym Mat 1994, 4(2), 111.
27. Aras, L.; Şen, Ş.; Kısakürek, D. Polymer 1995, 36, 3015.
28. Baştürkmen, M.; İşçi, H.; Kısakürek, D. Polym Int J 1993, 30, 387.
29. Kesici, N. M.S. Thesis, Middle East Technical University, 1997.
30. Kısakürek, D.; Aslan, A.; İşçi, H. Polymer 2000, 41, 5149.
31. Karabey, L. M.S. Thesis, Middle East Technical University, 1998.
32. Aydemir, K. M.S. Thesis, Middle East Technical University, 1999.
33. Şentürk, Ö. M.S. Thesis, Middle East Technical University, 2000.
34. Hunter, W. H.; Seyfried, L. M. J Am Chem Soc 1921, 43,151.
35. Ladd, M. F. C.; Perrins, D. H. G. Acta Crystallogr B 1980, 36, 2260.
36. Richardson, M. F.; Wulsberg, G.; Marlow, R.; Zayhonni, S.; Mclorhle, D.; Bhadid, K.; Gagliardi, J.; Farris, B. Inorg Chem 1993, 32, 1913.
37. Van Landschoot, R. C.; Van Hest, J. A. M.; Reedijk, J. Inorg Chim Acta 1980, 46, 41.
38. Kısakürek, D.; Binboğa, N.; Harrod, J. F. Polymer 1987, 28, 1767.
39. Boschke, F. L.; Fresenius, W.; Huber, J. F. K.; Pungur, F.; Rectinitz, G. A.; Simon, W.; West, J. Tables of Spectral Data for Structure Determination of Organic Compounds ^{13}C NMR, ^{1}H NMR, IR, MS, UV/VIS; Springer-Verlag: Berlin, 1983.
40. Marengo-Rullan, J. R.; Willett, R. D. Acta Crystallogr C 1986, 42, 1487.