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Synthesis of poly(ethyl vinyl ethers) containing two dipolar electronic systems and their properties

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Summary

5-Nitro-2-(2'-vinyloxyethoxy)benzylidenemalononitrile (**2a**), methyl 5-nitro-2-(2'-vinyloxyethoxy)benzylidenecyanoacetate (**2b**), 3-nitro-4-(2'-vinyloxyethoxy)benzylidenemalononitrile (**4a**), and methyl 3-nitro-4-(2'-vinyloxyethoxy)benzylidenecyanoacetate (**4b**) were prepared by the condensation of 5-nitro-2-(2'-vinyloxyethoxy)benzaldehyde (**1**) and 3-nitro-4-(2'-vinyloxyethoxy)benzaldehyde (**3**) with malononitrile or methyl cyanoacetate, respectively. Vinyl ether monomers **2a-b** and **4a-b** were polymerized with boron trifluoride etherate as a cationic initiator to yield poly(vinyl ethers) **5-6** having nitrooxybenzylidenemalononitrile and nitrooxycyanocinnamate, which is effective chromophore for second-order nonlinear optical applications. Polymers **5-6** were soluble in common organic solvents such as acetone and DMSO. T_g values of the resulting polymer were in the range of 70-81°C. Electrooptic coefficient (r_{33}) of the poled polymer films were in the range of 19-27 pm/V, which was improved by introducing of nitro group. Polymers **5-6** showed a thermal stability up to 300øC in TGA thermograms, which is acceptable for NLO device applications.

Introduction

Functional materials of nonlinear optical (NLO) activity based on organic compounds have long been extensively studied because of their potential applications in the field of telecommunications, optical signal processing, optical switching, etc (1-6). It is well known that organic and polymeric materials with highly dipolar electronic systems exhibit large NLO properties. The organic materials seem to be superior because of their higher nonlinear optical activity and faster response time than the inorganic ones. Among the organic materials the NLO polymers are receiving great attention, mainly because they offer many advantages such as mechanical endurance, light weight, chemical resistance, and good processability to form electrooptic devices (7). A potential NLO polymer must contain a highly polarizable conjugated dipolar electronic systems and these polymers have to be mechanically very strong and thermally stable with a high T_g. There are tremendous challenges in designing and synthesis of polymers of large NLO effects. Polyesters (8-10), polyurethanes (11), polyamides (12-13), and poly(phenyleneethynylenes) (14) containing the chromophoric main chain were prepared. Various polymers with the NLO-phores in the side chain such as poly(meth)acrylates (15

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-20), and polystyrene (21) were also reported. Recently we have prepared poly(alkyl vinyl ethers) containing *p*-oxybenzylidenemalononitrile or *p*-oxybenzylidenecyanoacetate as a NLO-phore (22). These poly(vinyl ethers) showed a thermal stability up to 300°C with a T_g of 60-70°C, which is too low for electrooptic applications. In this work we prepared four isomeric poly(vinyl ethers) containing nitrooxybenzylidenemalononitrile or nitrooxybenzylidenecyanoacetate, which is presumably effective NLO-chromophore in the side chain. We selected geminally substituted double bond as a NLO-phore because it is easy to synthesize and is more polar than monosubstituted one. We attached two electron-withdrawing nitro- and 1,1-dicyanovinyl- or 1-cyano-1-carbomethoxyvinyl groups onto the aromatic ring in the pendant substituent for enhancing T_g and thermal stability. Electrooptic (EO) coefficient, T_g, and thermal stability of the resulting poly(vinyl ethers) were compared with those of poly(vinyl ethers) without nitro group. All the thermal and EO properties were improved by introduction of nitro group. We now report the results of the initial phase of the work.

Experimental

Materials. The reagent grade chemicals were purchased from Aldrich and purified by either distillation or recrystallization before use. 2-Chloroethyl vinyl ether was distilled under vacuum. Malononitrile was recrystallized from water and distilled from phosphorus pentoxide. *N,N*-Dimethylformamide (DMF) was purified by drying with anhydrous calcium sulfate, followed by distillation under reduced pressure. 2-Iodoethyl vinyl ether was prepared according to the procedure previously described (22).

Measurements. IR spectra were taken on a Shimadzu FT IR-8201PC infrared spectrophotometer. ^{1}H NMR spectra were obtained on a Varian 300MHz NMR spectrometer. Elemental analyses were performed using a Perkin-Elmer 2400 CHN elemental analyzer. The glass transition temperatures (T_g) were measured on a DuPont 910 differential scanning calorimeter in a nitrogen atmosphere. DuPont 951 thermogravimetric analyzer with a heating rate of 10°C/min up to 700°C was used for the thermal degradation study of polymers under nitrogen. The electrooptic coefficient (r_{33}) of the corona poled film was measured by simple reflection technique. Melting points were measured in Buchi 530 melting point apparatus and are corrected. Viscosity values were obtained by using a Cannon-Fenske viscometer.

5-Nitro-2-(2'-vinyloxyethoxy)benzaldehyde **(1)**.

2-Hydroxy-5-nitrobenzaldehyde (16.7 g, 0.10 mol), anhydrous potassium carbonate (18.0 g, 0.13 mol), and 2-iodoethyl vinyl ether (25.7 g, 0.13 mol) were dissolved in 180 mL of dry DMF under nitrogen. The mixture was refluxed in an oil bath at 80°C for 10 h under nitrogen. The resulting solution was cooled to room temperature, diluted with 220 mL of water, and extracted with 300 mL of diethyl ether three times. The organic layer was washed with saturated aqueous sodium chloride solution, and dried with anhydrous magnesium sulfate. Rotary evaporation of diethyl ether gave crude product, which on vacuum distillation yielded 19.2 g (81% yield) of pure product **1**. Mp: 37-38 °C. ^{1}H NMR ($CDCl_3$) δ 4.10-4.48 (m, 6H, CH_2=, -O-CH_2-CH_2-O-), 6.47-6.54 (q, 1H, =CH-O-), 7.13-7.16 (m, 1H, aromatic), 8.40-8.44 (m, 1H, aromatic), 10.47 (s, 1H, -CHO). IR (neat) 3114, 3070 (=C-H), 2962, 2939, 2882 (C-H), 1684 (C=O), 1611, 1572 (C=C), 1510, 1348 (N=O) cm^{-1}.

5-Nitro-2-(2'-vinyloxyethoxy)benzylidenemalononitrile **(2a)**.

Piperidine (0.13 g, 1.5 mmol) was added to a solution of 5-nitro-2-(2'-vinyloxyethoxy)-

benzaldehyde **1** (6.64 g, 28 mmol) and malononitrile (1.98 g, 30 mmol) in 50 mL of *n*-butanol with stirring at 0°C under nitrogen. After stirring for 1 h at 0°C, the product was filtered and washed successively with cold n-butanol (80 mL), water (30 mL), and cold *n*-butanol (20 mL). The obtained pale yellow product was recrystallized from *iso*propyl alcohol to give 6.87 g (86% yield) of **2a**. Mp: 90-92 °C. ^{1}H NMR ($CDCl_3$) δ 4.13-4.47 (m, 6H, CH_2=, -O-CH_2-CH_2-O-), 6.47-6.54 (q, 1H, =CH-O-), 7.14-7.17 (d, 1H, aromatic), 8.22 (s, 1H, aromatic), 8.43-8.47 (m, 1H, aromatic), 9.04-9.05 (d, 1H, aromatic). IR (KBr) 3121, 3038 (=C-H), 2930 (C-H), 2237 (CN), 1628, 1611, 1585 (C=C), 1518, 1350 (N=O) cm^{-1}. Anal. Calcd for $C_{14}H_{11}N_3O_4$: C, 58.95; H, 3.88; N, 14.73. Found: C, 58.87; H, 3.82; N, 14.68.

Methyl 5-nitro-2-(2'-vinyloxyethoxy)benzylidenecyanoacetate **(2b)**.

Piperidine (0.17 g, 2.0 mmol) was added to a solution of 5-nitro-2-(2'-vinyloxyethoxy)-benzaldehyde **1** (7.11 g, 30 mmol) and methyl cyanoacetate (2.98 g, 30 mmol) in 45 mL of *n*-butanol with stirring at 0°C under nitrogen. The resulting solution was stirred for 1 h at 0°C and 10 h at room temperature. After cooling in an ice bath, the product was filtered and washed successively with cold *n*-butanol (30 mL), water (50 mL), and cold *n*-butanol (20 mL). The obtained pale yellow product was recrystallized from *iso*propyl alcohol to give 8.59 g (90% yield) of **2b**. Mp: 109-110 °C. ^{1}H NMR ($CDCl_3$) δ 3.95 (s, 3H, -OCH3), 4.10-4.45 (m, 6H, CH_2=, -O-CH_2-CH_2-O-), 6.46-6.53 (q, 1H, =CH-O-), 7.10-7.13 (d, 1H, aromatic), 8.37-8.41 (q, 1H, aromatic), 8.63 (s, 1H, aromatic), 9.07-9.09 (d, 1H, aromatic). IR (KBr) 3121, 3121, 3074, 3057 (=C-H), 2963, 2932 (C-H), 2230 (CN), 1720 (C=O), 1628, 1609 (C=C), 1518, 1317 (N=O) cm^{-1}. Anal. Calcd for $C_{15}H_{14}N_2O_6$: C, 56.61; H, 4.43; N, 8.80. Found: C, 56.53; H, 4.48; N, 8.87.

3-nitro-4-(2'-vinyloxyethoxy)benzaldehyde **(3)**.

4-Hydroxy-3-nitrobenzaldehyde (16.7 g, 0.10 mol), anhydrous potassium carbonate (18.0 g, 0.13 mol), and 2-iodoethyl vinyl ether (25.7 g, 0.13 mol) were dissolved in 180 mL of dry DMF under nitrogen. The mixture was refluxed in an oil bath at 80°C for 10 h under nitrogen. The resulting solution was cooled to room temperature, diluted with 220 mL of water, and extracted with 300 mL of diethyl ether three times. The organic layer was washed with saturated aqueous sodium chloride solution, and dried with anhydrous magnesium sulfate. Rotary evaporation of diethyl ether gave crude product, which on vacuum distillation yielded 19.4 g (82% yield) of pure product 3. Mp: 75-76 °C. ^{1}H NMR ($CDCl_3$) δ 4.08-4.47 (m, 6H, CH_2=, -O-CH_2-CH_2-O-), 6.46-6.53 (q, 1H, =CH-O-), 7.28 (s, H, aromatic), 8.35 (s, 1H, aromatic), 9.94 (s, 1H, -CHO). IR (neat) 3074 (=C-H), 2936, 2882 (C-H), 1684 (C=O), 1611, 1589 (C=C), 1522, 1346 (N=O) cm^{-1}.

3-Nitro-4-(2'-vinyloxyethoxy)benzylidenemalononitrile **(4a)**.

Piperidine (0.13 g, 1.5 mmol) was added to a solution of 3-nitro-4-(2'-vinyloxyethoxy)-benzaldehyde **3** (6.64 g, 28 mmol) and malononitrile (1.98 g, 30 mmol) in 50 mL of *n*-butanol with stirring at 0°C under nitrogen. After stirring for 1 h at 0°C, the product was filtered and washed successively with cold *n*-butanol (80 mL), water (30 mL), and cold *n*-butanol (20 mL). The obtained pale yellow product was recrystallized from *iso*propyl alcohol to give 6.63 g (83% yield) of **4a**. Mp: 100-102 °C. ^{1}H NMR ($CDCl_3$) δ 4.09-4.49 (m, 6H, CH_2=, -O-CH_2-CH_2-O-), 6.45-6.52 (q, 1H, =CH-O-), 7.28-7.31 (d, 1H, aromatic), 7.69 (s, 1H, aromatic), 8.24-8.28 (m, 2H, aromatic). IR (KBr) 3094, 3034 (=C-H), 2932, 2880 (C-H), 2232 (CN), 1616, 1585 (C=C), 1529, 1358 (N=O) cm^{-1}. Anal. Calcd for $C_{14}H_{11}N_3O_4$: C, 58.95; H, 3.88; N, 14.73. Found: C, 58.86; H, 3.84; N, 14.78.

Methyl 3-nitro-4-(2'-vinyloxyethoxy)benzylidenecyanoacetate **(4b)**.

Piperidine (0.17 g, 2.0 mmol) was added to a solution of 3-nitro-4-(2'-vinyloxyethoxy)-benzaldehyde **3** (7.11 g, 30 mmol) and methyl cyanoacetate (2.98 g, 30 mmol) in 45 mL of *n*-butanol with stirring at 0°C under nitrogen. The resulting solution was stirred for 1 h at 0°C and 10 h at room temperature. After cooling in an ice bath, the product was filtered and washed successively with cold n-butanol (30 mL), water (50 mL), and cold n-butanol (20 mL). The obtained pale yellow product was recrystallized from *iso*propyl alcohol to give 8.79 g (92% yield) of **4b**. Mp: 117-118 °C. ^{1}H NMR ($CDCl_3$) δ 3.94 (s, 3H, $-OCH_3$), 4.08-4.47 (m, 6H, $CH_2=$, $-O-CH_2-CH_2-O-$), 6.45-6.52 (q, 1H, =CH-O-), 7.22-7.30 (m, 1H, aromatic), 8.15 (s, 1H, aromatic), 8.31-8.38 (m, 2H, aromatic). IR (KBr) 3122, 3076, 3041 (=C-H), 2953, 2882 (C-H), 2226 (CN), 1717 (C=O), 1628, 1614, 1599 (C=C), 1520, 1352 (N=O) cm^{-1}. Anal. Calcd for $C_{15}H_{14}N_2O_6$: C, 56.61; H, 4.43; N, 8.80. Found: C, 56.56; H, 4.49; N, 8.86.

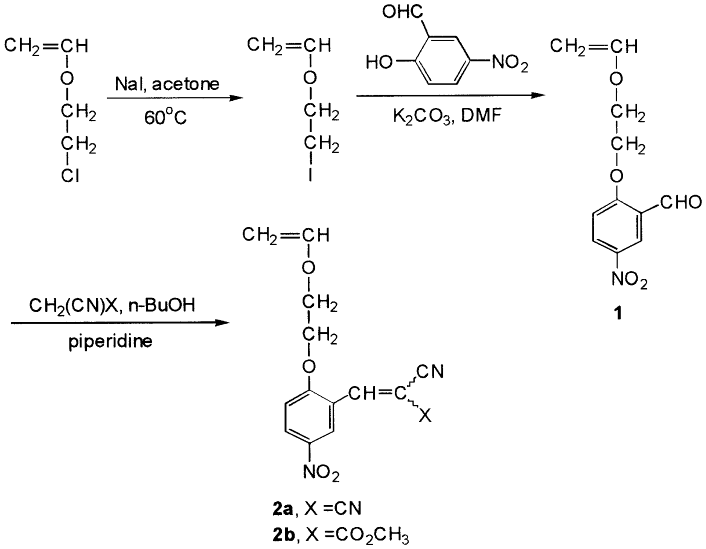
Cationic polymerization of monomers **2** *and* **4**

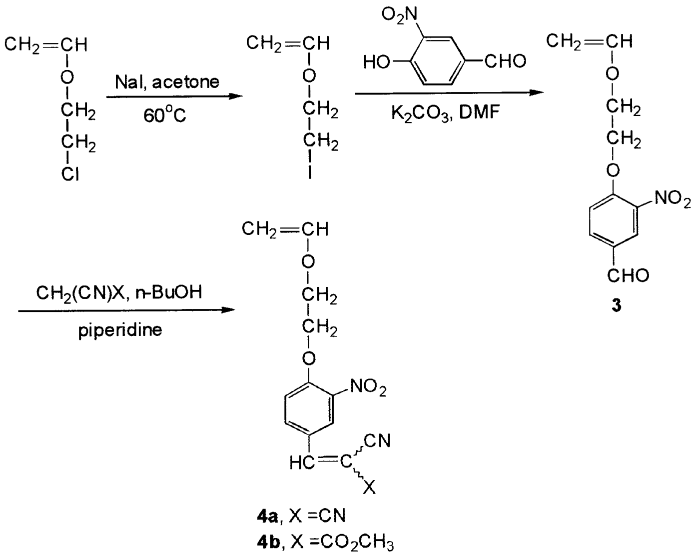
A representative cationic polymerization procedure (the case of **2a**) was as follows: A solution of **2a** (0.86 g, 3.0 mmol) in dichloromethane (3.0 mL) was placed in a rubber septum stopper capped glass ampule under nitrogen. The resulting solution was flushed with nitrogen for 20 min. The ampule was then placed in an ethanol-acetone bath kept at -60 °C under nitrogen, and 0.0034 mL (0.03 mmol) of boron trifluoride etherate was added to the solution. After 14 h the ampule was taken out and the polymerization mixture was poured into 500 mL of methanol. The precipitated polymer was collected and reprecipitated from acetone into methanol to give 0.74 g (86% yield) of polymer **5a**; η_{inh} = 0.26 dL/g (*c* 0.5 g/dL in acetone at 25 °C). **5a**: ^{1}H NMR (acetone-d_6) δ 1.50-2.14 (m, 2H, $-CH_2-$), 3.67-4.16 (m, 3H, $-CH_2-O-CH-$), 4.32-4.60 (m, 2H, $-Ph-O-CH_2-$), 7.23-7.54 (m, 1H, aromatic), 8.28-8.57 (m, 2H, aromatic), 8.82-9.02 (m, 1H, aromatic). IR (KBr) 3049 (=C-H), 2934, 2874 (C-H), 2231 (CN), 1612, 1593 (C=C), 1522, 1346 (N=O) cm^{-1}. Anal. Calcd for $(C_{14}H_{11}N_3O_4)_n$: C, 58.95; H, 3.88; N, 14.73. Found: C, 58.88; H, 3.94; N, 14.81. **5b**: ^{1}H NMR (acetone-d_6) δ 1.51-2.15 (m, 2H, $-CH_2-$), 3.92 (s, 3H, $-OCH_3$), 3.67-4.16 (m, 3H, $-CH_2-O-CH-$), 4.32-4.57 (m, 2H, $-Ph-O-CH_2-$), 7.26-7.52 (m, 1H, aromatic), 8.26-8.72 (m, 2H, aromatic), 8.96-9.14 (m, 1H, aromatic). IR (KBr) 3050 (=C-H), 2955, 2876 (C-H), 2226 (CN), 1736 (C=O), 1611, 1582 (C=C), 1522, 1346 (N=O) cm^{-1}. Anal. Calcd for $(C_{15}H_{14}N_2O_6)_n$: C, 56.61; H, 4.43; N, 8.80. Found: C, 56.70; H, 4.49; N, 8.85. **6a**: ^{1}H NMR (acetone-d_6) δ 1.50-2.08 (m, 2H, $-CH_2-$), 3.67-4.09 (m, 3H, $-CH_2-O-CH-$), 4.35-4.58 (m, 2H, $-Ph-O-CH_2-$), 7.40-7.61 (m, 1H, aromatic), 8.14-8.38 (m, 2H, aromatic), 8.39-8.55 (m, 1H, aromatic). IR (KBr) 3040 (=C-H), 2934, 2878 (C-H), 2230 (CN), 1616, 1591 (C=C), 1533, 1356 (N-O) cm^{-1}. Anal. Calcd for $(C_{14}H_{11}N_3O_4)_n$: C, 58.95; H, 3.88; N, 14.73. Found: C, 58.90; H, 3.93; N, 14.79. **6b**: ^{1}H NMR (acetone-d_6) δ 1.45-2.08 (m, 2H, $-CH_2-$), 3.89 (s, 3H, $-CH_3$), 3.60-4.06 (m, 3H, $-CH_2-O-CH-$), 4.32-4.56 (m, 2H, $-Ph-O-CH_2-$), 7.32-7.59 (m, 1H, aromatic), 8.12-8.40 (m, 2H, aromatic), 8.40-8.59 (m, 1H, aromatic). IR (KBr) 3040 (=C-H), 2934, 2876 (C-H), 2224 (CN), 1732 (C=O), 1603 (C=C), 1535, 1354 (N=O) cm^{-1}. Anal. Calcd for $(C_{15}H_{14}N_2O_6)_n$: C, 56.61; H, 4.43; N, 8.80. Found: C, 56.56; H, 4.48; N, 8.86.

Results and Discussion

Syntheses of monomers **2a-b** and **4a-b**

5-Nitro-2-(2'-vinyloxyethoxy)benzaldehyde (1) and 3-nitro-4-(2'-vinyloxyethoxy)benzaldehyde (**3**) were prepared by reaction of 2-iodoethyl vinyl ether with the corresponding 2-hydroxy-5-nitrobenzaldehyde and 4-hydroxy-3-nitrobenzaldehyde. 5-Nitro-2-(2'-vinyloxyethoxy)ben-zylidenemalononitrile (**2a**), methyl 5-nitro-2-(2'-vinyloxyethoxy)benzylidenecyanoacetate (**2b**), 3-nitro-4-(2'-vinyloxyethoxy)benzylidenemalononitrile (**4a**), and methyl 3-nitro-4-(2'-vinyloxyethoxy)benzylidenecyanoacetate (**4b**) were prepared by the condensations of **1** and **3** with malononitrile or methyl cyanoacetate, respectively (23). Compounds **2** and **4** were obtained in high yield and were purified by recrystallization in *iso*propyl alcohol for polymerization. The chemical structure of the compounds was identified by ^{1}H NMR, IR spectra, and elemental analysis. Monomers **2-b** and **4a-b** show olefinic protons at δ= 6.45-6.55 (q, 1H, vinylic) in their ^{1}H NMR spectra, respectively. The same monomers showed strong absorption bands at 2226-2237, 1717-1743, 1585-1628, and 1512-1529 cm-1 indicating the presence of nitrile, carbonyl,

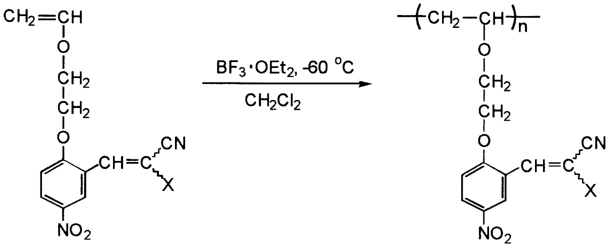




olefin, and nitro groups, respectively. Spectral data indicated that compounds **2b** and **4b** were a mixtures of the cis- and trans-isomers.

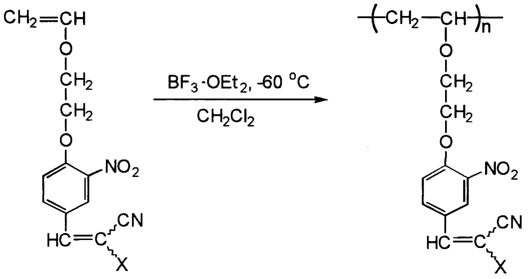
Cationic polymerization of monomers **2a-b** and **4a-b**

Isomeric vinyl ether monomers **2a-b** and **4a-b** were polymerized in solution at -60°C with boron trifluoride etherate as cationic initiator to yield the polymers **5a-b** and **6a-b**. The polymerization results are summarized in Table 1. Monomers **2a-b** and **4a-b** were readily polymerized to yield the polymers **5a-b** and **6a-b** in high yield. The chemical



2a, X =CN
2b, X =CO_2CH_3

5a, X =CN
5b, X =CO_2CH_3



4a, X =CN
4b, X =CO_2CH_3

6a, X =CN
6b, X =CO_2CH_3

Table 1. Cationic polymerization of **2** and **4** under various conditions

Monomer	Monomer/Solvent[a] (mol/1 L)	Initiator to Monomer (mol%)	Temp (°C)	Time (h)	Yield (%)	η_{inh}[b] (dL/g)
2a	1.0	1.0	-60	14	86	0.26
2a	0.8	0.8	-30	15	84	0.28
2b	1.0	1.0	-60	16	78	0.25
2b	0.8	0.8	-30	15	75	0.24
4a	1.0	1.0	-60	15	71	0.20
4a	0.8	0.8	-30	15	68	0.25
4b	1.0	1.0	-60	19	75	0.24
4b	0.8	0.8	-30	15	72	0.23

[a] Solvent: Dichloromethane.

[b] Inherent viscosity of polymer: Concentration of 0.5 g/dL in acetone at 25°C.

Table 2. Thermal and electrooptic properties of polymers **5-6**

Polymer	T_g, °C	Degradation temp, °C			Residue at 700 °C, %	r_{33}[a]
		5%-loss	20%-loss	40%-loss		
5a	75	292	349	523	1.7	22
5b	70	308	318	356	14.0	19
6a	81	289	343	543	3.3	27
6b	72	299	308	342	6.7	23
P(*o*-VEBM)[b]	59	307	350	389	21.4	13
P(*o*-VEBCA)[b]	60	314	351	370	9.3	11
P(*p*-VEBM)[b]	70	254	316	365	29.5	16
P(*p*-VEBCA)[b]	61	330	367	394	27.4	14

[a] EO coefficients were measured by simple reflection technique.

[b] **P(*o*-VEBM)** = Poly[*o*-(2-Vinyloxyethoxy)benzylidenemalononitrile];
P(*o*-VEBCA) = Poly[Methyl *o*-(2-Vinyloxyethoxy)benzylidenecyanoacetate];
P(*p*-VEBM) = Poly[*p*-(2-Vinyloxyethoxy)benzylidenemalononitrile];
P(*p*-VEBCA) = Poly[Methyl *p*-(2-Vinyloxyethoxy)benzylidenecyanoacetate].

structure of the polymers were confirmed by ^{1}H NMR, IR spectra, and elemental analyses. Spectral data indicated that the terminal olefin group did not participate in the vinyl ether polymerization.

Properties of polymers

The resulting polymers **5a-b** and **6a-b** were soluble in common solvents such as acetone, chloroform, dichloromethane, DMF and DMSO, but were not soluble in methanol and diethyl ether. The inherent viscosities of polymers were in the range of 0.20-0.30 dL/g. The thermal behavior of the polymers were investigated by thermogravimetric analysis (TGA) and differential scanning calorimeter (DSC) to determine the thermal degradation pattern and glass transition temperature (T_g). The results are summarized in Table 2. The polymers showed a double phase degradation pattern and a thermal stability up to 300°C in TGA thermograms. The glass transition temperature (T_g) of the polymers measured by DSC thermograms were around 70-81°C. These T_g values are higher than those for poly(ethyl vinyl ethers) containing *o*- or *p*-oxybenzylidenemalononitrile and oxybenzylidenecyanoacetate as a NLO-phore (60-70°C), probably due to the presence of polar nitro group. The electrooptic coefficient (r_{33}) of the corona poled polymer films **5-6** measured by simple reflection technique was in the range of 19-27 pm/V, which was improved by introducing of nitro group.

Acknowledgements

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