Synthesis and Thermal Behavior of Some Anthracene-Based Copolymers Obtained by Diels–Alder Cycloaddition Reactions

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ABSTRACT: New polymer structures have been synthesized via Diels–Alder cycloaddition of bisdiene compounds bearing two anthracene groups and different bisdienophiles, all containing bismaleimide or biscitraconimide functions. The monomers and polymers were characterized by FTIR, UV, and ^{1}H NMR techniques and compared with two models having a cycloadduct structure. The polymers were studied by thermogravimetric analyses. © 2007 Wiley Periodicals, Inc. J Appl Polym Sci 107: 846–853, 2008

Key words: bisanthracene compounds; bismaleimides; biscitraconimides; Diels–Alder copolymerization; thermal behavior

INTRODUCTION

Many reactions, very much known in organic chemistry, can be successfully extended in macromolecular chemistry to obtain new polymers with particular architectures and properties. Such an example is the Diels–Alder reaction or ''diene synthesis,'' where an electrophilically activated double or triple bond adds 1,4 to a conjugated diene so that the product is always a six-membered ring. This reaction is one of the most used reaction in organic chemistry to obtain mono-, bi-, and polycyclic compounds with bridge, and in the last years, it was extended to synthesis of the polymers.[1]

The Diels–Alder reaction involves a 2 + 4 cycloaddition between a diene and a dienophile compound. The application of Diels–Alder chemistry to polymer synthesis,[2–13] modification[14–17] and (reversible) cross-linking,[18–23] or polyphenylene and ribbon-like (two-dimensional) conducting polymers[24] has been reported. However, the lack of success in using at large scale of this method is limited by thermal reversibility of the cycloaddition and low molecular weight of the products.

The synthesis of polymers by Diels–Alder cycloaddition can be performed by a self-polymerization reaction (when both complementary functions are situated in the same molecule, an AB-type monomer) or by an intermolecular reaction (when bisdienes and bisdienophiles are used as comonomers, A-A and B-B comonomers). Anthracene and its derivatives are well known as fairly reactive dienes that easily participate to the Diels–Alder cycloaddition reactions with different dienophiles.[25] The self-polymerization of anthracene-based monomers by Diels–Alder cycloadditions can take place if the double or triple bond, electrophilically activated by presence of one or two carbonyl groups, i.e., acrylate, methacrylate, maleimide, citraconimide, propiolate, etc, is bounded at anthracene ring.[26–33] In some cases, this reaction can be intramolecular one, depending on length of the spacer connecting the diene and dienophile, when a wide variety of 9,12-bridged ethano- and etheno anthracenes have been obtained by cyclization of 9-substituted anthracenes.[26] The self-polymerization of A-B anthracene monomers by Diels–Alder cycloadditions has been reported for the first time by Simionescu and coworkers.[27–32] The following A-B type monomers have been studied in self-polymerization reactions: 1- and 2-anthryl maleimide,[29,33] 1-anthryl citraconimide,[31] 1-anthryl and 2-anthryl itaconimide,[32] and 9-anthrylmethyl methacrylate, 9-anthrylmethyl acrylate, 9-anthrylethyl acrylate and methacrylate,[27,28] and 9-anthrylmethyl propiolate.[30]

The first use of bisanthracene compounds in Diels–Alder copolymerization was reported by Meek et al.,[34–36] using bismaleimide compounds. Meador and coworkers,[37–40] has used an effective bisdienophile diepoxyanthracene in reaction with anthracene-end capped polyimide oligomers to obtain matrix resins for high-temperature applications. The polymers obtained have high softening points and good thermal oxidative stabilities, making them applicable for high-temperature composites.

TABLE I
Some Characteristics of Obtained Polymers

No.	Polymer	M_n (g mol^{-1})	Polydispersity	Yield (%)	IR spectra (cm^{-1})
1	**7**	8260	1.17	65.0	3400, 1705, 1660, 1500, 1395, 1245, 1155, 1100, 775, 455
2	**8**	10950	1.32	61.0	3400, 1710, 1660, 1500, 1395, 1245, 1160, 840, 700, 510
3	**9**	–	–	67.0	3450, 1705, 1660, 1500, 1250, 1050, 730, 500
4	**10**	–	–	63.0	3400, 1705, 1660, 1500, 1395, 1145, 1050, 840, 740, 550
5	**11**	6390	1.21	58.1	3450–3300, 3050, 2950, 1705, 1660, 1500, 1395, 1195, 1050, 740, 500
6	**12**	7850	1.24	61.1	3400, 3060, 2910, 1700, 1660, 1500, 1395, 1240, 1150, 740, 500
7	**13**	–	–	48.5	3450, 1705, 1660, 1500, 1195, 1050, 750, 500
8	**14**	3480	1.46	69.0	3400, 1705, 1660, 1550, 1445, 1395, 1290, 1250, 1145, 1050, 740, 595, 550

The present work is an extension of our previous interest on synthesis by Diels–Alder polycycloaddition reactions[27–32] of new polymer architectures applicable as thermostable coatings and optoelectronic materials using A-A and B-B monomer pairs, such as anthracene-based bisdienes and bismaleimide or biscitraconimide comonomers. A special attention is focused on the thermal stability of the copolymers due to the possibility of cycloadduct-type structures to depolymerize by a retro Diels–Alder reaction.

EXPERIMENTAL

Materials

9-Anthrylcarboxaldehyde was synthesized by a known method,[41] while 1,2-diaminoethane, 1,3-diaminopropane, maleic anhydride, citraconic anhydride, and p-toluenesulfonic acid are commercially products (Aldrich) and are used as received. The synthesized monomers were purified by recrystallization. Commercially, solvents (benzene, toluene, and N,N-dimethylformamide) (Fluka, Germany) were dried and purified by standard methods.

Synthesis of the monomers

Synthesis of the bisdiene monomers

Bis[(9-anthryl)-N,N-dimethylene]azometine monomer (**1**) and bis[(9-anthryl)-N,N-three-methylene]azometine monomer (**2**) were synthesized using a reported method[11] by a coupling reaction between 9-anthrylcarboxaldehyde and 1,2-diaminoethane, or 1,3-diaminopropane, respectively. The monomers were twice recrystallized from methanol as yellow needles.
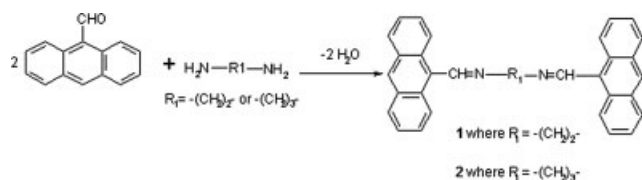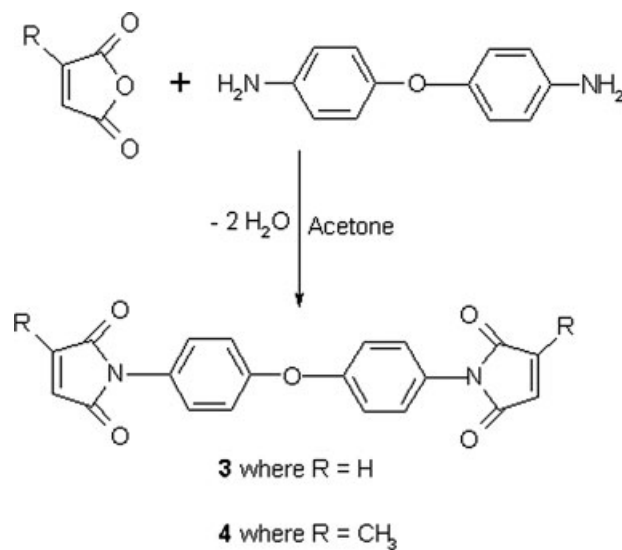

Scheme 1

Monomer (**1**)—yield: 85% and mp = 170–171°C; ^{1}H NMR (CDCl$_3$, ppm): 9.50 (s, 2H, —CH=N—), 8.60–7.35 (m, 18H, aromatic protons), 4.52 (s, 4H, —N—CH$_2$); UV (CHCl$_3$, λ_{max}): 262, 334, 352, 372, 388 nm. Monomer (**2**)—yield: 88% and mp = 139–140°C; ^{1}H NMR (CDCl$_3$, ppm): 9.56 (s, 2H, —CH=N—), 8.70–7.46 (m, 18H, aromatic protons), 4.23 (t, 4H, —N—CH$_2$), 2.57 (q, 2H, —(CH$_2$) —); UV (CHCl$_3$, λ_{max}): 262, 333, 353, 370, and 389 nm.

Synthesis of the bisdienophile monomers

Bismaleimide (**3**) and biscitraconimide (**4**) were synthesized by the reaction of maleic anhydride and citraconic anhydride, respectively, with 4,4'-diaminodiphenylether, according to the method reported by Dix et al.[42]
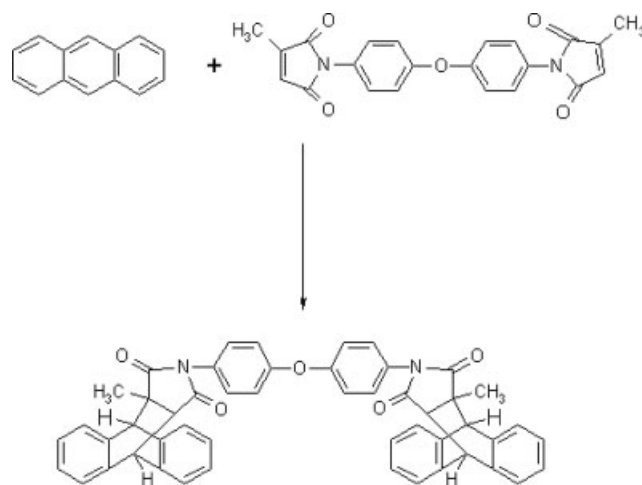
Bismaleimide (**3**) was recrystallized from toluene. Yield 68%, mp 178–183°C. Anal. Calcd for C$_{20}$H$_{12}$N$_2$O$_5$ (%): C, 66.66; H, 3.35; N, 7.77. Found: C, 65.93; H, 3.67; N, 7.38. IR (KBr), cm^{-1}: 1785, 1720 (C=O imide), 1250 (—O—). ^{1}H NMR (DMSO-d_6, ppm): 7.50–7.33 (d, aromatic protons ortho to imide),

3 where R = H

**4** where R = CH$_3$
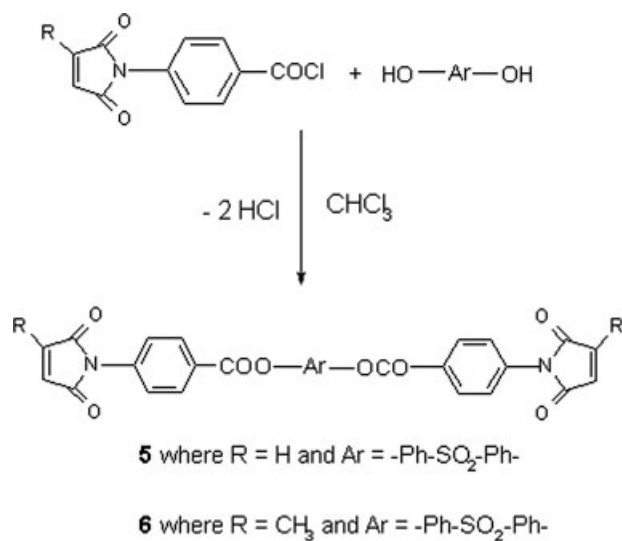
Scheme 2

Scheme 3

Scheme 5

7.22–7.08 (d, aromatic protons ortho to —O—), 6.80–6.72 (m, vinyl protons).

Biscitraconimide (**4**) was recrystallized from dichlorethane/ethanol. Yield 67%; mp 175–178°C; IR (KBr) cm^{-1}: 1780 and 1710 (C=O imide), 1250 (—O—). Anal. Calcd for $C_{22}H_{16}N_2O_5$ (%): C, 68.03; H, 4.15; N, 7.21. Found: C, 68.25; H, 4.61; N, 6.98. ^{1}H NMR (DMSO-d_6, ppm): 7.50–7.33 (d, aromatic protons ortho to imide), 7.22–7.08 (d, aromatic protons ortho to —O—), 6.80–6.72 (m, vinyl protons), 2.11–2.01 (m, aliphatic protons).

Bismaleimide (**5**) and biscitraconimide (**6**) were synthesized according to the method of Hoyt et al.[43]

Bismaleimide (**5**) was recrystallized from dichlorethane/ethanol. Yield 69%; mp 235–240°C. Anal. Calcd for $C_{34}H_{20}N_2O_{10}S$ (%): C, 62.96; H, 3.10; N, 4.31; S, 4.94. Found: C, 62.31; H, 2.92; N, 4.18; S, 5.21. IR (KBr) cm^{-1}: 1785 and 1725 (C=O imide), 1755 (C=O ester), 1330 and 1160 (—SO$_2$—). ^{1}H NMR (DMSO-d_6, ppm): 8.22–8.07 (d, aromatic protons, ortho to —COO), 8.11–8.00 (d, aromatic protons, ortho to —SO$_2$—), 7.63–7.48 (d, aromatic protons, ortho to maleimide and ortho to —O—), 7.16 (s, olefinic protons).

Biscitraconimide (**6**) was recrystallized from dichlorethane/ethanol. Yield 72%; mp 180°C. Anal.
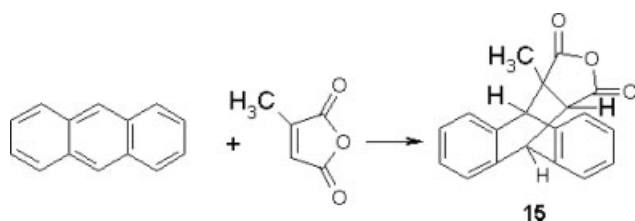
Calcd for $C_{36}H_{24}N_2O_{10}S$ (%): C, 63.90; H, 3.57; N, 4.13; S, 4.73. Found: C, 63.57, H, 3.82; N, 3.92; S, 4.49..IR (KBr) cm^{-1}: 1790 and 1720 (C=O imide), 1755 (C=O ester), 1330 and 1165 (—SO$_2$—). ^{1}H NMR (DMSO-d_6, ppm): 8.56–8.37 (m, aromatic protons, ortho to —SO$_2$—), 8.26–8.15 (m, aromatic protons, ortho to —COO), 8.00–7.65 (m, aromatic protons, ortho to imide and ortho to —O—), 7.09–7.00 (m, olefinic protons) and 2.25–2.00 (m, aliphatic protons).
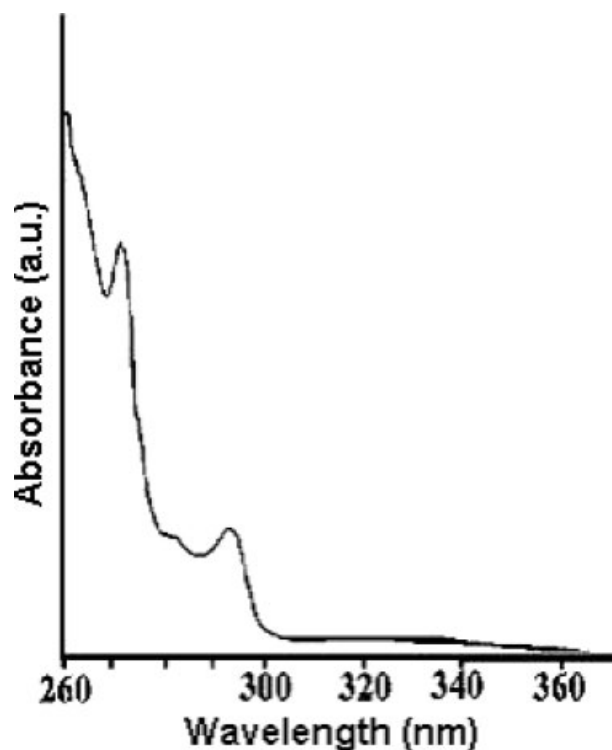

Scheme 4

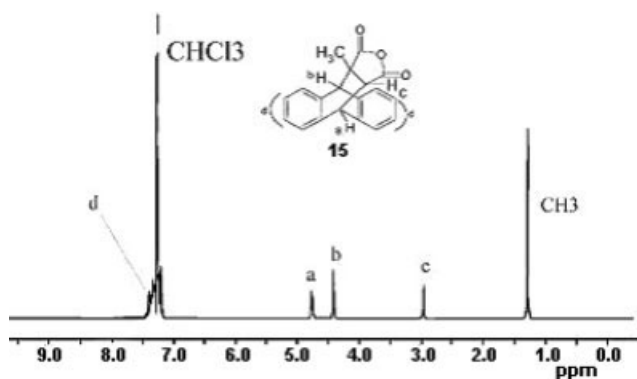**Figure 1** The UV spectra of the model **15**.

Figure 2 The ^{1}H NMR (CDCl$_3$) spectrum of the model **15**.

Diels–Alder polycycloaddition

An equimolar mixture containing 1 mmol bisdiene, 1 mmol bisdienophile, and 20 mL N,N-dimethylformamide was heated at 120°C under a nitrogen atmosphere for 20 h. After cooling at room temperature, the homogeneous dark solution was precipitated in water. The polymer was filtered, dried, and purified twice by reprecipitating in methanol from DMF solution. The yields and some structural characteristics of the synthesized polymers are presented in Table I.

Cycloadduct models synthesis

To elucidate the structure of the polymers, two model compounds (**15**) and (**16**) were synthesized. Compounds were obtained by thermal cycloaddition of citraconic anhydride to anthracene (1 : 1M ratio) and biscitraconimide (**4**) cycloaddition to anthracene (1 : 2M ratio), respectively. The reactions were performed in bulk at 110°C for 24 h and in the presence of traces of hydroquinone. After recrystallization from methanol, pure products were obtained.

(**15**): Yield 79%; mp 158–159°C. (**16**): Yield 82%; mp 181–182°C.

MEASUREMENTS

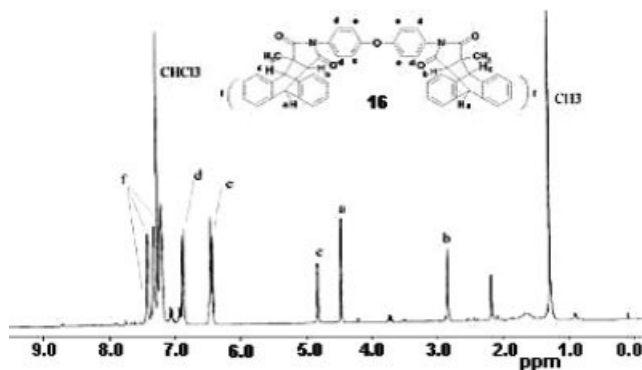FTIR spectra were recorded in KBr pellets on a Digilab-FTS 2000 spectrometer while UV–vis absorption

spectra were obtained on a Specord M42 Carl Zeiss Jena spectrophotometer (Jena, Germany) in chloroform solutions using 10-mm quartz cells. Melting points are uncorrected and were measured with a Boetius microscope (Dresden, Germany). ^{1}H NMR spectra were recorded on a Bruker NMR spectrometer Avance DRX 400 MHz (Rheinstetten, Germany), using CDCl$_3$ or DMSO-d_6 solutions as solvent, at room temperature (TMS as internal standard). The relative molecular weights were determined by gel permeation chromatography using a PL-EMD instrument (England), polystyrene standards for the calibration plot, and N,N'-dimethylformamide as solvent. Thermal gravimetric analysis (TGA) was performed on MOM (Budapest, Hungary) thermobalance apparatus, in air at a heating rate of 12°C/min.

RESULTS AND DISCUSSION

Design, synthesis, and characterization of bisdiene monomers

Two bisdiene monomers (**1**) and (**2**), containing an azomethine linkage as a spacer between anthracene nuclei, were synthesized by coupling reactions between 9-anthrylcarboxaldehyde and 1,2-diaminoethane, or 1,3-diaminopropane, respectively, (Scheme 1).
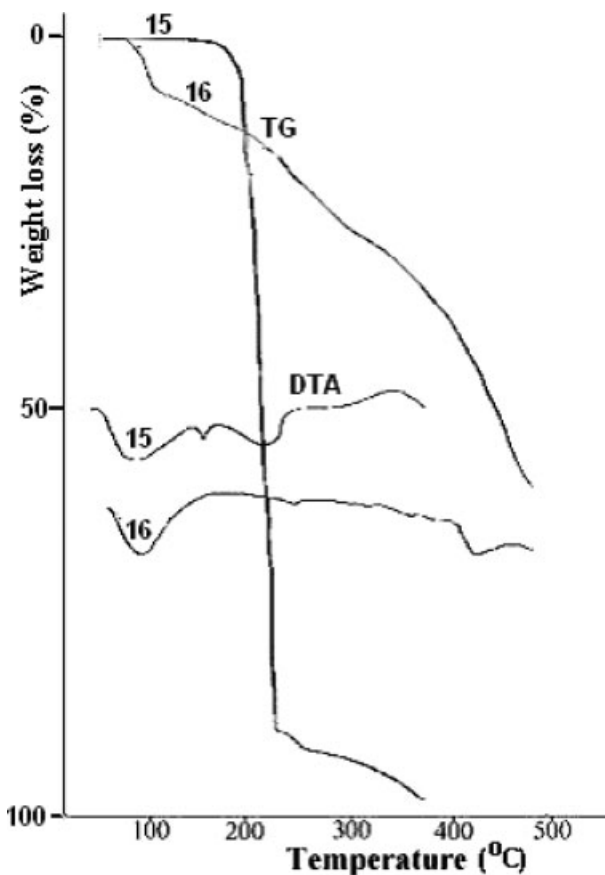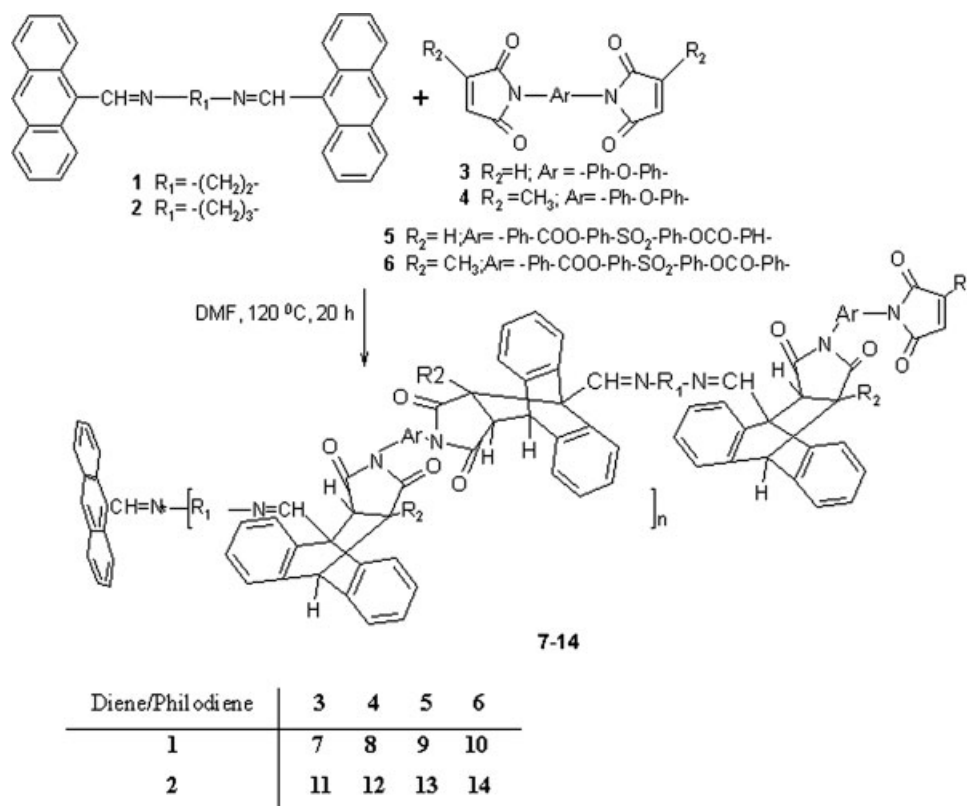

Figure 4 TGA and DTA thermograms of model compounds **15** and **16**.

Figure 3 The ^{1}H NMR (CDCl$_3$) spectrum of the model **16**.

Scheme 6

In the ^{1}H NMR spectra of both monomers, the multiplet situated in the range of 8.7–7.3 ppm is associated with anthracene protons while the singlet appearing at ∼ 9.5 ppm is assigned to CH=N protons. The peak signal that appears in the 4.52 and 4.23 ppm is attributed to the N—CH$_2$ groups in the monomer (**1**) and (**2**), respectively, while the aliphatic protons in the middle (CH$_2$) group of monomer (**2**) are the singlet at 2.59 ppm. Also, UV spectra show strong and characteristic absorptions of 9-anthryl substituted rings (260–390 nm).

Synthesis and characterization of the bismaleimide and biscitraconimide monomers

Bismaleimide and biscitraconimide monomers were synthesized according to the methods reported by Dix et al.[42] and Hoyt et al.[43] and are presented in Schemes 2 and 3.

The elemental analysis and ^{1}H NMR spectra of bismaleimides and biscitraconimides confirm the expected structures.

Synthesis of the Diels–Alder model compounds

To obtain more information about the structure of the polymers, two model compounds, **15** and **16**, were synthesized by cycloaddition reaction between

a monofunctional diene monomer (anthracene) and citraconic anhydride, or biscitraconimide **4** as dienophile (Schemes 4 and 5).

The IR spectra of **15** and **16** show peaks at 3410 cm^{-1} and 2990 cm^{-1} due to C—H from aromatic rings, at 1850 and 1790 cm^{-1} (the absorption characteristic of C=O stretching from imide) while the —O— linkage was observed at 1455 and 1220 cm^{-1}. The peaks at 1005, 950, 905, 750, 700, 550, and 505 cm^{-1} are relevant for the formation of cycloadducts, they being present only in 9,10-dyhydroanthracene structures, while bisanthracene monomers **1** and **2** exhibit characteristic absorptions due to the mono-substituted anthracene rings, νC—H aromatic at 730, 840, and 875 cm^{-1}. Also, UV spectroscopy can very well differentiate anthracene and dihydroanthracene structures. Thus, monomers **1** and **2** show characteristic absorptions of anthracene nucleus, 250–260 nm and 320–395 nm, while cycloadducts **15** and **16** showed absorption maxima characteristic to 9,10-dihydroanthracene structures (*ortho*-disubstituted benzenes) (λ_{max} at 270 and 295 nm), the absorptions situated between 320 and 390 nm have disappeared due to destruction of anthracene by cycloaddition) (Fig. 1).

The ^{1}H NMR spectrum of **15** and **16** are presented in Figures 2 and 3. Model **15** shows signals at 7.52–7.17 ppm (8 aromatic protons from dihydroanthracene structure), 4.76 ppm (dihydroanthracene proton),
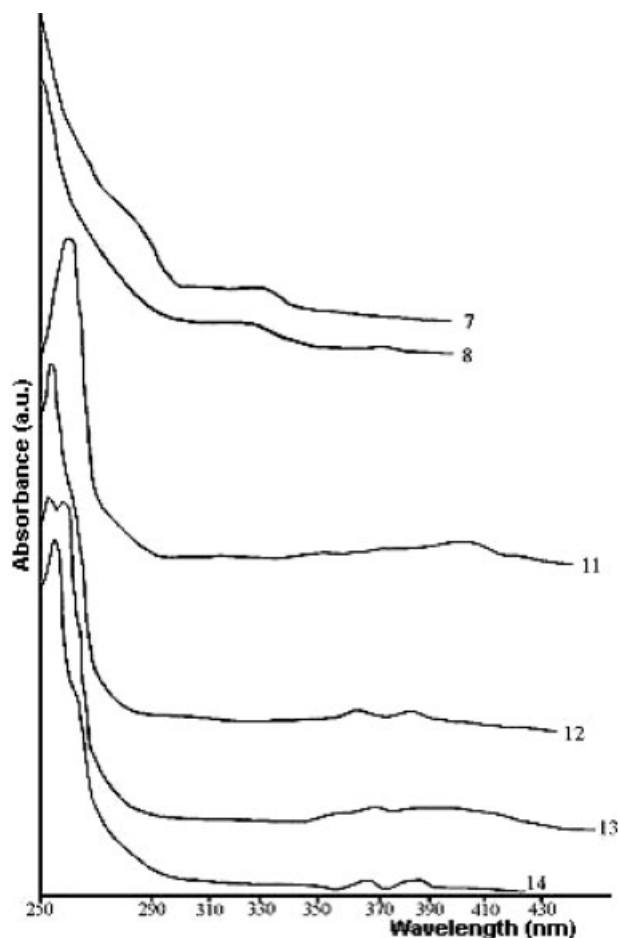
Figure 5 The UV spectra of the synthesized polymers.

4.40 ppm (dihydroanthracene proton), 3.0 ppm (CH from anhydride), and 1.26 ppm (protons from —CH$_3$).

Model **16** shows signals at 7.43–7.18 ppm assigned to aromatic protons, 6.90 (aromatic protons from dienophile, ortho to imide), 6.45 ppm (aromatic protons from dienophile, ortho to —O—), 4.82 ppm (dihydroanthracene proton), 4.46 ppm (dihydroanthracene proton), and 1.24 ppm (—CH$_3$ protons).

The thermal stability of the model compounds was evaluated by TGA, and Figure 4 presents typical TGA and DTA traces of these compounds performed in air atmosphere. The endotherm peak at 148°C is assigned to melting for model **15** followed by a retro Diels–Alder reaction with formation and evaporation of anthracene and citraconic anhydride. The retro Diels–Alder reaction in the case of model **16** is less evident, probably because the dienophile monomer is stable until 200°C. Both model compounds **15** and **16** afforded char yields 0% at 600°C.

Synthesis of polymers by Diels–Alder polycycloaddition

The reactions used for synthesis of copolymers are based on a Diels–Alder cycloaddition and are pre-

sented in Scheme 6. The reactions were performed in DMF and polymers were precipitated in methanol. The polymers are soluble in polar organic solvents such as DMF, THF, DMSO, and NMP, and Table I presents the yields and some characteristics of obtained polymers.

The first observation that can be made is related to low molecular weight, all products being oligomers due to the negative influence of the sterical hindrances and reversibility of the Diels–Alder reaction. There are only few examples where Diels–Alder polymerization has yielded to high-molecular-weight polymers, and in all cases, the irreversibility of the reaction was assured via subsequent elimination of a small molecule.

According to the UV, FTIR, and NMR spectra, polymers have resulted by addition of the dienophile to the mesopositions of anthracene nucleus and formation of 9.10-dihydroanthracene structures. Thus, the intensities of absorption bands at 260 and 320–390 nm, attributed to the π-π* transition in the anthracene nucleus, are very diminished and are assigned to the anthracene-end groups. In the same time, new absorption bands appear at 258, 266, 275,
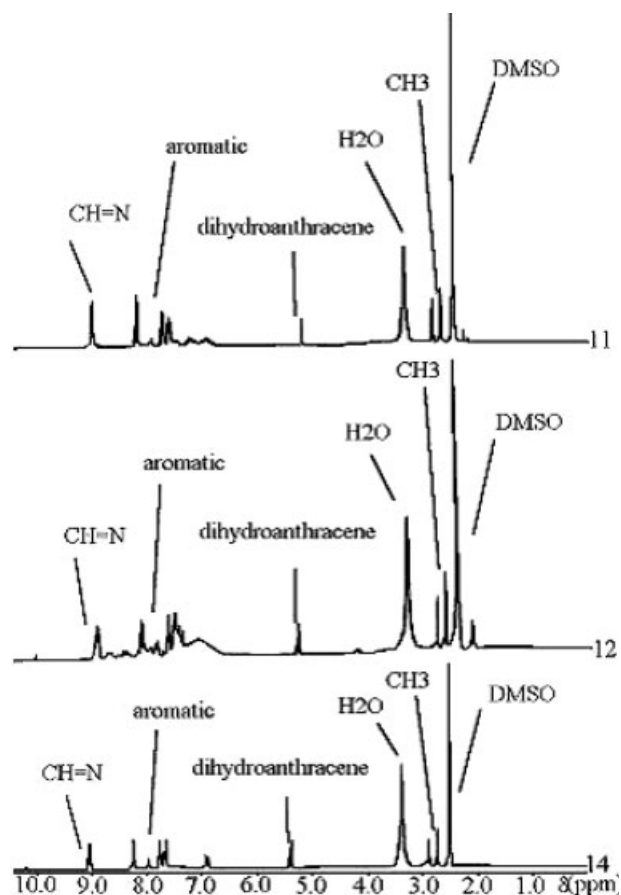

Figure 6 The ^{1}H NMR spectra of polymers **11**, **12**, and **14** in DMSO-d_6.

and 295 nm due to the formation of dihydroanthracene structure (Fig. 5).

The ^{1}H NMR spectra of three polymers are presented in Figure 6 and show a distinct signal above 9.0 ppm attributed to the azomethine proton and a signal at 4.5–5.0 ppm to the 10-dihydroanthracene protons. Also, the presence of signals attributed to anthracene and maleimide and citraconimide groups, respectively, as ends indicate that the products are oligomers, and molecular weight measurements confirm this conclusion.

The thermal stability of the polymers was evaluated by TGA. Figure 7 presents typical TGA traces for five polymers (**8**, **11–14**) in air atmosphere. The polymer initial decomposition temperature was determined by the intersection of the tangent to the steepest portion of the TGA curve with its strain-line before the polymer degradation. All polymers were stable up to 130–140°C in air atmosphere and the weight loss (2–4%) observed in this interval is associated with evaporation of humidity and solvent traces. They afforded a char yield 1–21% at 600°C. The decomposition of the polymers takes place in many stages. This fact can be explained based on structural peculiarities of each polymer. The DTA curves indicate, for all polymers, moderate endothermal effects in the first studies (until ~ 250°C) and then a strong exothermal effect. Based on thermal behavior of cycloadduct models **15** and **16**, one may

TABLE II
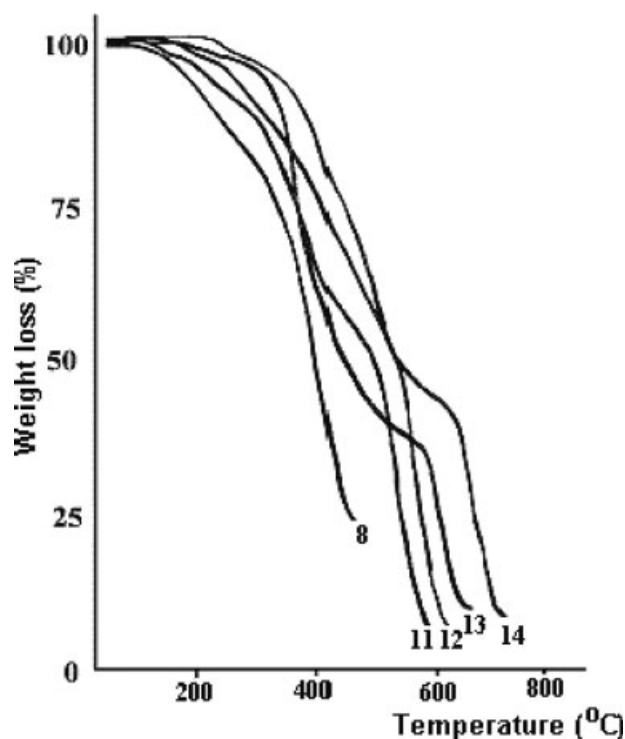### Thermogravimetric Characteristics of the Polymers

Polymers	IDT (°C)	$T_{10\%}$ (°C)	T_{dmax} (°C)	Y_c (%)
7	112	224	316	21
8	136	188	408	18
11	116	240	552	2
12	100	232	548	2
13	120	256	544	6
14	99	228	548	1

IDT, onset degradation temperature; $T_{10\%}$, temperature at weight loss 10%; T_{dmax}, temperature of the maximum decomposition; Y_c, char yields at 600°C.

assume that the degradation includes a retro Diels–Alder reaction and the rupture of —CH=N-linkages (endothermic processes) followed by oxidation of polymer chain fragments. Some thermogravimetric characteristics are showed in Table II.

CONCLUSIONS

The present work shows the feasibility of repetitive Diels–Alder addition as an alternate approach to obtain polymers with cycloadduct structure using bisanthracene monomers with bismaleimides and biscitraconimides. All polymerization products are oligomers due to limited solubility of the growing chains and the reversibility of the Diels–Alder reaction. Polymers have a limited solubility in strong polar solvents and a good thermal stability.

Figure 7 TGA and DTA traces of polymers **8**, **11–14** in air atmosphere.

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