Novel thermosetting diimides containing terminal propargyl groups

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A series of new thermosetting diimides containing terminal propargyl groups was synthesized via one-step high-temperature catalytic condensation of dianhydrides of a tetracarboxylic acid with *N*-Boc-(4-propargyloxy)aniline (APR) in the so-called "active medium", *viz.* molten benzoic acid. Structures of the obtained compounds were confirmed by ^{1}H NMR and IR spectroscopy. The effect of symmetrical and unsymmetrical structures of the middle bis-imide moiety obtained from dianhydrides of isomeric diphenyltetracarboxylic acid on physicochemical characteristics of the diimides was evaluated. A similar comparison of properties was also carried out for a pair of new propargyl-containing diimides synthesized from dianhydrides of symmetric and asymmetric structure: 4,4-biphenylenedioxydiphthalic and 3,4-biphenylenedioxydiphthalic dianhydrides, respectively. Thermal properties of thermosetting oligoimides were estimated using DSC and TMA methods. It was found that their thermal properties and solubility in organic solvents depend significantly on the structure of central bis-imide moiety.

Key words: polyimides, thermosetting oligomers, synthesis, dianhydrides, propargyl alcohol, binders, polymer composite materials.

Thermosetting oligomers containing terminal propargyl ether groups are of interest as promising binders for heat-resistant polymer composite materials (PCM).[1,2] They possess good adhesion towards glass and metals. In comparison with epoxy and maleimide resins, they are more water-resistant, exhibit better dielectric properties, are stable during storage, and have a high temperature of the degradation onset (>470 °C). As compared to oligomers containing acetylene and phenylacetylene groups, they can be produced using an accessible precursor, *viz.* propargyl alcohol. Propargyl-containing oligomers obtained from bisphenol A,[1] natural phenols,[3] terephthalic acid,[4] oligoetheretherketone,[5] triazine derivatives,[6] novolac resins,[7,8] and liquid crystals[9] have been already reported. In the case of heat-resistant polyimide matrices for PCM, they are mainly obtained from oligomers with ethynyl, phenylethynyl, *etc.* moieties.[10—14] At the same time, we consider propargyl-containing oligomers as obviously advantageous raw materials in comparison with oligomers of this type.

There are only few works reporting on the synthesis of oligoimides with terminal propargyl (PR) groups.[15—17] The mechanism for curing of propargyl-containing monomers has been explored in details using monofunctional model compounds.[15] It has been demonstrated that it includes the generation of unsaturated chromene cycle, polymerization at which occurs subsequently to form a spatial network. An oligoimide containing terminal propargyl groups has been previously[15] synthesized *via* a complicated route, *viz.* the reaction of propargyl bromide with a fluorine-containing oligoimide cotaining phenolic groups, which was preliminary obtained *via* cyclocondensation of dianhydride (dianhydride 6F) of 2,2-hexafluoropropylidenediphenyltetracarboxylic acid with 3-aminophenol. In another work,[17] 3-(4-aminophenyl)propan-1-yl was synthesized and used for the preparation of oligoimides containing terminal propargyl ether moieties. The authors at the first step have carried out the low-temperature polycondensation of 4,4-oxydianiline (ODA) with tetracarboxylic acid dianhydride in DMF in the presence

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of a propargyl-containing amine, which was followed by the thermal imidization of resulting polyamide acid in solution upon removal of water at 150 °C by azeotropic distillation with toluene.

We have recently[18] demonstrated the possibility to obtain reactive diimides containing PR groups according to the method developed previously for catalytic polycondensation of diamines and dianhydrides in a benzoic acid melt.[19] The process proceeded at 145 °C within 2 h without the use of any toxic solvents. We have synthesized a new series of reactive bisimides containing propargyl ether groups from three dianhydrides of different structures and 4-propargyloxyaniline. The results of evaluation of thermal and rheological properties of bisimides revealed the promising outlook of these compounds for developing heat-resistant binders for PCMs.

This work was aimed at the expansion of range of obtained reactive diimides (RDIs) and in particular, at the evaluation of the possibility to use the starting dianhydrides of a more «rigid» structure for their synthesis, as well as at the clarification of effect of the symmetry of arrangement of their reactive groups on some properties of RDIs and cured products. Commercially available symmetrical and asymmetrical dianhydrides of diphenyltetracarboxylic acid (3,4,3′,4′-DPDA and 2,3,3′,4-DPDA, respectively), as well as two new double-hinged anhydrides with a central biphenylene moiety (symmetrical 4,4-biphenylene-4,4-diphthalic anhydride (4,4-BPODA) and asymmetrical 3,4-BPODA) were selected as model objects. *N*-Boc-amino-4-(propargyloxy)benzene (APR) was used as the propargyl-containing monomer.

Results and Discussion

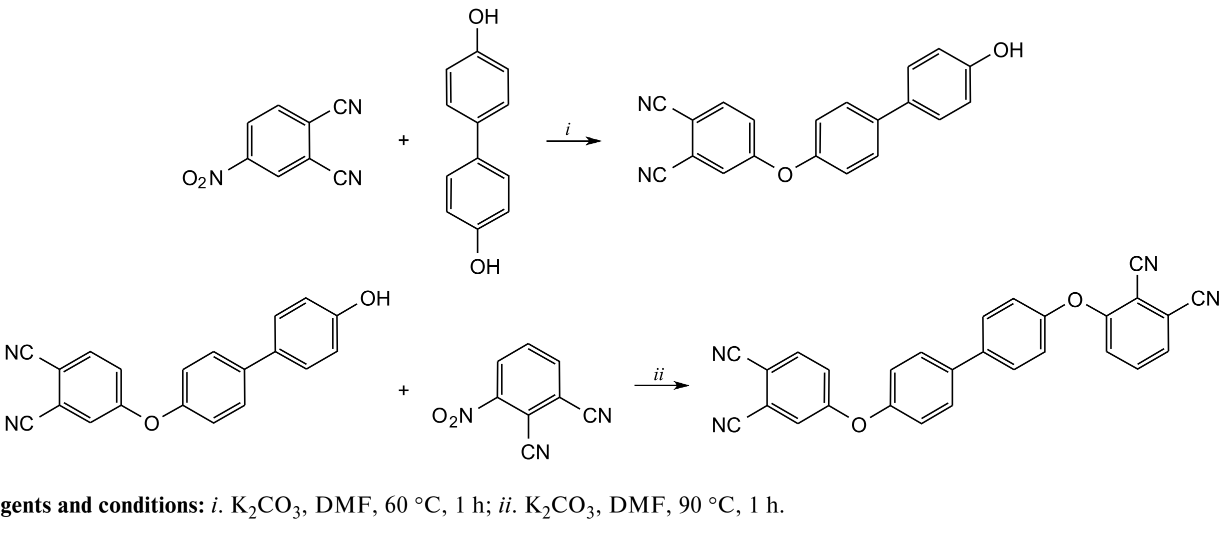
Schemes 1 and shows the synthesis of new unsymmetrical dianhydride 3,4-BPODA.

Symmetrical bis-dinitrile was obtained in one step at 90 °C and at the ratio of nitrophthalodinitrile (NPDN) to bis-phenol equal to 2 : 1. Unsymmetrical bis-dinitrile was synthesized in two steps (see Scheme 1). At the first one, the ratio of NPDN to bis-phenolate was 1 : 1, and the reaction was carried out at 60 °C. Etherification of the first hydroxy group of bis-phenolate makes less active the second one since the activation energy of that step is higher than that for the first hydroxy group. Exactly this fact allows one to obtain unsymmetrical dianhydride and subsequently unsymmetrical diimide. At the second step, 3-nitrophthalodinitrile was introduced into the system, and the temperature was elevated up to 90 °C (see Scheme 2). Phthalonitrile groups were saponificated to give the corresponding tetraacids. The isolated tetraacids were dried and dehydrated by refluxing in acetic anhydride to obtain the target dianhydrides (see Scheme 2).

The reaction of dianhydrides with APR gave the corresponding diimides with terminal propargyl groups (Scheme 3).

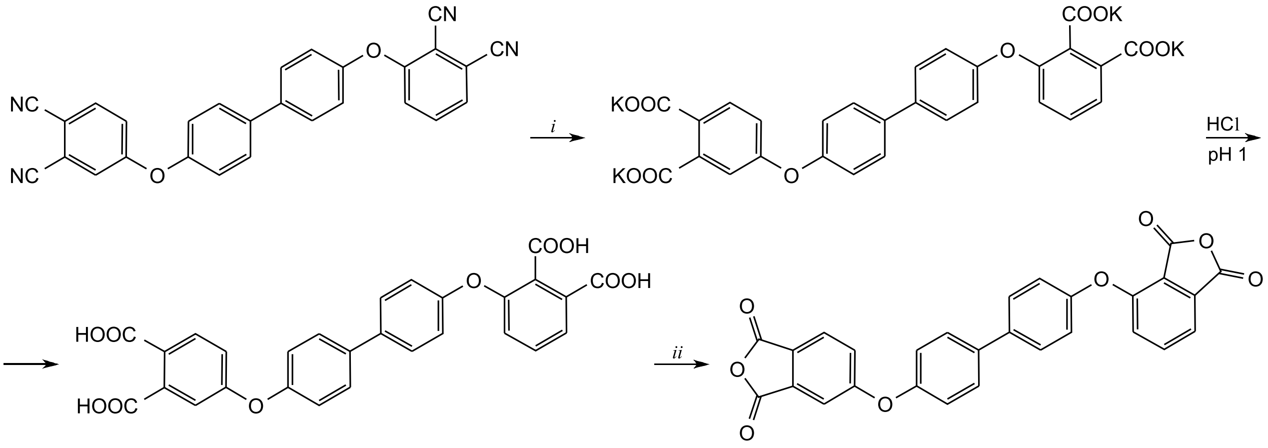
Four new reactive diimides with propargyl groups were synthesized (see Scheme 3). From the data on the solubility of RDI in organic solvents (Table 1),

Scheme 1



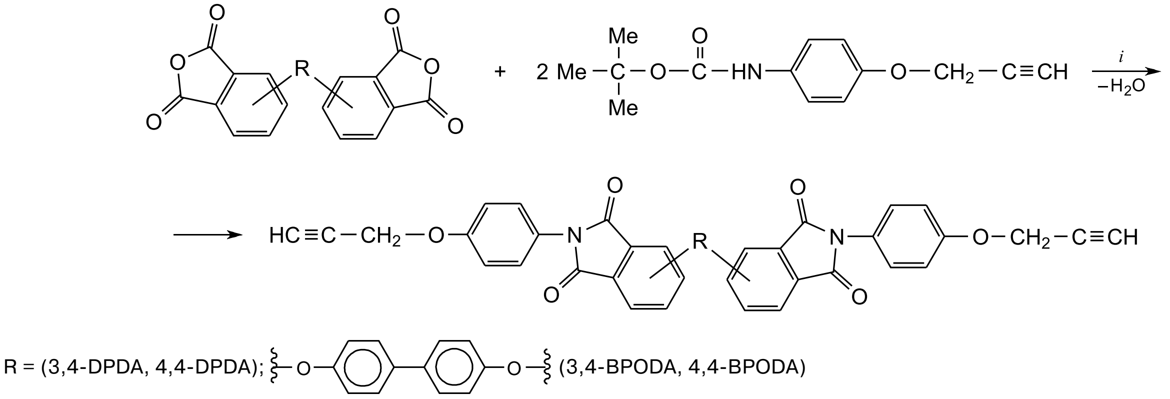
Reagents and conditions: *i*. K_2CO_3, DMF, 60 °C, 1 h; *ii*. K_2CO_3, DMF, 90 °C, 1 h.

Scheme 2



Reagents and conditions: *i*. KOH, 12 h; *ii*. $(MeCO)_2O$, 120 °C, 1 h.

Scheme 3



Reagents and conditions: *i*. Benzoic acid, $H_2C{=}\overset{Me}{C}{-}Me$, CO_2.

it follows that diimides with an asymmetric structure of the unit are soluble in amide solvents and DMSO. This allowed us to analyze their structure by ^1H NMR and GPC. Since diimides based on symmetrical dianhydrides are soluble in hot DMSO, their structures were also confirmed by ^1H NMR spectroscopy.

In all the ^1H NMR spectra (Figs. 1 and 2), there are signals (δ_H 3.59—3.64 and 4.87—4.89) from protons of the propargyl moiety in strong fields and signals from aromatic protons in weak fields (δ_H 7.0—8.0). The integral intensities were in agreement with the expected structures of synthesized RDIs.

Table 1. Solubility of diimides (10 mg) in organic solvents (0.5 mL)

Sample	Acetone	THF	Chloroform	DMF	DMAA	*N*-MP	DMSO
RDI 3,4-DPDA-PR	—	—	—	+	+	+	+
RDI 4,4-DPDA-PR	—	—	—	–/+*	–/+*	–/+*	–/+*
RDI 3,4-BPODA-PR	—	—	—	+	+	+	+
RDI 4,4-BPODA-PR	—	—	—	–/+*	–/+*	–/+*	–/+*

* Soluble in hot solvent. *N*-MP is *N*-methylpyrrolidone.

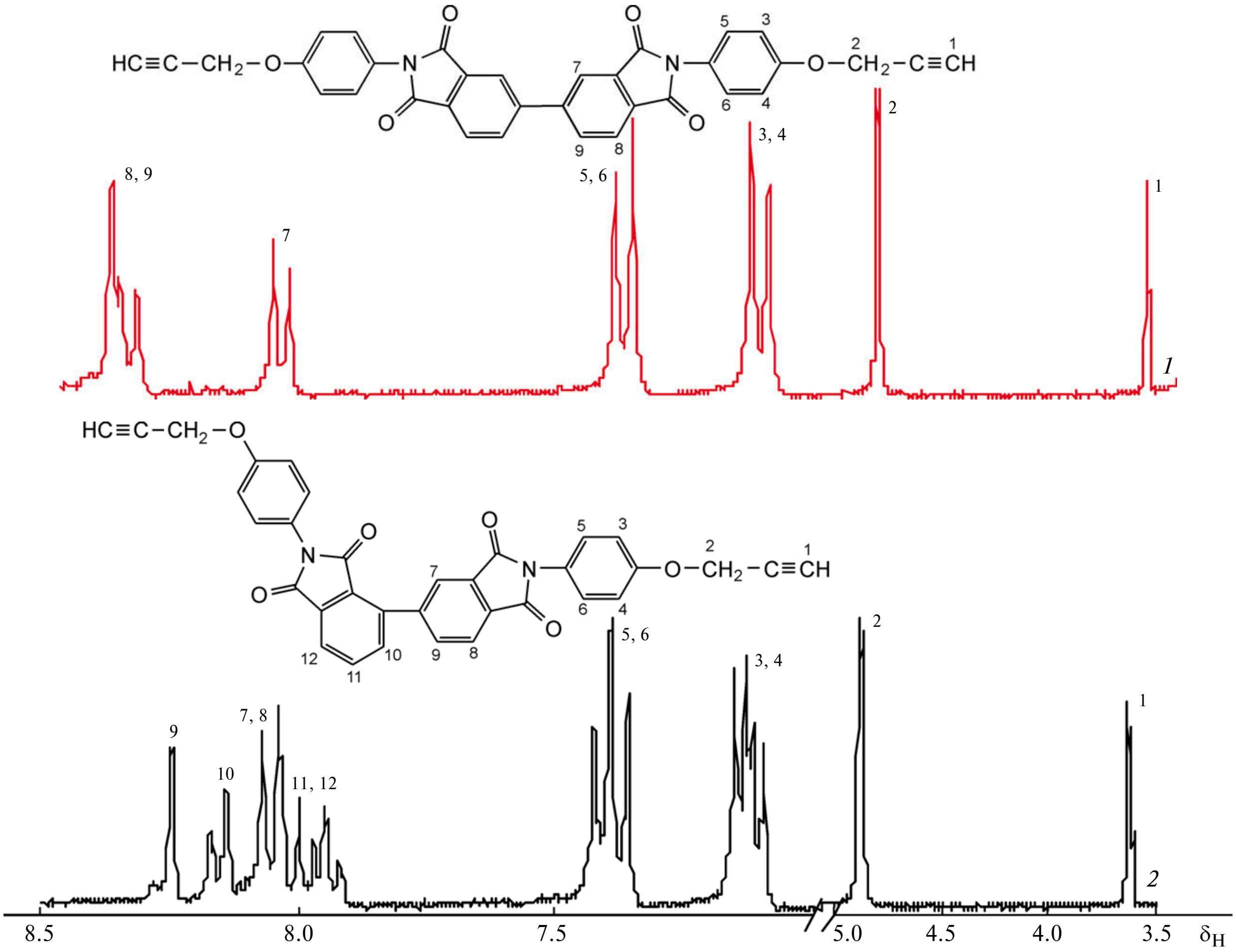


Fig. 1. ^{1}H NMR spectra recorded for diimides of RDI 4.4-DPDA-PR (1) and RDI 3.4-DPDA-PR (*2*).

The GPC data acquired for the RDI samples 3,4-DPDA-PR and 3,4-BPODA-PR confirmed that the synthesized diimides are individual compounds (Fig. 3).

Figure 4 shows the IR spectrum recorded for RDI 3,4-BPODA-PR. It contains absorption bands characteristic of C=O bonds in the imide cycle in the region of 1715 and 1780 cm^{-1} and a band of CH vibrations in the propargyrlic moiety in the region of 3260 cm^{-1}. As expected, this band was disappearing during curing due to the consumption of chromene cycle upon the polymerization. Similar spectra were obtained for the remaining RDIs.

Thermal properties of powdered samples of diimides were evaluated using TMA and DSC methods. The TMA thermograms recorded for the rod penetration mode in the sample contain deformation curves reaching 100% deformation at the temperatures of 260—300 °C (Fig. 5). The temperature of flow onset for asymmetric 3,4-isomers was lower by approximately 20 °C than that for diimides based on 4,4-isomers. The temperatures of 100% deformation for the obtained samples are listed in Table 2.

The DSC curves (first scan) recorded for samples of diimides clearly demonstrated exothermic effects related to the thermal curing process in the range of 250—320 °C (Fig. 6). According to the DSC data, the temperature of maximum heat flow rate increases in the series of RDI: 4,4-DPDA-PR < < RDI 3,4-DPDA-PR < 4,4-BPODA-PR < < 3,4-BPODA-PR. All the curves contain a distortion of the exothermic effect due to the superposition of a narrow, well-reproducible endothermic peak that can most likely be assigned to the melting of crystalline phase of RDI. The studied RDI samples probably possess a partially crystalline structure, and the curing process begins earlier than or simultaneously with the onset of melting. No changes were observed in the thermograms during the second scan.

As it turned out, the overlap of softening and onset temperatures does not exclude the possibility of processing diimides into monolithic tablets by heat

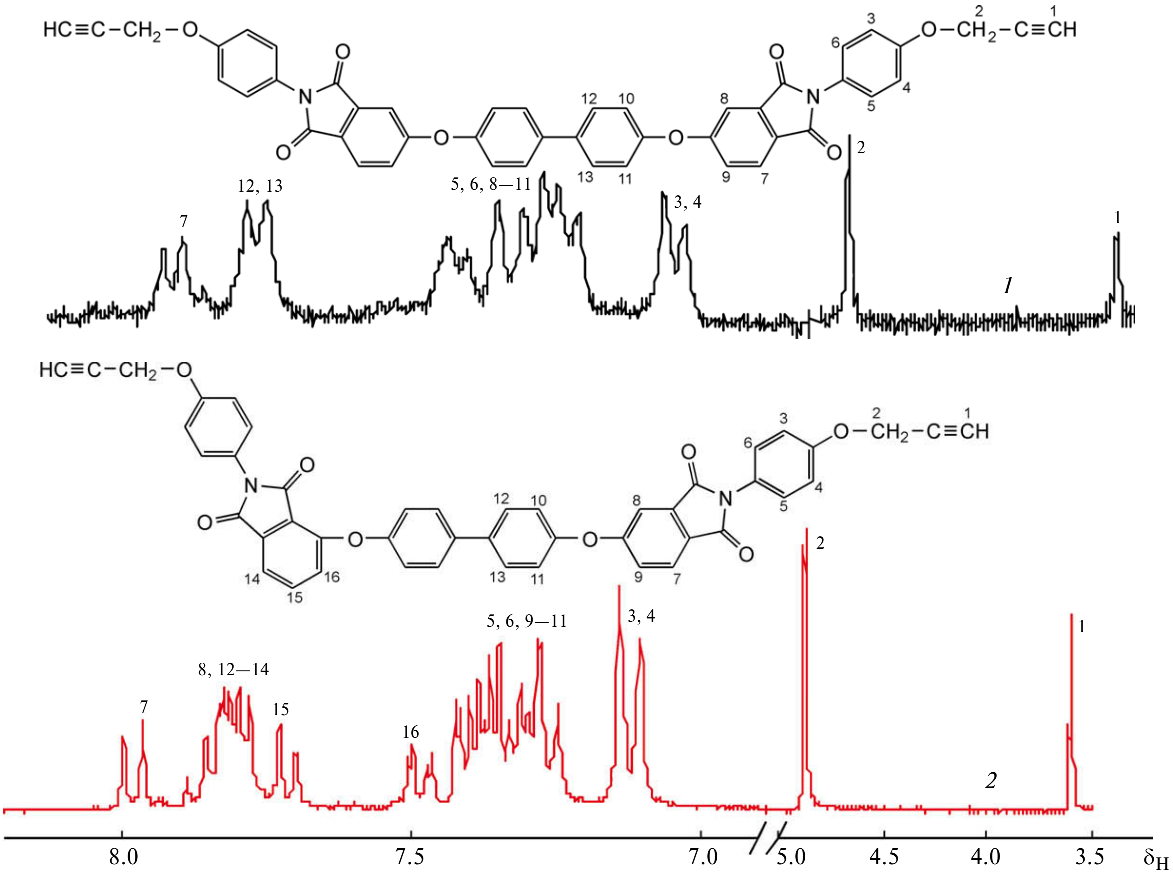


Fig. 2. ^{1}H NMR spectra recorded for diimides RDI 4.4-BPODA-PR (*1*) and RDI 3.4-BPODA-PR (*2*).

pressing under low pressure (10—30 kg cm^{-2}) at 200—320 °C. Samples in this case were obtained in the form of solid plates of thickness about 300 μm, which were difficult to separate from the substrate. It would apparently be reasonable to evaluate the physicomechanical properties for this series of oligoimides containing a longer chain.

In summary, the present work reported on the series of four new thermosetting diimides containing terminal propargyl groups with a «hard» and «semihard» middle dianhydride moiety, which was synthesized via the polycyclocondensation in the so-called «active medium», molten benzoic acid. One of the initial anhydrides (3,4-BPODA) was synthesized for the first time. Structures of the obtained compounds were confirmed by ^{1}H NMR and IR spectroscopy. The asymmetry of middle bis-imide moiety obtained from isomeric dianhydrides of diphenyltetracarboxylic acid (3,4-DPDA and 3,4-BPODA) provides solubility to these diimides in amide solvents. The considered diimides are partially crystalline oligomers, whose curing begins in the amorphous phase, while melting of the crystalline phase occurs directly during curing. Powders of the synthesized diimides can be processed by heat pressing at a temperature of 280—300 °C to yield plates of 1 mm thickness.

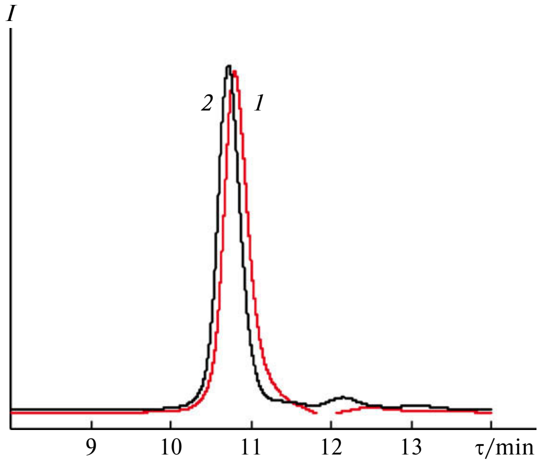


Fig. 3. GPC chromatograms for RDI 3.4-DPDA-PR (*1*) and RDI 3.4-BPODA-PR (*2*).

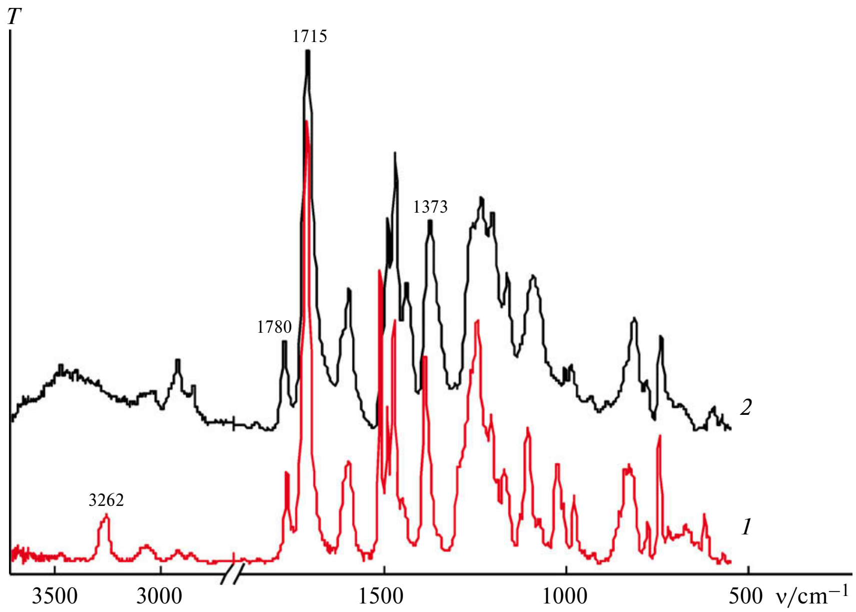


Fig. 4. IR spectra recorded for diimide RDI 3.4-BPODA-PR: powder from synthesis (*1*) and cured at 320 °C (*2*).

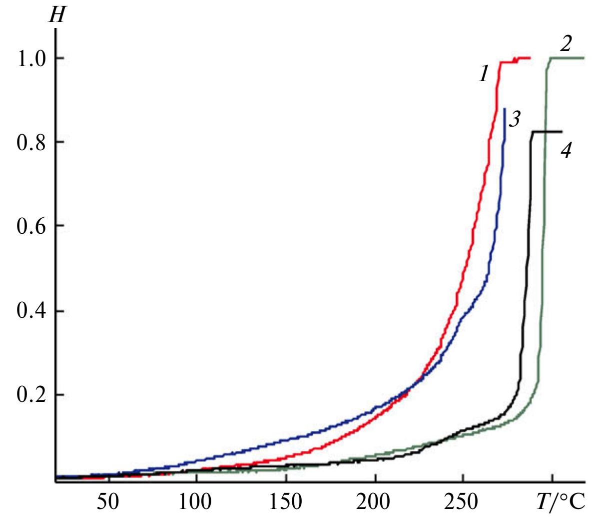


Fig. 5. TMA recorded for diimides RDI 3.4-BPODA-PR (*1*), RDI 4.4-BPODA-PR (*2*), 3.4-DPDA-PR (*3*), and 4.4-DPDA-PR (*4*).

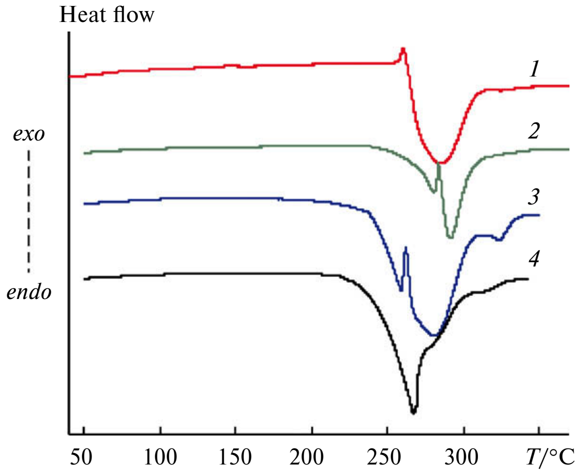


Fig. 6. DSC thermograms recorded for diimides RDI 3.4-BPODA-PR (*1*), RDI 4.4-BPODA-PR (*2*), 3.4-DPDA-PR (*3*), and 4.4-DPDA-PR (*4*).

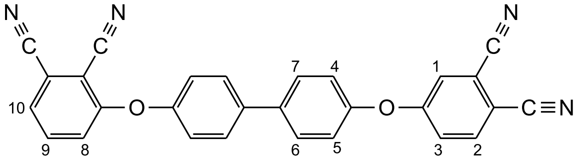
Table 2. Temperatures of the thermal deformation onset of the sample and melting of the crystalline phase of RDI

Sample	T_m	T_{cr}
	°C	
RDI 3,4-DPDA-PR	262	272
RDI 4,4-DPDA-PR	267	296
RDI 3,4-BPODA-PR	261	264
RDI 4,4-BPODA-PR	283	288

Experimental

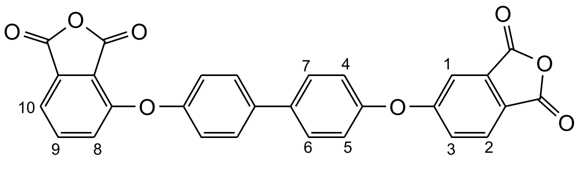
Materials. 3,4′-Diphthalic anhydride, 4,4′-diphthalic anhydride, 4,4′-dioxydiphenyl, 3- and 4-nitrophthalonitrile were obtained from Sigma-Aldrich and used without further purification. Dimethylformamide of pure grade was dried over P_2O_5 and purified by distillation *in vacuo* (b.p. 153 °C). Diethyl ether of pure grade (Ruskhim) was used without further purification. Benzoic acid of analytical grade (Ruskhim) was recrystallized from an ethanol—water mixture and dried *in vacuo* (m.p. 122 °C).

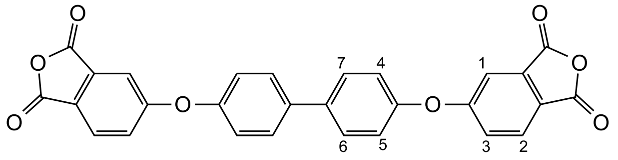
Synthesis of 3,4′-(4,4′-diphenyldioxy)diphthalic anhydride (3,4-BPODA). At the first step, 3,4′-(4,4′-diphenyldioxy)diphthalonitrile was synthesized. 4-Nitrophthalonitrile (16.0 g, 0.092 mol) and 4,4′-dioxydiphenyl (16.76 g, 0.09 mol) were loaded upon stirring in a three-necked flask equipped with a stirrer, reflux condenser, and thermometer and containing *N*,*N*-dimethylformamide (DMF) (120 mL). A solution of $K_2CO_3 \cdot 1.5H_2O$ (33.6 g, 0.20 mol) in water (30 mL) was added to the reaction mixture. The reaction mixture was vigorously stirred at 60—65 °C for 1 h, 3-nitrophthalonitrile (16.0 g, 0.092 mol) was added, the temperature was increased up to 90 °C, and stirring was continued under these conditions for another 1 h. Upon completion of the reaction, the reaction mixture was cooled to 5—10 °C and poured into cold water (300 mL). The resulting precipitate was filtered, washed with water (3×100 mL), and dried at 60 °C. to yield 3,4′-(4,4′-diphenyldioxy)diphthalonitrile (32.3 g, 82% of theoretical). The yield was 60%, beige crystalline powder, m.p. > 300 °C. Found (%): C, 77.80; H, 4.01; N, 11.39. $C_{28}H_{14}N_4O_2$. Calculated (%): C, 76.70; H, 3.22; N, 12.78. IR (KBr), ν/cm^{-1}: 2227 (CN), 1241 (C—O—C). ^{1}H NMR (300 MHz, DMSO-d$_6$, *J*/Hz), δ: 7.34 (d, 4 H, C(4)H, C(4′)H, C(5)H, C(5′)H, *J* = 8.79 Hz); 7.38 (d, 2 H, C(3)H, C(8)H, *J* = 7.82 Hz); 7.82 (d, 4 H, C(1)H, C(9)H, C(2)H, C(10)H, *J* = 8.55 Hz); 7.84—7.90 (m, 2 H, C(6)H, C(7)H).



At the second step, 3,4′-(4,4′-diphenyldioxy)diphthalic acid and subsequently 3,4′-(4,4′-diphenyldioxy)diphthalic anhydride were obtained similarly to the known procedure.[20] 3,4′-(4,4′-Diphenyldioxy)diphthalic anhydride (32.8 g, 93% of theoretical) was obtained as light beige crystalline powder, m.p. 140 °C. Found (%): C, 70.18; H, 2.88. $C_{28}H_{14}O_8$. Calculated (%): C, 70.30; H, 2.95. IR (KBr), ν/cm^{-1}: 1823, 1779 (C=O); 1260, 1125 (C—O—C). ^{1}H NMR (300 MHz, DMSO-d$_6$), δ: 7.03—7.07 (m, 2 H, C(1)H, C(3)H); 7.14—7.27 (m, 5 H, C(4)H, C(4′)H, C(5)H, C(5′)H, C(10)H); 7.5—7.55 (t, 1 H, C(8)H); 7.63—7.81 (m, 6 H, C(6)H, C(6′)H, C(7)H, C(7′)H, C(9)H, C(2)H).

4,4′-(4,4′-Diphenyldioxy)diphthalic anhydride (4,4-BPODA) was obtained similarly the known procedure.[20] Found (%): C, 70.20; H, 2.91. $C_{28}H_{14}O_8$. Calculated (%): C, 70.30; H, 2.95. IR (KBr), ν/cm^{-1}: 1830, 1775 (C=O); 1260, 1125 (C—O—C). ^{1}H NMR (300 MHz, DMSO-d$_6$), δ: 7.15—7.34 (m, 6 H, C(4)H, C(4′)H, C(5)H, C(5′)H, C(3)H, C(3′)H); 7.51—7.59 (m, 2 H, C(1)H, C(1′)H); 7.77—7.87 (m, 4 H, C(6)H, C(6′)H, C(7)H, C(7′)H); 8.08—8.12 (m, 2 H, C(2)H, C(2′)H).





Synthesis of the diimide RDI 3,4-DPDA-PR (general procedure). Benzoic acid (BA) (13.5 g) was loaded into a three-necked flask (*V* = 25 mL) equipped with a magnetic stirbar. The flask was heated at 145 °C, and after melting BA, 3,4′-diphthalic anhydride (3,4-DPDA) (0.5472 g, 1.86 mmol) was added. When dissolution of 3,4-DPDA was completed, *N*-Boc-4-propargyloxyaniline (APR) (0.9337 g, 3.76 mmol) was added. The synthesis of diimide was continued for 2 h upon slow purging with nitrogen at 145 °C and stirring at ω = 400 rpm. The liquid reaction mixture was poured onto a Petri dish. When the mass was solidified, the product was washed with diethyl ether 5 times to remove the BA. It was dried at room temperature for 22 h and at 25 °C *in vacuo* in an oven for 1 h. The yield of dry product was 60—70%, m.p. 262 °C.

IR (CsI), cm^{-1}: 3283 (C—H, propargyl moiety); 2130 (C≡C, propargyl moiety); 1765, 1708 (C=O, imide moiety); 1386, 730 (C—N, imide moiety). ^{1}H NMR (300 MHz, DMSO-d$_6$), δ: 3.62 (s, 2 H, C(1)H, C(1′)H); 4.87 (m, 4 H, C(2)H, C(2′)H); 7.1—7.15 (m, 4 H, C(3)H, C(3′)H, C(4)H, C(4′)H); 7.36—7.43 (m, 4 H, C(5)H, C(5′)H, C(6)H, C(6′)H); 7.92—8.17 (m, 5 H, C(7)H, C(8)H, C(10)H, C(11)H, C(12)H); 8.25 (s, 1 H, C(9)H).

Similar syntheses were carried out starting from 4,4′-diphthalic anhydride (4,4-DPDA), 3,4-BPODA, and 4,4-BPODA to give RDI 4,4-DPDA-PR, 3,4-BPODA-PR, and 4,4-BPODA-PR, respectively.

Diimide RDI 4,4-DPDA-PR. M.p. 268 °C. IR (CsI), cm^{-1}: 3286 (C—H, propargyl moiety); 2136 (C≡C, propargyl moiety); 1770, 1713 (C=O, imide moiety); 1387, 734 (C—N, imide moiety). ^{1}H NMR (300 MHz, DMSO-d$_6$), δ: 3.64 (s, 2 H, C(1)H, C(1′)H); 4.89 (m, 4 H, C(2)H, C(2′)H); 7.12—7.16 (m, 4 H, C(3)H, C(3′)H, C(4)H, C(4′)H); 7.39—7.42 (m, 4 H, C(5)H, C(5′)H, C(6)H, C(6′)H); 8.08—8.09 (m, 2 H, C(7)H, C(7′)H); 8.36—8.40 (m, 4 H, C(8)H, C(8′)H, C(9)H, C(9′)H).

Diimide RDI 3,4-BPODA-PR. M.p. 262 °C. IR (CsI), cm^{-1}: 3283 (C—H, propargyl moiety); 2130 (C≡C, propargyl moiety); 1765, 1708 (C=O, imide moiety); 1386, 730 (C—N, imide moiety). ^{1}H NMR (300 MHz, DMSO-d$_6$), δ: 3.62 (s, 2 H, C(1)H, C(1′)H); 4.87 (m, 4 H, C(2)H$_2$, C(2′)H$_2$); 7.1—7.15 (m, 4 H, C(3)H, C(3′)H, C(4)H, C(4′)H); 7.36—7.43 (m, 4 H, C(5)H, C(5′)H, C(6)H, C(6′)H); 7.92—8.17 (m, 5 H, C(7)H, C(8)H, C(10)H, C(11)H, C(12)H); 8.25 (s, 1 H, C(9)H).

Diimide RDI 4,4-BPODA-PR. M.p. 283 °C. IR (CsI), cm^{-1}: 3252 (C—H, propargyl moiety); 2136 ($C\equiv C$, propargyl moiety); 1767, 1713 (C=O, imide moiety); 1381, 760 (C—N, imide moiety). ^{1}H NMR (300 MHz, DMSO-d$_6$), δ: 3.59 (s, 2 H, C(1)H, C(1′)H); 4.86 (m, 4 H, C(2)H$_2$, C(2′)H$_2$); 7.10—7.14 (m, 4 H, C(3)H, C(3′)H, C(4)H, C(4′)H); 7.26—7.42 (m, 10 H, C(5)H, C(5′)H, C(6)H, C(6′)H, C(9)H, C(9′)H, C(10)H, C(10′)H, C(11)H, C(11′)H, C(15)H); 7.5 (s, 1 H, H(16)); 7.70—7.88 (m, 6 H, C(8)H, C(8′)H, C(12)H, C(12′)H, C(13)H, C(13′)H, C(14)H); 7.96—7.99 (d, 1 H, C(7)H).

IR spectra were recorded on an Equinox 55/S FTIR spectrometer (Bruker) in the range of 4000—400 cm^{-1}. The IR spectra were recorded on an Equinox 55/S FTIR spectrometer (Bruker) in the range 4000–400 cm^{-1}. The samples were prepared as pellets with CsI (the content of tested substance 1%). ^{1}H NMR spectra were recorded on a Bruker AM-300 instrument operating at the frequency of 300.13 MHz. Chemical shifts are reported relative to SiMe$_4$. DSC experiments were carried out on a DSC-500 instrument (Russia). GPC measurements were carried out on a Stayer instrument equipped with Phenogel columns (5000–70000) using DMF as the eluent at 60 °C and a flow rate of 1 mL min^{-1}. Polystyrene samples with a molecular weight of $(0.2—7.0) \cdot 10^4$ were used as the standard. The primary data were processed using the MultiChrom Version 1.5X software. Thermomechanical analysis (TMA) was performed on a UIP-70M device with computer signal recording in the rod penetration mode into the tablet at a temperature rise rate of 10 °C min^{-1} and a load of 0.7 kg cm^{-2}.

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Animal Testing and Ethics

No human or animal subjects were used in this research.

Conflict of Interest

The authors declare no competing interests.

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