



# SYNTHESIS OF POLYAMIDE-IMIDES BY THE INTERACTION OF TEREPHTHALOYL CHLORIDE AND 3,3',4,4'-DIPHENYL ETHER TETRACARBOXYLIC ACID DIANHTRIDE WITH VARIOUS DIAMINES

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Cite this: *INEOS OPEN*,  
2022, 5 (6), 170–176  
DOI: 10.32931/io2226a

Received 9 June 2023,  
Accepted 1 August 2023

<http://ineosopen.org>

## Abstract

The effect of temperature, the order and duration of the interaction of terephthaloyl chloride (0.5 mol) and 3,3',4,4'-diphenyl ether tetracarboxylic acid dianhydride (0.5 mol) with 9,9-bis(4-aminophenyl)fluorene or 2,2'-bis(trifluoromethyl)benzidine (1.0 mol) in NMP on the properties of the resulting polyamide-imides was studied. The molecular weights and yields of the resulting polymers were shown to vary in a wide range ( $M_w = 64\text{--}438$  kDa, yields 85–97%) and depend on the reaction conditions. The polyamide-imides obtained possess high heat resistance ( $T_g = 260\text{--}360$  °C), thermal stability ( $T_{10\%} \sim 500$  °C) and form high-strength films ( $\sigma = 122\text{--}160$  MPa,  $E = 1.3\text{--}1.9$  GPa,  $\varepsilon = 9\text{--}30\%$ ).

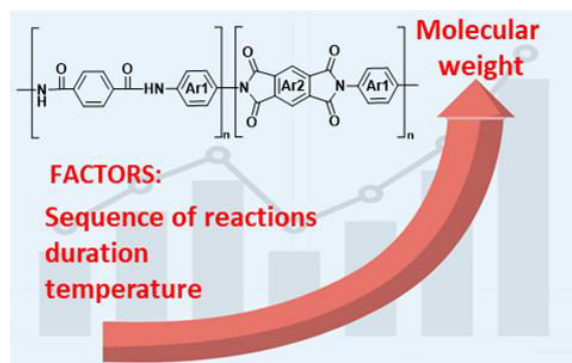
**Key words:** polyamide-imides, polycondensation, terephthaloyl chloride.

## Introduction

Polyamide-imides (PAIs) bearing both amide bonds and imide rings in their structures combine the valuable properties of polyimides and polyamides. PAIs have high heat resistance, are highly soluble in organic solvents, form high-strength flexible films, and have good adhesion to various substrates [1–3]. They are promising objects for the production of membranes [4–6], adhesives [7, 8], protective coatings for electrical wires [9–12] and optical fibers [13, 14], as well as in integrated optics [15, 16], electronics [17, 18], *etc.*

The molecular design of PAIs is diverse and is determined by a wide variety of monomers available for their synthesis, such as diamines and dianhydrides of tetracarboxylic acids, carboxylic acid chlorides, diisocyanates, *etc.* [19]. One of the main methods for the synthesis of PAIs is the low-temperature polycondensation of diamines with trimellitic anhydride chloride (TMAC) [19]. The unsymmetrical structure of TMAC along with the different reactivities of the anhydride and acid chloride groups can lead to products in low yields with low molecular weights [2, 19]. Terephthaloyl chloride (TPC) with highly active acid chloride groups in the *para*-positions is more promising for the production of high molecular weight polymers [20]. A literature survey, encompassing patent literature, revealed certain interest in the PAIs based on TPC [13, 21–23]. However, most of the reports deal with the practical application of these polymers and do not concern the peculiarities of their formation. At the same time, understanding these relationships is of great importance from the viewpoint of producing the polymers with the desired physicochemical properties.

The purpose of this study was to optimize the low-

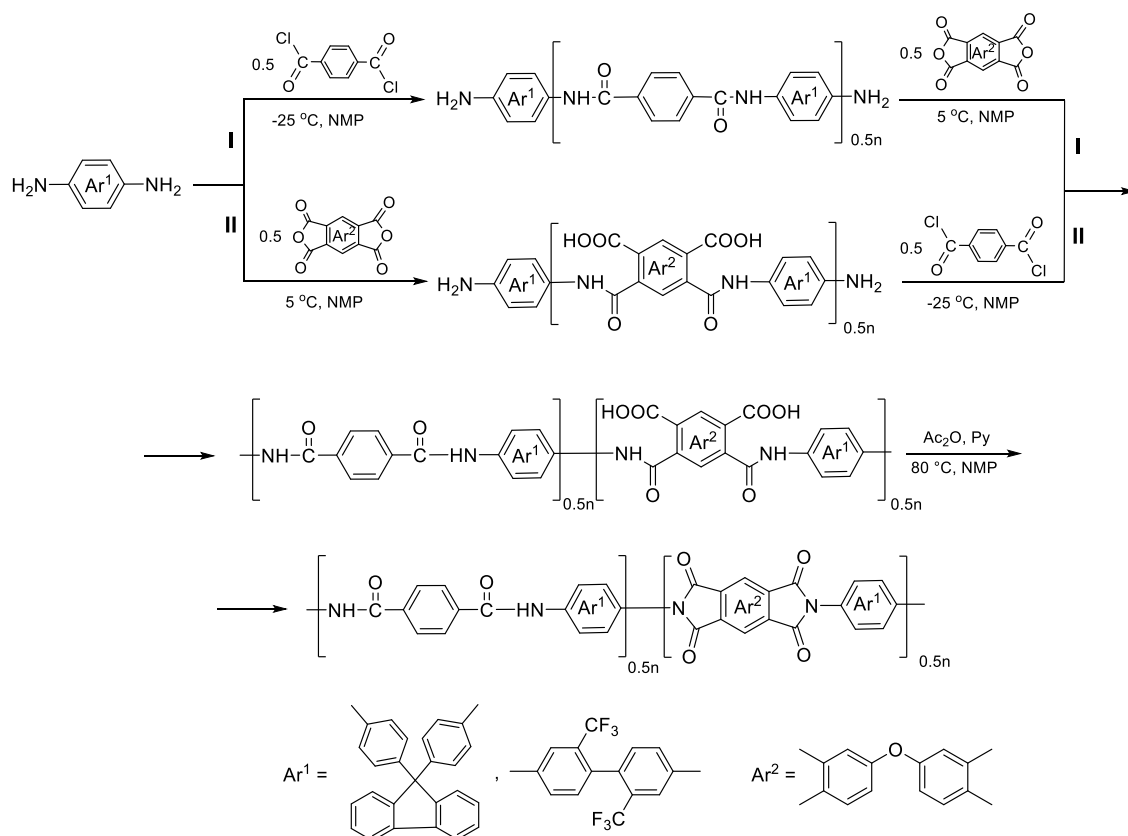


temperature polycondensation of 2,2'-bis(trifluoromethyl)benzidine (TFDB) or 9,9-bis(4'-aminophenyl)fluorene (FDA) with 3,3',4,4'-diphenyl ether tetracarboxylic acid dianhydride (ODPA) and TPC. The effect of the order of monomer introduction into the reaction mixture, temperature and duration of the process on the yields and molecular weights of the resulting PAIs is discussed.

## Results and discussion

The synthesis of PAIs from diamine, TPC, and ODPA consists of three sequential reactions: the reaction of the diamine with TPC/ODPA, the subsequent reaction of the intermediate obtained with ODPA/TPC leading to the formation of a poly(amic acid) (PAAA), and the final imidization of PAAA to PAI (Fig. 1). Taking into account the extensive experience in the production of poly(amic acids) [24, 25], polyimides [24–28], as well as aromatic polyamides [29], the technological parameters of the synthesis, namely, the method for introducing the components, reaction temperature, *etc.* should affect the molecular weight of the resulting PAAA, its stability and, ultimately, the yield and molecular weight of the PAI.

It is known that one of the ways to regulate the molecular weight of a poly(amic acid) is to control the conditions of the acylation [30]. Therefore, at the first stage, we studied the effect of the order of introducing the monomers into the reaction mixture on the yield and molecular weight of the resulting PAI. In the first method, solid TPC was added portionwise to a solution of diamine in NMP cooled to  $-25$  °C (Fig. 1, I). The choice of this mode was dictated by the high reactivity of di-



**Figure 1.** Synthesis of PAIs.

carboxylic acid chlorides, in particular, towards aprotic amide solvents [31]. At this temperature, TPC is insoluble in NMP, which minimizes their possible interaction. At the second stage of the synthesis, in order to reduce the probability of local overheating as a result of an exothermic reaction between ODPA and amino groups and the formation of dianhydride complexes with the solvent [30], ODPA was also added gradually. According to the second method (Fig. 1, II), ODPA was first added to the diamine solution, and then TPC was introduced. In both methods, the reaction proceeded heterogeneously until the monomers (TPC or ODPA) were fully consumed. Therewith, the viscosity of the reaction mixture rapidly increased. The cyclization of the resulting PAAAs was carried out under the action of a mixture of acetic anhydride and pyridine.

As can be seen from the results presented in Table 1, method II provided the PAIs in higher yields and with higher molecular weights (Fig. 1). Due to the fact that the low-temperature polycondensation of diamines with dicarboxylic acid chlorides in most cases leads to the formation of high molecular weight polyamides [31], the relatively low yields and molecular weights of the polymers obtained by method I are likely to be associated with a large contribution of side reactions [32]. Perhaps, the

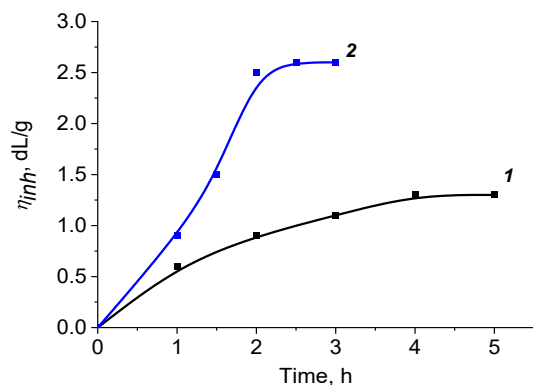
addition of ODPA at the second stage leads to local overheating, resulting in the partial cyclization of PAAA with the release of water and subsequent hydrolysis of amic acid groups [30]. It is also possible that this method facilitates the formation of macrocycles to a greater extent [33].

The duration of the reaction between the diamine and ODPA has a significant effect on the molecular weight of the resulting PAI (hereinafter, PAI was synthesized by method II). For the considered polymers, according to Fig. 2, the maximum values of inherent viscosity ( $\eta_{inh}$ ) are achieved in 4 (FDA:ODPA:TPC) and 2 h (TFDB:ODPA:TPC) after the formation of a homogeneous solution. Moreover,  $\eta_{inh} = 2.6$  dL/g for the TFDB:ODPA:TPC, and this value is more than twice the corresponding value for FDA:ODPA:TPC (1.2 dL/g). Cardo diamines are less basic [34]; therefore, in this case, the production of high molecular weight PAAAs is complicated.

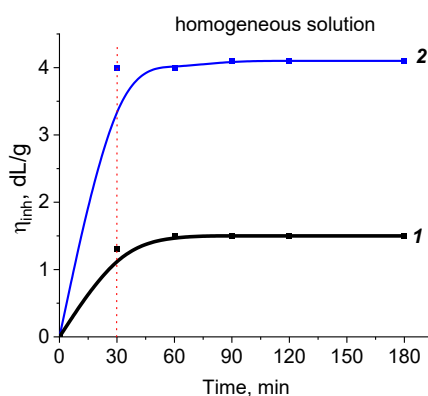
In contrast, the values of  $\eta_{inh}$  for the PAAA and PAI weakly depend on the duration of the reaction of the amic acid with TPC. Already at the stage of formation of a highly viscous homogeneous solution, the PAAAs with almost the maximum achievable molecular weight are formed (Fig. 3). It is noteworthy that the observed increase in the viscosity of the reaction mixture, which turns into a gel over time, is not reflec-

**Table 1.** Dependence of the PAI yield and inherent viscosity on the synthesis method

PAI	Method	Order of monomers	Yield, %	$\eta_{inh}$ , dL/g
FDA:TPC:ODPA	I	1) FDA, 2) TPC, 3) ODPA	85	0.3
FDA:ODPA:TPC	II	1) FDA, 2) ODPA, 3) TPC	96	0.9
TFDB:TPC:ODPA	I	1) TFDB, 2) TPC, 3) ODPA	90	0.8
TFDB:ODPA:TPC	II	1) TFDB, 2) ODPA, 3) TPC	98	2.6



**Figure 2.** Dependence of the inherent viscosity of the PAIs FDA:ODPA:TPC (1) and TFDB:ODPA:TPC (2) on the duration of the reaction between ODPA and the diamine. The reaction time at the second step was 2 h.

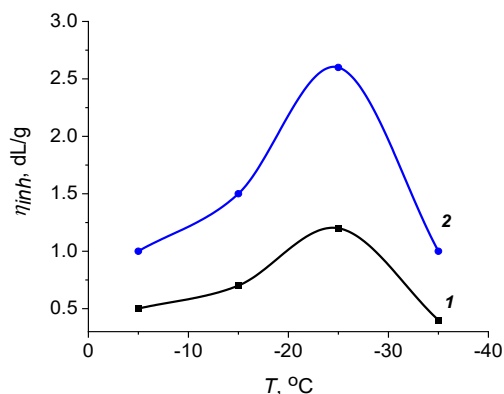


**Figure 3.** Dependence of the inherent viscosity of the PAAAs FDA:ODPA:TPC (1) and TFDB:ODPA:TPC (2) on the duration of the reaction between the amic acid and TPC at  $-25\text{ }^{\circ}\text{C}$ . The duration of the interaction of ODPA with FDA/TFDB was 4/2 h.

ted in the values of  $\eta_{\text{inh}}$  of the resulting PAAAs. This behavior of solutions, as in the case of poly(amic acid)s, is caused by the orientation of macromolecules and the formation of strong hydrogen and other intermolecular bonds. The inherent viscosities of the PAIs FDA:ODPA:TPC (1.2 dL/g) and TFDB:ODPA:TPC (2.6 dL/g) appeared to be lower than those of the corresponding PAAAs (1.5 dL/g and 4.1 dL/g), which is stipulated by the structural changes and possible partial hydrolysis of the PAAA during imidization.

As can be seen from Fig. 4, the dependence of the inherent viscosity of the PAIs FDA:ODPA:TPC and TFDB:ODPA:TPC

on the reaction temperature of the intermediate amic acid with TPC has an extreme character. The highest molecular weight of the PAI is achieved at  $-25\text{ }^{\circ}\text{C}$ , while the formation of the PAAA occurs at the interface. The reaction at  $-15\text{ }^{\circ}\text{C}$  leads to a significant decrease in the molecular weight of the resulting polymers. This is apparently due to the contribution of side reactions, since, at the mentioned temperatures, TPC is already soluble in NMP. Reducing the reaction temperature to  $-35\text{ }^{\circ}\text{C}$  also does not contribute to an increase in the molecular weight of the resulting polymer, presumably, due to a decrease in the reactivity of the compounds and a significant increase in the viscosity of the reaction mixture.



**Figure 4.** Dependence of the inherent viscosity of the PAIs FDA:ODPA:TPC (1) and TFDB:ODPA:TPC (2) on the temperature of the reaction between the amic acid and TPC for 2 h. The duration of the interaction of ODPA with FDA/TFDB was 4/2 h.

By varying the reaction temperature and time, random copolymers with a wide range of molecular weights were obtained. As can be seen from Table 2, the results of GPC analysis correlate with the  $\eta_{\text{inh}}$  values of the PAIs. The molecular weights of the fluorene-containing PAIs were expectedly lower than those of the PAIs based on TFDB. The maximum value of  $\eta_{\text{inh}}$  for FDA:ODPA:TPC was 1.2 dL/g ( $M_w = 241\text{ kDa}$ ), which exceeds the viscosity values of the fluorene-containing homo- and copolyamide-imides obtained previously with TMAc ( $\eta_{\text{inh}} = 0.44\text{--}0.88\text{ dL/g}$ ) [22, 35]. The use of more reactive TFDB ensures an increase in the molecular weights of the resulting polymers to  $M_w = 203\text{--}438\text{ kDa}$  and  $\eta_{\text{inh}} = 1.5\text{--}2.6\text{ dL/g}$ . It is worth noting that the polycondensation of TFDB with TPC and 3,3',4,4'-biphenyltetracarboxylic dianhydride provides a poly(amic acid) with the maximum value of  $\eta_{\text{inh}}$  equal to 1.22 dL/g, which was achieved in more than 24 h of the reaction at room temperature [36].

**Table 2.** Molecular weight characteristics and solubility of the resulting PAIs and the mechanical properties of the films

PAI	Molecular weight characteristics				Solubility <sup>a</sup>			Films		
	$\eta_{\text{inh}}$ , dL/g	$M_n$ , kDa	$M_w$ , kDa	$M_z$ , kDa	S <sup>b</sup>	Acetone	$\sigma$ , MPa	E, GPa	$\epsilon$ , %	
FDA:ODPA:TPC	0.5	22	64	138	+	–	122	1.4	10	
	0.9	30	153	415	+	–	130	1.9	9	
	1.2	57	241	712	+	–	135	1.9	10	
TFDB:ODPA:TPC	1.5	63	203	500	+	±	140	1.4	25	
	2.6	133	438	1027	+	±	160	1.3	30	

<sup>a</sup> solubility was evaluated for a 10 mg sample of the polymer in 1 mL of the solvent; "+" soluble at room temperature, "±" soluble when heated; "–" insoluble;

<sup>b</sup> S – solvent: N-methyl-2-pyrrolidone; N,N-dimethylacetamide; N,N-dimethylformamide; dimethyl sulfoxide; m-cresol; pyridine.

The structure and purity of the PAIs obtained were confirmed by IR spectroscopy and elemental analysis. According to the elemental analysis data (see the Experimental section), certain found values of the carbon content were slightly lower than the calculated ones, while the hydrogen contents were slightly higher. This is probably due to the hygroscopicity of the resulting polymers [37, 38]. The IR spectra of the PAIs (Fig. 5) show the characteristic absorption bands corresponding to the functional groups: 1782 and 1728  $\text{cm}^{-1}$  (asymmetric and symmetric imide C=O stretches), 1669  $\text{cm}^{-1}$  (amide I), 1525  $\text{cm}^{-1}$  (amide II), 1370  $\text{cm}^{-1}$  (–C–N–C– imide stretches), 1255, 1172–1093  $\text{cm}^{-1}$  (C–F stretches in the  $\text{CF}_3$  group). The absence of a broad band in the region typical for stretching vibrations of the O–H bond of carboxy groups, in contrast to the spectra of the corresponding PAAAs (Fig. 5, 1' and 2'), unambiguously confirms the formation of completely imidized macromolecules.

It was found that the resulting PAIs, regardless of  $M_w$ , easily dissolve at room temperature in NMP, DMA, DMF, DMSO, *m*-cresol, and pyridine (Table 2), and TFDB:ODPA:TPC, in addition, is soluble in acetone. The high solubility of the PAIs is explained by the presence of bulky side cardo or  $\text{CF}_3$  groups, which prevent the close packing of macromolecules and reduce interchain interactions [39, 40]. The films cast from the resulting polymers have high values of tensile strength (122–160 MPa) and tensile modulus (1.3–1.9 GPa), while the samples of the PAIs based on TFDB are more deformable ( $\epsilon \sim 25\text{--}30\%$ ) compared to the cardo analogs ( $\epsilon \sim 10\%$ ). The thermal properties of the PAIs were evaluated using TMA and TGA methods. It was found that the heat resistance of the PAI largely

depends on the nature of the diamine in use (Fig. 6, 1). For the cardo polymer FDA:ODPA:TPC  $T_g = 360$  °C, while for TFDB:ODPA:TPC the value of glass transition point is 100 °C lower. The bulky fluorene units prevent the free rotation of the segments of a polymer chain, thereby increasing its rigidity [41] relative to that of the more mobile macromolecules with trifluoromethyl groups [42]. A similar trend was earlier observed for (co)polyamide-imides of the general formula  $\text{TFDB}_n\text{:FDA}_m\text{:TMAC}$ , for which the value of  $T_g$  increased with an increase in the content of FDA [22]. All the PAIs obtained have high thermooxidative stability ( $T_{10\%} \sim 500$  °C) (Fig. 6, 2), which is comparable to the thermal stability of polyimides or slightly inferior to them [43].

## Experimental section

### Materials

2,2'-Bis(trifluoromethyl)benzidine (>98%,  $M_p = 188$  °C), 9,9-bis(4'-aminophenyl)fluorene (>98%,  $M_p = 236\text{--}237$  °C), 3,3',4,4'-diphenyl ether tetracarboxylic acid dianhydride (>98%,  $M_p = 226$  °C) were purified by sublimation under vacuum (20 Pa) at the temperatures close to their melting points. Terephthaloyl chloride (>98%,  $M_p = 83$  °C) was distilled under vacuum. Pyridine (99%) and acetic anhydride (98%) were used as received without further purification. Monomers and cyclizing agents were purchased from TCI or Acros Organics. NMP (99%) was kept for two days over  $\text{CaH}_2$  and then distilled under vacuum.

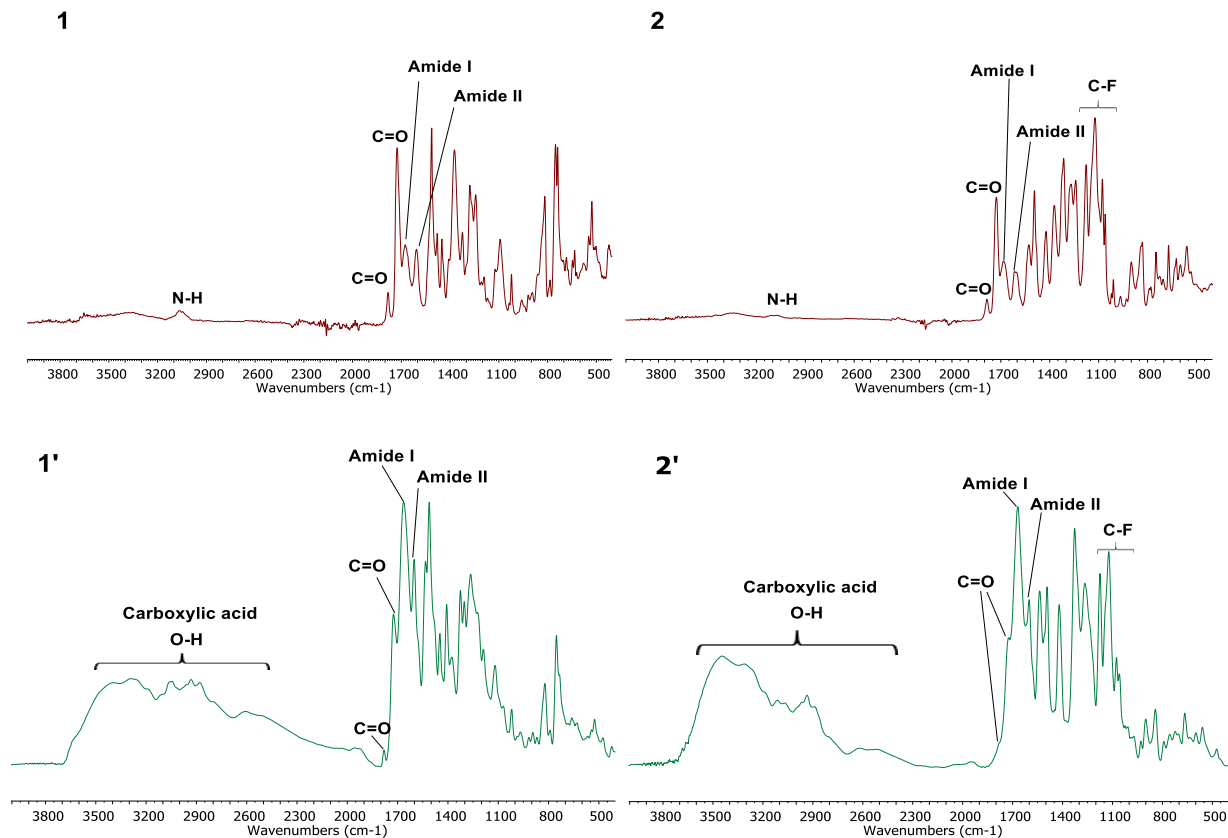
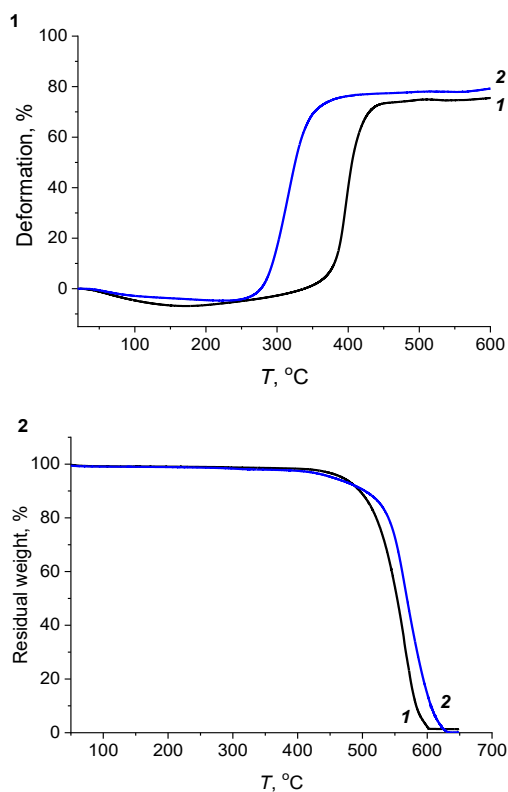


Figure 5. IR spectra of the PAIs FDA:ODPA:TPC (1) and TFDB:ODPA:TPC (2), and the corresponding PAAAs (1') and (2').



**Figure 6.** TMA and TGA curves (**1** and **2**) for FDA:ODPA:TPC (curve **1**) and TFDB:ODPA:TPC (curve **2**).

## Syntheses

### Synthesis of polyamide-imides

**Method I.** To obtain the poly(amide amic acid) FDA:TPC:ODPA, a three-neck flask (250 mL) equipped with a mechanical overhead stirrer and an argon outlet adapter was charged with 3.2905 g (0.0094 mol) of FDA and 22 mL of dry NMP. The monomer was dissolved upon stirring under an argon atmosphere at room temperature. The resulting solution was cooled with a mixture of acetone and liquid nitrogen to  $-25\text{ }^{\circ}\text{C}$ , and then 0.9605 g (0.0047 mol) of solid TPC and 1 mL of NMP were added portionwise. The reaction mixture was stirred at  $-25\text{ }^{\circ}\text{C}$  until the formation of a homogeneous solution and for further 1–2 h at  $-25\text{ }^{\circ}\text{C}$ . Then the temperature of the reaction mixture was increased to  $5\text{ }^{\circ}\text{C}$ , and 1.4655 g (0.0047 mol) of ODPa and 1 mL of NMP were added portionwise. After the formation of a homogeneous solution, stirring was continued for another 5 h at  $5\text{ }^{\circ}\text{C}$ . The temperature of the reaction mixture was gradually increased to room temperature ( $23\text{--}25\text{ }^{\circ}\text{C}$ ). A cyclizing mixture was added to the poly(amide amic acid) solution: 2.7 mL (0.0283 mol) of pyridine and 3.6 mL (0.0377 mol) of acetic anhydride. The reaction mixture was stirred at room temperature for 1 h, left overnight without stirring, and then stirred at  $80\text{ }^{\circ}\text{C}$  for 4 h. The resulting viscous solution was cooled to room temperature, NMP was diluted to 5% concentration, and the polymer was isolated in the form of fibers by precipitation into a five-fold volume of water. The polymer was filtered and rinsed repeatedly with distilled water, then with methanol in a Soxhlet apparatus for 48 h. The product was dried in air for 12 h followed by drying in a vacuum chamber at  $80\text{ }^{\circ}\text{C}$  for 12 h, and finally at  $200\text{ }^{\circ}\text{C}$  and 1 mmHg within 2 h.

**Method II.** To obtain the poly(amide amic acid) FDA:ODPA:TPC, a three-neck flask (250 mL) equipped with a mechanical overhead stirrer and an argon outlet adapter was charged with 3.2905 g (0.0094 mol) of FDA and 22 mL of dry NMP. The monomer was dissolved upon stirring under an argon atmosphere at room temperature. The resulting solution was cooled to  $5\text{ }^{\circ}\text{C}$ , and then 1.4655 g (0.0047 mol) of ODPa and 1 mL of NMP were added portionwise. The reaction mixture was stirred at  $5\text{ }^{\circ}\text{C}$  until the formation of a homogeneous solution and for further 1–5 h. Then the reaction mixture was cooled to  $-25\text{ }^{\circ}\text{C}$  and 0.9605 g (0.0047 mol) of solid TPC and 1 mL of NMP were added portionwise. After the formation of a homogeneous solution, stirring was continued for another 2–4 h at  $-25\text{ }^{\circ}\text{C}$ . Subsequently, the imidization, isolation, and purification of the polymer were carried out similarly to method I.

FDA:ODPA:TPC (yield: 96%). Anal. Calc. for  $\text{C}_7\text{H}_{44}\text{N}_4\text{O}_7$ : C, 80.73; H, 4.00; N, 5.09. Found: C, 79.19; H, 4.41; N, 5.09%. IR ( $\nu/\text{cm}^{-1}$ ): 1777 (asym C=O of imide), 1721 (sym C=O of imide), 1672 (amide I), 1603 (amide II), 1369 (C–N).

TFDB:ODPA:TPC (yield: 98%). Anal. Calc. for  $\text{C}_{52}\text{H}_{24}\text{F}_{12}\text{N}_4\text{O}_7$ : C, 59.88; H, 2.11; N, 5.37; F, 21.88. Found: C, 59.48; H, 2.46; N, 5.48; F, 21.53%. IR ( $\nu/\text{cm}^{-1}$ ): 1781 (asym C=O of imide), 1723 (sym C=O of imide), 1681 (amide I), 1611 (amide II), 1368 (C–N), 1173–1056 (C–F in  $\text{CF}_3$ ).

## Methods

The FTIR spectra were recorded on a Bruker Vertex 70v FTIR-spectrometer.

The inherent viscosities ( $\eta_{\text{inh}}$ ) of the polymers were determined by the viscometric analysis. The flow time of a solution of 0.05 g sample in 10 mL of NMP was measured in the Ostwald viscometer at  $25\text{ }^{\circ}\text{C}$ . For some PAAAs, the inherent viscosities were estimated as a function of the reaction time. For this purpose, the samples taken at certain time intervals during the synthesis were diluted with dry NMP to  $C = 0.5\text{ g/dL}$ , and then the solution flow time was determined.

The molecular weight characteristics of the polymers were assessed by GPC on a Waters Alliance GPCV 2000 instrument equipped with a Waters DRI detector. The column set (Styragel HT6E) was eluted with NMP polymer solutions ( $C = 1.0\text{ mg/mL}$ ) at a rate of 1.0 mL/min at  $80\text{ }^{\circ}\text{C}$ . The resulting data were calibrated using monomodal polystyrene standards (Polymer Standards Service).

The solubility of the copolymers was tested in various organic solvents at a polymer concentration of 0.01 g/mL.

The glass transition temperature ( $T_g$ ) was defined using thermomechanical analysis on a TMA Q-400 device at a fixed load of 0.034 MPa and a heating rate of  $5\text{ }^{\circ}\text{C/min}$ . The thermogravimetric analysis was carried out in air on an STA 6000 PerkinElmer instrument at a heating rate of  $10\text{ }^{\circ}\text{C/min}$ .

The PAI films were prepared by casting a 10 wt % solution of the corresponding polymer in NMP onto a glass substrate, followed by evaporation of the solvent at  $70\text{ }^{\circ}\text{C}$ . The thickness of the resulting films was controlled using an applicator (gap thickness 600–800  $\mu\text{m}$ ). Then the resulting films (40–50  $\mu\text{m}$ ) were removed from the glass substrate and dried under vacuum (2 mm Hg) at  $70\text{ }^{\circ}\text{C}$  for 6 h,  $200\text{ }^{\circ}\text{C}$  for 2 h, and, finally, at  $230\text{ }^{\circ}\text{C}$  for 0.5 h. The mechanical properties of the films

obtained were assessed using at least five samples (40×2×0.05 mm) on a Polyani dynamometer (Hungary) at a stretching rate of 0.033 mm/s at room temperature. The average values of tensile strength ( $\sigma$ , MPa), tensile modulus ( $E$ , GPa), and elongation at break ( $\varepsilon$ , %) were calculated from the measurement results.

## Conclusions

The effect of the order of interaction of diamines with terephthaloyl chloride/3,3',4,4'-diphenyl ether tetracarboxylic acid dianhydride, temperature and reaction duration on the yields and molecular weights of the resulting PAIs was studied. It was shown that the PAIs with the highest molecular weight ( $M_w = 241\text{--}438$  kDa) and quantitative yields can be formed during the primary interaction of the diamine with ODPa at 5 °C and the subsequent reaction of the intermediate amic acid with TPC at  $-25$  °C. The variation of the synthesis conditions and the chemical structures of the monomers opens the way to the synthesis of PAIs with controlled molecular weight, thermal, and mechanical properties. The resulting polymers hold great promise for the production of highly heat resistant films and coatings, membranes, etc.

## Acknowledgements

This work was performed with financial support from the Ministry of Science and Higher Education of the Russian Federation (agreement no. 075-03-2023-642) using the equipment of the Center for Molecular Composition Studies of INEOS RAS.

The authors are grateful to E. S. Afanasyev, M. G. Ezernitskaya, and E. M. Chaika for the TMA, IR spectroscopic studies, and mechanical testing of the polymers.

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