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RESEARCH ARTICLE

Influence of the structure of phosphorus(III)-containing oligoester(meth)acrylates on the physical and mechanical properties, thermal stability, and combustion mechanisms of cured polymers

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Abstract

Objectives. The work sets out to investigate the influence of the structure of spacer in structure of phosphorus(III)-containing oligoester(meth)acrylates on physical and mechanical properties of polymers and their combustibility.

Methods. The physical and mechanical properties of polymers were determined using the following: DMA 242 E Artemis dynamic mechanical analyzer (*NETZSCH*, Germany); universal testing machine for standard tests on materials (*ZwickRoell Group*, Germany); GT-7045-HMH(L) impact test machine (*Gotech Testing Machines, Inc*., Taiwan); Q-1500 D derivatograph of the Paulic–Paulic–Erdey system (thermogravimetry, *IOM*, Hungary); Oxygen Index Module device for determination of burning behavior by plastic flammability testing according to oxygen index (*Concept Equipment*, United Kingdom); GT-7045-HMH(L) device for determination of the Vicat softening temperature (*Gotech Testing Machines, Inc*., Taiwan); SFT-110XW supercritical fluid extractor (*Supercritical Fluid Technologies, Inc*., USA) for supercritical fluid extraction with carbon dioxide.

Results. The influence of the spacer structure in the structure of phosphorus(III)-containing oligoester(meth)acrylates on the dynamic mechanical and physicomechanical properties of polymers was established. Comparative assessment of the impact of the spacer structure on properties of polymers was carried out in terms of their heat stability (thermogravimetric analysis) and combustibility (measurement of limited oxygen index). It is established that polymers having balanced physical and mechanical properties can be obtained by introducing spacer characteristics into the oligomer structure. Polymers obtained on the basis of phosphorus(III)-containing oligoester(meth)acrylates with spacers demonstrate considerable resistance to impact strength tests.

Conclusions. The achieved results testify to the possibility of obtaining polymers on the basis of phosphorus(III)-containing oligoester(meth)acrylates with spacer attributes that possess increased resistance to impact strength and thermal stability tests at an insignificant decrease in their combustibility.

Keywords

spacer, phosphorus-containing polymers, dynamic mechanical analysis, DMA, limited oxygen index, coupled combustion model

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НАУЧНАЯ СТАТЬЯ

Влияние строения фосфор(III)-содержащих олигоэфир(мет)акрилатов на физико-механические свойства, термическую стабильность и механизмы горения отвержденных полимеров

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Аннотация

Цели. Исследование влияния строения спейсера в структуре фосфор(III)-содержащих олигоэфир(мет)акрилатов на физико-механические свойства полимеров и их горючесть.

Методы. Физико-механические свойства полимеров определяли с помощью динамического механического анализатора DMA 242 E Artemis (*NETZSCH*, Германия); универсальной машины для испытаний (*ZwickRoell Group*, Германия); копера GT-7045-HMH(L) (*Gotech Testing Machines, Inc*., Тайвань); дериватографа Q-1500 D системы Паулик–Паулик–Эрдей (термогравиметрические исследования, *MOM*, Венгрия); установки «Oxygen Index Module» для определения кислородного индекса пластмасс — Fire Testing (*Concept Equipment*, Великобритания), прибора Копер GT-7045-HMH(L) для определения температуры размягчения по Вика (*Gotech Testing Machines, Inc*., Тайвань); сверхкритического флюидного экстрактора SFT-110XW (*Supercritical Fluid Technologies, Inc*., США) для флюидной экстракции сверхкритическим диоксидом углерода.

Результаты. Установлено влияние строения спейсера в структуре фосфор(III)-содержащих олигоэфир(мет)акрилатов на динамические механические и физико-механические свойства полимеров. Сделана сопоставительная оценка влияния строения спейсера на свойства полимеров, их термостабильность (проведен термогравиметрический анализ) и горючесть (измерен кислородный индекс). Установлено, что введение спейсера в структуру олигомера приводит к получению полимеров со сбалансированными физико-механическими свойствами. Полимеры на основе фосфор(III)-содержащих олигоэфир(мет)акрилатов со спейсерами обладают значительно большим сопротивлением ударным нагрузкам.

Выводы. Достигнутые результаты свидетельствуют о возможности получения полимеров на основе фосфор(III)-содержащих олигоэфир(мет)акрилатов со спейсером, обладающих повышенным сопротивлением ударным нагрузкам и термостойкостью при незначительном снижении их горючести.

Ключевые слова

спейсер, фосфорсодержащие полимеры, динамический механический анализ, кислородный индекс, сопряженная модель горения

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INTRODUCTION

A compendium of research into the synthesis of phosphorus-containing acrylates, including the characteristics of their curing and an assessment of the properties of the resulting materials can be found in [1]. Further development of the described direction is presented in the works [2–4]. However, a more recent round of research is associated with establishing the possibility of synthesizing monomers with spacers, which in turn determines the relevance of publishing a series of articles on this topic.

In a previous publication, the results of a study on the synthesis of spatially separated phosphorus-containing oligoester (met)acrylates (a phosphorus-containing polymerizable compound, PPC) were presented along with an evaluation of the effect of the spacer structure on the kinetics of their photo-curing [5].

The materials presented in the present article, which are a continuation of earlier research based on B.A. Buravov's dissertation[1], are devoted to the study of the physical and mechanical properties of cured phosphorus-containing oligomeric(meth)-acrylates with spacers based on trivalent phosphorus. The synthesized monomers are envisaged for use as raw materials for the manufacture of products having reduced flammability by laser stereolithography (LSL) and digital light processing (DLP), as well as 3D printing methods used e.g. in automotive, aviation and railway technology applications. In this connection, it is important to evaluate the effect of the spacer structure on the physicomechanical properties of cured materials in order to predict possible options for the use of end products. Therefore, the effect of the spacer and its structure on the properties of cured polymers was evaluated using the dynamic mechanical analysis (DMA) method.

The present study investigates the temperature variation of the complex dynamic mechanical modulus of phosphorus(III)-based polymers containing oligoester methacrylates. The influence of the spacer structure on the modulus of accumulation of mechanical losses is estimated along with the damping factor of polymers. As a rule, these results on temperature changes in the plastic-elastic properties of the material allow us to get an idea of the structure of the formed polymer and assess the boundary conditions of its application. The establishment of these areas has been facilitated by the implementation of Cole–Cole diagrams, a method that has been instrumental in the analysis of the balanced properties of cured polymers.

EXPERIMENTAL

The properties of polymers were studied using PPCs that differ in terms of their structure, in particular, concerning the presence of a spacer in the structure of the initial monomer. The chemical formulas of compounds PPC-1, PPC-2, and PPC-3 are shown in Fig. 1.

The structure of PPC-4 is similar to that of PPC-2 and PPC-3, but one of the functional ether methacrylate groups has been replaced with an allyl group. The synthesis of PPC-4 was carried out similarly to that of PPC-2 and PPC-3, but in this case, when loading the reagents, one mole of glycidyl methacrylate (GMA) out of the four used in the synthesis of PPC-3 and PPC-4 was replaced with one mole of allyl glycidyl ether [6].

The synthesized compounds are summarized in Fig. 2, which shows the differences in the structure of the compounds, as well as the types of compounds used in the synthesis to form a particular functionality in the product.

Polymer samples used later for testing were obtained in molds by curing PPCs in the presence of 0.5% by weight of the photoinitiator phenyl-bis(2,4,6-trimethylbenzoyl)-phosphine oxide (phenyl-bis(2,4,6-trimethylbenzoyl)-phosphine oxide (BAPO) under the influence of ultraviolet (UV) radiation in the UV Exposure Lab Chamber (*SPDI UV*, USA). Here, the radiation range of the mercury lamp is 200–400 nm, while the power is 62 W/cm^2 at a curing time in the chamber of 25 min. The exposure time of the samples following extraction from the molds is 10 min.

UV-cured polymer samples were subjected to sol–gel analysis by fluid extraction with supercritical carbon dioxide (using a supercritical fluid extractor SFT-110XW, *Supercritical Fluid Technologies, Inc.*, USA). The results of the analysis indicate almost complete binding of monomers during polymerization (Table 1). The degree of curing was 100.0, 99.4, 98.0% for PPC-1, PPC-2, and PPC-3, respectively.

Table 1. Sol–gel analysis results

Samples	Gel fraction content, wt %	
	At 18°C	At 40°C
PPC-1	100.0	–
PPC-2	99.4	99.98
PPC-3	98.0	100.00

Note: the ratio of PPC/BAPO = 99.5 : 0.5 wt %. Conditions: pressure is 1300 psi, time is 3 h.

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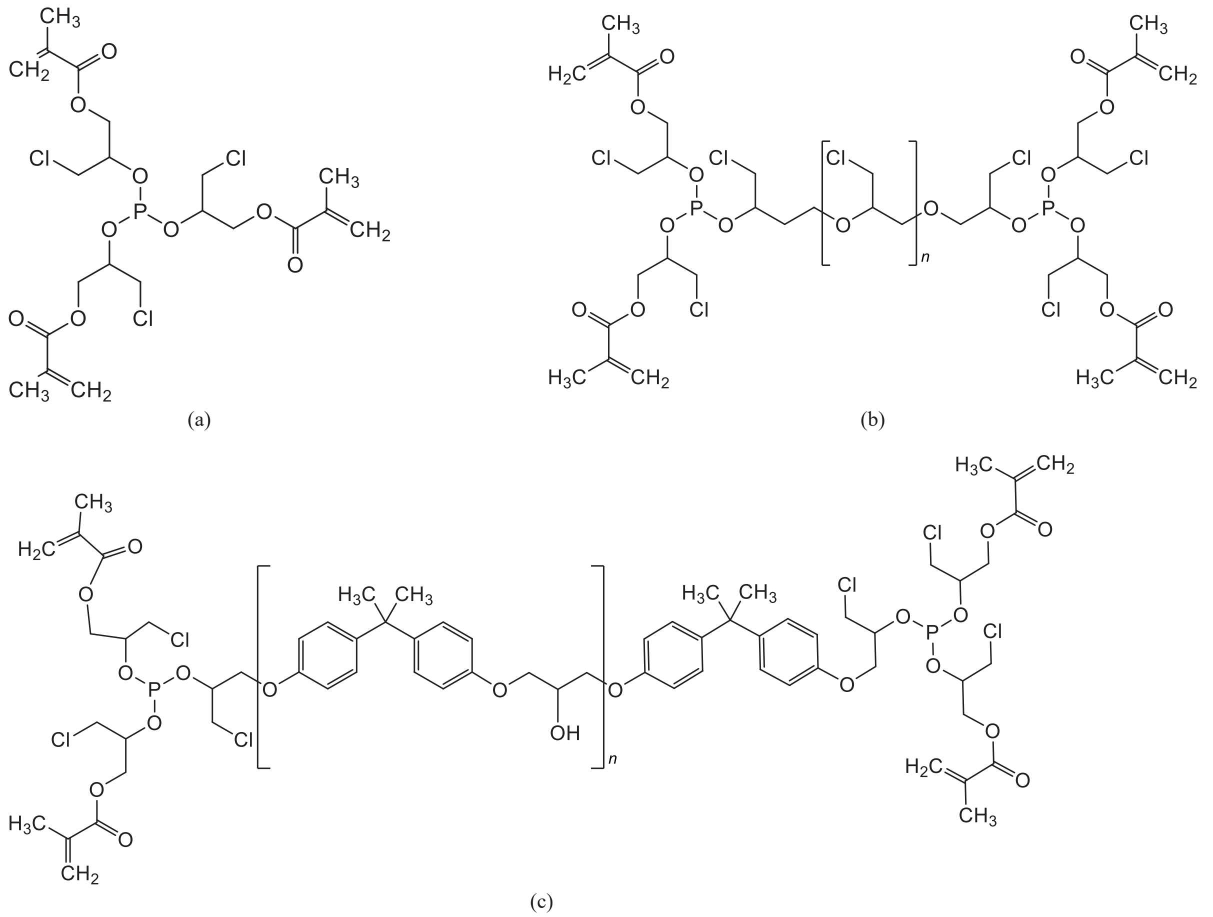
[1] Buravov B.A. Synthesis and properties of polymerization-capable phosphorus-containing oligomers with a spacer in the structure. Cand. Sci. Thesis (Chem.). Volgograd; 2020, 161 p. (In Russ).



(a) (b)

(c)

Fig. 1. Structure of (a) PPC-1; (b) PPC-2; (c) PPC-3

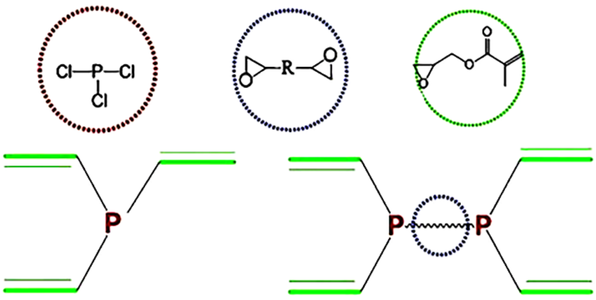


Fig. 2. Generalized structural chemical formulas and the starting reagents for their production: (a) PPC-1, without spacer; (b) PPC-2 and PPC-3, with the different spacer structure

The results of the sol–gel analysis indicate that the presence of spacer in the structure of the initial compound practically does not limit the conversion of the oligomeric methacrylate fragment into a spatially cross-linked material.

The study of the effect of the spacer and its structure on the properties of cured polymers was carried out under dynamic loading in accordance with GOST R 57916-2017[2] using the DMA method on a DMA 242 E Artemis device (*NETZSCH*, Germany) under double-arm bending conditions using the equipment and techniques supplied to the device.

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[2] GOST R 57916-2017 (ISO 6721-5:1996). National Standard of the Russian Federation. Plastics. Determination of dynamic mechanical properties. Part 5. Flexural vibration. Non-resonance method. Moscow: Standartinform; 2017 (in Russ.).

RESULTS AND DISCUSSION

Dynamic flexural modulus of elasticity (E')

The dynamic modulus of elasticity (E') is a storage modulus that represents the energy stored in a sample under sinusoidally applied load. According to GOST R 56801-2015[3], the modulus of elasticity, which is proportional to the maximum energy stored during the loading cycle, serves as a measure of the stiffness of a viscoelastic material. The temperature dependence of changes in the stiffness of the studied cured polymers with and without different spacer structures is shown in Fig. 3. The summarized data is presented in Table 2. The analysis of the above data makes it possible to compare the rigidity and degree of structuring of the polymer matrix [7, 8].

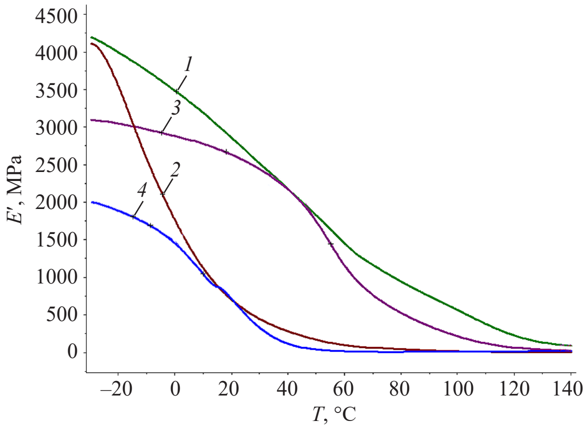

Fig. 3. Temperature dependence of the modulus of elasticity E': (*1*) PPC-1; (*2*) PPC-2; (*3*) PPC-3; (*4*) PPC-4

The elastic modulus of all materials studied has three temperature-dependent areas:

- low-temperature area of the vitreous state;
- temperature area of the transition period, i.e., the area of rapid relaxation processes in the polymer matrix under the influence of applied loads, indicating that the material is in a transitional glassy-viscoelastic state;
- high-temperature area representing a plateau of highly elastic state of the material.

It can be seen from the presented data that the PPC-1 sample having the highest rigidity (E' = 4200 MPa*)* at a temperature of −25°C is the one whose monomer does not contain a spacer in the structure. The RPC-2 sample containing an aliphatic spacer (product residue E-181) is characterized by a slightly lower initial hardness value (E' = 4120 MPa). The PPC3 polymer, containing an aromatic spacer (ED-20 epoxy residue), has even lower rigidity (E' = 3090 MPa). A significantly lower initial stiffness value (E' = 2000 MPa) is obtained in the cured product sample (PPC-4) with an aromatic spacer in which one of the four methacrylate groups is replaced by an allylic one. This group, which is characterized by a lower reactivity compared to methacrylates, probably does not fully enter into photopolymerization reactions that act as a defective group, which ultimately leads to a decrease in crosslinking density. The compound PPC-4 (Fig. 4) was additionally synthesized by us under conditions similar to the preparation of PPC-1, PPC-2, and PPC-3 to identify the effect of defect formation in the structure of crosslinked polymers.

Table 2. Results of dynamic mechanical analysis of structured samples

Parameter	PPC-1	PPC-2	PPC-3	PPC-4
E'_{max}, MPa	4200	4120	3090	2000
E'_{min}, MPa	88	12	30	9
$T_{E_{min}}$, °C	140	70	140	120
T_g, °C, E'_{max}	−5	−20	35	0.0
E_a, kJ/mol	283.0	568.8	283.7	203.4

Note: E' is the dynamic modulus of elasticity under bending; E'_{max}, E'_{min} is the maximum and minimum value of the dynamic modulus of elasticity under bending; $T_{E_{min}}$ is the temperature at the minimum value of the modulus of elasticity under bending; T_g, °C, E'_{max} is the glass transition temperature determined at the maximum temperature of the dynamic modulus of elasticity; E_a is the activation energy of the viscous flow.

We should note significant differences in the response of material properties to the effect of temperature (Fig. 3). The sample without a spacer (curve *1*) probably has a homogeneous globular supramolecular structure, which determines a monotonous decrease in the value of the dynamic modulus of elasticity in the temperature range from −25 to +114°C. At the same time, the slight inflection in the region of 60°C is probably due to the release of interglobular bonds of the cured material, which differ little in terms of structure and chemical nature of the formed chemical bonds inside the globules.

The introduction of a soft spacer into the structure of the PPC-2 polymer, consisting of an aliphatic low-molecular-weight unit with oxygen in its main chain (Fig. 3, curve *3*), has practically no effect on the initial value of the dynamic modulus of elasticity compared

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[3] GOST R 56801-2015 (ISO 6721-1:2011). National Standard of the Russian Federation. Plastics. Determination of dynamic mechanical properties. Part 1. General principles. Moscow: Standartinform; 2016 (in Russ.).

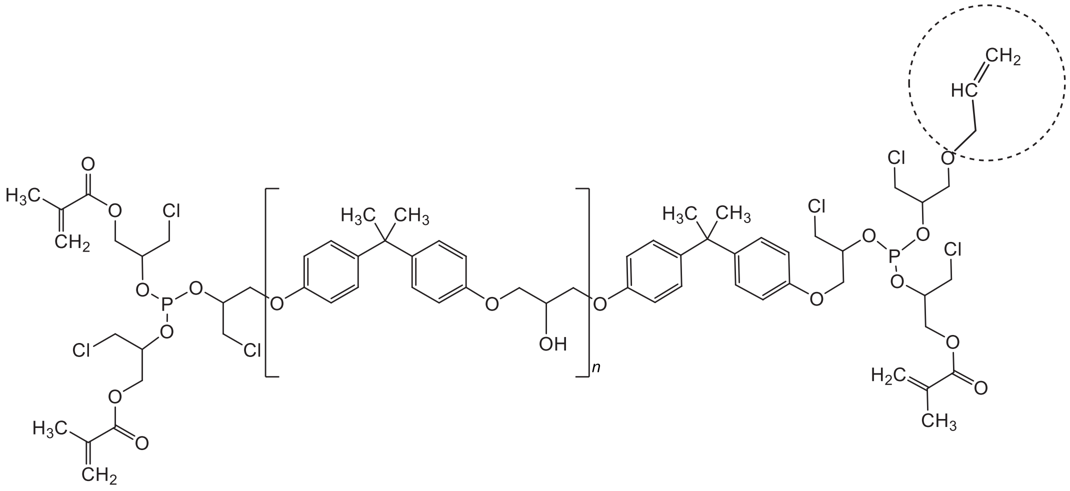

Fig. 4. Structure of PPC-4

with the PPC-1 sample without a spacer (Fig. 3, curve *1*). However, with a relatively small increase in temperature, the soft spacer exerts a plasticizing effect, which manifests itself in a fairly active and significant decrease in the dynamic modulus of elasticity of the PPC-2 sample without any observed transient phenomena [9], as is observed in other samples (PPC-1, PPC-3, PPC-4).

The dependence of the dynamic modulus of elasticity on the temperature of a polymer containing a volumetric hard aromatic spacer (PPC-3), a residue from ED-20 epoxy resin, has a pronounced bend in the range of 40°C (Fig. 3, curve *2*), which can be explained by an increase in the mobility of the globules of the formed matrix of the crosslinked polymer connected to each other an aromatic structure spacer. Accordingly, the E' value of the PPC-3 polymer matrix at temperatures from −25 to +40°C is determined by the combined effect of the spacer structure and the crosslinked globular structure of the polymer. An increased effect of the presence of a spacer above the specified temperature is expressed by a significant decrease in E' compared to the indicator of the PPC-1 polymer without a spacer.

The replacement of one of the four methacrylate fragments with a less reactive allylic fragment (Fig. 4, dotted line) significantly affects the elastic properties of the polymer matrix at low temperatures from −30 to +20°C. This effect, which is determined by a lower conversion of allyl groups during their polymerization, is confirmed by calculations of the crosslinking density shown below (Table 4). The introduction of a less reactive group leads to a decrease in the stiffness of the globules and consequent reduction in the initial values of the modulus of elasticity to 2000 MPa at −25°C, which shifts the transition state characteristic of PPC-3 to 0°C.

This assumption is confirmed by the values of the activation energy E_a of the viscous flow of polymers, which are determined by the results of DMA studies (Table 2). PPC-1 and PPC-3 have almost identical values of activation energies (283.0 and 283.7 kJ/mol, respectively). The increased activation energy of the viscous flow of PPC-2, which is almost twice as high as that of PPC-1 and PPC-3, can probably be explained by the flexibility of the spacer containing articulated oxygen in the main chain and presence of a polar substituent in the side chain [10].

For the PPC-4 polymer, there is a decrease in the activation energy from 283.7 to 203.4 kJ/mol, which indicates a significant decrease in the packing density of the globules of the resulting material. This was confirmed by calculations of the total crosslinking density of cured polymers (Table 4).

Dynamic loss modulus (E'')

The dynamic loss modulus (E'') is the energy dissipated as heat during the test cycle, and is determined by the area under the temperature dependence curve (Fig. 5) [11]. In fact, this is a characteristic of the mechanism of diffuse relaxation.

The investigated temperature dependence of the loss modulus of samples with spacers of different structures contained in the initial products retains the tendency of stiffness changes discussed above. Table 3 summarizes the research results.

The graphical and tabular data show the effect of the spacer in the polymer matrix on the value of the loss modulus and the temperature change of its maximum, which characterize the structural changes of the polymer.

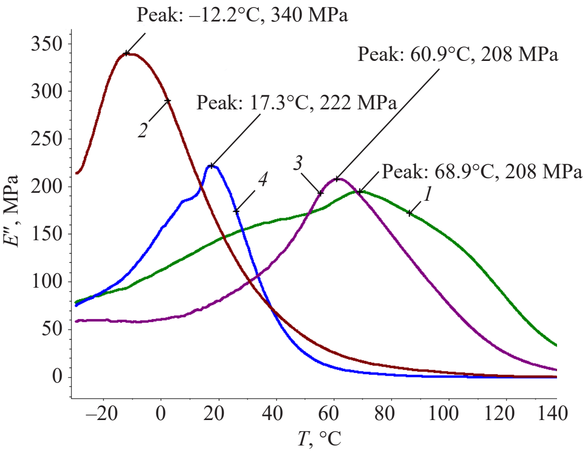

Fig. 5. Temperature dependence of the dynamic loss modulus E'': (*1*) PPC-1; (*2*) PPC-2; (*3*) PPC-3; (*4*) PPC-4

Table 3. Summary data by loss modulus

Parameter	PPC-1	PPC-2	PPC-3	PPC-4
$E''_{\max}$ MPa	195	340	208	222
T_g, °C, $E''_{\max}$	69	−12	60	17

The results obtained indicate that the polymer PPC-1, which does not have a spacer, has minimal dissipation of loading energy, characterized by a high packing density of polymer chains of the methacrylate fragment. Polymers having a volumetric aromatic spacer (PPC-3 and PPC-4) have relatively little difference in $E''_{\max}$ value. The presence of an aliphatic spacer in PPC-2 significantly increases the amount of stress energy dissipation during bending (E'' = 340 MPa) at sufficiently low temperatures characterized by a phase transition at lower temperatures ($T_g = -12$°C), which is probably due to the plasticizing effect of the mobile aliphatic spacer.

It should be noted that the shape of the temperature dependence of the loss modulus of the spacer samples differs significantly from the shape of the temperature dependence of the loss modulus of the comparison sample. The dependence of the loss modulus of PPC-1 has the form of a large halo over a wide temperature range. This behavior of the material can be explained in terms of a set of structures having a wide range of molecular weight distribution of polymer globules, as well as close values of strained interglobular and intraglobular chemical bonds, which are comparable both in terms of nature and strength.

The introduction of a low-reactive allyl group (PPC-4) has a significant effect on the properties of the material. As compared to PPC-3, the temperature of the maximum loss modulus decreases from 60 to 17°C, while the value of E'' increases from 208 to 222 MPa.

Thus, the introduction of spacers into the oligomer structure has a significant effect on the loss modulus. Polymer globules without a spacer have low mobility and consequently reduced tendency to dissipate energy from the applied load, which ultimately should make the material brittle. The presence of an aliphatic spacer, which likely acts as a soft crosslinking between the resulting high-molecular globules, significantly increases the loss modulus due to the plasticizing effect. The presence of a hard aromatic spacer exerts a negligible influence on the value of the indicator, concurrently constraining the temperature range of its maximum. This phenomenon can be elucidated by the diminished values of the molecular mass distribution of interconnected globular structures, which are circumscribed by the size of the monomer molecule.

Damping factor ($tg\delta_f$)

The magnitude of the values of the damping coefficient $tg\delta_f$ (damping factor) is an indicator of the relationship between the formed polymer phases of the material. The interaction, adhesion (penetration) between the phases, including their compatibility, as well as the effect of the curing method of the composite material, can be determined by the temperature dependencies of the indicator, whose maximum is used to determine the temperature (T_g) of the transition of the material to a viscoplastic state [12].

Figure 6 shows the temperature dependencies of the damping coefficient $tg\delta_f$. From the presented data, it can be seen that $tg\delta_f$ takes on large values with increasing temperature to reach a maximum in the region of the phase transition to the viscoelastic state.

It should be noted that $tg\delta_f$ has lower values at temperatures below T_g for all samples, since the segments of the chain of the cured material are not in a labile, but in a sedentary state. As the temperature increases, the chain segments, while maintaining the overall structure, become more mobile, which leads to an increase in the values of the indicator. Thus, the higher the extreme value of $tg\delta_f$, the higher the degree of molecular mobility [12].

Comparing the maximum values of $tg\delta_f$ of the studied samples of cured polymers based on PPC-1 (0.376) and PPC-3 (0.381), it is possible to make an assumption about their almost equal molecular mobility and relatively close values of phase transition temperatures (127 and 114°C, respectively). Considering the structure of the compounds, it can be stated that the aromatic spacer is inactive in polymer structures, which provides them with rigidity by limiting the mobility of globular segments [13].

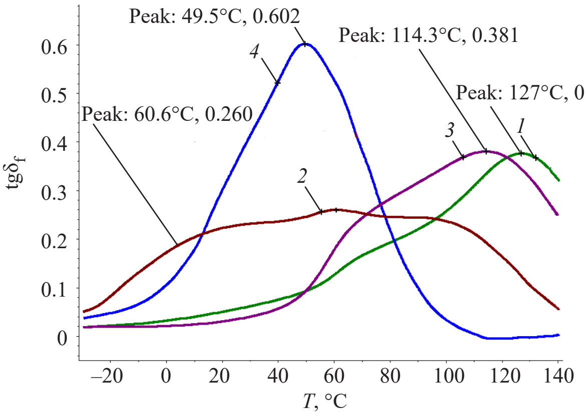

Fig. 6. Temperature dependence of $tg\delta_f$ structured phosphorus-containing oligomers: (*1*) PPC-1; (*2*) PPC-2; (*3*) PPC-3; (*4*) PPC-4

The temperature dependence of the PPC-4 damping coefficient, which is characterized by a lower crosslinking density (Fig. 6), has the maximum indicator value (0.6) compared to the others; this maximum manifests at a temperature of 50°C, which is 77 and 65°C lower than that of the samples PPC-1 and PPC-3, respectively. This may be attributed to the lower density of intra-globular PPC-4 crosslinking.

The crosslinking density [14, 15] was calculated according to the formula (1):

$$\nu = \frac{E'_r}{3 \cdot R \cdot T_r}, \tag{1}$$

where ν is the crosslinking density, mol/m^3; E'_r is the value of the dynamic modulus of elasticity under bending E' (J) at T_g + 30°C, the index r means the rubbery (elastic) state/area; T_r is the absolute temperature corresponding to T_g + 30°C; R is the universal gas constant, J/(K·mol).

The results of calculating the total crosslinking density are presented in Table 4.

Table 4. Summary data on the damping factor

Parameter	PPC-1	PPC-2	PPC-3	PPC-4
$tg\delta_{f\,max}$,	0.38	0.26	0.38	0.60
T_g, °C, $tg\delta_f$	127	60	114	50
ν, mol/m^3	15473	10129	8018	4510

The temperature dependence of the PPC-2 damping coefficient has a rather blurred halo over a wide temperature range (from −30 to +140°C), while it practically does not change at temperatures from 20 to 110°C, having a slightly pronounced maximum at 60°C. This indicates the high viscoplastic properties of the polymer, which should provide high values of impact resistance as confirmed by additional studies (Fig. 7).

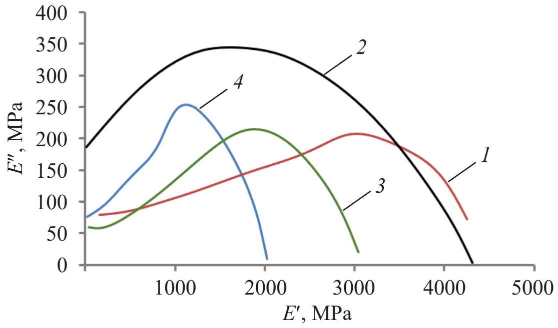

Fig. 7. Cole–Cole diagrams of UV-cured composites: (*1*) PPC-1; (*2*) PPC-2; (*3*) PPC-3; (*4*) PPC-4

The Cole–Cole diagram

Figure 7 shows the Cole–Cole graph, a tool for studying structural changes occurring in crosslinked polymers, which is important when comparing the effect of a spacer on the viscoelastic properties of polymer materials [16] since ultimately determining their physicomechanical properties. The semicircular character of the Cole–Cole graph, which is constructed in coordinates of the change in the loss modulus from the modulus of elasticity, can be used to characterize the degree of balance of the properties of the crosslinked polymer composite [17]. The imperfection of the semicircular shape indicates the structural heterogeneity of the formed material. On the other hand, the proximity of the shape of the dependence to the ideal sector of the circle indicates the balance of the structures composing the polymer matrix [14] over a wide temperature range. The summarized data is given in Table 5.

From the presented graphical data, it can be seen that the shape of the dependence of the loss modulus on the elastic modulus of a polymer containing no spacer is far from the shape of the ideal sector of a circle, whose maximum is shifted to the region of large values of the elastic modulus (Fig. 7, curve *1*). The introduction of a spacer of different structures brings the Cole–Cole diagram to a more perfect form (curves *2* and *3*), shifting the maximum of the loss modulus to the region of lower values of the elastic modulus close to its average value. Since the diagram characterizes the interaction of the structures of the formed crosslinked polymer, the introduction of a spacer can be concluded to leads to the formation of a more perfect structure [18] and consequent improvement in physicomechanical properties, including resistance to shock loads. This assumption was confirmed by further comparative tests of the studied materials (see below).

Table 5. Summary data obtained from the Cole–Cole diagram

Parameter	PPC-1	PPC-2	PPC-3	PPC-4
$E''_{\max}$ (Cole–Cole), MPa	206	344	214	252
E' at $E''_{\max}$ (Cole–Cole), MPa	3056	1612	1943	1125
$\frac{1}{2}E'_{\max}$, MPa	2100	2060	1545	1000

The replacement of one methacrylate group with an allyl group that is relatively less reactive to polymerization under the studied conditions [19] leads to a significant change in the Cole–Cole diagram (Fig. 7, curve *4*) and a decrease in characteristic parameters (Table 5). The shape of curve *4* deviates towards a less perfect one compared to the analogues of PPC-2 and PPC-3 (curves *2* and *3*), while the maximum loss modulus decreases and shifts to the region of lower values of the modulus of elasticity. This indicates an increase in the defect structure of the formed polymer matrix of the crosslinked material when a less reactive allyl group is introduced into the oligomer.

Summarizing the above and comparing the results of the DMA studies, we can conclude that the elastic properties of cured polymers are significantly affected by the introduction of a spacer into the oligomer structure. While the introduction of an aliphatic spacer leads to the formation of a material with increased elastic properties without changing the modulus of elasticity at low temperatures, it also provides greater globular mobility of the cured material. The introduction of an aromatic spacer leads to a decrease in the modulus of elasticity at relatively low temperatures, while maintaining the rigidity of the cured polymer at higher temperatures. In both cases, a more balanced structure of the cured polymer is formed.

The incorporation of low-reactive functional groups has been demonstrated to exert a substantial influence on the supramolecular structure of the material, consequently affecting the physicomechanical properties of the formed crosslinked matrix of the cured polymer.

The findings suggest that the incorporation of spacers within the structures of cured compounds facilitates the intentional fabrication of materials having predetermined viscoelastic properties, which is a process of particular significance for brittle phosphorus-containing materials.

Impact of spacer composition and morphology on a polymer material's physicomechanical properties, its heat, and fire resistance

In order to compare the results of DMA with the physicomechanical properties of polymers obtained in the presence of initiators of UV-cured materials based on PPC-1, PPC-2 and PPC-3, their strength under static bending, resistance to shock loads, heat resistance and fire resistance were investigated. In addition, decomposition models of PPC1, PPC-2, and PPC-3 and kinetic parameters of the ongoing macrostage reactions were obtained. The results of the conducted studies are shown in Figs. 8–10 and Tables 6–10.

Impact strength test results

The study of the effect of a spacer in the structure of oligomers on the impact resistance of polymers based on them was carried out using the incised iodine method in accordance with GOST 19109-2017 (ISO 180:2000)[4]. The obtained values of impact strength (Fig. 8) are fully consistent with the results of the conducted DMA studies.

It can be seen from the presented data that the cured polymer containing a soft aliphatic spacer (PPC-2) is characterized by higher values of impact resistance than the sample containing a hard spacer comprising the remainder of the ED-20 epoxy resin (PPC-3), which is probably due to the greater dissipation of impact energy by the moving part of the spacer.

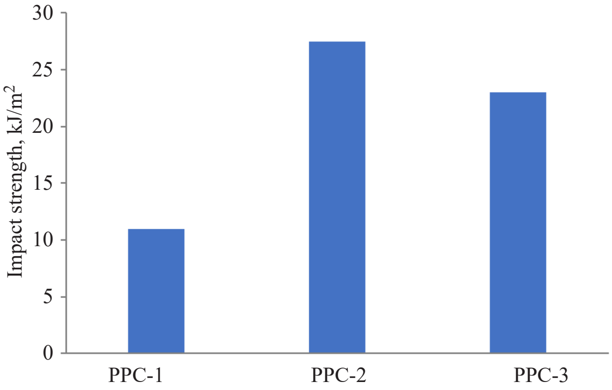

Fig. 8. Impact strength PPC1, PPC-2, and PPC-3

The introduction of spacers into the structure of oligomers is shown to increase the impact strength of cured polymers by 2.5–3 times, with the soft spacer having a more significant effect. This fact is in

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[4] GOST 19109-2017 (ISO 180:2000). Interstate Standard. Plastics. Method for determination of Izod impact strength. Moscow: Standartinform; 2018 (in Russ.).

good agreement with the results of the DMA studies presented above.

The values of the modulus of elasticity determined in the study of the resistance of materials to static bending showed compliance with the DMA studies (Table 6) subject to consideration of the compliance of the temperature of the physicomechanical tests.

Table 6. Resistance to bending stress

Parameter	PPC-1	PPC-2	PPC-3
E, GPa	2.57	0.49	1.92
dl at fracture, %	2.6	8.4	2.4
$F_{break.}$, MPa	60	29.5	40.5

Note: E is Young's modulus; dl is elongation at fracture, %; F_{break} is breaking load, MPa.

RRS-1, which is characterized by a maximum value of Young's modulus E at bending (2.57 GPa), collapses with significantly greater applied forces (60 MPa) at the lowest deflection value (2.6%). The introduction of a soft spacer (PPC-2) leads to a decrease in the Young's modulus to 0.45 GPa (more than 5.5 times), a decrease in destructive loading to 29.5 MPa; at the same time, deflection increases by more than 3 times (8.4%) during fracture. The introduction of a hard spacer into the structure of the material (PPC-3) is accompanied by a decrease in its Young's modulus to 1.92 GPa with a 30% decrease in fracture stress and increased deflection values (3.4%).

The results confirm that the plastic properties of cured materials can be significantly enhanced by the introduction of spacers. Thermoplastic properties of polymers determined by the Vicat method according to GOST 15088-2014[5] (Table 7) also indicate an increase in the viscoplastic properties of cured samples containing a spacer in the oligomer structure. Increasing the load does not significantly change the softening temperature of PPC-1, while a decrease in softening temperatures is observed with increasing load for samples with a spacer (PPC-2 and PPC-3).

The results obtained indicate that the introduction of a spacer into the oligomer structure makes it possible to obtain a polymer material with enhanced viscoplastic properties, which is not typical for phosphorus-containing polymer materials and usually restricts their use in compounds.

Table 7. Vicat softening temperature

Test conditions	PPC-1	PPC-2	PPC-3
	Softening point, °C		
Load 10H	231	242 (0.5 mm)*	243
Load 50H	228	170	121

* Immersion of the indenter 0.5 mm into the sample at a temperature of 242°C.

Thermal stability

Considering that phosphorus-containing oligoester methacrylates should have a reduced flammability, it is of interest to evaluate the effect of the spacer on the thermal stability and flammability of polymers.

The thermal stability of the cured samples was evaluated using a Paulic–Paulic–Erdey derivatograph Q–1500 D (*IOM*, Hungary). The research results are shown in Fig. 9 and in Table 8. It can be seen that the samples differ in terms of heat resistance, the dynamics of the destruction process, and the amount of coke residue at 400°C. The presence of an aliphatic spacer in the PPC-2 sample slightly reduces the temperature of the beginning of decomposition of the material (~140°C) compared with PPC-1 (~145°C). The increased temperature of the onset of decomposition to ~190°C caused by the introduction of an aromatic spacer into the structure of the oligomer (PPC-3) indicates an increase in its heat resistance.

It is noted that the thermal degradation of PPC-1 and PPC-2 polymers proceeds by similar mechanisms. This is evidenced by similar dynamic processes occurring in the first stages of the destruction of materials, with the initiation of the process at temperatures of 140–150°C. A similar stage for the PPC-3 sample begins only at 160°C. In this case, the peak of the maximum decay occurs at 320°C, whereas for samples of PPC-1 and PPC-2 it occurs at 260°C. The observed fact indicates an increase in the thermal stability of the polymer when an aromatic spacer is introduced into it.

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[5] GOST 15088-2014. Interstate Standard. Plastics. Thermoplastic materials. Determination of Vicat softening temperature (VST). Moscow: Standartinform; 2014 (in Russ.).

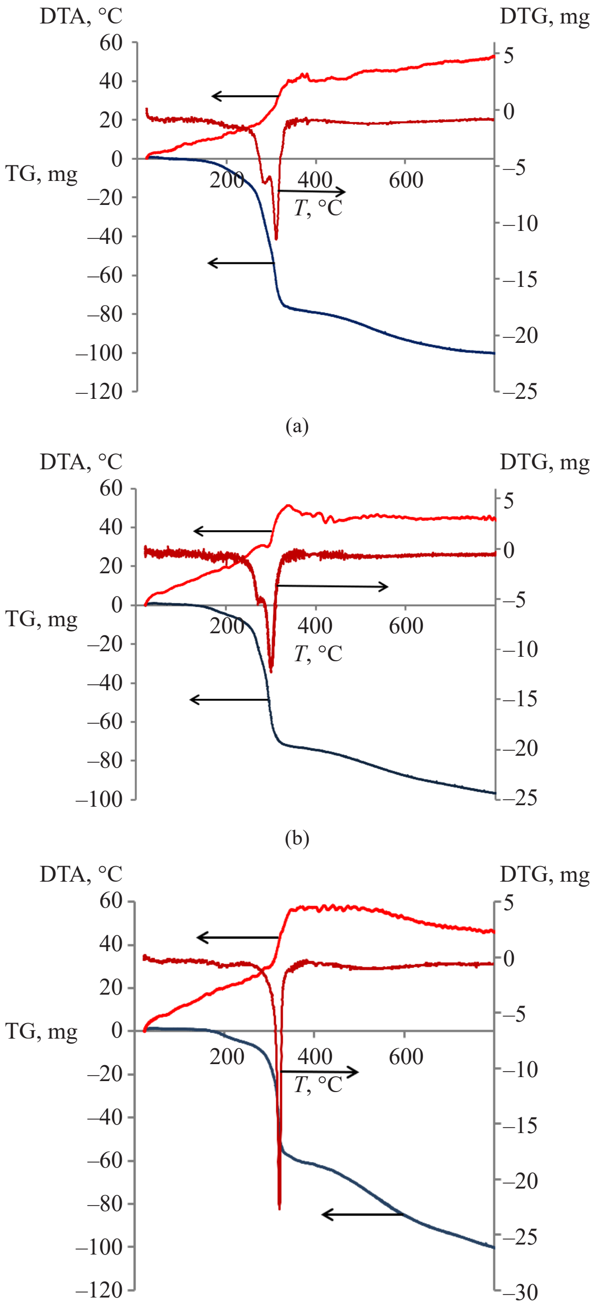

Fig. 9. Derivatograms of cured materials: (a) PPC-1; (b) PPC-2; (c) PPC-3. Heating rate 10°C/min. DTA — differential thermal analysis, °C; TG — thermogravimetry, mg; DTG — derivative thermogravimetry, mg/min

Table 8. Results of derivatographic studies

Samples	$T_{initial}$, °C	T_5, °C	T_{10}, °C	T_{50}, °C	Coke at 400°C, wt %
PPC-1	150	200	236	305	29
PPC-2	140	207	248	300	34
PPC-3	190	249	289	330	44

Note: $T_{initial}$ is the temperature of the beginning of destruction; T_5 is the temperature of 5% mass loss; T_{10} is the temperature of 10% mass loss; T_{50} is the temperature of 50% mass loss.

Table 9. Scheme of the polymer degradation model of PPC-1, PPC-2, PPC-3 and kinetic parameters of the proceeding reactions of macrostages

Reaction	*A*, 1/s	E_a, kJ/mol	*n*	Comment	Range *T*, °C
PPC-1					
RPC-1 → a-liquid	–	–	–	Phase change	~140–300
a-liquid → 0.84 b-liquid + gas	$6\cdot10^{12}$	156	0.32	Pyrolysis and evaporation	215–290
b-liquid → 0.29 c-liquid + gas	$1\cdot10^{13}$	168	0.42	Pyrolysis and evaporation	240–325
c-liquid → 0.1 a-char + gas	$1\cdot10^{2}$	75	1.1	Degradation	330–685
PPC-2					
PPC-2 → 0.98 a-liquid + gas	$5\cdot10^{6}$	77.7	2.96	Phase change	120–300
a-liquid → 0.82 b-liquid + gas	$1\cdot10^{13}$	155	0.3	Pyrolysis and evaporation	217–275
b-liquid → 0.23 c-liquid + gas	$1\cdot10^{15}$	186	0.65	Pyrolysis and evaporation	250–315
c-liquid → 0.1 char + gas	–	–	–	Degradation	~400–600
PPC-3					
PPC3 → a-liquid	–	–	–	Phase change	~150–250
a-liquid → 0.44 a-char + gas	$5\cdot10^{8}$	124	0.03	Evaporation	250–325
a-char → 0.1 b-char + gas	$8.6\cdot10^{4}$	115	2.89	Degradation	430–700

It should be noted that the thermal degradation of the PPC-3 polymer, which is characterized by a single stage without the formation of intermediate intermediates, probably occurs due to a more thermostable aromatic spacer. The greater heat resistance of PPC-3 is also evidenced by the amount of coke residue formed at a temperature of 400°C and amounting to 44% of the initial mass. For samples PPC-1 and PPC-2, this indicator corresponds to 29 and 34 wt %, respectively.

The study of thermal decomposition processes of PPC1, PPC-2, and PPC-3 at temperatures up to 800°C was carried out according to thermogravimetric analysis using the Reaxfire/gpyro program (2023)[6] within the framework of a decomposition model followed by the release of decomposition products into the gas phase (evaporation/sublimation). The scheme of the PPF decomposition model and the kinetic parameters of the ongoing macrostage reactions are presented in Table 9. The model considered four (for PPC-1 and PPC-2) and three (for PPC-3) sequential reactions (macrostades) of thermal decomposition of a pseudocomponent (in Table 9 they are designated a-, b-, c-liquid) along with the formation of new unidentified pseudocomponents. In this model, liquid can be understood as the solid state of a pseudocomponent that is consumed without

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[6] Generalized model of pyrolysis of combustible solids. URL: https://github.com/reaxfire/gpyro. Accessed July 07, 2024.

forming a liquid layer (sublimation). Figure 10 shows the nature of the main peaks of the reaction rates of the second and third stages of the decomposition of PPC, comprising an acute peak and a sharp front of the termination of the reaction.

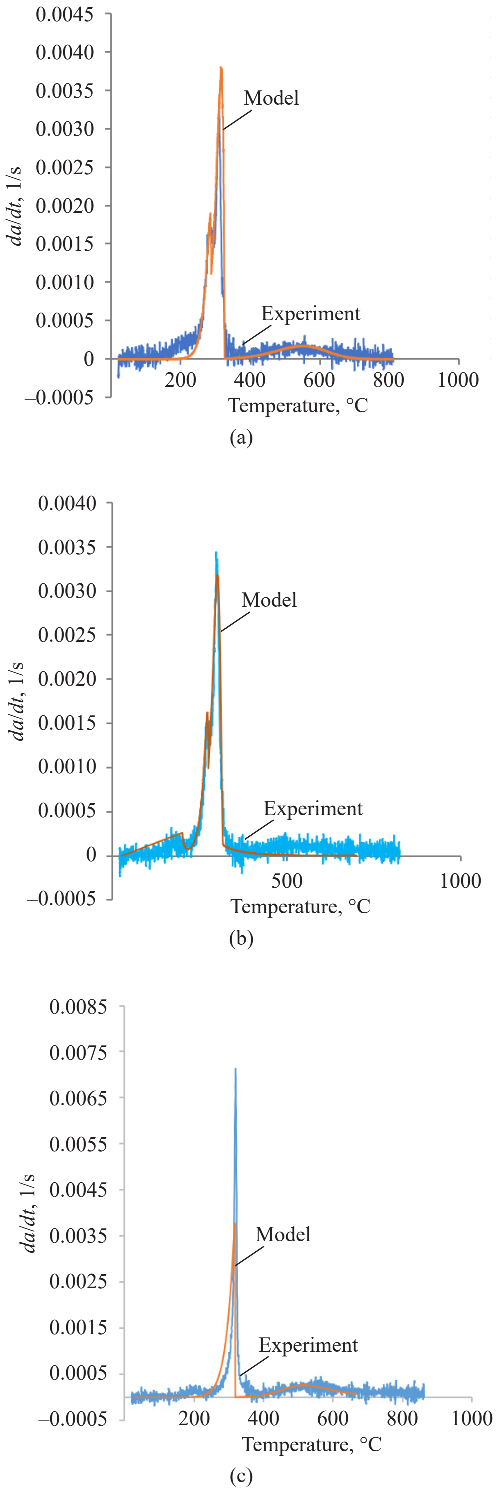

Fig. 10. Comparison of experimental data and simulation of thermal degradation kinetics:
(a) PPC-1; (b) PPC-2; (c) PPC-3

The simulation results satisfactorly describe the experimental data presented in Fig. 10. The reaction rate is represented as:

$$\frac{da}{dt} = k \cdot (1 - a)^n, \tag{2}$$

where a is the fraction of the product formed; n is the reaction order; the reaction rate constant k is represented in Arrhenius equation $k = A \cdot \exp(-E_a / RT)$ (A is the preexponent, E_a is the activation energy of the reaction).

From formula (2), it can be seen that weakly concentration-dependent reactions occur when the reaction index is fractional (n = 0–1). An example is the thermal decomposition of minerals [20]. At $n = 0$, evaporation occurs, while at $n = 1$, a reaction involving one component occurs. When $n > 1$, reactions involving several components occur, as is often observed during the thermal decomposition of polymers [21]. Kinetic parameters for the first stage of decomposition of PPC-1 and PPC-3, which proceed at a low rate, have not been determined. The first stage, according to the proposed model, is associated with a change in the phase of matter and a small gas output (mass loss). It can be assumed that the first stage is associated with the transition of the polymer from a solid state to a softened one, in which the process of surface destruction of the interglobular region takes place. In the case of PPC-1 and PPC-2, the second and third stages are characterized by significant weight loss (60 wt %). In the framework of the proposed model, this is most likely due to the decomposition of the polymer and the formation of a monomer (for example, methyl methacrylate), which enters the gas phase without decomposition (evaporation) and involving partial decomposition of the spacer. This assumption is based on the low values of the reaction index for PPC-1 and PPC-2 (0.32–0.65). In the case of PPC-3 in the second stage, $n = 0.03$ is very close to zero, i.e., the main process of mass loss is evaporation.

The obtained kinetic data can be used to create a combustion model for the samples studied.

Determination of the flammability of cured phosphorus(III)-containing polymers

The thermal decomposition curves PPC-1 and PPC-2 (Fig. 10), showing the release of combustible products into the gas phase and the process of flame propagation, are close to each other and thus should not affect the values of the limiting oxygen index (LOI). However, with approximately the same phosphorus content, the chlorine content in PPC-1, which is a halogen and acts as a flame retardant, is significantly higher (1.45 times) than in PPC-2, which reduces the LOI for PPC-2 from 28 to 24 units. The amount of phosphorus and chlorine in the PPC-2 and PPC-3 polymers is very close, but the

Table 10. Results of flammability studies of photocured polymers

System components		Wt %		LOI, %
		P	Cl	
PPC-1	BAPO (0.5%)	5.5	18.7	28.0
PPC-2	BAPO (0.5%)	5.7	12.9	24.0
PPC-3	BAPO (0.5%)	5.2	11.8	27.0

LOI of PPC-3 is 3 units higher than that of PPC-2, which can be explained by the higher thermal stability of the aromatic spacer in PPC-3 (the maximum decomposition temperature of PPC-3 is 20°C higher than that of PPC-2) and smaller quantity of gases released (the remaining gases are larger at 400°C).

The flammability of crosslinked polymers was assessed by the LOI determination method (GOST 21793-76[7]). The results are presented in Table 10.

It can be seen from the presented data that the polymer without a spacer has the maximum LOI value. Samples with spacers have slightly lower LOI values.

Thus, it was found that the introduction of a spacer into the oligomer structure has a significant effect on the course of thermal degradation of cured polymers. The introduction of an aromatic spacer into the oligomer structure significantly increases the thermal stability of polymers. The presence of an aliphatic spacer in the structure of the oligomer reduces the temperature of the beginning of decomposition of the material, but leads to an increase in the coke residue in the end compared with the PPC-1 polymer.

CONCLUSIONS

It has been established by DMA that the introduction of an aliphatic spacer is accompanied by a decrease in the glass transition temperature of polymers from −5 to −20°C, the introduction of an aromatic spacer increases the glass transition temperature of the polymer to 35°C. The aliphatic spacer reduces the transition temperature to the viscoelastic state by 67°C, and the aromatic state by 13°C. The impact strength of the samples with spacers in the structure is 2.5–3 times higher than the impact strength of the reference sample without a spacer. Polymers with an aliphatic spacer have the most balanced properties, as confirmed by the Cole–Cole diagrams.

Derivatographic studies have established that the aliphatic spacer in the PPC structure increases the temperature of loss of 5% of the mass by 7°C, while the aromatic space leads to a corresponding loss by 49°C, as well as increasing the coke residue from 29 to 34 and 44 wt %, respectively. The LOI of polymers containing an aromatic spacer is 27, while that of the aliphatic spacer is 24.

Thus, it has been established that the introduction of spacers into the structure of phosphorus-containing polymerizable oligomers makes it possible to obtain polymers with reduced flammability and enhanced viscoelastic characteristics providing high resistance to shock loads.

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Authors' contributions

B.A. Buravov—experimental data processing, writing the text of the article.

A. Al-Hamzawi—consultation on experimental procedures.

R.B. Gadzhiev—literature analysis, formalization of the reference list.

S.A. Orlova—literature analysis, formalization of the reference list.

L.Yu. Donetskova—experiment execution, data analysis, data processing.

S.M. Solomakhin—experiment execution, data analysis, data processing.

S.V. Borisov—consultation on rheological properties of binders.

O.S. Fomenko—translation of the abstract into English.

S.A. Trubachev—experiment execution, data analysis, data processing.

A.A. Paletsky—experiment execution, data analysis, data processing.

A.G. Shmakov—experiment execution, data analysis, data processing.

O.I. Tuzhikov—consultation on chemistry of phosphorus-containing compounds, as well as planning, methodology, and implementation of research.

O.O. Tuzhikov—research idea, consultation on experimental procedures, writing the text of the article.

The authors declare no conflicts of interest.

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[7] GOST 21796-76. State Standard of the USSR. Plastics. Method for determination of the Oxygen Index. Moscow: Izdatelstvo standartov; 1976 (in Russ.).

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