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CROSS-LINKING OF EPOXY-OLIGOESTERIC MIXTURES IN THE PRESENCE OF FLUORINE-CONTAINING EPOXY RESINS

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Abstract. Cross-linking of epoxy-oligomeric mixtures based on ED-20 industrial epoxy dianic resin and TGM-3 oligoesteracrylate in the presence of fluorine-containing peroxy oligomers has been studied. Oligomers used for the cross-linking have been obtained *via* chemical modification of peroxy derivative of ED-24 epoxy resin with alcohols-telomers by general formula $HOCH_2–(CF_2–CF_2)_nH$, where *n* is 3 or 4. The thermal stability of peroxy groups in the initial material, fluorine-containing peroxy oligomers and stability of cross-linked films have been examined using derivatographic methods. The effect of temperature, process time and mixture composition on the gel-fraction content and hardness of obtained films has been established.

Keywords: cross-linking, epoxy, peroxy, fluorine-containing, alcohol-telomer, gel-fraction, hardness, films.

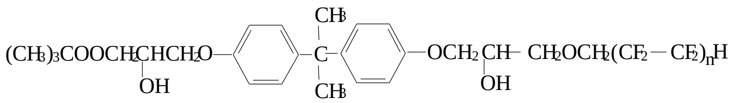
1. Introduction

It has been shown earlier [1] that (3-aminopropyl)triethoxysilane (APTES) may be used for the improving of properties of films based on the mixtures of ED-20 industrial epoxy resin, peroxy derivative of ED-20 epoxy resin and TGM-3 oligoesteracrylate. The films on the basis of the mentioned compounds are formed in the presence of polyethylenepolyamine (PEPA) stepwise, first at room temperature for 24 h and then at heating to 403 K. In the presence of APTES formed under the mentioned conditions the films are characterized by improved adhesion to the surface of silicate glass [1].

Peroxy derivative of ED-20 epoxy resin in such a mixture acts as a binding material for molecules of ED-20 epoxy resin and TGM-3 oligoesteracrylate. Moreover, peroxide molecule is added at room temperature using PEPA. The heating decomposes –O–O– bonds in the peroxide molecule forming free radicals which further initiate grafted polymerization of TGM-3 to the matrix. The latter is formed using molecules of ED-20 industrial epoxy resin and peroxy derivative of ED-20. Bonding of epoxy and oligoestracrylate molecules leads to the impossibility of oligoesteracrylate "bleeding" in the operational processes and in such a way preserves its operational characteristics [1]. APTES of formula $H_2N(CH_2)_3Si(OC_2H_5)_3$ attaches to the main matrix based on ED-20 epoxy resin due to the presence of amine group in its molecule and improves adhesion of polymeric films to the glass surface due to the presence of etoxysilyl groups. Thus it should be noted that properties of the mixtures based on the industrial epoxy resin are improved if all components in the mixture are chemically bounded between each other.

To improve the thermal properties of the films based on epoxy resins and their resistance to aggressive media some authors [2] use compounds with fluorine atoms.

On the basis of epoxy resins we synthesized oligomers containing fluorine atoms together with labile –O–O– bond [3-5]. The simultaneous presence of peroxy groups and fluorine atoms in the molecule of oligomers of general formula



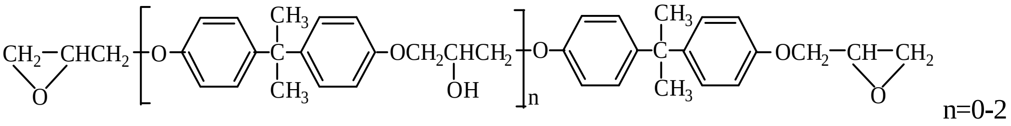
allows them to be a cross-linking agent (–O–O– bond) and modifier (fluorine atoms) in the cross-linking processes of epoxy-oligomeric mixtures.

This work deals with polymeric networks formed as a result of epoxy-oligoesteric mixtures cross-linking in the presence of fluorine-containing epoxy resins.

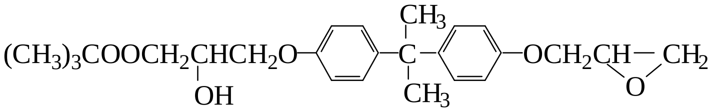
2. Experimental

2.1. Starting Reagents

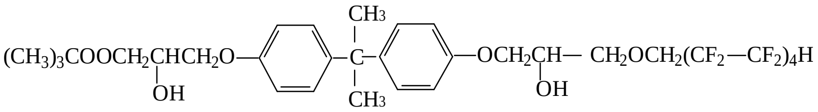
Ed-20 industrial epoxy dianic resin was used with molecular mass (M_n) of 390 g/mol and epoxy number (*e.n.*) of 20.0 %. Its formula is:



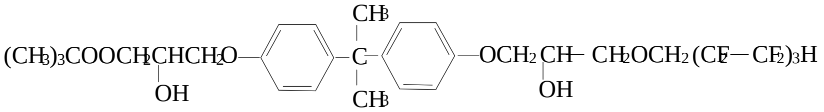
Peroxy derivative of ED-24 epoxy resin (PDER-24) was obtained according to the procedure described in [6]. Its M_n is 420 g/mol; *e.n.* is 12.0 % and active oxygen content ([O_{act}] is 3.5 %. The formula is as follows:



Fluorine-containing peroxy oligomer (FPO-I) and fluorine-containing peroxy oligomer (FPO-II) were synthesized as described in [4]. Their formulas are given below and their characteristics are given in Table 1.



FPO-I



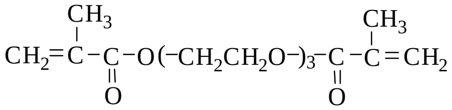
FPO-II

Table 1

FPO characteristics

FPO symbols	M_n, g/mol	[O_{act}], %	Yield, %
FPO-I	800	2.7	83.6
FPO-II	722	2.1	81.6

The molecular mass of oligoesteracrylate (TGM-3) is 280 g/mol. Its formula is:



$$CH_2{=}\overset{CH_3}{\overset{|}{C}}-\underset{\underset{O}{\|}}{C}-O(-CH_2CH_2O-)_3-\underset{\underset{O}{\|}}{C}-\overset{CH_3}{\overset{|}{C}}{=}CH_2$$

Polyethylene polyamine (PEPA) was used as received without additional purification.

2.2. Experimental Procedure

Thermographic investigations of oligomers were carried out using "Paulic-Paulic-Erdei" derivatograph Q-1500D in a dynamic mode in the air with heating rate of 2.5 K/min. The samples weight was 300 mg. The device sensitivity was 100 mcV according to the scale of differential-thermal analysis (DTA), 100 mg – to the thermogravimetric scale (TG) and 500 mcV – to the differential thermogravimetric scale (DTG). The samples were heated to 523 K. Alumina was used as a standard.

Thermographic investigations of composites were carried out using "Paulic-Paulic-Erdei" derivatograph Q-1500D in a dynamic mode in the air with heating rate of 10.0 K/min. The samples weight was 100 mg. Device sensitivity was 250 mcV according to the scale of differential-thermal analysis (DTA), 100 mg – to the thermogravimetric scale (TG) and 500 mcV – to the differential thermogravimetric scale (DTG). The samples were heated to 523 K. Alumina was used as a standard.

Epoxy-oligoesteric mixtures cross-linking. Composites were prepared by pouring of prepared mixture on the glass plates of standard size. The mixture was prepared by mixing of components with PEPA hardener and then cross-linked for 1–10 days at room temperature or at heating for 75 min. After cross-linking the films were examined at M-3 pendulum device using the procedure described in [8] to determine their hardness (*H*, rel. units). The gel-fraction content (*G*, %) was determined using extracting of grinded samples by acetone in Soxhlet apparatus for 14 h at heating [7].

3. Results and Discussion

3.1. Thermal Stability of Fluorine-Containing Peroxy Oligomers

Thermal stability of oligomers containing labile –O–O– bonds is an important characteristic essentially affecting the possibility of their application in the process of epoxy-oligoesteric mixtures cross-linking. Peroxy oligomers based on epoxy resins without fluorine atoms are capable of decomposition by –O–O– bonds [9] at 293–303 K. The oligomers synthesized by us were compared with known compounds based on epoxy resins, without fluorine atoms. The presence of fluorine atoms in the molecule of compound with –O–O– bond is supposed to affect thermal stability of peroxy groups. Information about temperature conditions for –O–O– decomposition in the molecule of fluorine-containing peroxide oligomers would provide the possibility of their correct application in the cross-linking of epoxy-oligoesteric mixtures.

Thermal stability of fluorine-containing peroxy oligomers was studied using "Paulic-Paulic-Erdei" derivatograph Q-1500D by the method described in Subsection 2. Oligomer FPO-II was chosen for the investigations. The curves obtained during the DTG and DTA analyses are represented in Fig. 1. Peroxy derivative of ED-24 epoxy resin (PDER-24) without fluorine atoms, which was the initial compound for the synthesis of FPO-II (Fig. 2), was used for the comparison.

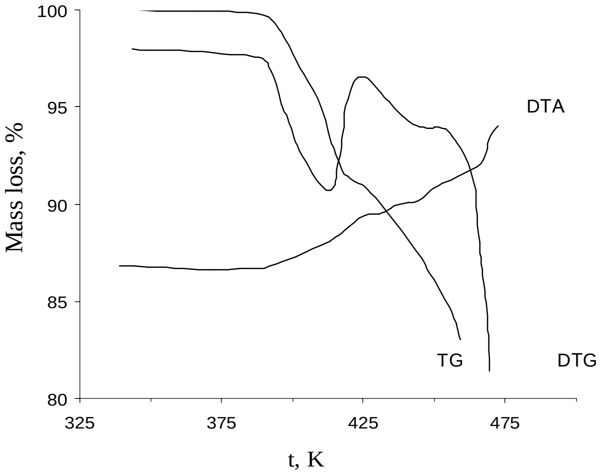


Fig. 1. FPO-II derivatographic curves

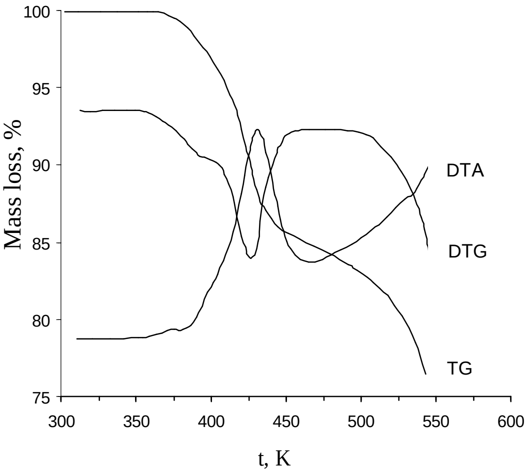
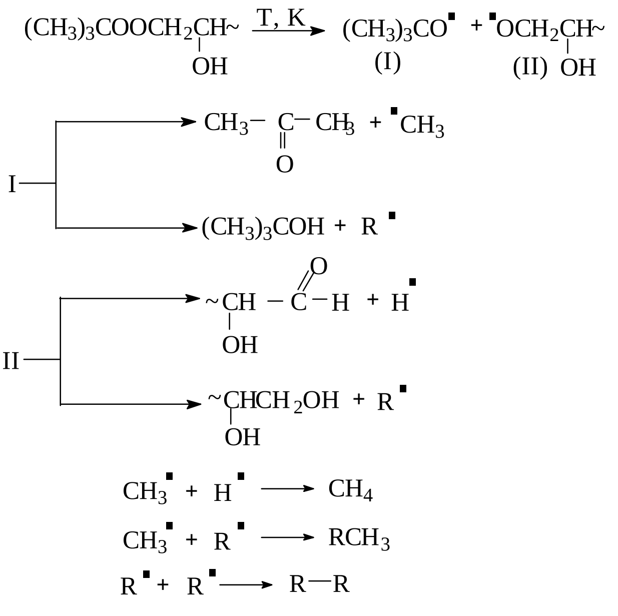


Fig. 2. PDER-24 derivatographic curves

We can see from Fig. 2 that the beginning of mass loss for PDER-24 takes place at 363 K. For FPO-II (Fig. 1) such beginning is observed at higher temperature (383 K). The obtained results show that the presence of fluorine atoms in the FPO-II molecule favors the stabilization of peroxy groups in the molecule of oligomer based on dianic epoxy resin. For PDER-24 the greatest intensity of mass loss is observed at 423 K (Fig. 2). At the same time for FPO-II (Fig. 1) there is no clear extreme at DTG curve. The mass loss of this oligomer becomes more intensive. To our mind, it means that the simultaneous presence of –O–O– bonds and fluorine atoms in the FPO-II molecule leads to the complex radical processes which gradually transfer to thermal destruction of the whole sample.

Analyzing TG curve (Fig. 1) one can see that mass loss of FPO-II is 7.9 % within the temperature range of 383–425 K. The FPO-II molecule contains peroxy fragment $(CH_3)_3COOCH_2\underset{\displaystyle OH}{\underset{|}{CH}}-$ in its structure, whose decom-position according to [10] may be expressed as follows:



$$(CH_3)_3COOCH_2\underset{OH}{\underset{|}{CH}}\sim \xrightarrow{T,\,K} \underset{(I)}{(CH_3)_3CO^{\bullet}} + \underset{(II)}{{}^{\bullet}OCH_2}\underset{OH}{\underset{|}{CH}}\sim$$

$$I \rightarrow CH_3-\underset{O}{\underset{\|}{C}}-CH_3 + {}^{\bullet}CH_3$$

$$I \rightarrow (CH_3)_3COH + R^{\bullet}$$

$$II \rightarrow \sim\underset{OH}{\underset{|}{CH}}-\overset{O}{\overset{/\!/}{C}}-H + H^{\bullet}$$

$$II \rightarrow \sim\underset{OH}{\underset{|}{CH}}CH_2OH + R^{\bullet}$$

$$CH_3^{\bullet} + H^{\bullet} \longrightarrow CH_4$$

$$CH_3^{\bullet} + R^{\bullet} \longrightarrow RCH_3$$

$$R^{\bullet} + R^{\bullet} \longrightarrow R-R$$

The theoretical value of possible mass loss owing to the decomposition of peroxy fragment and separation of volatile products (*tert*-butyl alcohol, acetone and methane) according to the above-mentioned scheme is 8.0 % for FPO-II. It is in agreement with experimental results (7.9 % according to derivatographic investigations).

The mass loss of PDER-24 is 16.9 % within the temperature range of 368–458 K (Fig. 2). Taking into account products which are formed as a result of such oligomer decomposition and its molecular mass, the calculated theoretical mass loss should be 17.0 %. The obtained calculated data for FPO-II and PDER-24 and the results of derivatographic investigations coincide with each other and indicate the effect of fluorine atoms on the decomposition of peroxy groups in FPO-II.

3.2. Cross-linking of Epoxy-Oligoesteric Mixtures

Cross-linking of epoxy-oligoesteric mixtures in the presence of fluorine-containing peroxy oligomers was studied taking polymeric mixture consisting of ED-20 resin and TGM-3 oligoestracrylate as an example. FPO-I and FPO-II were studied as fluorine-containing peroxy oligomers. PEPA was a hardener. Mixtures with PDER-24 instead of FPO were studied for comparison. The composition of epoxy-oligoesteric mixtures is given in Table 2.

Cross-linking of epoxy-oligoesteric mixtures was studied using the procedure described in Subsection 2 and the obtained results are given in Table 3.

The data of Table 3 show that the introduction of compounds containing peroxy groups (composites II–VI) into epoxy-oligoesteric mixture decreases both gel-fraction content and films hardness at cross-linking for 24 h at room temperature. The same regularity is observed even at heating to 403 K for 30 min. The further increase of cross-linking time to 75 min almost equalizes the values of gel-fraction content but increases hardness of films containing both PDER-24 (composite II and FPO-II (composites III, IV and V). Comparing composites with different quantity of FPO-II (20 mas % for composites IV and V and 10 mas % for composite III) the decrease of gel-fraction content is observed.

Table 2

Composition of epoxy-oligoesteric mixtures

Mixture component	Component content in the mixture, mas %					
	I	II	III	IV	V	VI
ED-20	100.0	90.0	90.0	80.0	70.0	90
FPO-II	–	–	10.0	20.0	30.0	–
FPO-I	–	–	–	–	–	10.0
PDER-24	–	10.0	–	–	–	–
TGM-3	10.0	10.0	10.0	10.0	10.0	10.0
PEPA	14.0	13.8	12.6	11.2	9.80	12.6

Table 3

Dependence of gel-fraction content and film hardness upon cross-linking time for composites I–VI at 403 K

Number of composites given in Table 2	Index	Value of index for cross-linking time, min.					
		24 h room temperature	15	30	45	60	75
I	*G*	88.6	93.4	96.1	96.5	97.7	98.9
	H	0.43	0.84	0.95	0.95	0.96	0.96
II	*G*	84.8	85.7	89.9	96.2	96.9	97.2
	H	0.38	0.99	0.93	0.98	0.97	0.96
III	*G*	77.5	90.2	92.0	98.3	98.4	99.0
	H	0.22	0.91	0.98	0.96	0.96	0.96
IV	*G*	77.2	86.6	89.1	90.2	92.2	95.7
	H	0.24	0.86	0.87	0.91	0.91	0.96
V	*G*	72.7	86.2	89.2	96.1	93.5	95.0
	H	0.26	0.78	0.91	0.92	0.92	0.98
VI	*G*	82.0	98.1	99.3	99.6	99.7	99.7
	H	0.33	0.91	0.93	0.94	0.94	0.96

Note: *G* – gel-fraction content, %; *H* – film hardness, rel. units

Cross-linking of epoxy-oligoesteric mixtures at room temperature (Table 3) takes place due to the forming of only ED-20 resin three-dimensional structure in the presence of PEPA hardener. Oligomers FPO-II and TGM-3 do not participate in the process at room temperature. The heating of such mixtures decomposes labile –O–O– bonds in both FPO-II and PDER-24 molecules. Free radicals, which are formed, initiate three-dimensional polymerization of TGM-3 molecules. In the composite I cross-linking of TGM-3 takes place due to thermal polymerization.

For composites III and IV peroxy oligomers are not included in the polymeric matrix structure but in the cross-linked network based on TGM-3, *i.e.* interpenetrating polymer network is formed. The composite II, compared with the composites III, IV and V, contains oligomer which has peroxy groups together with free epoxy groups. The latter ones take part in the polymeric network forming already at the stage of cross-linking at room temperature. It means that at the composite II cross-linking at room temperature for 24 h three-dimensional structure containing PDER-24 molecules is formed. Then free peroxy groups of polymeric matrix decompose and graft TGM-3 molecules to the cross-linked polymeric structure obtained at room temperature. Thus, at the forming of polymeric structure with the participation of the composite II the unified three-dimensional network rather than "network in network" is formed.

Comparing mixtures with fluorine-containing oligomers different by their nature (composites II and VI) one can see that the films obtained by the addition of FPO-I (composite VI) have more insoluble products and higher hardness after cross-linking at room temperature for 24 h. The further heating to 403 K for 75 min results in polymeric films forming with the same values of gel-fraction content and hardness. This indicates that the nature of initial fluorine-containing oligomer has practically no influence on the above-mentioned values.

Taking the composite IV as an example we studied the effect of temperature on the gel-fraction content and film hardness (Fig. 3).

We can see from Fig. 3 that heating to 363 K does not increase gel-fraction content but gradually increases film hardness. It means that reactions leading to the molecules enlargement take place at such temperature. The result is the increase of film hardness but formed films have linear structure and therefore they are soluble in organic solvents. The mentioned phenomenon takes place at the absence of radical processes. Figs. 1 and 2 confirm the stability of peroxy groups in FPO-II oligomer at 363 K. The increase of the temperature to 383 K sharply increases the quantity of insoluble products as well as film hardness. This fact indicates that 383 K is the temperature at which peroxy groups decompose resulting in the three-dimensional polymerization of TGM-3 oligoesteracrylate. The same results were obtained at 403 K.

3.3. Thermal Stability of Cross-linked Mixtures

As it has been shown above, mixtures containing molecules of oligomers with fluorine-containing fragments form polymeric films with sufficiently high gel-fraction content and hardness. On the other hand it is known [2] that the presence of compounds with fluorine atoms in the polymeric mixture essentially affects the properties of obtained products. The effect of fluorine-containing oligomer on thermal stability of obtained films was studied using "Paulic-Paulic-Erdei" derivatograph Q-1500D as described in Subsection 2. Composites I, II and III (Table 3) were taken for investigations. The obtained results are represented in Figs. 4-6 and Table 4.

According to the data of differential thermogravimetric analysis (DTG curve, Fig. 4) the appearance of endothermic effect is observed at DTA curve in the low-temperature area. This effect corresponds to the softening processes of the mixtures. For the composite I without peroxy oligomers the first stage of intensive mass loss is within the temperature range of 543–663 K with the maximum rate (DTG curve) at 613 K. The appearance of a well-defined exothermal effect (DTA curve) with the maximum at 636 K corresponds to the first stage. The exothermal effect indicates the proceeding of thermooxidative processes of space network non-cross-linked fragments. At this stage the mass loss is 67 %.

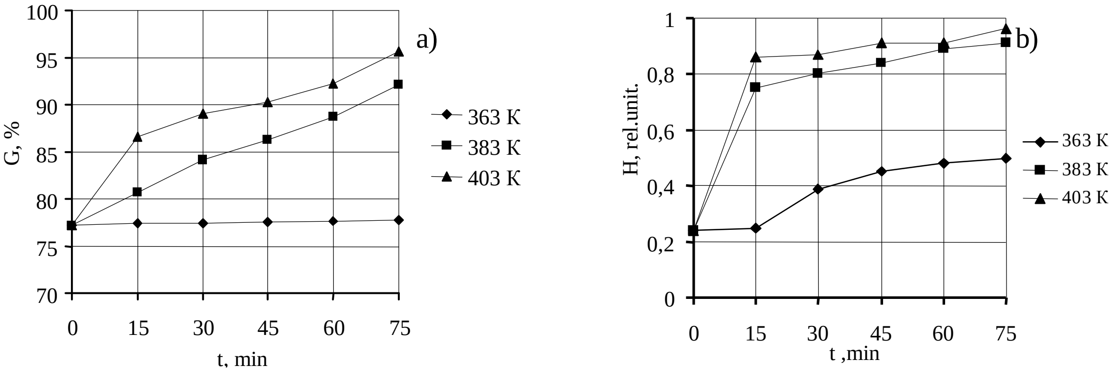


Fig. 3. Dependence of gel-fraction content (a) and film hardness (b) upon cross-linking time and temperature for composite IV

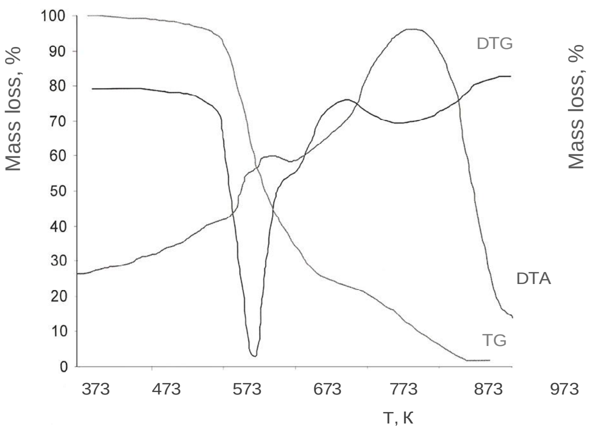


Fig. 4. Composite I derivatographic curves

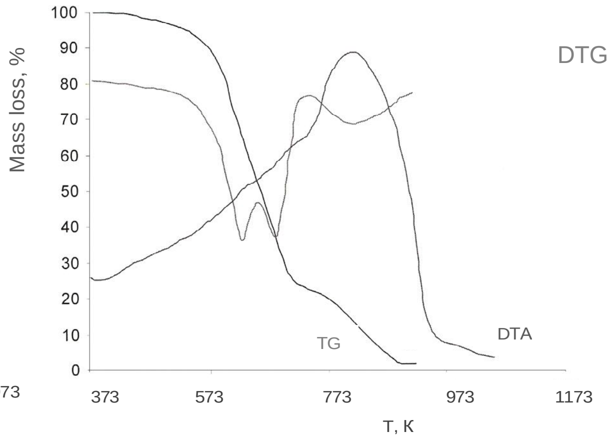


Fig. 5. Composite II derivatographic curves

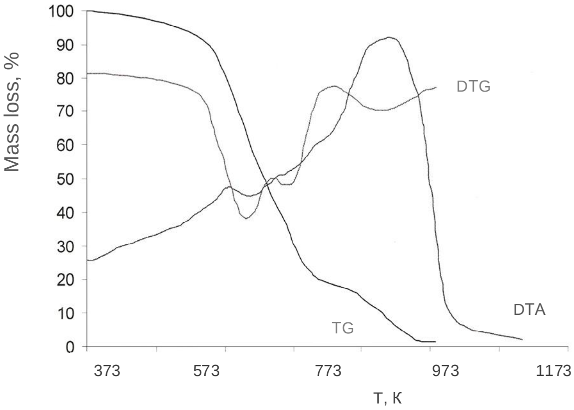


Fig. 6. Composite III derivatographic curves

Table 4

Results of complex thermogravimetric and differential analyses of composites I–III

Number of composites given in Table 2	Stage	Temperature range, K	The mass loss, %	Maximum rate of mass loss at temperature of, K
I	1	543–663	67	613
	2	663–731	11	–
II	1	533–653	48	628
	2	653–738	30	678
III	1	533–643	58	613
	2	613–728	20	668

The second stage of thermooxidative destruction of the composite I proceeds within the temperature range of 663–731 K. The mass loss in this case is 11 %. Organic residues of the sample burn at the temperatures above 731 K. This process is accompanied by the appearance of a definite exothermal effect at DTA curve with the maximum at 828 K.

The first stage of thermooxidative destruction of the composite II with PDER-24 (Fig. 5) proceeds within the temperature range of 533–653 K with the maximum rate at 628 K. It is characterized by the appearance of an indefinite exothermal effect at DTA curve, indicating the density of the composition's space network, which displays lower ability to thermooxidative processes as compared with the composite I. The mass loss at this stage is 48 %.

The second stage of destruction is within the temperature range of 653–738 K. The mass loss is 30 %. Organic residues of the sample burn at the temperatures above 738 K. This process is accompanied by the appearance of a definite exothermal effect with the maximum at 823 K.

For the composite III, which contains ED-20 resin, TGM-3, PEPA and fluorine-containing oligomer (Fig. 6), the first stage of thermooxidative destruction is within the temperature range of 533–643 K with the maximum rate at 603 K. The mass loss is 58 %. This stage is accompanied by the appearance of a definite exothermal effect at DTA curve with the maximum at 588 K. It indicates a sufficiently high intensity of thermooxidative processes at the first stage. The reason is that composite structure is not dense in the presence of FPO-II fluorine fragment ("network in network"). The mass loss is 22 %. The oxidation of composite residues takes place at the temperatures above 728 K and is accompanied by exothermal effect at DTA curve with the maximum at 813 K.

The composite II contains PDER-24, *i.e.* it contains epoxy groups and –O–O– bonds at the same time. In this case PDER-24 attaches to cross-linked matrix based on ED-20 resin owing to the interaction between epoxy groups. Free peroxy groups which form free radicals at heating are grafted to the main matrix of oligoesteracrylate molecules. This phenomenon is not observed in the composite III but interpenetrating polymer network is formed as was mentioned above. It means that structured molecules of FPO-II and TGM-3 oligoesteracrylate are located between cells of the cross-linked fragment based on ED-20.

The results of thermogravimetric analysis allow to calculate the kinetic parameters of composites thermooxidative destruction within the temperature range of 533–738 K. The calculations were made according to the methodology described in [11, 12], uisng program package Mathcad 2001 Professional based on the modified kinetic equation in the complex with least-squares method:

$$\frac{dW}{W_k dt} = \ln\frac{z}{q} - \frac{E}{R_T} + n\ln(1 - \frac{W}{W_k})$$

where W – mass loss of the sample at temperature T determined by TG curve; W_k – general mass loss of the sample at this stage, mg; T – absolute temperature, K; E – activation energy, kJ/mol; n – reaction order; z – preexponential factor; q – heating rate of the sample, dg/s.

The results given in Table 5 show the lowest thermal stability for the composite I. In the investigated temperature range this composite is characterized by the greatest values of the destruction rate effective constant and loss mass.

Table 5

Calculation results of kinetic parameters of composites I–III thermooxidative destruction

Number of composites given in Table 2	Temperature range, K	E, kJ/mol	n	z, s^{-1}	$k \cdot 10^{-3}$, c^{-1}
I	543–731	127	2.02	$1.89 \cdot 10^{10}/60$ $3.2 \cdot 10^{8}$	11.64
II	533–738	75	1.20	$3.03 \cdot 10^{10}/60$ $5.1 \cdot 10^{3}$	3.49
III	533–728	62	1.22	$0.25 \cdot 10^{10}/60$ $0.42 \cdot 10^{3}$	3.37

The composites II and III are characterized by lower values of the destruction rate effective constant and loss mass and have higher thermal stability compared with the composite I. It should also be noted that destruction of the composites II and III proceeds by the similar mechanism. Types of TG curves, close values of effective activation energy and values of reaction order, which are fractional and higher than one, testify to this fact. The decomposition is induced and proceeds by complicated mechanism. Moreover, within the temperature range of 533–465 K thermal stability of the composite III is higher compared with that of the composite II. The effective destruction constant is lower.

To conclude it should be stressed that the introduction of oligomers with fluorine-containing atoms into epoxy-oligoesteric mixture allows to increase thermal stability of the films based on the above-mentioned mixtures.

4. Conclusions

1. Fluorine-containing peroxy oligomers may be used as active additives to epoxy-oligoesteric mixtures based on ED-20 industrial dianic epoxy resin and TGM-3 oligoesteracrylate. During cross-linking while heating fluorine-containing peroxy oligomers cause three-dimensional polymerization of TGM-3 molecules. Cross-linked film has an interpenetrating polymer network structure, where oligomer is in the structure of cross-linked oligoesteracrylate. ED-20 resin is cross-linked in the presence of polyethylenepolyamine.

2. Thermal stability of peroxy groups in fluorine-containing oligomer is higher by approximately 20 K compared with that of peroxy groups in the initial oligomer without fluorine atoms.

3. The introduction of oligomers containing fluorine atoms in the amount of 10-30 mass parts to epoxy-

oligoesteric mixture allows to increase thermal stability of the films based on the above-mentioned mixture.

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СТРУКТУРУВАННЯ ЕПОКСИ-ОЛІГОЕСТЕРНИХ СУМІШЕЙ В ПРИСУТНОСТІ ФЛУОРВМІСНИХ ЕПОКСИДНИХ СМОЛ

Анотація. *Вивчена можливість структурування епокси-олігоестерних сумішей на основі промислової епоксидної діанової смоли ЕД-20 та олігоестеракрилату ТГМ-3 в присутності флуорвмісних пероксидних олігомерів. Використані для структурування флуорвмісні пероксидні олігомери отримані внаслідок хімічної модифікації пероксидної похідної епоксидної смоли ЕД-24 спиртами-теломерами загальної формули* $HOCH_2$-(CF_2-CF_2)$_n$H, *де n=3 або 4. Дериватографічними дослідженнями вивчено термічну стабільність пероксидних груп у вихідному продукті, флуорвмісних пероксидних олігомерах та стабільність структурованих плівок. Встановлено вплив температури, тривалості процесу та складу суміші на вміст гель-фракції та твердості отримуваних плівок.*

Ключові слова: *структурування, епоксид, пероксид, флуорвмісний, спирт-теломер, гель-фракція, твердість.*