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Synthesis of Styrene - Based Copolymers and Study of their Thermal and Thermo - Oxidative Stability

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ABSTRACT: Copolymers of heterocyclic ethers of methacrylic acids with styrene were synthesized at low conversions. Copolymerization constants were determined, and their thermal and thermo-oxidative stability was studied.

KEYWORDS: monomer, styrene, copolymer, relative activity constants, azeotrope, specific activity and polarity parameters.

I. INTRODUCTION

By copolymerization of heterocyclic derivatives of methacrylic monomers with styrene, new copolymers with a range of valuable physical and chemical properties can be synthesized. This allows for directed chemical modification due to nitrogen, oxygen, halogen and sulfur-containing heterocyclic fragments in the macrocycle during the synthesis of thermo and light-stable polymers [1-3].

For this purpose, as starting compounds were selected following monomers: methyl methacrylate benzoxazolone (BOMA), 6-chloro-benzoxazolphenylethynyl (6-Cl-BOMMA), 6-bromobenzimidazole-methyl methacrylate (6-Br-BOMA), benzoxazolphenylethynyl (BOTMA), phthalimidomethyl (FEMA), benzotriazolyl-methacrylate (BTMA) and styrene.

In order to determine the effect of the composition of the initial monomer mixture on the composition of the copolymer, the reaction was performed at different mole ratios with a total concentration of 0.8 mol/l comonomers and the initiator $[DAC] = 3 \cdot 10^{-3}$ mol/l. The composition of copolymers and the relative activity constants of monomers were determined at low degrees of transformation, by determining the amount of nitrogen by elemental analysis. As can be seen, in the case of styrene, the formation of an azeotropic point is not observed (table.1). This is due to the significantly greater activity of radicals formed from molecules of heterocyclic esters (met)acrylic acids compared to styrene radicals.

II. THE MAIN FINDINGS AND RESULTS

As can be seen from table.1. the composition of the copolymers benzoxazolone-methacrylate, 6-хлорбензоксазолтионилметилакрилата, 6-bromobenzimidazole of immediatedata, benzoxazolinone with styrene, the corresponding curve comes above the azeotrope, indicating that over the whole range of initial ratios of monomers the composition of the copolymer is enriched with links of heterocyclic esters of methacrylic acids. This is probably due to the significantly greater activity of the molecules of heterocyclic ethers of methacrylic acids compared to styrene,

Table 1

Dependence of the composition of copolymers of heterocyclic ethers of methacrylic acids with styrene on the initial ratio of monomers

$C_M = 0,8$ моль/л, $C_{II} = 0,005$ моль/л, $T = 343K$

Initial ratio of monomers, %		The yield of copolymer, %	Nitrogen content, %	The composition of the copolymer mol, %	
M ₁	M ₂			M ₁	M ₂
1	2	3	4	5	6
Benzoxazolonylmethylmethacrylate-styrene					
10	90	7,48	2,19	20,39	79,61
20	80	7,75	3,18	39,44	76,56
30	70	8,05	3,90	45,25	54,85
50	50	8,08	4,81	63,91	36,09
70	30	8,47	5,40	80,28	19,62
90	10	8,23	5,87	92,25	7,75
6-chlorobenzoxazolonylmethylmethacrylate-styrene					
10	90	6,78	1,94	18,63	81,37
20	80	6,97	2,81	31,04	68,96
30	70	7,81	3,39	41,75	58,85
50	50	7,84	4,16	60,15	39,85
70	30	6,65	4,73	75,52	21,48
90	10	7,31	5,12	94,72	5,280
6-bromobenzimidazole II methyl methacrylate-styrene					
10	90	3,6	1,64	15,77	84,23
30	70	5,8	2,93	36,64	63,36
50	50	6,3	3,69	56,09	43,91
70	30	7,2	4,22	75,50	24,50
90	10	7,82	4,57	92,57	7,420
Benzoxazoltionmethylmethacrylate-styrene					
10	90	6,8	1,83	16,76	83,23
20	80	6,5	2,96	31,69	68,21
30	70	7,5	3,60	42,75	57,25
50	50	7,8	4,43	60,81	39,18
70	30	6,7	5,07	79,30	20,70
90	10	7,2	5,48	94,13	5,870

Phthalimidomethyl – styrene					
10	90	5,43	1,68	15,02	84,08
20	80	6,17	2,66	27,06	73,94
30	70	6,75	3,43	39,17	60,83
50	50	8,03	4,37	58,39	41,61
70	30	9,10	5,04	76,08	23,02
90	10	10,2	5,51	92,57	7,430
1	2	3	4	5	6
Benzothiazolylsulfenamide – styrene					
10	90	6,80	2,21	20,00	80,00
20	80	7,30	3,40	34,81	65,19
30	70	7,70	4,13	46,02	53,98
50	50	7,55	5,20	66,48	33,52
70	30	7,80	5,93	84,50	15,50
90	10	8,10	6,25	93,70	6,300

that is, a growing molecular chain with a terminal Monomeric link m_1 attaches both "its" and "someone else's" radical in the acts of growth, preferring to attach only "someone else's".

Copolymerization constants were calculated using the Mayo-Lewis differential equation [3,4] and the Ezrielev and Roskin analytical method [5]. According to the calculations, the copolymerization constants are less than one, which indicates the formation of an azeotrope.

The found values of copolymerization constants for heterocyclic esters of methacrylic acids with styrene show that in copolymerization reactions, the more active component is heterocyclic esters (met)acrylic acids (table.1.) the values of copolymerization constants show that both types of formed radicals react faster with another monomer than with their own, and the effect of alternating monomer links is observed in copolymers. One of the reasons for this phenomenon is known to be the difference in the polarity of monomers and radicals.

Table 2
Parameters of copolymerization of heterocyclic estersmethacrylic acids with styrene

Monomers	r_1	R_2	$r_1 r_2$	$1/r_1$	$1/r_2$	Q_1	e_1
BOMMA-styrene	1,50	0,41	0,6150	0,670	2,430	1,1700	-0,1000
6-Cl-BOMMA-styrene	1,35	0,45	0,6070	0,740	2,222	0,4200	-0,0800
6-Br-BOMMA-styrene	1,20	0,53	0,6360	0,830	1,880	3,2300	-0,1270
BOTMMA-styrene	1,36	0,52	0,7000	0,735	1,923	0,3296	0,2928
FIMMA-styrene	1,23	0,59	0,7257	0,813	1,694	2,6000	-1,3500
BTMMA-styrene	0,76	0,45	0,7920	0,568	2,220	0,3100	1,2800

It is known that chain growth in the copolymerization reaction is possible only if the resulting terminal radical is able to react not only with the monomer from which it was formed, but also with the "foreign" monomer. Therefore, the factors that determine the composition of the copolymer, first of all, include the reactivity of monomers and their radicals. The relationship between the structure of the monomer and the reactivity from reactions with free radicals is determined by the steric, resonant and polar effects. The semi-quantitative q-e scheme proposed by Alfrey and price is the most common for estimating the resonant and polar effects [6,7]. To evaluate the composition and structure of

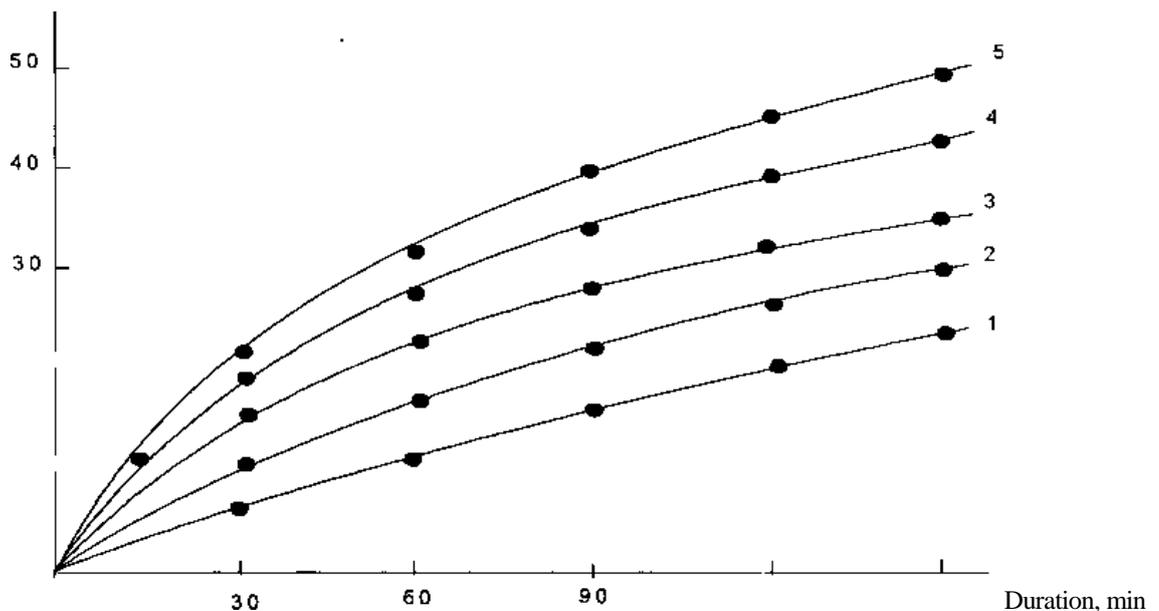
copolymers, the relative activity constants of monomers, the numerical values of r_1 and r_2 , as well as the parameters of specific activity and polarity are determined, which are shown in table 2. The calculated values of copolymerization constants indicate that macroradicals ending in links of heterocyclic ethers of methacrylic acids tend to react more actively with their monomer than with styrene $r_1 > 1$; $r_2 < 1$ ($r_1 \cdot r_2 < 1$), which indicates the tendency of monomer links to alternate in the copolymer chain.

Based on the obtained values of copolymerization constants, the specific activity factors (Q1) and the polarity of the radical addition product (E1) were calculated using this scheme. The Q2 and E2 values for styrene are 0.74 and 0.40, respectively. As can be seen from table 2, the introduction of links of heterocyclic ethers of methacrylic acids into the structure of the copolymer increases the values of Q and e. The increase in polarity seems to be determined by the electrodonor character of the heterocyclic group and the increase in the polarizability of the double bond of the monomer.

Copolymerization of heterocyclic ethers of methacrylic acids with styrene was performed at a temperature of 353 K in order to determine the effect of reaction time on the yield of a copolymer of different compositions (Fig. 1). As can be seen from the figure, the yield of copolymers increases with increasing reaction time. Based on experimental data, it was found that the rate of copolymerization increases with an increase in the concentration of interaction of heterocyclic ethers of methacrylic acids in the initial mixture of monomers.

The increase in speed seems to be due to the participation of heterocyclic groups in initiation acts and chain transfer reactions, which facilitates the formation of radicals, Hence, with an increase in the number of active centers, the speed of copolymerization increases. In this case, the characteristic viscosity of copolymer solutions decreases. The decrease in the intrinsic viscosity of copolymers with increasing content of heterocyclic esters of methacrylic acid in the starting mixture can be explained by the transfer and open circuit to the monomer taking place with the participation of molecules of heterocyclic esters of methacrylic acids.

Thus, experimental results have shown that heterocyclic derivatives of methacrylic monomers do not inhibit the copolymerization reaction, but on the contrary, actively enter into it, which makes it possible to obtain a copolymer with stabilizing fragments in wide ranges of initial concentrations of comonomers.



T=353 K Fig. 1. Dependence of the yield of the copolymer benzoxazolthionylmethylene ether methacrylic acid with styrene on the duration of the reaction at different initial ratios of monomers: 1 - 90:10; 2 - 25:75; 3 -50: 50; 4 -75 : 25; 5 - 90 : 10. [DAK]= $3.79 \cdot 10^{-3}$ mol/l. The solvent is dioxane. T=353 K



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The effectiveness of the use of polymer materials in the national economy largely depends on the preservation of the properties of polymers in the conditions of processing and operation. Increasing the time of reliable operation of polymers is equivalent to the production of many hundreds of thousands of tons of additional products. In this regard, the research of destruction processes, the establishment of the mechanism of polymer decomposition under the interaction of various factors and the development of ways to increase their stability are relevant. Polymer stabilization thus becomes one of the most efficient ways to save labor costs, natural resources, and energy.

It is known from the literature that increasing the thermal stability of polystyrene can be achieved by introducing into their macromolecule stabilizing links that perform a different role depending on their structure. Thus, the effectiveness of some monomers of stabilizers is due to the implementation of the "foreign link" effect, which leads to slowing down the destruction process [8-15].

Of particular interest is the possibility of increasing the thermal stability of polymers by introducing Monomeric links in their chains with a structure close to that of the stabilizing object. This applies, in particular, to monomers containing active sulfur and nitrogen atoms in the heterocycle, due to their participation in the destruction of hydroperoxide and peroxide groups formed in the process of destruction and are the cause of the beginning of the chain depolymerization process [9]. The introduction of a small amount of monomer stabilizer in the macromolecule of the main polymer leads to increased thermal stability and at the same time prevents migration, volatilization, and leaching of the stabilizer.

The thermal and thermo-oxidative stability of polymers and copolymers was studied by thermogravimetric analysis, chromatographic and monometric methods, as well as changes in their molecular weights during degradation [16-22].

Results of dynamic thermogravimetry of polystyrene and their copolymers with insignificant (0.5-3.0 wt.%) amounts of heterocyclic ethers of methacrylic acids, show that the modified samples have a higher temperature resistance than polystyrene. The beginning of thermal decomposition is shifted to the region of higher temperatures. The stabilizing properties of synthesized stabilizers are most effective when the polymer structure contains 0.5-1.0 wt.% of heterocyclic esters of methacrylic acids.

Table 3 shows the experimental results of TGA samples of homopolymers and copolymers and their compositions. As can be seen from the table, the introduction of nitrogen -, oxygen -, sulfur -, and halogen - containing heterocyclic fragments into the polymer chain contributes not only to an increase in the temperature of the beginning of mass loss of samples (10%), but also to the temperature of maximum decomposition.

The maximum rate of decomposition is also shifted in the region of higher temperatures, compared to unstabilized samples. This seems to be due to the blocking effect of the kinetic chain of decomposition by benzoxazoltionylmethacrylate links.

The most powerful stabilizing effect is provided by Monomeric links of benzoxazoltionylmethacrylate than known analogues, as in the case of a copolymer with styrene. Apparently, during thermooxidation destruction, the stabilizing effect of heterocyclic links that have thion groups is associated with the formation of low-activity compounds when chain processes break down, with the destruction of macromolecules. The apparent activation energies of thermooxidative destruction were calculated by the Reich method of double logarithmization using dynamic thermogravimetry [23].

Analysis of volatile products of thermal and thermo-oxidative degradation of stabilized polystyrene samples by mass spectroscopy and electron-paramagnetic resonance showed that, in fact, the main monomer is formed during thermal decomposition, as well as a benzoxazoltion radical and CO₂ and CO. The formation of benzoxazoltion radicals in the process of thermal degradation was confirmed by EPR spectroscopic studies.

Table 3

Parameters of thermo-oxidative degradation of styrene Homo-and copolymers during non-isothermal oxidation in air at a rate of heating 5⁰ / min.

Content of the stabilizer congestion, mass. %	Decomposition temperature at 100 % mass loss, K	Temperature of the maximum decomposition rate, K	mass loss at maximum decomposition rate,%	The energy of thermo-oxidative degradation kJ/mol
1	2	3	4	5
Composition of polystyrene with 2-mercaptobenzoxazole				
0,5	607	644	84	175,01 ± 2,2
1,0	605	641	86	174,12 ± 1,8
2,0	604	639	87	173,25 ± 1,4
3,0	603	637	91	172,93 ± 1,7
The copolymer BAIMAK – styrene				
0,5	666	719	23	254 ± 1,6
1,0	553	715	38	238 ± 1,5
2,0	508	697	57	242 ± 1,1
3,0	595	688	69	234 ± 1,4
Composition of polystyrene with benzoxazolone				
1,0	601	684	71	215 ± 1,2
2,0	596	675	84	222 ± 1,6
3,0	593	656	89	183 ± 1,7
Copolymer of 6-Cl-BAIMAK – styrene				
0,5	572	723	24	267 ± 1,8
1,0	664	715	37	256 ± 1,1
2,0	657	703	52	231 ± 2,2
3,0	501	690	64	223 ± 1,4
4,0	512	678	79	226 ± 1,5
Composition of polystyrene-6-Cl-BOMEMAK 1,0 601 686 74 210 ± 1,3				
1,0	601	686	74	210 ± 1,3
2,0	604	671	80	206 ± 1,6
3,0	600	649	93	217 ± 1,2
Copolymer benzoxazolonylmethylene ether acrylic acid-styrene				
0,5	666	693		237,9
1,0	685	721		241,5
2,0	643	684		229,8
3,0	628	680		223,4
1	2	3	4	5
Copolymer 6-brombenzoxazolonylmethylene ether acrylic acid-styrene				
0,5	677	702		243,2
1,0	696	727		247,4
2,0	654	690		235,5
3,0	639	686		229,7
Copolymer benzoxazolonylmethylene ether acrylic acid-styrene				
0,5	671	698		240,5
1,0	688	725		245,6
2,0	646	687		232,0
3,0	631	683		226,6
Copolymer 6-brombenzoxazolthionylmethylene ether acrylic acid-styrene				
0,5	682	706		238,4

1,0	698	732		250,0
2,0	657	695		235,7
3,0	642	689		228,6
Benzothiazolylsulfenamide ether copolymer, methacrylic acid – styrene				
0,5	579	692	36,96	218,0
1,0	581	685	52,22	204,0
2,0	575	671	57,80	201,0
4,0	573	657	66,30	207,0

The effect on the process of thermal-oxidative degradation of polystyrene, a monomer of phthalimidomethylmethacrylate, which was added to the polymer in the form of a conventional mechanical mixture (table 4), was studied. As you can see, a small addition of phthalimidomethylmethacrylate to polymers increases the temperature of the beginning of decomposition of polystyrene-by 48-610. Moreover, the increase in the content of phthalimidomethyl in the composition leads to an increase of onset temperature of decomposition and the maximum speed of the development process.

The greatest interest was the introduction of phthalimido methyl methacrylate links into the polymer chains of polystyrene. For this purpose, copolymers of phthalimidomethylmethacrylate were synthesized with the specified monomers containing 0.5 – 3.0% FIMMA links. Since the process was carried out to deep degrees of transformation (89-95 %), the composition of the copolymers practically corresponded to the composition of the original monomer mixture. Table 3-4 shows the dependence of mass loss when copolymers with phthalimido methyl methacrylate are heated in air. As can be seen, the addition of phthalimido methyl methacrylate links to copolymers leads to a significant stabilizing effect. The initial rate of decomposition is shifted in the region of higher temperatures compared to unmodified samples, which allows us to conclude that intramolecular stabilization is highly effective.

The results show that not only the temperature of the beginning of decomposition, the maximum rate of development of the process, but also the activation energy of stabilized samples is higher than that of unstable polymers.

Table 4

Parameters of thermal-oxidative degradation of the polystyrene composition with phthalimidomethyl methacrylate in air at a heating rate of 5⁰ S/min.

Content FIRM mass. %	PS	
	T began to decompose, K	T max speed and mass loss, K
0,0	570	606
0,5	618	657
1,0	625	676
2,0	627	685
3,0	631	694

Table 5

Parameters of thermal oxidative degradation of styrene copolymers with phthalimide methyl ether methacrylic acids in the air with a heating speed of 5⁰ minutes

Content Links' FEMME, %	Temperature Beginnings Decomposition, To	Temperature Maximum Speed potters The Masses, To	Energy Oxidation Noi of destruction, kJ/mol
FIMALAC a copolymer of : styrene			
0,0	570	606	213,0
0,5	633	685	228,2

1,0	648	689	234,6
1,5	652	695	238,5
2,0	671	698	242,7
2,5	689	722	256,8
3,0	695	726	246,3

In principle, there is an increase in the activation energy of decomposition with an increase in the content of phthalimidomethylmethacrylate in the copolymer. Individual results that fall out of this pattern can be explained by experimental errors. It is interesting to compare the data in tables 4 and 5. As can be seen from the comparison of results, copolymers exhibit a greater stabilizing effect compared to the mechanical mixture, which indicates a higher efficiency of intramolecular stabilization.

The introduction of a stabilizer not only shifts the temperature of the beginning of decomposition, but also helps to preserve the molecular weight. Studies of changes in the viscosity of copolymers depending on the content of the last link phthalimidomethylene ether methacrylic acid. The results are shown in table 25. As can be seen from the results, samples with different content of phthalimidomethylene ether methacrylic acid in the copolymer have approximately the same characteristic viscosity and after destruction retain this parameter in different ways. The decrease in the characteristic viscosity for all copolymers depends on the content of phthalimidomethylmethacrylate in them. Moreover, the more of them in the copolymer, the smaller the difference between the values of the characteristic viscosity and after destruction.

Table 6

The dependence of the reduced viscosity of solutions of copolymers phthalimide methylene ether methacrylic acid with styrene on the content of FIMEMAC links in them

Content FIMALAC in the copolymer, %	The copolymer FIMALAC:styrene		
	Temperature, K	Reduced viscosity, DL / g	
		Source	After the destruction
0,0	573	1,74	1,07
0,0	593	1,74	0,65
0,0	620	1,74	0,17
0,5	573	1,73	1,03
0,5	593	1,73	0,80
0,5	620	1,73	0,65
1,0	573	1,72	1,09
1,0	593	1,72	0,94
1,0	620	1,72	0,75
2,0	573	1,70	1,21
2,0	593	1,70	1,06
2,0	620	1,70	0,82
3,0	573	1,68	1,32

3,0	593	1,68	1,12
3,0	620	1,68	0,87
5,0	573	1,66	1,45
5,0	593	1,66	1,28
5,0	620	1,66	0,93

Therefore, the presence of links phthalimidomethyl in the copolymer contributes to the preservation of the molecular mass of the samples prevents the leakage of products of thermal and oxidation decomposition of copolymers of methyl methacrylate with phthalimidomethyl when the content of 0,5-5,0% show that the main degradation products are the monomers of heterocyclic methacrylate, also detected a small amount of carbon oxides CO₂ and co, the formation of which occurs, apparently, when the destruction of the polystyrene.

Table 6 shows the dependence of the amount of released methacrylate monomer on the content of phthalimidomethylmethacrylate in the copolymer. The amount of released methacrylate decreases significantly with an increase in the content of phthalimidonaphthylmethacrylate in the copolymer, which is in accordance with the above studies and indicates effective stabilization [24].

III. CONCLUSION

Analysis of volatile products of thermal-oxidative degradation of stabilized polystyrene samples by mass-, IR - spectroscopy and chromatography showed that, indeed, a monomer is released during decomposition. In addition, such products as benzaldehyde, benzoic acid, carbon oxides are formed, which indicates the oxidation of styrene molecules.

Thus, the introduction of a small number of links of heterocyclic ethers of methyl methacrylate in the composition of polystyrene contributes to a significant increase in their resistance to thermal-oxidative degradation.

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