

**METHODS FOR THE SYNTHESIS OF AROMATIC OLIGESTERS**

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Abstract

Due to the fact that the problem of creating thermostable polymers with high fire resistance and good physical and mechanical properties is very relevant today for various fields of technology, depending on the structure of the starting materials, the production of copolyesters and block copolyesters based on oligosulfoneketone, oligosulfone, oligoketone and oligoformal. It is important from a scientific and practical point of view to study the laws of formation, to determine the relationship between the structure, composition and properties of copolymers.

Keywords: oligosulfoneketones (OSK), oligoketones (OC), oligosulfones (OS), dimethyl sulfoxide (DMSO), degree of polycondensation, disodium salt, dipolar solvent.

Introduction

Polymer materials such as polyarylate, polysulfone, and polyketone are widely used in various fields of modern industry. Each of them is described with certain advantages. At the same time, they have some disadvantages. Recently, in order to embody the positive qualities of different classes of polymers in one material, oligomers that keep the links of polymers of this class in their chain have been widely used.

For example, in order to improve certain properties of polysulfone or polyether ketones, a number of block copolymers have been created and their properties have been studied. They are important for various branches of modern industry as heat-resistant materials with construction and electrical insulation functions.

Nowadays, oligosulfonic ketones (OSK), oligoketones (OK) and oligosulfones (OS) with different structure and degree of polycondensation have been synthesized in order to obtain high molecular weight, fire-resistant, high physico-mechanical properties copolymers of polysulfone and oligoformals.

The synthesis of oligosulfoneketone, oligoketone, oligosulfone and oligoformals was carried out by high-temperature polycondensation in an aprotic dipolar solvent - dimethylsulfoxide (DMSO) in an inert gas (nitrogen) atmosphere.

Some properties of aromatic oligoethers - oligosulfone, oligoketone and oligoformals are presented in tables 1-4.



1 – Table Properties of aromatic oligoketones

Oligoketones	Polycondensation level	η dl/g	Output, %	Softening temperature, °C	Calculated mol. mass	Amount of OH groups, %**	
						Calculated	Found
OK – 1D	1	0,04	98	129-135	635	5,35	5,40
OK – 3D	3	0,08	98	139-146	1148	2,35	2,10
OK – 5D	5	0,12	98	147-152	2261	1,50	1,60
OK – 7D	7	0,14	98	154-157	3074	1,11	1,05
OK – 10D	10	0,16	99	160-165	4293	0,79	0,82
OK – 20D	20	0,18	99	167-175	8358	0,40	0,45
OK – 1F	1	0,03	98	196-200	815	4,17	4,20
OK – 5F	5	0,06	98	210-215	2801	1,21	1,20
OK – 10F	10	0,11	99	237-244	5284	0,64	0,65
OK – 20F	20	0,16	99	255-260	10249	0,33	0,30

* Numbers in the designation of oligosulfones – the average value of the degree of polycondensation
D – dian derivatives, F – phenolphthalein derivatives

** Found by Werley titration

2 – Table Properties of aromatic oligosulfones

Oligosulfones	Degree of polycondensation	η dl/g	Output, %	Softening temperature, °C	Calculated mol mass	Amount of OH groups, % **	
						Calculated	Found
OS – 1D	1	0,02	98	85-88	671	5,00	5,08
OS – 3D	3	0,06	98	106-147	1556	2,32	2,36
OS – 5D	5	0,11	98	155-162	2441	1,39	1,32
OS – 7D	7	0,12	99	173-178	3326	1,02	1,06



OS – 10D	10	0,13	98	177-183	4654	0,73	0,78
OS – 20D	20	0,24	98	185-186	9078	0,38	0,32
OS – 1F	1	0,02	98	202-205	851	4,00	4,03
OS – 10F	10	0,14	99	262-269	5645	0,60	0,64
OS – 20F	20	0,25	99	292-300	10970	0,31	0,29

* The numbers in the designation of oligoketones are the average value of the degree of polycondensation n, D - dian derivatives, F - phenolphthalein

** Verley titrlash orqali topilgan

3– Table Properties of aromatic oligosulfonketones

Oligosulfone-ketones	Degree of polycondensation	η , dl/g	Output, %	Softening temperature, °C	Calculated molar mass	Amount of OH groups, %**	
						Calculated	Found
OSK – 1D	1	0,06	98	137-143	1077	3,15	3,20
OSK – 3D	3	0,11	98	145-150	2775	1,22	1,20
OSK – 5D	5	0,15	98	152-157	4473	0,76	0,80
OSK – 7D	7	0,18	99	164-168	6171	0,55	0,53
OSK – 10D	10	0,19	98	170-176	8718	0,39	0,30
OSK – 20D	20	0,22	99	179-194	17209	0,20	0,18
OSK – 1F	1	0,04	98	198-206	1347	2,52	2,62
OSK – 5F	5	0,06	98	224-240	5464	0,62	0,60
OSK – 10F	10	0,11	99	256-263	10609	0,32	0,35
OSK – 20F	20	0,13	99	263-271	20900	0,16	0,20

* Numbers in the designation of oligosulfone ketones - the average value of the degree of polycondensation n, D - dian derivatives, F - phenolphthalein

** Werley found by titration



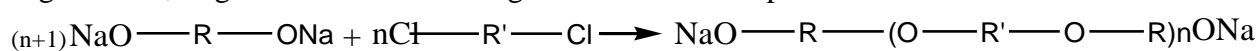
4 – Table Properties of aromatic oligoformals.

Oligoformals	Degree of polycondensation	η , dl/g	Output, %	Softening temperature, °C	Calculated molar mass	Amount of OH groups, %**	
						Calculated	Found
OF – 1D	1	0,07	98	44-46	470	7,2	7,8
OF – 5D	5	0,08	98	48-52	1430	2,3	2,6
OF – 10D	10	0,18	97	54-57	2631	1,2	1,6
OF – 20D	20	0,23	97	140-145	5034	0,7	0,9
OF – 1F	1	0,07	98	48-51	650	5,2	5,6
OF – 5F	5	0,08	98	68-72	1970	1,7	1,9
OF – 10F	10	0,19	97	103-106	3622	0,9	1,3
OF – 20F	20	0,25	97	155-160	6925	0,5	0,9

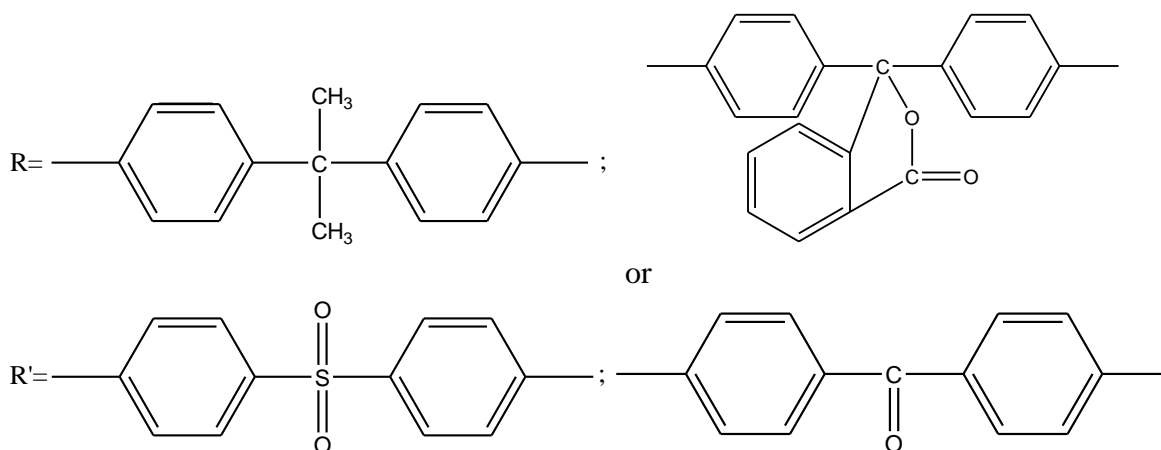
Numbers in notation of oligoformals - the average value of the degree of polycondensation n, D - dian derivatives, F - phenolphthalein

** Werley found by titration

Disodium salt is formed at the first stage of interaction in a 2:1 molar ratio of NaOH solution and bisphenol. Then, a reaction is carried out between diphenolate and the corresponding diaryl halide by means of high-temperature polycondensation. The general scheme of the synthesis of oligosulfone, oligosulfonketone and oligoketones can be expressed as follows:

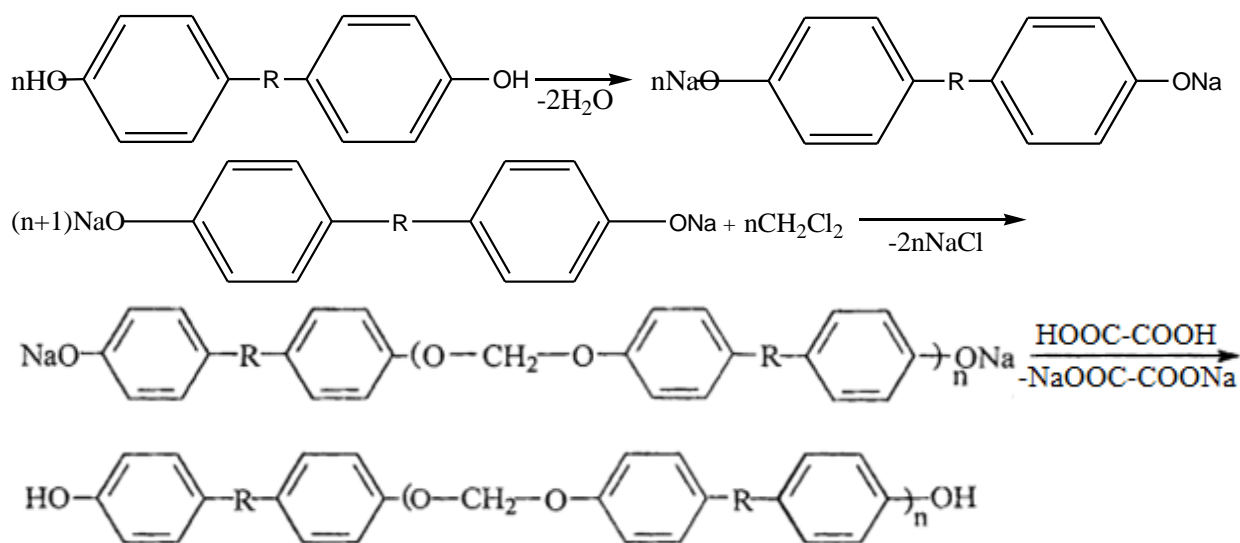


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Or an equimolecular mixture of them (1 : 1)

Aromatic oligoformals were obtained according to the following general scheme:



The structure of oligosulfonketone, oligosulfone and oligoketones is confirmed by elemental analysis results in Table 5 and IR spectroscopy. In IR spectra $930, 1015, 1045 \text{ cm}^{-1}$ to simple ether bonds, $1290, 1365, 1385, 1415 - 1485, 2930 - 2980 \text{ cm}^{-1}$ to the isopropylidene group of dian residue (in the case of dian oligomers), $1750 - 1780 \text{ cm}^{-1}$ corresponding to lactone group (for phenolphthalein oligomers), $3600-3200 \text{ cm}^{-1}$ to hydroxyl groups, $1150-1170, 1315, 1245-1295, 1320 \text{ cm}^{-1}$ to sulfonyl group (for oligosulfones) and $1610-1650 \text{ cm}^{-1}$ to ketone group the presence of absorption bands indicates the formation of oligosulfonketone, oligosulfone and oligoketones.

5 – Table Elemental analysis of oligosulfones, oligoketones and oligosulfonketones

Oligoethers	Calculated, %			Found, %		
	C	H	S	C	H	S
OS - 1D	75,01	5,80	4,50	75,20	5,71	4,78
OS - 5D	73,80	5,20	6,57	74,06	5,02	6,59
OS - 10D	73,56	5,11	6,89	73,59	5,07	7,03
OS - 20D	73,42	5,06	7,06	73,54	5,09	7,18
OS - 1F	73,40	4,03	3,77	73,69	4,27	3,79
OS - 10F	72,35	3,82	5,68	72,39	3,91	5,72
OS - 20F	72,26	3,80	5,85	72,30	3,76	5,91
OK - 1D	81,20	6,01	–	81,36	6,03	–
OK - 5D	82,35	5,62	–	82,41	5,60	–
OK - 10D	82,53	5,54	–	82,56	5,49	–
OK - 20D	82,63	5,50	–	82,66	5,47	–
OK - 1F	78,12	4,20	–	78,15	4,16	–
OK - 10F	79,56	4,08	–	79,60	4,04	–
OK - 20F	79,56	4,07	–	79,62	4,03	–
OSK - 1D	78,04	5,61	2,97	78,22	5,55	3,00
OSK - 5D	77,86	5,32	3,58	77,92	5,28	3,66
OSK - 10D	77,84	5,27	3,67	77,76	5,22	3,71
OSK - 20D	77,82	5,25	3,72	77,72	5,20	3,82
OSK - 1F	75,77	4,04	2,37	77,65	4,00	2,45
OSK - 5F	75,82	3,95	2,93	75,84	3,90	3,11
OSK - 10F	75,84	3,93	3,02	75,79	3,89	3,09
OSK - 20F	75,86	3,92	3,06	75,81	3,85	3,16



* The numbers in the designation of oligoesters are the average value of the degree of polycondensation p, D – dian, F – phenolphthalein deriv.

The structure of the obtained oligoforms is also confirmed by the results of elemental analysis (table 6).

6 – Table Elemental analysis of oligoforms

Oligoformals *	Calculated, %		Found, %	
	C	H	C	H
OF-1D	79,46	6,88	79,29	6,83
OF-5D	79,80	6,76	79,75	6,80
OF-10D	79,88	6,74	79,93	6,81
OF-20D	79,93	6,73	79,77	6,89
OF-1F	75,92	4,35	75,80	4,40
OF-5F	76,21	4,29	76,15	4,37
OF-10F	76,28	4,29	76,23	4,31
OF-20F	76,31	4,28	76,27	4,29

* Numbers in notation of oligoformals – The average value of the degree of polycondensation, D – dian, F – phenolphthalein deriv.

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