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ABSTRACT

of the dissertation for the degree of Doctor of Philosophy

PROPERTY AND RESEARCH OF PROPANTHRIOL-BASED NEW GENERATION EPOXY (MET) ACRYLATE OLIGOEFIRES AND HYBRIDED EPOXIDE COMPOSITIONS

Speciality:2304.01 - Chemistry of MacromoleculesField of science:Chemistry

Applicant:

Raisa Ispandiyar Ismayilova

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The dissertation work was carried out in the laboratory of "Polyester and Polycarbonate Materials" of the Institute of Polymer Materials of the Azerbaijan National Academy of Science

Scientific supervisor:	Doctor of chemical sciences, professor Aga Mammad Mustafayev
Scientific advisor:	Corresponding member of ANAS, Doctor of chemical sciences, professor Bakhtiyar Ajdar Mammadov
Official Opponents:	Doctor of Chemical Sciences, professor Ogtay Hummat Akbarov Doctor of Chemical Sciences, professor Minaver Jafer Ibrahimova Doctor of Philosophy in Chemistry,docent Elnara Telman Aslanova

Dissertation Council ED.1.28 of the Supreme Attestation Commission under the President of the Republic of Azerbaijan operating at the Institute of Polymer Materials of the Azerbaijan National Academy of Sciences

Chairman of the Dissertation council:



Doctor of Chemical Sciences, docent Nushaba Ismayil Gurbanova

Doctor of Philosophy in Chemistry, docent Khayala Vagif Allahverdiyeva

Doctor of Chemical Sciences, Professor Najaf Tofig Gahramanov

GENERAL CHARACTERISTICS OF THE WORK

Relevance and development of the topic. Epoxy oligomers have high adhesion, strength, chemical resistance, etc. It is superior to other representatives of synthetic polymers in terms of its properties. They are widely used as binders in the production of high-strength polymer compositions, paints, adhesives, castings and sealing compounds. However, in some cases the application of epoxy oligomers is limited. Thus, the high viscosity of epoxy oligomers, their resistance to heat and combustion, the fragility of polymers obtained after hardening, etc. in some cases limits their ability to compile. One of the effective ways to regulate and purposefully change the properties of epoxy oligomers is their chemical modification. In this regard, the search for multi-purpose modifiers and their synthesis based on multilayer propanthriol is one of the current problems.

The use of non-toxic chemicals and compounds obtained as byproducts in various industries of the chemical industry as raw materials in other chemical syntheses is also noteworthy. In this regard, the increase in the number of scientific researches in the field of obtaining a wide range of new valuable products from propanthriol, which is used as a raw material in the production of many important chemical compounds, is due to the production of biopropanthriol from vegetable oils. Thus, as biofuel production expands, the amount of propanthriol obtained as a by-product also increases.

Given the above, the synthesis of different functional groups of epoxy(met)acrylate oligo esters based on propanthriol triglycerides and polyoxychloropropylene triglycerides and (met)acrylic acids and the creation of new hybrid epoxy compositions based on them is one of the scientific and practical problems of macromolecular chemistry.

Object and subject of research. The object of research is the synthesis of branched, functionally structured epoxy(met)acrylate oligoefers based on propanthriol and α -monochloropropandiol. The subject of the research is the production of hybridized epoxy

compositions based on synthesized epoxy(met)acrylate oligoesters, the study of their hardening process and the study of antimicrobial properties.

Goals and objectives of the study. The purpose of the study is to study the reactions of propanetriol-, polyoxychlorpropylenetriglyceride and monochlorpropandiol diglicide esters with (met)acrylic acids, to determine the new conditions of high viscosity of various modifiers containing low-viscosity epoxy(met)acrylate oligoester consists of obtaining hybrid epoxy compositions and determining their properties

The following studies have been conducted to achieve this goal:

- From the reaction of propanthriol and monochloropropandiol with 3-chlorine-1.2-epoxy-propane development of methods for the synthesis and effective production of diglicid oligo esters of propanthriol, polyoxychloropropylene-triglycidyl and monochloropropandiol, which have a tri- and diepoxide cycle at the end of the chain;
- Obtaining PEPA-based hybrid compositions based on polyoxychlorpropylene triglyceride oligo ether and ED-20 epoxy resin, studying their dependence on the composition of a number of physical and mechanical properties;
- Synthesis, structure and composition of new low-viscosity modifiers based on polyoxychlorpropylene triglyceride ether and (met)acrylic acids containing epoxy(met)acrylate oligoesters of different functional groups;
- Obtaining new hybrid epoxy compositions with high performance based on synthesized epoxy(met)acrylate oligoesters and ED-20 resin and investigation of the hardening process in the insulated system with the participation of polyethylene polyamine (PEPA), diaminodiphenylsulfone (DADFS) and 1.4.5.6.7.7-hexabrombi-cyclo-[2.2.1]-hept-5-en-2.3-dicarboxylic acid anhydride (BED-anhydride) by the method of Differential Thermal Analysis.

Research methods. All syntheses were carried out in the laboratory at the Institute of Polymer Materials of the Azerbaijan National Academy of Sciences using existing methods. The IR spectrum of the obtained substances was recorded on a "Spekord 75-IR" device in a suspension prepared in Vaseline oil. The ¹H NMR spectrum was recorded on a Brooker Avance 300 MHz spectrometer in CDCl3 and DMSO. Thin-layer chromatography (NTX) UV-254 was performed on Silufol board, using UV-rays to clearly see the formed spots. Column chromatography was performed on Merck silica gel. Element analysis was performed on a Carlo Erba analyzer.

The processes of the compositions obtained from the synthesized epoxy(met)acrylate oligoesters base resin ED-20 and hardening mixtures hardening process on Derivatograph, hardening degree on Soxhlet apparatus, density by hydrostatic drawing on the Westphalian-Mor scale, thermophysical and thermomechanical measurements were carried out in the Kepler's consistometer, physical and mechanical tests were carried out in laboratory test machines. Dielectric measurements were made on an E-6-13 teraometer and a digital capacitance meter at a frequency of 1 kHz.

The main provisions of the defense.

- Synthesis of mono- and di- epoxy(met)acrylate oligoesters from the reaction of propanthriol and polyoxychloropropylene triglycer esters with (met)acrylic acids;
- Obtaining and discussion of the results of the study of the process of obtaining and solidification of hybridized epoxy compositions based on epoxy(met)acrylate oligoesters of propanthriol and polyoxychlorpropylene and ED-20 epoxy resin;
- Results obtained from the reaction of α-monochloropropandiol with diglycid ether with (met)acrylic acid to obtain monoepoxy, diepoxy mono- (met)acrylate ether;
- Research of epoxy(met)acrylate oligoesters based on propanthriol and polyoxychloropropylene as a modifierantipyrene in ED-20 epoxy resin and obtaining a refractory composition, studying the hardening process of the

compositions and determining the properties and their discussion.

Scientific novelty of the research. Propanthriol-based epoxy oligoesters with a tri- and diglicid cycle at the end of the molecule, including epoxy(met)acrylate esters, were synthesized, compositions were obtained from their mixtures with ED-20 epoxy resin, and the solidification process was studied.

- The triglycer ester of polyoxychloropropylene was obtained from the interaction of propanthriol with 3-chlorine-1.2epoxypropane, and mono- and di- epoxy(met)acrylate oligoesters were synthesized as a result of esterification of (met)acrylic acids with it. The composition and structure of the synthesized epoxy(met)acrylate oligoesters were confirmed by IR-, ¹H NMR-spectral and elemental analysis.
- Hybridized compositions with permeable polymer mesh structure were obtained from mixtures of polyoxychlorpropylene triglyceride oligoefir and ED-20 epoxy resin, their physical-mechanical and dielectric properties were studied. It was found that the density, mesh density, physical and mechanical properties of hybrid compositions are higher than the analogous properties of materials obtained from the same conditions of triglyceride oligo esters and ED-20 resin.
- Synthesized epoxy(met)acrylate oligoesters have been studied in ED-20 resin as a modifier-antipyrene. Polyethylene polyamine, diaminodiphenylsulfone and 1.4.5.6.7.7-hexabrombicyclo[2.2.1]-hept-5-en-2.3-dicarboxylic acid anhydride and insulating system were used as hardeners. Analysis of the physical and mechanical properties of the obtained compositions shows that the physical and mechanical properties of the compositions obtained in the presence of epoxy(met)acrylate PEPA, DADFS, BED-anhydride and insulated in an insulated system prevail in all cases. All compositions hardened with BED-anhydride have both high heat resistance and self-extinguishing properties.

 The antifungal activity of synthesized epoxy(met)acrylate oligo esters was studied by dilution and it was determined that they have high bactericidal properties.

Theoretical and practical significance of the research. Conditions for obtaining low-viscosity, new epoxy resins containing different functional groups of epoxyacrylate oligomers based on (met)acrylic acids of propanetriol polyoxychlorpropylene triglyceride and α -monochloropropandiol esters have been developed. It has been determined that these resins can be used in the production of new hybridized epoxy compositions for various purposes with high performance.

Personal presence of the author. Compilation of the literature review, to determine the purpose of the work,to carry out most of the experimental research, to summarize them, to compile articles and theses. belongs to the author.

Approbation and application of research. 31 scientific works, including 6 articles, 25 theses were published on the materials of the dissertation.

The results of the dissertation were presented at the following conferences and symposiums: Republican Scientific Conference dedicated to the International Year of Chemistry "Disposal and use of industrial waste", June 29-30, (Sumgayit-2011); Republican Scientific Conference dedicated to the 85th anniversary of Academician TN Shakhtakhtinsky, October 27-28, (Baku-2011); XIV International scientific and technical conference "Scienceintensive chemical technologies-2012" May 21-25, Yasnaya Polvana-Kulikovo Pole, (Tula-2012); II Republican Scientific Conference "Modern problems of chemistry of monomers and polymers" October 31-November 1, (Sumgavit-2012); Conference on fundamental and applied problems of macromolecular chemistry. Republican Scientific Conference dedicated to the 75th anniversary of Academician AA Efendiyev, June 27-28, (Sumgavit-2013); Scientific Conference dedicated to the 105th anniversary of Academician MF Nagiyev (Baku-2013); Third All-Russian Scientific Conference (with international participation): "Successes in synthesis

and complex formation" The conference is dedicated to 55-PFUR, 21-25, (Moscow-2014); XV International April Scientifik Conference "High-Tech in Chemikal Engineering-2014" Zvenigorod September 22-26, (Moskow-2014); Republican scientific-practical conference dedicated to the 100th anniversary of Academician S.C.Mehdivev, December 2-3, (Baku-2014); Republican scientific conference "Lubricants, fuels, special liquids, additives and reagents" dedicated to the 50th anniversary of the Institute of Chemistry of Additives named after Academician A. Gulivev, October 13-14, (Baku-2015); III Republican Conference on Modern Problems of Monomers and Polymer Chemistry, November 05-06, (Sumgavit-2015); "International Scientific Conference on Actual Problems of Modern Chemistry and Biology", dedicated to the 93rd anniversary of National Leader Heydar Aliyev, May 12-13, (Ganja-2016); Republican scientific conference on "Organic synthesis and composite materials in macromolecular chemistry", dedicated to the 50th anniversary of the Institute of Polymer Materials of ANAS, October 20-21, (Sumgavit-2016); Collection of Materials XIII Numanov readings. Achievements of Chemical Science for 25 years of State Independence of the Republic of Tajikistan, November 23, (Dushanbe-2016); International scientific and technical conference "Petrochemical synthesis and catalysis in complex condensed systems", dedicated to the 100th anniversary of Academician B.K. (Baku-2017); International Zevnalov. June 29-30, scientific conference "Functional monomers and special polymer materials: Problems, Perspectives and practical views", November 15-16, (Sumgavit-2017); Scientific conference "Nagiyev readings" dedicated to the 110th anniversary of Academician Nagiyev (Baku-2018); II International Scientific Conference of Young Researchers dedicated to the 95th anniversary of national leader Heydar Alivev. April 27-28, BEU, (Baku-2018); The International Scientific Conference "Actual problems of modern chemistry" Dedicatel to the 90th anniversary of the Academician Y.H.Mammadaliyev institute of petrochemical processes. October 2-4, (Baku-2019); International scientific conference "Prospects for innovative development of chemical technology and engineering", dedicated to the 70th anniversary of Sumgayit, November 28-29, (Sumgayit -2019); Ministry of Education of the Republic of Azerbaijan Sumgayit State University (SSU), conference proceedings, April 15-16, №1, (Sumgayit, 2021).

Name of the organization where the dissertation work is carried out. The dissertation work was carried out in accordance with the plan of research work of the laboratory "Polyester and polycarbonate materials" of the Institute of Polymer Materials of ANAS (State registration N_{0} 0111 Az 2151).

The structure and scope of the dissertation. The dissertation is reflected in the 169 page computer text: introduction 8 pages. (14.641 characters), three chapters, literature review; (first chapter) 37 pages. (63.541 characters), methods of conducting experiments (second chapter) 23 pages. (28.704 characters), discussion of research (third chapter) 64 pages. (91.062 characters), results 2 pages. (2.249 characters), it includes a list of references from 247 sources 28 pages, 21 pictures and 14 tables, and has the volume of 253.838 characters (excluding pictures, tables, bibliography and appendices).

In the introductory part, the relevance of the topic, the goals and objectives of the dissertation, scientific novelty, practical significance are substantiated, information on approbation, structure, volume and publications are given, as well as the essence of the chapters is briefly presented.

In the first chapter, scientific and scientific-technical information devoted to the study of the synthesis properties of epoxyacrylate oligomers, curing reactions and curing products is analyzed, systematized and summarized.

The second chapter covers the raw materials and their properties, the methods of experimental research and the research methods used.

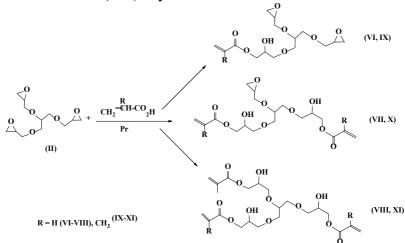
The third chapter presents the synthesis of epoxy acrylate oligo esters of propanthriol and the preparation, study and discussion of the obtained results of hybridized epoxy compositions of ED-20 epoxy resin with these compounds. Synthesis and some kinetic properties of reaction of unsaturated polyethylated with bicyclic structure are given.

The results reflect the main findings of the research.

MAIN CONTENT OF THE WORK

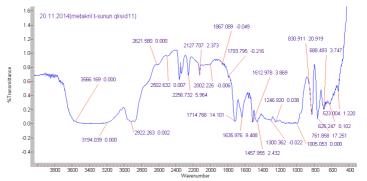
1. Synthesis of propanthriol epoxyacrylate oligoesters and production and research of hybridized epoxy compositions based on ED-20 epoxy resin

In order to develop effective methods for obtaining new epoxy(met)acrylate oligoesters, the reaction of propanthriol epichlorohydrin was carried out, first propanriol triglyceride (II) ester was obtained and its epoxy(met)acrylate oligoesters were condensed by condensation with (met)acrylic acids:



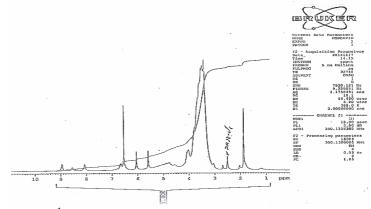
The structural formula of the synthesized compound (X) was confirmed by IR and ¹H NMR spectroscopy. In the IR- spectrum, 1635 cm⁻¹ has an absorption band corresponding to a double bond. The absorption band at frequencies 1246 and 688-761 cm⁻¹ indicates the presence of the C-O-C bond, and the characteristic absorption band at 1715-1720 cm⁻¹ indicates the presence of the carboxyl group

of the ether fragment. A weak band at a frequency of 956 cm⁻¹ confirms the presence of an epoxy ring. A wide absorption band at 3460 cm^{-1} in the IR- spectrum indicates the presence of a hydroxyl group in the compound (pic.1.).



Picture 1. IR spectrum (X) of propanetriol monoepoxide hydroxide (met) acrylate oligo ether.

In the ¹H NMR spectrum of the X compound, signals in the form of multiplets at 1.75 m.h. correspond to the protons of the methyl group, and at 5.2 m.h., corresponding to the protons of the double bond, to the protons of the ether fragment and at 2.64-2.73 m.h., signals corresponding to the epoxy groups of this compound were recorded in the form of a group of low intensity (pic.2).

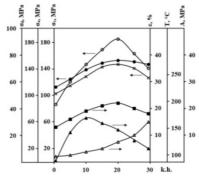


Picture 2. ¹H NMR spectrum (X) of propanetriol monoepoxide hydroxide(met)acrylate oligoether.

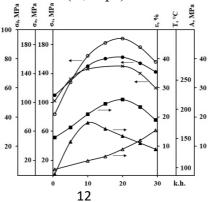
2. Physico-mechanical properties of composite materials based on epoxy acrylate oligoesters and ED-20 epoxy resin

Synthesized epoxy(met)acrylate oligoesters were used as modifiers for VI and VII ED-20 epoxy resin.

Picture 3 and 4 present the results of the study of the physical and mechanical properties of the composite materials. It can be seen from the given curves that the inclusion of the synthesized compounds in the resin significantly improves the properties of the compositions. The maximum values of strength properties are observed mainly when epoxyacrylates are added to ED-20 in the amount of 20 k.h:



Picture 3. Propanthriol's effect on the properties of composite materials based on ED-20 of the amount of diepoxymonohydroximonoacrilat oligoeffect (VI): \circ -strength limit (σ d, MPa); the strength limit (s, MPa); \bullet Strength limit (σ s, MPa); Δ -relative stretch (ϵ , %); \blacktriangle -heating (T, °C); \blacksquare Adhesion (A, Mpa).

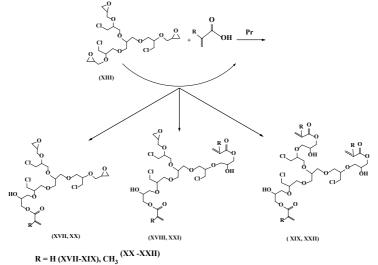


Picture 4. The amount of VII oligomerine affects the properties of composition materials based on ED-20: strength limit (σ d, MPa); the strength limit (s, MPa); \bullet Strength limit (σ s,MPa); Δ -relative stretch (ϵ ,%); \blacktriangle -heating (T, °C); \blacksquare Adhesion (A, MPa).

It was determined that the degree of hardening of the studied samples is not less than 98-99%. Thus, the physical and mechanical properties of compositions obtained in the presence of epoxy(met)acrylates are superior to compositions based on ED-20 resin and PERA, due to the participation of functional groups in epoxy(met)acrylate oligomers in the solidification process.

3. Obtaining and research of hybridized epoxy compositions based on polyoxychloropropylene triglyceride oligo ester XIII and ED-20 resin

In order to obtain lowviscosity epoxyacrylate resins with aliphatic structure containing chlorine atoms, polyoxychloropropylene triglyceride ester was treated with acrylic and methacrylic acids and corresponding epoxyacrylate oligoesters XVII, XXI with 83-94% yield were obtained:

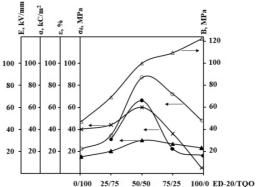


Hybrid compositions of permeable polymer mesh type were obtained from mixtures of polyoxychloropropylene triglyceride

oligoester (TGO) and ED-20 resin, their physical-mechanical and dielectric properties were studied.

It was found that TQO resin hardens at a lower rate than ED-20 resin and the life of the compositions based on it is longer. TQO resin hardened at 323 K becomes highly elastic, and the amount of modulus of elasticity in this area is practically unchanged up to 523 K. This quantity reaches a maximum when the ED-20 / TQO ratio is 50/50 in the reciprocal penetration network (CI).

When TQO / ED-20 = 50/50, the physical and mechanical parameters of the obtained compositions (strength, deformation and impact viscosity) also receive maximum values (Fig. 5). It should be noted that the prices of these quantities are significantly higher for both TCO and ED-20-based materials. This fact can be attributed to the high density of structures and the mutual penetration of different types of chains.



Picture 5. The amount of TQO affects the physical and mechanical properties of composites based on epoxy layers: \circ -strength limit (σ d, MPa); x-relative stretch (ϵ ,%); • Durability (α , kC/m2); Δ (B, MPa); \blacktriangle -electric durability (E, kV/mm).

The obtained QNTs retain high dielectric properties, and in the optimal composition the dielectric strength of the compositions is higher than the dielectric strength of each component separately.

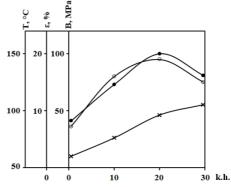
Thus, QNT-type hybrid binary mixtures from ED-20 and TQO homopolymers allow to increase their main performance by

maintaining the high dielectric properties of transition epoxy compositions.

Picture 6 and 7 show the results of the effect of the amounts of polyoxychlorpropylene epoxy acrylate oligo esters XVII, XVIII on the properties of ED-20 resin. It turns out that the maximum values of hardness and heat resistance are recorded in compositions containing 17 k.h of oligo ethers XVII and XVIII. As the amount of relative elongation modifier increases, it increases continuously, increasing the oligo ether to 20 k.h. The maximum value of heat resistance is 150 and 160° C.

4. The process of hardening of polyoxychloropropylene epoxy acrylates with PEPA XVII-XVIII and ED-20 resin

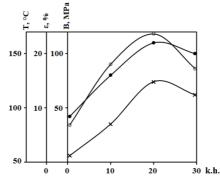
The synthesized epoxyacrylate oligomers XVII, XVIII were tested in ED-20 resin as antipyrene-modifiers. Polyethylene polyamine (PERA) was used as a binder:



Picture 6. Effect of XVII on the properties of composite materials based on ED-20 epoxy resin: •-sensitivity (T, °C); x-relative stretch (ε , %); o-strengthening (B, MPa).

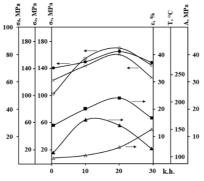
In the samples considered in XVII, the maximum value of hardness is relatively high and is 120 MPa, the heat resistance is 160° C and the relative elongation is 15.0%. It was also shown that the epoxy materials obtained and hardened by the inclusion of synthesized XVII, XVIII oligoethers in the epoxy resin also show the

fire resistance properties, which is due to the presence of chlorine atoms in the modifiers.



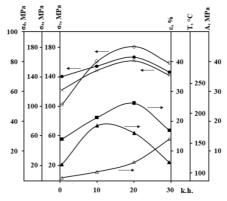
Picture 7. Influence of the amount of oligomer XVIII on the properties of composite materials based on ED-20 epoxy resin: \bullet -heating (T,°C); x-relative stretch (ε ,%); \circ -strengthening (B, MPa).

Picture 8 and 9 show the physical and mechanical characteristics of XX and XXI epoxyacrylates and composition materials based on ED-20 epoxy resin, depending on the composition.



Picture 8. Influence of the amount of XX oligomer on the properties of composite materials based on ED-20 epoxy resin: \circ -strength limit (σ d, MPa); the strength limit (s, MPa); \bullet Strength limit (σ s, MPa); Δ -relative stretch (ϵ , %); \blacktriangle -heat continuity (T, °C); \blacksquare Adhesion (A, MPa).

A comparison of the corresponding dependencies shows that they vary according to approximately the same regularity, but the samples obtained on the basis of XXI oligoether are characterized by relatively high values of tensile strength. In these samples, the maximum values of strength are obtained in the amount of 20 k.h of oligo ether, and the heat resistance in the amount of 10 k.h of epoxyacrylate is 175° C.



Picture 9. Influence of the amount of XXI oligomer on the properties of composite materials based on ED-20 epoxy resin: \circ -strength limit (σ d, MPa); the strength limit (s, MPa); \bullet Strength limit (σ s, MPa); Δ -relative stretch (ϵ , %); \blacktriangle -heating (T, °C); \blacksquare Adhesion (A, MPa).

Thus, with the inclusion of different types of synthesized oligoethers in the composition of ED-20 resin, it is possible to obtain a wide range of composite materials characterized by high physical and mechanical properties with practical application as special purpose construction materials.

5. Application of 1.4.5.6.7.7 - hexabrombicycle [2.2.1] - hept-5-en-2.3 - dicarboxylic acid anhydride as a hardener

Relevant compositions for obtaining refractory polymeric materials based on synthesized XXI oligoether and ED-20 were fortified with 1.4.5.6.7.7 – hexabrombicyclo [2.2.1] -hept-5-en-2.3 – dicarboxylic acid anhydride (BED) (tab. 1).

Table 1. Some physical and mechanical properties of composite materials obtained after 1.4.5.6.7.7–hexabrombicyclo [2.2.1]-hept-5-en-2.3-dicarboxylic acid anhydride (accelerator UP-606/2) hardening of mixtures of ED-20 epoxy resin and epoxy(met)acrylates (XX and XXI) in different compositions.

ED-20:	Н	ardness lin	nit,	Adhesi-	Rela-	Heat	Fire						
Compound:		Mpa		on to	tive	resis-	resis						
hardener: accelerator	In ten- sion	In bend- ing	In com- pres- sion	the steel, MPa	strech, %	tance accor- ding to Vika, ⁰ C	tance, Sec						
	Compound XX												
90:10:70:1	79	144	155	21	3.7	165	19						
80:20:70:1	91 154		163	23 5.1 147		147	Turns off						
		•	Compour	nd XXI									
90:10:70:1	81	152	158	19	3.7	175	21						
80:20:70:1	92 159 17		172	25	6.8	147	Turns off						
70:30:70:1	81 137		155	18	18 12.7 11		Turns off						
			ED-	20									
100:70:1	54±4	135±5	180±5	15	2.3	123	Turns						
							on						

It was found that increasing the amount of modifier in all samples to 30 k.h., the deformation strength and adhesion properties of the composition increase significantly. The maximum limit of their mechanical properties is recorded in the amount of 20 k.h of the modifier, and compositions hardened with BED-anhydride have a self-extinguishing property.

6. Investigation of the process of solidification of polyoxychlorpropylene epoxy(met)acrylate oligoether in an insulated system

Modification of epoxy resin with polyoxychlorpropylene epoxy(met)acrylate (PEM) resins was carried out, laws and products of hardening process were studied. The solidification process was carried out at room temperature for 24 hours, then at 60° C for 20 hours and finally at 150° C for 5 hours, the degree of solidification of the samples was determined by extraction in acetone or benzene in a Soxhlet apparatus. The amount of non-reactive epoxy resin and anhydride in extraction with acetone and styrene in extraction with benzene was determined (Table 2).

Regardless of its composition, only 65-70% of the resin is converted into a tar material during cold hardening. The combination of individual reagents is not uniform: the reaction involves the entire amount of styrene, and most of the maleic anhydride and PEM resin (75-80%), while ED-20 resin does not participate in the solidification process. It was found that 40% of the amount of PEM reacts when the mixture is heated to 60° C, but when heated to 150° C, the amount of polymer built is more than 96% of the total mass.

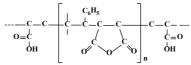
Table 2. Indications of	f harden	ing of	ED-20 ep	oxy ol	ligo	ether
with polyoxychlorpropylene	epoxy	(met)	acrylate	resin	in	"cold
environment" and in insulated	d system	l				

50	The degr	ee of com	bination o %	Combinati- on of	The amount of insoluble concentration, %			
Hardening	PEM	EM ED-20 styrene resin		Maleic anhydride				
Ha					a baggy polymer, %	In Ace-	With Ben-	
						tone	zene	
Ι	81/68	0/0	99/91	96/90	67/61	72/65	73/66	
II	86/82	38/32	99/97	99,5/96	78/75	77/76	76/78	
III	89/90	94/92	99/99	99/99	93/92	94/95	94/95	

Note: Structure of PEM composition at speed,%: PEM- 45.9; epoxy resin ED-20-22.8; styrene-22.8; (MA) - 7.7.

Divide: structure of composition, in%: PEM-48.4; Epoxy resin ED-20 -21.1; styrene -22.5; maleic anhydride (MA) - 7.9.

The results show that the transition of the resin to a threedimensional lattice structure in the presence of maleic anhydride goes through the process of obtaining a copolymer as an intermediate, ie the structure formed from epoxy(met)acrylate resin is joined as a bridge by copolymer styrene and maleic anhydride manganese:



It has been experimentally confirmed that copolymerization (up to 98%) of styrene maleic anhydride occurs in parallel in the initial stage of solidification. 53-55% of PEM reacts. Under more severe conditions (at 150° C), the conversion of PEM increases (up to 75%). At this stage, there is a high probability that the polyoxy-chlorpropylene epoxy(met)acrylate chains would join together by double bonding.

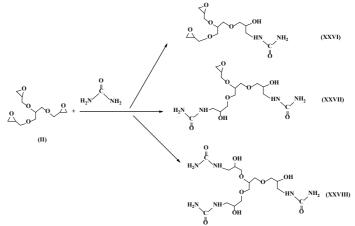
The possibility of such reactions was tested in two model systems. In one of them, the process was carried out by removing maleic anhydride and in the other by removing PEM. When the mixture is carried out in the cold state with the removal of PEM, a linear copolymer of styrene and maleic anhydride is obtained, which is soluble in acetone but insoluble in benzene. In this case, 93% of styrene and 86% of maleic anhydride react, ED-20 does not react and does not form a lattice structure.

It was found that when the temperature is increased, the fraction of maleic anhydride and maleic anhydride in the copolymer hardens the epoxy resin, and the yield of the three-dimensional product increases up to 79%. Maleic anhydride does not react with ED-20 resin "cold" in the extracted mixture. Its hardening starts at 60°C. Under these conditions, glycide and carboxyl groups react in equivalent amounts. When the temperature is raised to 150°C, the number of glycide groups that react increases, which is probably due to their interaction with the dual hydroxyl group.

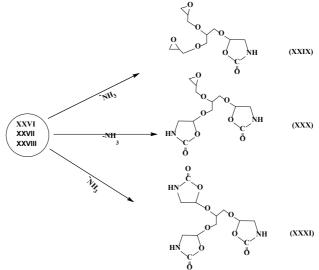
7. Synthesis of polyoxypropylene substitute urea and 1.3-oxazolidine-2-one

The reaction of urea with propanriol triglyceride ether was carried out at the melting temperature of urea without solvent (130-

135°C) and in the presence of dimethylformamide at 100-110 °C. The course of the reaction was monitored by thin-layer chromatography. It was determined that, depending on the molar ratio of propanetriol glycide ether and urea, mono- XXVI, di- XXVII and tri- XXVIII substituted derivatives are obtained:



When the synthesized compounds are heated to 160-180⁰ degrees Celsius XXVI-XXVIII, polyoxypropylene-substituted oxazolidine-2 is obtained:



Polyoxypropylene-oxazolidine-2 is released from XXVI-XXVIII when heated in the presence of XXIX-XXXI, DMFA.

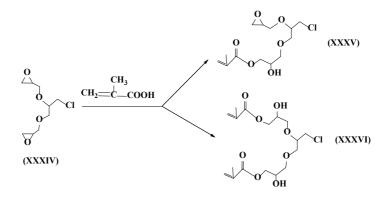
Determination of physical and mechanical properties of a mixture of 1.3-oxazolidone-2 and ED-20 epoxy resin after heating shows that no chemical reaction occurs between 1.3-oxazolidone-2 and epoxy resin. Therefore, 15% polyethylene polyamine was added as a binder to the mixture obtained from 1.3-oxazolidone-2 and ED-20 resin. Physico-mechanical properties of the compositions after hardening show that 1.3-oxazolidine-2-ten fragment oligoesters can be used as plasticizers-modifiers for ED-20 epoxy resin (Table. 3).

Table 3. Physico-mechanical properties of polyoxypropylene-oxazolidine-2-on oligoethers based on XXIX, XXX and ED-20epoxy resins and PEPA-reinforced compositions

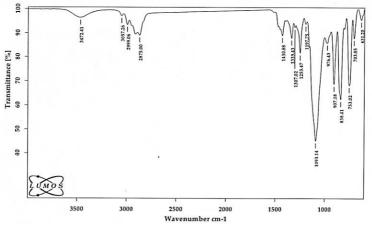
ED-20:	Tensile	Adhesion	relative	Heat resistance							
Compound:	strength,	to the steel	elongation	according to							
XXIX, XXX	Mpa	MPa	%	Vika ⁰ C							
hardener: k.h.											
Compound XXIX											
90:10:20	77	20	3.5	165							
80:20:20	84	24.7	6.3	178							
70:30:20	80	24	8.4	171							
	С	ompound XX2	X								
90:10:20	78	21	4,4	175							
80:20:20	90	19	6.5	183							
70:30:20	85	14	11.7	176							
		ED-20									
100:20	51.4	14	2.3	119							

7. Synthesis and study of methacrylate oligoesters based on α -monochlorine propanediol

The widespread use of epoxy acrylates in industry highlights the development of efficient methods for obtaining new epoxyacrylate oligomers. In this regard, the optimal conditions for the production of mono- and diepoxy(met)acrylate oligoesters from the esterification of α -monochloropropandiol with diepoxide ether and methacrylic acid and some kinetic regularities were determined. The following oligo esters were synthesized by condensation of glycide ester XXXIV with methacrylic acid synthesized on the basis of monochloropropandiol:

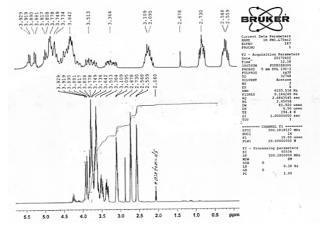


The absorption bands of 1197 and 703-753 cm⁻¹ in the IR spectrum of the XXXIV compound prove the existence of C-O-C and C-Cl bonds. The characteristic absorption band at a frequency of 3472 cm^{-1} proves the presence of a hydroxyl group (pic. 10).



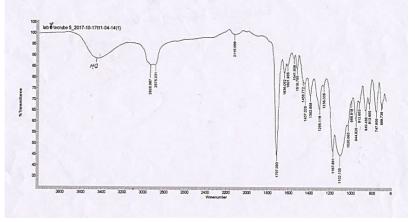
Picture 10. IR- spectrum of XXXIV α -monochloropropandiol diepoxide oligoester.

The structure of this compound was also confirmed by ¹H NMR spectral analysis (pic.11.) Thus, in the ¹H NMR spectrum of this compound, along with the signals of protons of chloride hydrons initially taken, signals corresponding to the methyl group at 1.75 m.h, protons of the vinyl group at 5.2 m.h. and protons of the ether fragment at 4.16 m.h. were recorded.



Picture 11. ¹H NMR spectrum of XXXIV α -monochloropropandiol diepoxide oligoester.

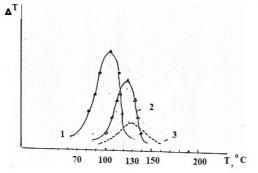
Absorption band corresponding to 1636 cm⁻¹ double bond was recorded in the IR- spectrum of XXXV compound (pic. 12).



Picture 12. IR- spectrum of XXXV α -monochloropropandiol epoxy(met)acrylate oligomer.

Absorption bands 1167 and 747 cm⁻¹ indicate the presence of C-O-C and C-Cl bonds, and characteristic absorption bands at 1707cm⁻¹ indicate the presence of a carboxyl fragment of the ester group. A weak strip of 912 cm⁻¹ indicates the presence of an epoxy ring in the XXXV compound. A wide absorption band of 3465 cm⁻¹ in the IR-spectrum indicates the presence of a dual hydroxyl group in the XXXV compound.

The process of fixing the synthesized α -monochloropropandiol epoxymethacrylate oligofer with XXXV PEPA was also studied. When comparing the DTA curve corresponding to the PEPA curing process of a-monochloropropandiol epoxymethacrylate ether with the DTA curve of the ED-20 resin + PEPA curing process (Pic. 12), it is clear that the added modifier slightly changes the curing temperature (curves 1 and 2). The peak is recorded at 100^oC, while the hardening peak in the reference sample is observed at 110^oC, and the hardening processes are completely completed at 130, 145, 160^oC, respectively. When a modifier is added, the reaction is more intense, ie a chemical reaction takes place between the modifier and PEPA



Picture 13. Fragments of DTA curves during solidification of epoxy compositions.

ED-20+ PEPA + Compound XXXV; 2- ED-20+PEPA;

Some indicators obtained from the study of thermooxidative destruction of hardened compositions are presented in Table 4.

Structure of	T ₁₀ , ⁰ C	T20, ⁰ C	T ₅₀ , ⁰ C	E _{active} ,	$\tau_{1/2}$,
composition				kC/mol	min
ED-20	3000	3200	3400	210.5	42.8
PEPA					
ED-20					
PEPA	280^{0}	300^{0}	320^{0}	195.5	40.0
Compound					
XXXV					

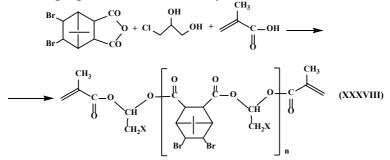
 Table 4. Thermal properties of compositions

 ^{0}C ; t_{1/2}- half-life, min.

 $T_{10, 20, 50}$ - is the temperature of loss of 10, 20 and 50% of the mass of the substance, respectively.

8. Synthesis and some kinetic properties of bicyclic unsaturated polyester

In order to develop a method for the synthesis of new unsaturated polyesters containing bromine and chlorine atoms, the interaction of 5.6-dibromonorbene-dicarboxylic acid anhydride, 1-chlorine-2.3-propanediol and methacrylic acid was studied:

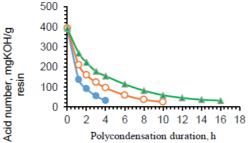


Polycondensation was carried out in a nitrogen medium at a temperature of 140, 160 and 180° C in the ratio of 0.5: 0.5: 1.1 moles of dibromonorbene-dicarboxylic acid anhydride, 1-chlorine-2.3-propanediol and methacrylic acid. It was found that the polycondensation process begins at 120° C and takes place at a fairly

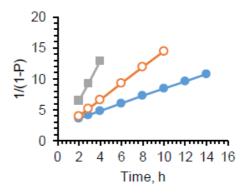
high rate. Thus, the study of the depth of the process at different temperatures shows that the number of acids corresponding to 1 g of polyester and equal to 30-40 mg of KOH is achieved in 10 hours at 140° C, 8 hours at 160° C and 3 hours at 180° C. 2.5 hours are sufficient to complete the reaction at 180° C.

During the reaction of dibromonborne-dicarboxylic acid anhydride with 1-chlorine-2.3-propanediol and methacrylic acid at temperatures of 140, 160 and 180^oC, the process takes place at a high rate, as can be seen from the time-varying curves of acid number and the rate of consumption of monomers (pic.14). At the initial stage, the kinetic curves are linear. The values of the rate constant of the reaction at different temperatures were calculated according to the speed of the initial stage. Picture 15 shows the time dependences of the degree of conversion found for the decrease in acid number. These dependencies are linear in nature and indicate that the reaction performed is carried out by the second-order kinetic equation.

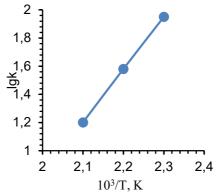
Thus, it was determined that the polycondensation reaction under the interaction of dibromonorbenic-dicarboxylic acid anhydride, methacrylic acid and 1-chlorine-2.3-propanediol is characterized by the second-order equation, and the values of the rate constants of the reaction determined by the kinetic curves consists of $1,18 \cdot 10^{-2} \, 1 \cdot \text{mol}^{-1} \cdot \text{min}^{-1}$ at 140^{0} C, $2,14 \cdot 10^{-2} \, 1 \cdot \text{mol}^{-1} \cdot \text{min}^{-1}$ at 160^{0} C and $6,20 \cdot 10^{-2} \, 1 \cdot \text{mol}^{-1} \cdot \text{min}^{-1}$ at 180^{0} C. Using the logarithmic form of the Arrhenius equation, the activation energy determined by the dependence of the logarithms of the values of the velocity constants at different temperatures on the inverse of the temperature is 63.1. kC·mol⁻¹ (Picture. 16).



Picture 14. Kinetic curves of the reaction of dibromonorbenicdicarboxylic acid anhydride 1-chlorine -2.3-propanediol and methacrylic acid: •- at 140° C; •- at 160° C; •- at 180° C.



Picture 15. Reaction time dependence of polycondensation rate of 1-chlorine-2.3-propanediol, methacrylic acid and dibromonorbene-dicarboxylic anhydride: **—** at 140^oC; **—** at 160^oC; **—** at 180^oC.



Picture 16. Temperature dependence of the logarithm of the rate constant (k) of the polycondensation reaction of 1-chlorine-2.3-propanediol, methacrylic acid and dibromonorbene-dicarboxylic anhydride.

Unsaturated polyester obtained on the basis of dibromnorbornen-dicarboxylic acid anhydride, 1-chlorine-2.3-propanediol and methacrylic acid with a yield of 90-95%, is dark

brown in solid state, softening temperature is $65-70^{\circ}$ C. Some indicators of synthesized unsaturated polyester XXXVIII are given in Table 5.

Table 5. Properties of hardened resin modified withdibromnorbornen-dicarboxylic acid anhydride

Amount	Properties	Values
1	Average molecular weight	1559
2	Density, kg / m ³	1473
3	Viscosity, sP	237
4	Acid amount, mq KOH / q	35±5
5	Gelatinization time, min.	180
	(Styrene content is 40% when methyl ketone	
	hydroperoxide is 3% and accelerator is 8%.)	

The process of hardening of unsaturated polyester XXXVIII was carried out in the presence of activating system, accelerator at 20-30^oC for 24 hours, and then at 80^oC for 10 hours. The amount of non-reactive reagents (styrene and unsaturated polyester) in the solidification process was determined by extraction with hot acetone for 10 hours using a Soxhlet apparatus.

The properties of hardened unsaturated polyester are presented in Table 6. Examination of the refractory properties of the samples by the Fire-Tube method showed that the hardened resin has a selfextinguishing ability.

		1 2
Amount	Properties	Values
1	Styrene content ,%	40
2	Hardness in compression, MPa	112
3	Bending strength, MPa	37
4	Hardness according to Brinell, kgf / mm2	24
5	fire resistance, ⁰ C	120
6	Free combustion time, seconds	Turns off
7	Weight loss,%	1.7

Table 6. Some indicators of hardened unsaturated polyesters.

Thus, the synthesized unsaturated polyester casting compound, which is characterized by high physical-mechanical and selfextinguishing ability, can be used as a binder for laminate and pressed plastics.

9. Investigation of antimicrobial properties of some synthesized propanthriol-containing epoxy (met) acrylate oligoesters

The study of antifungal activity of synthesized substances was carried out in the Laboratory of Microbiological Biotechnology of the Institute of Microbiology of ANAS. The following substances were selected for the study: Polyoxychlorpropylene diepoximonohydroxymonoacrylate XVII, polyoxychlorpropylene monoepoxide dihydroxyacrylate XVIII, polyoxychlorpropylene diepoximonohydroxymonomethacrylate XX, polyoxychlorpropylenemono-epoxide dihydroxydimethacrylate XXI, 3-chloropropyl-1,2-diepoxy-propyl ether XXXIV.

st	me,	The substance under study																			
Culture test	Exposure time, (min)	XVII				XVIII				XX			XXI				XXXIV			/	
Cult	Expo (1	2	3	4	1	2	3	4	1	2	3	4	1	2	3	4	1	2	3	4
Asper-	20	-	-	Ŧ	+	-	I	±	+	I	Ħ	+	+	1	-	Ŧ	+	-	-	-	-
gillus	40	-	-	-	-	-	-	±	+	-	-	±	+	-	-	±	+	-	-	-	±
flavus	60	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
	80	-	-	-	±	-	-	-	-	-	-	-	±	-	-	-	-	-	-	-	-
А.	20	-	-	±	+	-	-	±	+	-	-	±	+	-	-	-	±	-	-	-	±
niger	40	-	-	±	+	-	-	-	±	-	-	-	-	-	-	-	-	-	-	-	+
	60	-	-	±	+	-	-	-	±	-	-	-	-	-	-	-	-	-	-	-	±
	80	-	-	-	+	-	-	-	±	-	-	-	-	-	-	-	-	-	-	-	±
Candi-	20	-	-	±	+	-	1	-	±	-	-	-	+	-	-	-	±	-	-	-	-
da	40	-	-	±	+	-	-	-	-	-	-	-	-	-	-	-	+	-	-	-	-
albi-	60	-	-	+	+	-	-	-	-	-	-	+	+	-	-	-	+	-	-	-	-
cans	80	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
Penicil	20	-	-	Ŧ	+	-	-	-	+	1	-	1	1	1	-	Ŧ	+	-	-	-	-
-lum	40	-	-	-	±	-	-	-	±	-	-	-	+	-	-	-	-	-	-	-	-
Cyclo-	60	-	-	-	±	-	-	-	±	-	-	-	-	-	-	-	+	-	-	-	-
piym	80	-	-	-	±	-	-	-	±	-	-	-	-	-	-	-	-	-	-	-	+

 Table 7. Antibacterial effect of compounds

Note: Note: "+" *indicates full,* " \pm " - *partial endings,* "-" *indicates no endings.*

The study of antifungal activity of the presented substances was studied by the method of dilution. It has been shown that these substances have antifungal activity, but the effect of this effect depends on both the duration of exposure and the concentration of the substance being tested. Thus, as a result of dilution of 1% alcohol solution of the tested substance in the ratio of 1:10 and 1: 100 during the whole exposure, antifungal activity is sharply observed and no growth is observed in any variant.

Subsequent dilutions, in some cases, partial growth is also recorded.

Thus, in conclusion, it can be noted that the antifungal activity of compounds XVIII, XXI and XXXIV is relatively high and their use for these purposes is appropriate.

RESULTS

- 1. Epoxy(met)acrylate resins with a triglyceride cycle at the end of the molecule based on propanthriol and polyoxychlorpropylene triglyceride oligoesters and (met)acrylic acids were synthesized, and their composition and structure were determined. Hybridized epoxy compositions based on synthesized epoxy(met)acrylate resins were obtained.
- 2. Epoxy(met)acrylate oligo esters containing chlorine atom were synthesized. It was shown that by changing the molar ratios of the raw materials and the conditions of the reaction, the process can be directed mainly to the production of mono-, di- and tri (met)acrylate oligo esters. The synthesized chlorine-containing epoxy(met)acrylate oligo esters were tested as a modifier in ED-20 epoxy resin, and compositions with high physical-mechanical and self-extinguishing properties were obtained.
- 3. Hybridized compositions were obtained from a mixture of polyoxychlorpropylene triglyceride oligo ether and ED-20 epoxy resin, their physical-mechanical and dielectric properties were determined.

- 4. A composition based on polyoxychloropropylene epoxy(met)acrylate oligo esters and epoxy resin in ED-20 was obtained and their hardening process with polyethylene polyamine (PEPA), N,N¹-diaminodiphenylsulfon, bromendic anhydride and properties of related materials were studied.
- 5. The process of UV- curing of synthesized polyoxychloropropylene epoxy (met) acrylate oligo ether was studied. It was found that the double bonds contained in epoxy(met)acrylate oligoesters polymerize to form a thin layer of transparent coating.
- 6. Condensation reaction of urea with propanriol triglyceride ester was carried out, the reaction successively synthesized propanthriol mono-, di- and tri carbamide oligoesters. High-yield polyoxypropylene-substituted oxazolidine-2 was obtained by heating the synthesized carbamates in DMFA and applied as a modifier in ED-20 epoxy resin. Compositions based on synthesized polyoxypropylene-substituted oxazolidin-2-on ED-20 epoxy resin were prepared and its hardening process with PEPA was studied by differential-thermal analysis method.
- 7. Diepoxide ester of monochlorpropandiol was obtained from the reaction of monochlorpropandiol with 3-chlorine-1.2-epoxy-propane, and epoxy(met)acrylate oligoester of propanediol was synthesized from its reaction with (met)acrylic acids. Self-extinguishing compositions with high physical and mechanical properties have been developed on the basis of epoxy(met)acrylate oligoether.

The main content of the dissertation is published in the following scientific articles and theses:

 Mustafaev, A.M., Mustafaev, S.G., Huseynov, I.A., Ismayilova, R.I. [et al.]. Synthesis and properties of epoxymethacrylate based on three-beam triglycidyl oligoether // Republican Scientific Conference dedicated to the International Year of Chemistry, -Sumgayit: - June 29-30, -2011, - p.22-24.

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Address: AZ5004, Sumgayit city, S.Vurgun street, 124.

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