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SYNTHESIS, POLYMERIZATION AND INVESTIGATION OF PROPERTIES OF CYCLOPROPANE-CONTAINING MONOMERS WITH VARIOUS SUBSTITUENTS

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AUTOABSTRACT

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of doctor of philosophy in chemistry (PhD in Chemistry)

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The dissertation has been carried out at the laboratory of "Photosensitive and optically transparent polymer materials" of the Institute of Polymer Materials of the Ministry of Science and Education of the Republic of Azerbaijan.

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GENERAL CHARACTERISTIC OF THE WORK

The actuality of theme and the degree of its development. Progress in new fields of science and technology can be achieved by creation of products with the required characteristics, the process of transition from traditional materials to new, multifunctional ones with the creation of materials with complex properties, which can be adjusted. Such materials include, first of all, the polymer materials, with their properties, chemical composition, structure and size of macromolecules. Besides taking into account all of the above-mentioned characteristics, one of the important factors can be considered the determination of the composition and structure of the compounds introducing into the composition of newly created materials, i.e., macromolecules.

The main purpose of the dissertation was the synthesis of a special class of polymers[1] differing with specific properties for substitution of the traditional polymer materials currently used in the fields such as aviation, space technology, machine construction, electrical engineering and microelectronics for polymer materials with higher properties: heat resistance[2], high strength, higher wear resistance, chemical and radiation resistance[3], etc.

The object and subject of the investigation. The object of the investigation is the synthesis of new styrene monomers containing cyclopropane with various substituents and the preparation of homopolymers and copolymers with some known monomers based on them, the study of their structure, composition

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[1] Гулиев, К.Г.Синтез и свойства циклопропансодержащих оптически прозрачных / К.Г.Гулиев, С.Б.Мамедли, А.М.Гулиев // Журн. Прикл. химии, – 2010, Т.83, №11, – с.2020-2023.

[2] Гулиев, К.Г. Синтез и свойства эпоксисодержащих полициклопропил-стиролов / К.Г.Гулиев, Г.З.Пономарева, А.М.Гулиев // Высокомолек. соед., – 2007. Т. 49Б. № 8, – с. 1577-1581.

[3] Гулиев, К.Г. Сополимеризация 2-хлорзамещенных п-циклопропил-стиролов со стиролом и изучение свойств полученных сополимеров / К.Г.Гулиев, Г.З.Пономарева, А.М.Гулиев // Журн. Прикл. хи¬мии, – 2005. Т.78. №2, – с. 316-319.

and properties. The subject of the research work was the study of the physical-chemical, mechanical, photosensitive and optical properties of transparency of the synthesized monomers and polymers obtained on the basis of these monomers. In this regard, among the various types of macromolecular compounds, the polyfunctional polymers are in the center of the greatest attention for solving the tasks we face. The main feature of such polyfunctional polymers is that their macromolecules consist of fragments and rings with functional groups.

Purpose and tasks of the investigation. The purpose of the work is the development of the scientific foundations of the preparation of new functional polymers and study of the regularities and reaction products of synthesis, homopolymerization and radical binary copolymerization of new cyclopropane-containing functional polymers with styrene, methyl methacrylate and glycidyl methacrylate, development of methods of preparation of the reactive polymer materials with a complex of specific properties, containing functionally substituted cyclopropane groups and fragments on the basis of chloromethyl, phenoxy, chlorophenoxy, diethylaminomethyl in the side chain. The resulting high-molecular compounds combine properties such as photosensitivity, optical transparency and biological activity and have wide application possibilities in the irreplaceable fields of science and technology.

For achievement of this purpose, the following investigations have been carried out:

- The monomers of *para*-(2-substituted) cyclopropylstyrol have been synthesized. The compounds with chloromethyl, phenoxy, chlorophenoxy, and diethylaminomethyl functional groups int position 2 of the cyclopropane ring have been obtained from these compounds.
- The homopolymerization of the synthesized monomers has been carried out, the general and kinetic regularities of the homopolymerization process have been studied and some kinetic parameters have been determined. The copolymerization reactions of these monomers with styrene,

methyl methacrylate and glycidyl methacrylate as a binary system have been also investigated and the compositions and structures of the obtained copolymers, including their microstructures, have been determined.

- The properties of photosensitivity, physical-mechanical, elastic, optical transparency and biological activity of the synthesized copolymers, depending on their composition and structure, have been studied.

Methods of investigation. The results of the investigations carried out on the topic of the dissertation work have been obtained using the following methods of physical-chemical analysis: infrared spectroscopy (Agilent Cary 630 FTIR), dilatometry (dilatometer manufactured on the basis of the IIRT-1 device), Vicat heat resistance, and the thickness of the obtained films has been measured using a microinterferometer "LINNIK MMI-4". The optical density of the copolymer solutions was measured on FEK-56M photoelectrocolorimeter and the resists exposition has been carried out using an ISM-3U electron microscope. DRT-220 mercury lamp was used as a light source during the investigation of photosensitive properties, and a RTN–105 radiation thermelement was used for measurement of the light intensity.

Scientific novelty of the investigation. The synthesis of new cyclopropane-containing styrene monomers with various substituents, their radical homopolymerization in a block and solution in the presence of radical, initiator, binary copolymerization reactions with styrene, methyl methacrylate, and glycidyl methacrylate. New reactive polyfunctional high-molecular compounds sensitive to influence of UV beams, optically transparent, highly adhesive, impact-resistance and other properties have been obtained and the following scientific innovations have been achieved.

Depending on the conditions for preparation of the synthesized compounds, their compositions and structures, including microstructure have been studied, the general and kinetic reagularities of the reactions, and copolymerization constant values have been determined. It has been shown that the radical homo- and copolymerization of newly obtained *para*-(2-substituted) cyclopropyl styrene occurs due to the opening of the double bond in the vinyl group. The cyclopropane ring with various substituents in *para*-positionof the aromatic ring of the synthesized initial monomers remains unaffected in the the side chain of macromolecule.

İt has been established during the investigation that the introduction of functional cyclopropyl fragment into *p*-position of the benzene ring of the styrene molecule increases their activity in copolymerization reactions in comparison with styrene, methyl methacrylate and glycidyl methacrylate. Thus, the introduction of electron–donor groups in position-2 of the cyclopropane ring leads to a decrease in this effect, and the introduction of electron-acceptor groups – to its increase.

It has been established that the copolymers synthesized on the basis of these new monomers have high optical transparency and the ability of cross-linking under the influence of rays of various nature, i.e. photosensitivity properties. It has been shown that by adjusting the molar quantity of cyclopropyl fragments in a macromolecular chain, the copolymers with close indices of photosensitivity, but essentially differing by elasticity and adhesive properties can be synthesized.

Theoretical and practical significance of the investigation. In the dissertation work, the effective methods of the synthesis of new polyfunctional reactive homo- and copolymers based on functionally substituted cyclopropyl styroles have been developed, which are of great importance for the modern microelectronic and optoelectronic industries, sensitive to light beams, optically transparent, showing high adhesion, elasticity, strength and other properties. The lithographic properties of the synthesized homo- and copolymers open up the possibility of their use in microelectronics as high-quality polymer photoresists. It has been established that the developed negative polymer resists can be used in integral microschemes of manufacturing technology and in electronic lithography processes. **Positions submitted for defense:**

- Synthesis monomers of *para*-(2-substituted) cyclopropyl styrene, structural properties, general regularities of the radical homo- and binary polymerization reactions (styrene, methyl methacrylate, glycidyl methacrylate), the composition, structure and properties of the obtained polymers.
- Kinetic regularities of the homopolymerization reactions of cyclopropane-containing polyfunctional styrene monomers, the order of activity of monomer, activation, kinetics and other determined parameters of monomers.
- Determination of the mechanism of the radical copolymerization reactions of the synthesized monomers with monomers of styrene, methyl methacrylate and glycidyl methacrylate, the results of experiments and conclusions based on them.
- The dependence of photosensitivity, optical transparency, high adhesive and elastic properties of the obtained polymers on the composition, structure and nature of functional groups of macromolecules.

Approbation and application of the investigation. 21 scientific papers, including 7 articles, 14 conference materials have been published on the topic of the dissertation. The main results of the dissertation work were reported and discussed at the following International and Republic Scientific Conferences: International Scientific-technical Conference "Polymer Composites and Tribology" (Gomel-Belarus, June 27-30, 2017), Functional monomers and special polymer materials: Problems, prospects and practical views (Sumgait, November 15-16, 2017), Conference dedicated to 120th anniversary of Surkhay Akhundov (Baku, 2017), Scientific Conference "Nagiyev's Reading" (Baku, 2018), International Scientific-practical Conference "Innovative prospects of the development of the oil refining and petrochemistry" dedicated to 110th anniversary of Academician V.S.Aliyev (Baku, October 9-10, 2018), International Scientific-technical Conference "Polymer Composites and Tribology" (Gomel-Belarus, 2019), 6th International

Caucation Symposium on Polymers and Advanced Materials (Georgia, July 17-20, 2019), International Scientific-practical Congress on "Actual problems of Medicine 2020", dedicated to the 90th anniversary of the Azerbaijan Medical University (Baku, December 19-20, 2020), The International Conference:Modern Problems of Theoretical and Experimental Chemistry devoted to 90th anniversary of honored scientist academician Rafiga Aliyeva, Baku State University (Baku, September 29-30 2022), Abstracts of VIII All-Russian Conference with international participation "Technical Chemistry. From theory to practice", dedicated to the 300th anniversary of the Russian Academy of Sciences (Perm, September 9-13, 2024).

Name of the organization, where the dissertation work has been done. The dissertation work has been carried out at the laboratory “Photosensitive and optically transparent polymer materials” of the Institute of Polymer Materials of the Ministry of Science and Education of the Republic of Azerbaijan

Structure and volume of the dissertation. The dissertation consists of an introduction, 4 chapters, conclusions, and a list of 131 cited references. The materials of the dissertation have been reflected on 159 computer text, including 33 figures, 13 tables. The total volume of the dissertation is 211529 symbols: introduction – 17120, I chapter – 47129, II chapter – 25536, III chapter – 49161, IV chapter – 67883, conclusions – 4700 symbols.

The first chapter of the work has been devoted to the analysis of literature data on the preparation of styrene-containing polyfunctional polymers and the manufacture of negative-type resists based on them, as well as methods for preparation of the photosensitive polymer materials containing styrene and cyclopropane and the analysis of scientific and patent investigations on their structural features and properties.

In the second chapter the physical-chemical characteristics of the initial substances used for carrying out of the investigations, synthesis of monomers of *para*-(2-substituted)cyclopropyl styrene, determination of *Q-e* parameters of the obtained monomers, study of of the composition, structure and properties of the synthesized homo- and copolymers, and also the methods of determination of the main parameters of kinetic regularities of the carried out reactions have been described.

In the third chapter the investigation of the method of preparation of homo- and copolymers on the basis of synthesized new cyclopropane-containing polyfunctional styrene monomers as a result of their radical homopolymerization and copolymerization reactions has been shown.

In the fourth chapter, the possibilities of using newly synthesized newhomo- and copolymers have been considered. Thus, the physical-mechanical, heat-physical, adhesive, photosensitive and optical properties of the synthesized homo- and copolymers have been studied. Taking into account the high operational indices of the obtained polymers, their use in such important and modern fields as aviation, space technology, electrical engineering and microelectronics has been recommended.

1. MAIN CONTENT OF THE DISSERTATION SYNTHESIS AND HOMOPOLYMERIZATION OF MONOMERS OF 2-CHLOROMETHYL 1-(P-VINYLPHENYL)CYCLOPROPANE, 2-PHENOXYCARBONYL 1-(P-VINYLPHENYL) CYCLOPRANE, 2-CHLOROPHENOXYCARBONYL 1-(P-VINYLPHENYL)CYCLOPPANE AND 2-DIETHYLAMINO-METHYL 1- (P-VINYLPHENYL) CYCLOPROPROPANE

The polymer materials possessing properties of photosensitivity and optical transparency are widely used in various fields of modern science and technique, especially in microelectronics and optics, which are in great demand. Currently, the use of polymers and materials made on their basis for modern electronics, and also optically transparent polymers in the optical field is very important problem for polymer chemistry. Thus, the resist materials made from polymer materials, including optical details, substitute many valuable materials necessary for other fields of industry, and, most importantly, silver compounds, which are considered the most economically efficient material for the microelectronic industry.

A brief review of scientific data devoted to the development of convenient methods for synthesis of functionalized polymers, especially functionalized polystyrene, for solving these problems, the well-thought-out choice of their properties, functional groups, and a study of application possibilities showed that the most convenient method for creation of functionalized polystyrene derivatives for various purposes with new, higher operational indices and a complex of special properties is the method possessing with great synthetic capabilities.

Naturally, p-(2-functionallysubstituted)cyclopropyl styrene has special properties and attracts much attention as a monomer for the synthesis of polyfunctional polymers. In this regard, some research works are known in the scientific literature on radical homo- and copolymerization of a number of their representatives **[100-107].**

It should be noted that the carried out purposeful investigation has been directed namely to the preliminary purposeful selection of monomers of various functional groups and the improvement of photosensitivity, optical transparency, physical-mechanical, heat-physical properties in cyclopropane-containing polymers on their basis and other necessary properties (lithographic and at the same time, one of the important adhesive property of resist material).

Thus, the preparation of the polymer products with various functional groups in the side chain of macromolecules obtained on the basis of synthesized monomers has been assumed. It should be noted that for the synthesis of such proposed polyfunctional substituted polystyrenes, the monomers of *p*-(2-functionally substituted) cyclopropyl styrene have been synthesized for the first time and the polymers based on them have been obtained by radical homopolymerization, their general regularities and structural properties of the obtained products have been studied. The obtained scientific results have been investigated and compared with some data of the scientific literature in this field.

Synthesis of the compounds of 2-ethoxycarbonyl 1-(p-vinylphenyl) cyclopropane, 2-hydroxycarbonyl 1-(p-vinylphenyl) cyclopropane, 2-chloromethyl 1-(p-vinylphenyl) cyclopropane. The main purpose of the dissertation was the synthesis of new functionalized cyclopropyl styrenes, the study of the general regularities of the carried out reactions and the possibilities of their practical use. For this purpose, the methods of the synthesis of styrene monomers containing cyclopropane with various functional substituents have been initially developed, and further the optimal conditions of the carried out reactions have been determined. 1,4-divinylbenzene, primarily produced in the industry and ethyldisoacetate synthesized by us have been used as the initial materials for the synthesis of *para*-(2-substituted) cyclopropyl styrene monomers. For carrying out this reaction, the reaction of 1,4-divinylbenzene with freshly distilled ethyldiazoacetate in a medium of pentane in the presence of anhydrous $CuSO_4$ catalyst and as a solvent (diluent) at temperature 323°C for ~4 h has been carried out and p-(2-ethoxycarbonyl) cyclopropyl styrene (ECCPS) has been synthesized with yield 95%.

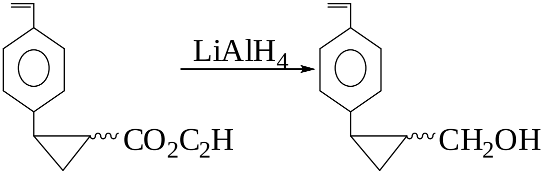
The reaction conditions have been described in detail in the experimental part.

The course of this reaction takes place according to the scheme presented below:

The main purpose during the reaction is the preparation of the reaction product (ECCPS) with high yield and prevention of the occurrence of side reactions (formation of esters of maleic and fumaric acid or cyclopropanation of both vinyl groups in 1,4-divinylbenzene) or prevention their formation, a fourfold excess mole content of 1,4-divinylbenzene relative to ethyldiazoacetate is taken. The optimal reaction conditions have been determined by gradually adding diazoacetate drop by drop into the reaction medium and simultaneously maintaining a temperature of 318-323 K.

It has been detected that temperature has a great influence on the carrying out of this reaction. Thus, keeping the temperature higher 323 K creates conditions for additional side reactions. Thus, during carrying out of the reaction at temperature of 353 K, both vinyl groups of 1,4-divinylbenzene are opened, creating conditions for the simultaneous formation of ethyl esters of malein and fumar. It has been determined that at ratio of the components 1:2 included in the initial reaction and rapid transfer of diazoacetate into the reaction medium with the thermocatalytic method, the reaction proceeds in parallel in both directions, i.e. in the direction of preparation of substances A and B. It should also be noted that in all cases it is preferable to obtain the substance A, i.e, A > B.

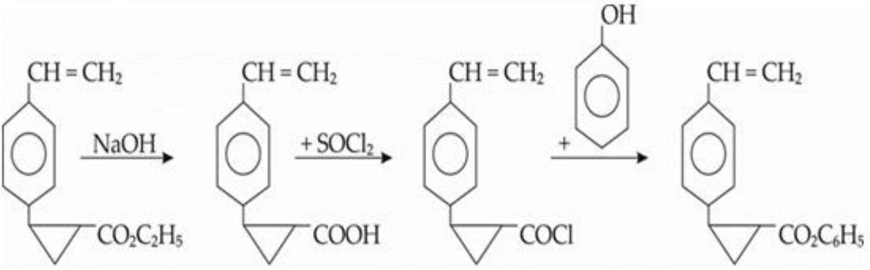
With the aim of preparation of cyclopropane monomer with hydroxyl group in 2-position, it has been developed the method of preparation of compound of *para*-(2-hydroxymethyl) cyclopropyl styrene (HMCPS) with 85% yield by reduction of the synthesized *para*-(2-ethoxycarbonyl) cyclopropyl styrene in an anhydrous medium of diethyl ether through $LiAlH_4$ at room temperature for 4 h.

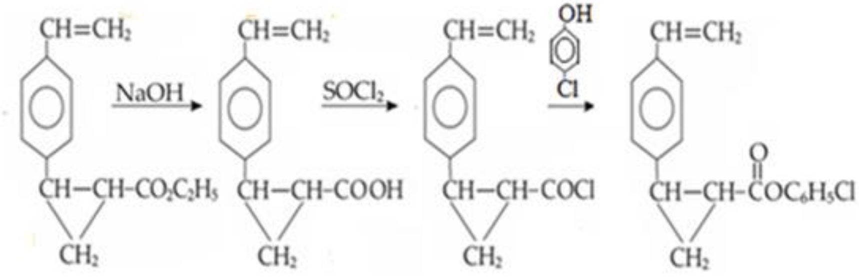


The synthesis of monomer of 2-chloromethyl 1-(p-vinylphenyl) cyclopropane (CVCP) has been synthesized by the interaction of PCl_3 with HMCPS, respectively. The synthesis proceeds according to the following reaction scheme:

It has been established that this reaction was carried out at low temperature, i.e. at negative temperature.

Synthesis of the compounds of 2-phenoxycarbonyl 1-(p-vinylphenyl) cyclopropane, 2-chlorphenoxy-carbonyl 1-(p-vinylphenyl) cyclopropane. The synthesis of the compounds of 2-phenoxycarbonyl-1-(p-vinylphenyl) cyclopropane (PhCVCP) and 2-chlorophenoxycarbonyl-1-(p-vinylphenyl) cyclopropane (CCVCP) opens up wide possibilities for the creation of new types of polymers, both in biological properties and as important monomers of polyfunctional compounds, obtaining highly biological, optical and photosensitive polymers owing to them. As a result of the saponification reaction of the previously synthesized ethoxycarbonylcyclopropyl styrene (ECCPS), the compound of hydroxycarbonylcyclopropyl styrene (HCCPS) has been synthesized, as a result of the interaction of thionyl chloride with it, HCCPS chloroanhydride has been obtained, respectively. The synthesis of these compounds is usually carried out by the following reaction.





The spectra (IR and NMR) of each of these synthesized compounds have been taken and the structure of these compounds has been determined **(Fig 1.1. and 1.2.).**

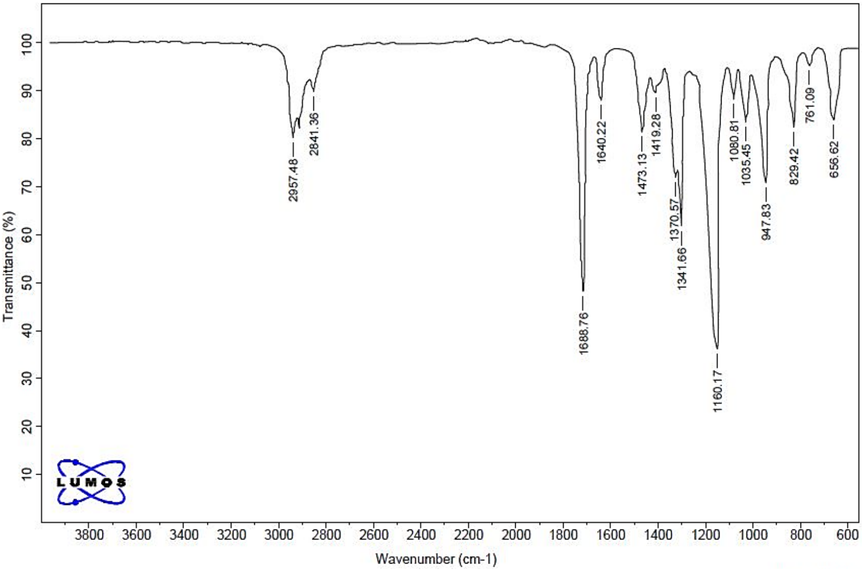


Fig. .1.1. IR spectrum of CCVCP monomer

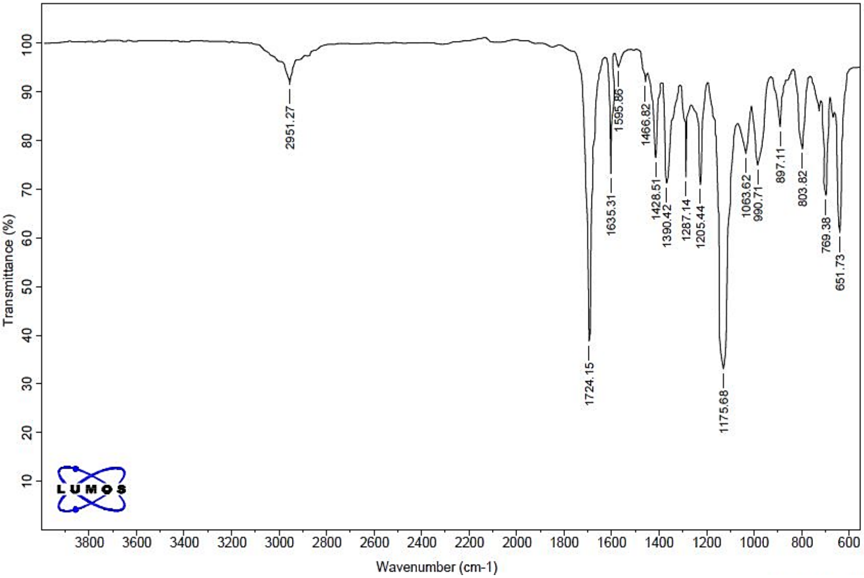


Fig. .1.2. IR spectrum of PhCVCP monomer

The absorption bands at 1635 cm^{-1} -1640 cm^{-1} and 1035 cm^{-1} - 1053 cm^{-1} in the molecules of PhCVCP and CCVCP compounds indicate the availability of both a vinyl group and a cyclopropane ring. In addition, the absorption band at 656 cm^{-1} in CCVCP molecule indicates the availability of a chlorine atom in this molecule **(Fig. 1.1.)**

It can be seen from the IR spectra that all functional groups in the composition of molecules keep their characteristic absorption bands. Thus, in all cases, in the IR spectrum, the intensive absorption bands in the field of 1500 and 1600 cm^{-1} characteristic for vinyl group in the benzene ring, 700 and 760 cm^{-1} characteristic for C-O group in the benzene nucleus, 1635 cm^{-1}, 947 and 990 cm^{-1} characteristic for (-CH_2=CH-) group, 1150 and 1205 cm^{-1} characteristic for –C-O- group, at the same time 1710 and1724 cm^{-1} characteristic for >C=O are observed.

The absorption band at 1710 cm^{-1}, the second absorption band (1724 cm^{-1}) can be considered as isomers belonging to PhCVCP and CCVCP monomers in *trans*-, and *sis*-position. It should be noted that the values of these observed absorption bands in the esters of saturated carboxylic acids are considerably lower than the values of the absorption bands of the carbonyl group (1750-1740 cm^{-1}).

Thus, after determination of the fact that the synthesized compounds of PhCVCP and CCVCP really consist of two geometric isomers, it became necessary to separate these geometric isomers due to the necessity for more detailed study of the peculiarities of their structure during their separation. For this purpose, it was possible to isolate PCVCP compound by a method of crystallization at negative temperatures (223K)**.** As a result, *trans*- and *cis*-geometric isomers of PhCVCP compound have been separately divided, and some of their physical-chemical indices have been determined and analyzed.

As mentioned above, the PMR spectra of all synthesized PhCVCP and CCVCP compounds have been taken and identified for further clarification of their structure. The resonance proton signals characteristic for both the vinyl group (δ=5.20÷5.68 ppm) and the cyclopropane ring (δ=0.75-1.65 ppm) have been detected **(Fig 1.3** and **1.4.)**. At the same time, the signals corresponding to the protons of the benzene ring (δ=7.0-7.5 ppm) are observed in the NMR spectra of these compounds.

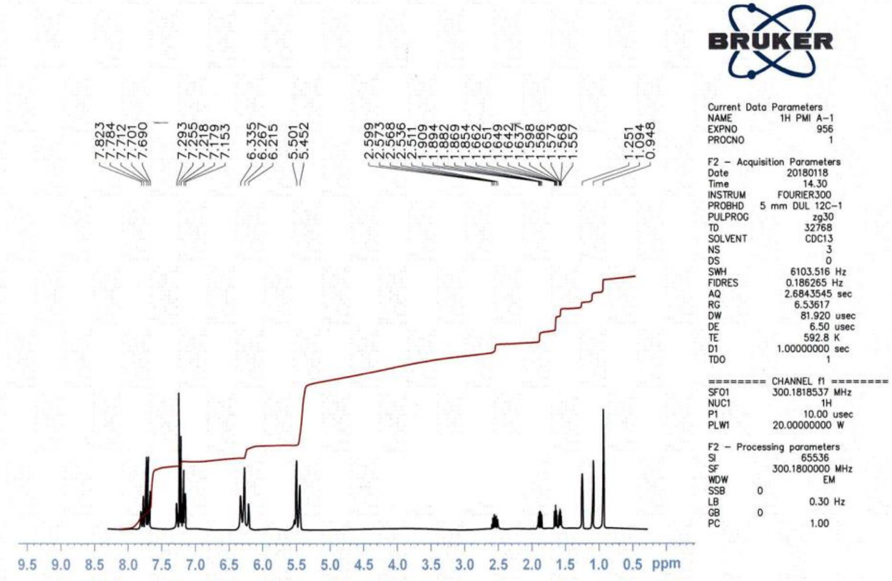


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Fig. 1.3. NMR spectrum of PhCVCP monomer

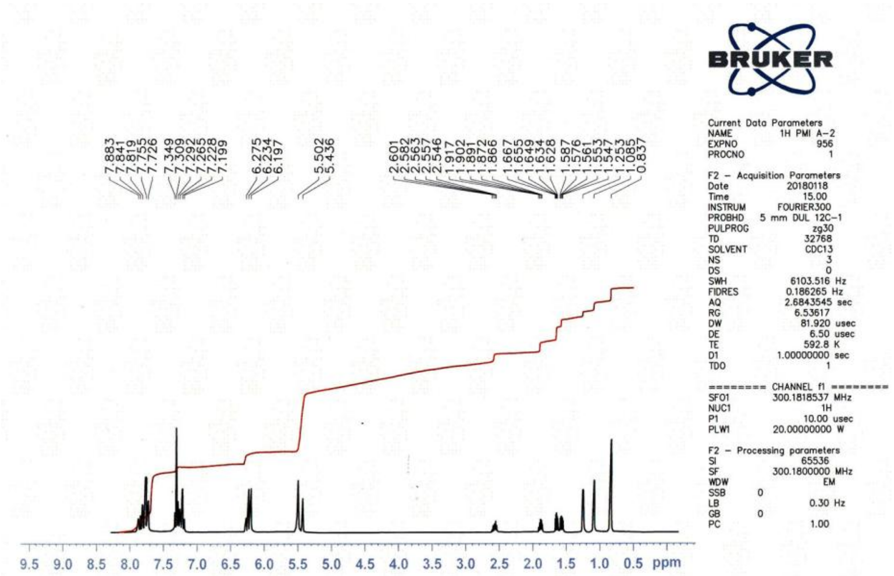


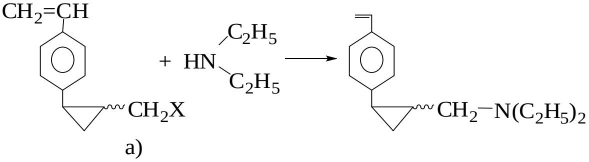
Fig. 1.4. NMR spectrum of CCVCP monomer

In all spectra, the absorption bands at 1170, 1160, 1080, 1110, and 1240 cm^{-1} belongs to (-c-o-c-) of the ether group. The absorption bands corresponding to ~1710 and 1720 cm^{-1} are very close and are observed in the shape of scissors. It has been also determined that this fact refers to both *trans*- and *cis*- stereoisomers.

Synthesis of monomer of 2-diethylaminomethyl-1-(p-vinylphenyl)cyclopropane. Cyclopropane-containing compounds have many special properties and are capable of meeting the modern industrial and technical requirements. Some of their representatives are used in medicine as drug, and in agriculture as insecticides and fungicides. On the other hand, in recent years, some representatives of cyclopropane-containing compounds are successfully used as monomers in polymerization processes.

The synthesis of such compounds was mainly due to the achievement of two upcoming goals**.** The first of them has been based not only on the study of the biological properties of these compounds, but also on the creation of highly resistive polymer materials with a special complex of properties on the basis of their radical polymerization.

For the synthesis of cyclopropane-containing compounds similar to open-chain amine compounds, we have established the possibility of synthesis of cyclopropane-containing amine compounds (2-diethylaminomethyl 1-(p-vinylphenyl) cyclopropane (DAVCP) correspondingly, as a result of the interaction reaction of halogen (CH_2X) fragment compounds with amines. The reaction is carried out according to the following scheme.



X=Cl, Br

Radical homopolymerization of synthesized p-(2-functional substituted) cyclopropyl styrene. As mentioned above, the monomers of 2-phenoxycarbonyl 1-(p-vinylphenyl) cyclopropane (PhCVCP) and 2-chlorophenoxycarbonyl 1-(p-vinylphenyl) cyclopropane (CCVCP), containing an active double bond in the form of a vinyl group in a molecule, which is easily subjected to the polymerization and having cyclopropane ring have been synthesized as initial monomers used for radical homopolymerization.

The homopolymerization reaction of the cyclopropane-containing monomers synthesized by us in the presence of the AIBN initiator has been carried out in mass or solvents (benzene, toluene, dioxane), closed glass ampoules, in a medium of nitrogen or helium, in the temperature range 333-353°C. K, within 4-5 h. In all cases, it has been established in study of the regularities of the polymerization process of monomers under selected conditions that the degree of conversion has been carried out in the range of ~10-12%.

These homopolymers, usually precipitated and dried, are white solid substances in appearance and macromolecular compounds.

In this case, the yield of each monomer involved in the polymerization process has been determined separately at different time intervals. Based on the obtained values, the graphics of the dependence of yield on time have been constructed **(Fig. 1.5).**

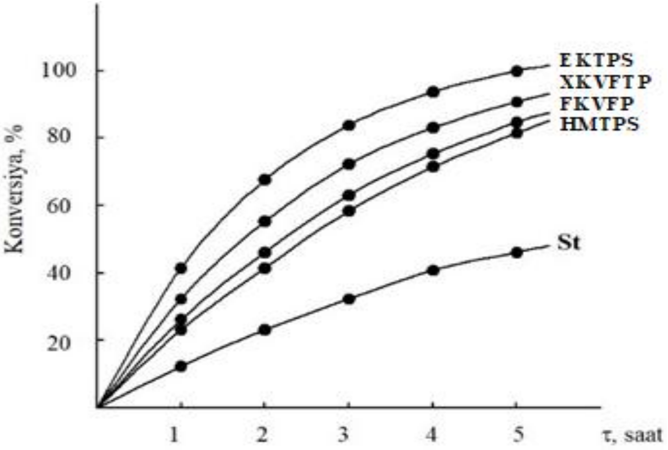


Fig. 1.5. Dependence on time of the degree of deep conversion of monomers of p-(2-functionally substituted)cyclopropyl styrene in the presence of AIBN, at temperature 70°C and in mass

It can be seen from the graphics that the polymerization reactions rate for these compounds is relatively different. It has been established that the reason for this regularity is the availability in the synthesized compounds of the above-mentioned electron-transfer coupling system of the electronic influence of cyclopropane ring substituents on the double bond. It was established as a result of the investigation that the most active monomer is ETKTPS monomer.

A number of investigations have been carried out for solution of some problems arising during the polymerization process of these p-cyclopropylstyrene substituted compounds.

Under the action of UV rays, the polymer containing CVCP is easily structured, and the coating obtained on its basis has a small defect and becomes insoluble.

The investigation of the photochemical structuring process of the polymer is described in. It should also be noted that due to the availability of groups (cyclopropane and C-Cl) strongly absorbing light energy, the synthesized polymer is photosensitive and undergoes photochemical conversions leading to the formation of a netlike structure at irradiation with UV rays. The photoreactive fragments have been investigated in thin layers of 15-150 mg at various polymer concentrations. In the UV spectrum, the polymers show absorption bands at 296 and 300 nm. The influence of radiation on a photosensitive polymer has been also investigated by measuring it in the UV spectrum.

Methodology of study of the kinetics of the radical polymerization reactions of functionally substituted cyclopropyl styrene. It is known that during polymerization of functionally substituted vinyl monomers, on the basis of which functional polymers are obtained, the steric hindrances due to large functional groups arise. In this case, depending on the nature of the substituent in the vinyl group, an increase or decrease of the electron density occurs. So, the monomers in compounds with -J-effective substituents are strong electron acceptors, and in compounds with +J-effective substituents are electron donors. It should also be noted that such monomers, in which the vinyl group is located near the volume substituent, are polymerized even worse

For study of the polymerization ability of functionally substituted monomers, we have compared the polymerization process with the results obtained during the polymerization of both styrene itself and para-substituted styrenes structurally close to these compounds. At the same time, the influence of various factors on the reactivity of the initial monomers has been clarified. Undoubtedly, the results obtained during these investigations are useful for the development of the theory of radical polymerization of *para*-substituted styrenes in general.

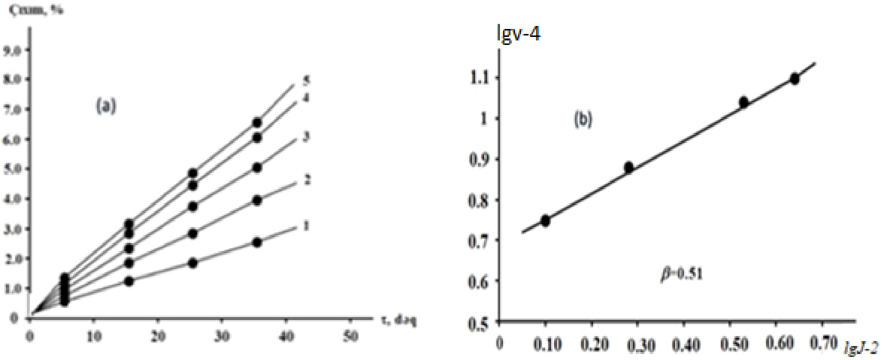


Fig. 1.6. Dependence of the yield on the time and the polymerization reaction rate on the concentration of the initiator (AIBN)

a) Kinetic curves of polymerization of PhCVCP monomer at various initiator concentrations: $[M]_0$=1.0 mol/l, T=340К, [AIBN], mol/l: 0.06 (1), 0.05 (2), 0.04 (3), 0.03 (4); 0.02 (5);

b) Logarithmic dependence of the initial polymerization rate of PhCVCP monomer on the initial concentration of the initiator.

As the results obtained from experiments show, the phenoxycarbonyl-substituted cyclopropyl styrene monomer showing a strong electron-acceptor influence due to the three-membered cyclopropane and phenyl ring is very easily polymerized under conditions of radical initiation.

The homopolymerization process of 2-phenoxycarbonyl 1-(p-vinylphenyl)cyclopropane (PhCVCP) has been carried out in a benzene solution at a monomer concentration of 0.16-1.39 mol/l, in the temperature range of 65-70°C.

In this case, the following process parameters have been determined: the composition on monomer and initiator, and also the activation energy.

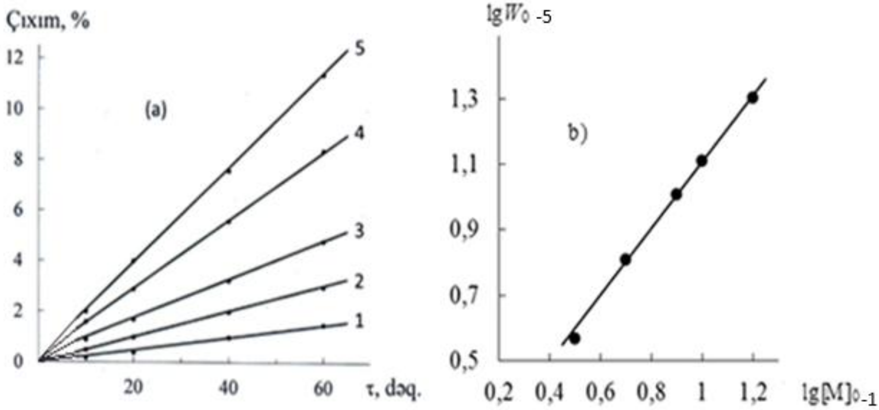


Fig. 1.7. Dependence of the yield on the time and the polymerization reaction rate on the concentration of the initiator. a) Kinetic curves of polymerization of PhCVCP monomer at various initiator concentrations: T=335 К, [ABIN]$_0$=0.03 mol/l, [M]$_0$, mol/l, 1.3 (1), 1.0 (2), 0.70 (3), 0.45 (4) və 0.20 (5);
b) Logarithmic dependence of the initial polymerization rate of PhCVCP monomer on the initial concentration of the initiator.

We find the quantities α and β from the graphics. Thus, the found values of β (reaction order on initiator) and α (reaction order on monomer) are common for the radical polymerization process and therefore do not require the further discussion.

Besides the orders determined on the monomer and initiator, k values (determined on the ratio of the rate constants of separate stages) and activation energy (determined on the temperature dependence of the polymerization rate) have been determined. The reaction order of the polymerization process per monomer (determine by the dependence of lgW0 on lg[FTPS]0) is equal to one. The reaction order on the initiator is assumed to be equal to 0,5 in determination based on the dependence of lg[AIBN] on lgW0. The activation energy value was 82.7 kJ/mol **(Fig. 1.8.).**

It has been established that the found values of the orders on the monomer and initiator, as well as the activation energy, correspond to the values of vinyl monomers obtained by a method of free radical polymerization known from the literature.

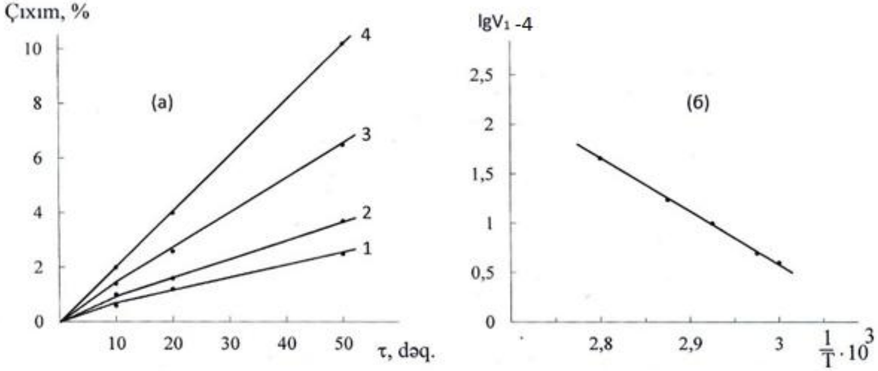


Fig. 1.8. Dependence of the yield on the time and polymerization reaction rate on temperature.

a) Kinetic curves of polymerization of PhCVCP monomer at various temperatures:: $[M]_0$=2.0 mol/l, [ABİN]$_0$=0.05 mol/l, T, K: 340 (4), 335 (3), 330 (2) və 325 (1); b) – dependence of lgk on reverse temperature

With the aim of investigation of some properties of the obtained poly-p-(2-functionally substituted)cyclopropyl styrenes, and also elucidation of the influence of substituents on the polymerization process, their polymerization process has been carried out under the same conditions. The polymerization has been carried out in a dilatometer under selected standard conditions: monomer concentration – 1 mol/l, initiator concentration –10^{-3} mol/l. For quantitative estimation of the reactivity, the initial rates have been determined at low monomer conversion depths (up to 12%) and the effective polymerization constants have been calculated on the basis of $V = k \cdot [M] \cdot [J]^{1/2}$ equation.

Investigation of the characteristic viscosity, some physical-mechanical, heat-physical and other properties of polymers on the basis of cyclopropane-containing monomers. One of the important problems was the study of the solubility of the initially obtained polymers with the aim of processing of the synthesized polymers. It has been established that these cyclopropane-containing polymers being highly soluble in many solvents: benzene, $CHCl_3$, CCl_4, toluene, dimethylformamide, etc., they are insoluble in water, alcohols and other similar solvents. At the same time, it should be noted that poly-HMCPS is slightly soluble in methyl and ethyl alcohols.

As is known, one of the important indices of the polymers is their molecular weight. These indices can be determined by a method of gel-penetrating chromatography (GPC), as well as by the characteristic viscosity, i.e., by the Mark-Kung-Huawink method.

2. INVESTIGATION OF THE REGULARITIES OF COPOLYMERIZATION REACTIONS OF THE SYNTHESIZED MONOMERS WITH STYRENE, METHYL METHACRYLATE AND GLYCIDYL METHACRYLATE, THEIR PRODUCTS AND DETERMINATION OF APPLICATION POSSIBILITIES

The selection of comonomers for copolymerization of the synthesized monomers of CVCP, PhCVCP, CCVCP and DAVCP was advisable with the aim of study of the regularities of the copolymerization reactions, investigation of the formed reaction products, as well as to express an opinion on the mechanism of the copolymerization process and make scientific conclusions based on them. So, styrene (St), methyl methacrylate (MMA) and glycidyl methacrylate (GMA) have been taken as standard monomers for copolymerization reactions, the regularities of this process have been investigated, the influence of many factors on the composition, structure and properties of the formed multifunctional copolymers has been clarified, at the same time the development of effective methods of preparation of copolymers of controlled composition possessing the required properties has been achieved. The use of existing St, MMA, GMA as monomers has been stipulated by the favorability of the investigation of the carried out radical copolymerization process on the one hand, and on the other hand –

their low cost.

Copolymerization of 2-chloromethyl-1-(p-vinylphenyl) cyclopropanes, 2-phenoxycarbonyl-1-(p-vinyl-phenyl)cyclopropane, 2-chlorophenoxycarbonyl-1-(p-vinyl-phenyl) cyclopropane and 2-diethylaminomethyl-1-(p-vinyl-phenyl)cyclopropane with styrene. As mentioned earlier, one of the important problems of the dissertation work is the synthesis of a new class of p-(2-phenoxycarbonyl, chlorophenoxycarbonyl, chloromethyl, diethylmethyl)-substituted cyclopropane-containing cyclopropyl styrene (CVCP, PhCVCP, CCVCP and DAVCP), determination of regularities of binary radical copolymerization reactions with styrene as a standard monomer and analysis of the results of the investigation of the obtained products. This selection allows to study easily the regularities of substitution of functional groups in the cyclopropane ring in copolymerization reactions with styrene (St) and determine the influence on the composition, structure and properties of the obtained polyfunctional copolymers, and at the same time to develop the effective methods of preparation of copolymers of regulated composition showing the required properties.

The conditions of carrying out of the copolymerization reactions of DAVCP system+ St and composition of the synthesized copolymers (T = 343 K, exciter AIBN, solvent – benzene) are given in **Table 2.1**.

Table 2.1.

DAVCP+St copolymerization constants and Q-e parameter values

Quantity of monomers in the initial mixture (mol %)		**Quantity of copolymer mol, %**		**Copolymerization constants and Q-e parameters**				**Microstructure of copolymer**		
M_1	M_2	m_1	m_2	r_1	r_2	Q_1	e_1	Lm_1	Lm_2	R
10	90	14.58	85.42	1.00	0.58	3.1	-1.4	1.10	5.02	37.71
25	75	31.62	68.38					1.33	2.63	48.36

50	50	53.38	46.62					2.04	1.50	52.61
75	25	76.29	23.71					4.21	1.14	47.38
90	10	97.13	2.87					10.69	1.04	15.69

Based on the copolymerization constants values, found in the Tables (r1=$_1$.00; r_2=0.58 for DAVCP+St system), it can be said that CCVCP and DAVCP monomers show themselves as more active monomers in the radical copolymerization reactions than styrene

Copolymerization of 2-chloromethyl 1-(p-vinylphenyl) cyclopropane, 2-phenoxycarbonyl 1-(p-vinylphenyl) cyclopropane, 2-chlorophenoxycarbonyl 1-(p-vinylphenyl) cyclopropane, 2-diethylaminomethyl 1-(p-vinylphenyl) cyclopropane with methyl methacrylate. In the IR spectra of the polymer synthesized on the basis of comonomers of DAVCPs with MA, two intensive peaks in the field of 1720-1730 cm^{-1} are clearly visible **(Fig. 2.1.).**

In this field of absorption, the peaks have the form of scissors. The peaks observed in this scissor form (1720-1730 cm^{-1}) prove the availability of two carbonyl groups in the macromolecule. One of them probably characterizes the >C=O group in PhCVCP and CCVCP molecules, and the other corresponds to >C=O group in methyl methacrylate.

It has been established that the absorption bands corresponding to the wavelength of 1720 cm^{-1} belong to the carbonyl group of monomers of poly-PhCVCP and poly-CCVCP, and 1730 cm^{-1} belongs to >C=O group of polymethyl methacrylate.

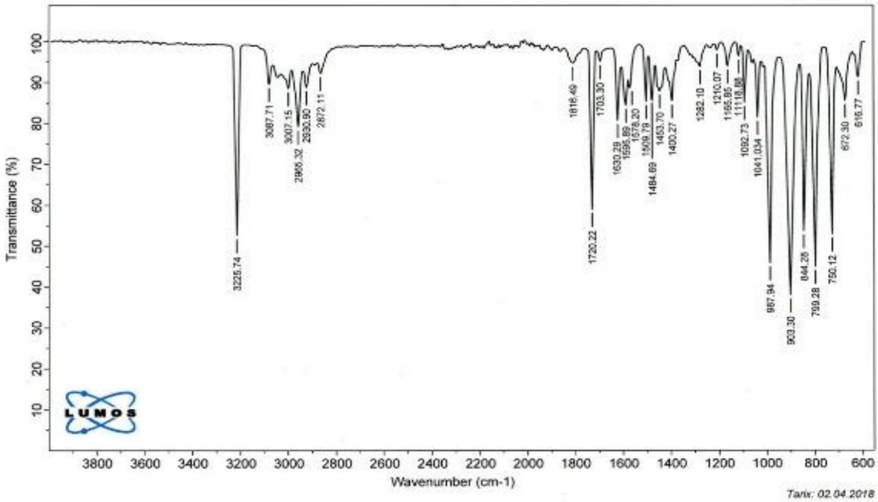


Fig. 2.1. IR spectrum of DAVCP-co-MMA copolymer.

In the IR spectra of the synthesized copolymers, the absorption bands corresponding to the valence vibrations of C-H bond in the -CH_2- and -CH- groups are 2930÷2945 cm^{-1} and 2860÷2975 cm^{-1}, at the same time, the absorption bands characterizing the aliphatic CH_3 group are also observed in the frequency range of 2980 cm^{-1} and 2800-3000 cm^{-1} connected to each other.

As an example, some physical-mechanical and heat-physical properties of the synthesized copolymers of CCVCP+St and CCVCP +MMA and their corresponding homopolymers are presented in the following table **(Table 2.2.)**.

Table 2.2.

Some physical-mechanical and heat-physical properties of CCVCP+St and CCVCP+MMA copolymers and their corresponding homopolymers

Indices	CCVCP +St	CCVCP +MMA	Poly-St	Poly-MMA	Poli-CCVCP
Light transmission	1.56	1.60	1.59	1.49	1.6
Vicat heat-	125	120	103	115	130

resistance °C					
Brinell hardness, kg·mm^{-2}	18.0	15.0	14	13	20.5
Relative impact strength, kg·cm·cm^{-2}	18.0	18.8	18	12	21.0
Tensile strength, MPa	34.6	52.0	35.0	60.0	45.0
Adhesion strength, MPa	4.2	5.4	-	-	8.3
Water absorption, 24 h	0.08	0.16	0.1	0.3	0.08

Note: the copolymers have been synthesized from a mixture of monomers at ratio M_1:M_2=40:60 (mol%), i.e. containing ~60 MMA links.

Indeed, the data shown in **Table 2.2** indicate that the synthesized copolymers have high strength, adhesion, physical-mechanical and other indices. So, these copolymers practically maintain their transparency at the same level even after heating at a temperature 373 K for 2 h.

The high adhesion strength and special impact resistance parameters of the synthesized cyclopropane-containing copolymers in comparison with both PSt and PMMA allow them to be used in the manufacture of contact lenses, optical devices and separate details of apparatus.

Investigation of the copolymerization reactions of chloromethyl 1-(p-vinylphenyl)cyclopropane, 2-phenoxycarbonyl 1-(p-vinylphenyl)cyclopropane, 2-chlorophenoxycarbonyl 1-(p-vinylphenyl)cyclopropane and 2-diethylaminomethyl 1-(p-vinylphenyl)cyclopropane with glycidyl methacrylate and study of the properties of the synthesized copolymers.

For full achievement of the purpose set in the investigated dissertation work, it was necessary to study the regularities of the binary radical copolymerization reactions of the synthesized p-(2-substituted)cyclopropyl styrene with glycidylmethacrylate and to investigate the obtained products. The styrene derivatives containing various cyclopropane-containg functional fragments: PhCVCP, CCVPCP, CVCP and DAVCP have been used as new monomers in copolymerization.

The occurrence of copolymerization process in the investigated systems has been confirmed by data of the turbidimetric titration analysis method. The availability of a single curve on the turbidimetric titration curves indicates that in the the system the copolymer and not a mixture of two homopolymers has been obtained (**Fig.2.1**).

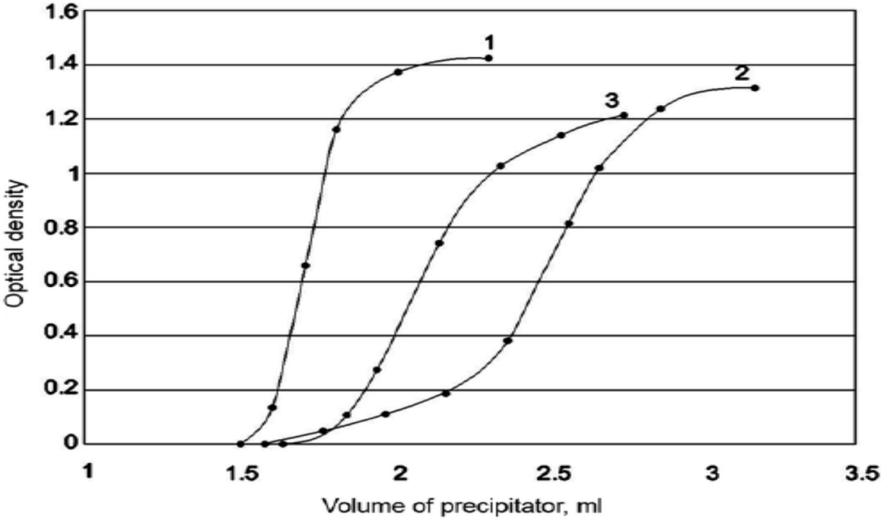


Fig 2.1. Turbidimetric titration curves of a copolymer containing CVCP co-GMA (1-10% GMA in the initial mixture, t=70°C, τ= 2 h; 2-50% GMA in the initial mixture, t=70°C, τ = 2 h, 3-75% GMA in the initial mixture, t=70°C, τ=4 h.

During carrying out of the copolymerization process, the content of CVCP and GMA rings is differed in the copolymers formed in any composition of the initial monomer mixture and changes from 8.95 to 91.05 mol. Thus, it is clear that the initial monomer composition is the main factor determining the properties of copolymers. It has been established that the composition of the formed copolymers depends on the composition of the initial monomer mixture.

In the UV spectrum of copolymers, they belong to the absorption bands connected with π→π ransitions from photoreactive fragments and located around 292 and 300 nm, respectively. The influence of irradiation on photosensitive polymers has been investigated by measuring changes in the UV spectrum. The intensity of absorption in the various irradiation intervals (**Fig. 2.2**) is in the ratio 59.35:40.65, respectively, which indicates changes in the UV spectrum.

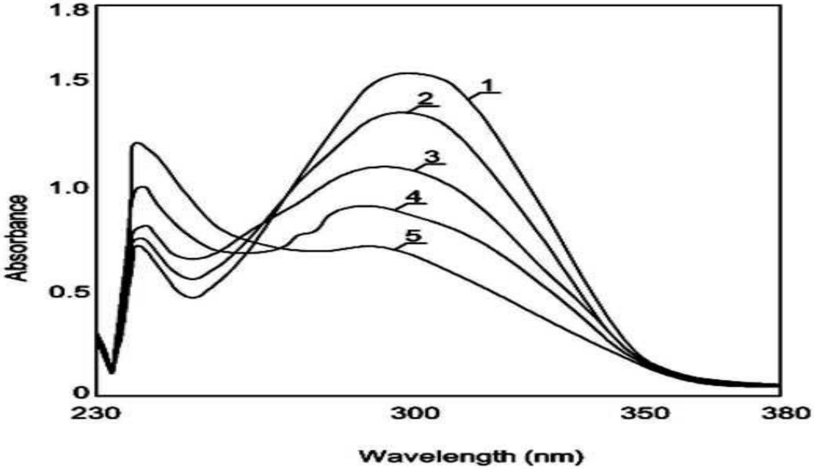


Fig 2.2. Change of the absorption in the UV spectrum of the composition prepared from the copolymer coating of poly CVCP-co-GMA at ratio of the copolymer [59,35:40,65], respectively. 1-5 in t=5,10,15,20,25 seconds of radiation, respectively.

It is seen from the character of the change in the UV spectra of the copolymer that in the early stages of irradiation, the change rate observed at a maximum in the range of 292-300 nm depends linearly on the irradiation time, but after 30 and 60 seconds the process decelerates and almost completely disappears within 5 minutes of irradiation.

The intensive course of the photochemical processes in copolymers has been stipulated by the availability of strong absorbing groups in them, such as glycidyl, cyclopropane, carbonyl, chlorine atoms, etc., which increase the sensitivity of copolymers to ultraviolet rays and cause photochemical conversions, which in turn leads to the crosslinking of polymer chains.

Under the influence of UV radiation, the copolymer based on CVCP and GMA is easily subjected to structuring, as a result of which the polymer coatings are insoluble and have low microdeficiency. The copolymers capable of photoreaction have been investigated in thin layers with mass of 15-150 mg at various concentrations.

It has been established that the sensitivity of the samples in the short-wavelength part of the spectrum strongly depends on the coating thickness. So, with the coating thickness, the three-dimensional structure formed after irradiation has the form of a loose net with large holes, which swells strongly during exposure and is compressed on the polymer layer druing, causing the appearance of folds and cracks. It has been established that the best results are obtained when working with coatings with thickness of 0,2-03 mcm.

CONSLUSİONS

1. The methods of the synthesis of *p*-cyclopropyl styrenes with various substituents (ethoxycarbonyl, hydroxymethyl, chloromethyl, phenoxycarbonyl, chlorphenoxycarbonyl and diethylaminomethyl) in *para*-position of the cyclopropane ring and the aromatic ring of styrene have been developed. It has been established that ethoxycarbonylcarbene, formed as a result of the reaction of ethyldiazoacetate with *p*-divinylbenzene in the presence of catalyst ($CuSO_4$) is regioselectively connected with one of the vinyl groups of *para*-divinylbenzene due to electrophilic combination with formation of *para*-(2-ethoxycarbonyl)cyclopropyl styrene compound. At the following

stages, the various chemical conversions have been carried out and hydroxymethyl-, chloromethyl-, phenoxycaronyl-, chlorophenoxycarbonyl- and diethylaminomethyl-substituted cyclopropane-containing monomers have been synthesized. It has been established as a result of GLC and spectral (IR and NMR) analysis that these compounds consist of a mixture of 2 geometric isomers. **[1,2,3,4,5].**

2. The regularities of the copolymerization reactions of these monomers in the presence of radical initiators, structural characteristics and composition of the obtained homopolymers have been established. It has been shown that the homopolymerization of *p*-(2-substituted)cyclopropyl styrenes in mass and in solvent is carried out by opening the double bond of the vinyl groups of the molecule, as a result of which the polymers capable of dissolving in a number of organic solvents, remaining untouched by the macromolecule together with cyclopropane substituents are obtained. **[1, 2].**
3. It has been established that the activity of these synthesized monomers in homopolymerization reactions has been closely connected with the nature of substituents. It has been shown that a monomer with an electron-acceptor substituent behaves as a more active monomer in homopolymerization than a monomer with an electron-donor nature. It has been established that the observed activity has been stipulated by a more stable growing macrodical due to the transfer of the inducing action of the substituent through cyclopropane and benzene rings. **[3,4].**
4. The kinetic regularities of polymerization of the synthesized monomers have been studied, the activation energy and the the process order on components (0,5 by initiator, 1,0 on monomer order) have been determined, it has been established that these processes are characteristic for radical polymerization, and the process is subjected to the regularities of polymerization of vinyl monomers. **[4, 5].**
5. The regularities of binary copolymerization reactions of *para*-(2-substituted)cyclopropyl styrene with various vinyl monomers

(styrene, methyl methacrylate, glycidyl methacrylate) in the presence of AIBN have been investigasted, the copolymerization constants (r_1 and r_2) for selected binary systems, Q-e parameters, molecular weight parameters, their composition and structural peculiarities, monomer composition of the macromolecule have been determined. It was found that the relative activity of these cyclopropane-containing monomers (M_1) with various types of substituents in copolymerization reactions is higher than the activity of styrene, MMA, GMA monomers ($r_1 > r_2$). It has been also established that this activity has been connected with the specific structure of the cyclopropane ring. **[6, 7, 8, 9].**

6. It has been established using UV spectra that these synthesized compounds confirm the possibility of formation of a single bound system and show that the cyclopropane ring is a part of this bound system. On the results of the analysis of spectral (IR and NMR) analyses, the copolymerization of *para*-(2-substituted) cyclopropyl styrenes with the used comonomers, as in the case of homopolymerization, occurs only due to of vinyl groups. The molecular weight parameters of the obtained copolymers have been determined by gel chromatographic method. It has been established that the obtained polymers practically consist of copolymers. At this time, it has been also found out that the comonomers cannot form homopolymers separately. **[6, 7].**
7. It has been established due to the linear structure of the obtained homo- and copolymers and the availability of various reactive fragments on the side of macromolecules that these polymers possess a complex of high physical-mechanical, heat-physical and other properties. It has been established that these homo- and copolymers are very processed, practically do not create difficulties in preparation of products in any form. It has also been shown that in comparison with polystyrene, such cyclopropane-containing polymers have higher adhesion, plasticity and impact resistance.
   [6, 7].

8. It has been revealed that the availability of photosensitive fragments in the macromolecular chain facilitates the preparation of photoresists based on them, and these polymers are easily structured under the action of UV rays and form resistive materials of a negative type. For the latter ones, the corresponding conditions of high photosensitivity, other lithographic indices and their preparation for use in solid and thin polymer coatings with lower microdefects and high adhesive properties have been determined. **[8, 9].**

Recomendation

The synthesized cyclopropane-containing polymers can be used as a negative-type resistive material in the making of integral microschemes in microelectronics, and MMA and styrene copolymers – in optoelectronics as optically transparent polymer materials.

List of published scientific papers on the topic of the dissertation:

1. Guliyev, K.G. Copolymers based on (p-vinylphenyl) cyclopropylmethylchlorocinnamate and methyl methacrylate /K.G.Guliyev, A.İ.Sadıgova, A.M.Aliyeva [et al.] // Russian Journal of Applied Chemistry, -New York: -2017, V. 90, № 2, -p.262-266.
2. Гулиев, К.Г., Садыгова, А.И., Алиева, А.М. [и др.] Синтез фоточувствительных полимеров на основе 2-замещённых циклопропилстиролов // Международная научно-техн. конф. «Полимерные композиты и трибология», -Гомель: -27-30 июня, -2017. - с. 114.
3. Quliyev, K.Q., Sadıqova, A.İ., Məmmədli, S.B. [və b.] Tsiklopropan tərkibli birgəpolimerlər əsasında foto- və optiki şəffaf polimerlərin sintezi //Funksional mono-merlər və xüsusi xassəli polimer materiallar problemlər,perspektivlər və praktiki baxışlar. Beynəlxalq elmi konfrans, -Sumqayıt: -15-16 noyabr, -2017, -s. 125.

4. Гулиев, К.Г., Садыгова, А.И., Гулвердашвили, Ц.Д. [и др.] Синтез и бактерицидные свойства 2-замещенных циклопропилстиролов. //prof. Surxay Axundovun 120 illik yubileyinə həsr olunur, -Bakı: -2017, s. 246-252.
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6. Гулиев, К.Г. Синтез 2-хлорметил-1-(п-винилфенил) циклопропана и его сополимеризация со стиролом / К.Г.Гулиев, Г.З.Пономарёва, А.И.Садыгова [и др.] // Журнал прикладной химии, – 2018. Т. 91. Вып. 2, – с. 105-110.
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9. Гулиев, К.Г., Садыгова, А.И., Алиева, А.М. [и др.] Синтез и полимеризация феноксикарбонил п-циклопропилстирола // Международная научно-техн. конф. «Полимерные композиты и трибология», -Гомель: -25-28 июня, -2019. - с. 42.
10. Guliyev, K.G., Sadıgova, A.İ., Gulverdashvili, Ts.D. Synthesis of optical transparent polymeron the basis of p-chlorophenoxycarbonylcyclopropyl styrene // 6th International Caucasian Symposium on Polymers and Advanced Materials, Georgia: -17-20 July, -2019, p.43.

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15. Sadigova, A.İ. Copolymers on the basis of (P-chlorophenoxycarbonyl) cyclopropyl styrene with styrene and methyl methacrylate // Azerbaijan Chemical Journal, Baku: – 2022, № 3, – p. 45-50.
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17. Sadıgova A.İ. Compliance Study of Copolymerization Process of 2-Chloromethyl-1-(P-Vinylphenyl) Cyclopropane with Methyl Methachrylate // Journal of Baku Engineering University, Chemistry and Biology, Baku: -2023, Vol:7, №:1, -

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