DOI: 10.6060/ivkkt.20196202.5764

УДК: 678.762.3

# СИНТЕЗ И ИССЛЕДОВАНИЕ СВОЙСТВ ЭЛАСТОМЕРСОДЕРЖАЩИХ КОМПОЗИЦИЙ НА ОСНОВЕ СООЛИГОМЕРОВ ПОЛИОКСИПРОПИЛЕНГЛИКОЛЯ И 4,4'-ДИФЕНИЛ-МЕТАНДИИЗОЦИАНАТА

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***Проведено исследование в целях получения высокоадгезионного и высокоэластичного покрытия клеящей и уплотняющей композиции. Получены соолигомеры полиоксипропиленгликоля и 4,4'-дифенил-метандиизоционата, изучена кинетика сополиконденсации. Установлено, что реакция уретанообразования протекает по миграционной полимеризации, основанной на присоединении к атому азота подвижных водородных атомов полиэфира. Методом ИК-спектроскопии исследована морфология синтезированных соолигомеров. Показано, что совмещение соолигомера с бутилкаучуком (сополимером изобутилена и изопрена) увеличивает эластичность системы, снимает усадку и повышает термо-, химическую стойкость. С целью получения адгезионного и высокоэластичного композиционного материала изучены смеси полиуретанового соолигомера с бутилкаучуком (БК), имеющего высокие показатели теплостойкости, эластичности, химической стойкости в агрессивных средах. Показано, что при смешении полиуретанового сополимера с бутил каучуком происходит образование полимерной системы, макромолекулы которой, механически взаимодействуя между собой, образуют так называемые взаимопроникающие сетки. Указанная композиция также наиболее устойчива в агрессивных средах, что делает ее применение перспективным. Сравнение данных дифференциально-термического анализа соолигомера и композиции соолигомер+бутил каучука показывает, что полученная композиция термически более устойчива, чем соолигомер. В работе решена важная научно-техническая проблема повышения качества адгезионных композиций. В результате проведенного исследования разработаны рекомендации по улучшению свойств адгезионных композиций, конкурентоспособных по отношению к существующим аналогам, по рецептуре строения, технологии получения и использования адгезионных композиций. Полученные указанным путем композиции могут быть использованы в производстве полимерных изделий для нефтяной, машиностроительной промышленности.***

**Ключевые слова:** химическая стойкость, полиуретановый соолигомер, бутилкаучук, полимеризация, теплостойкость

# SYNTHESIS AND STUDY OF PROPERTIES OF ELASTOMER-CONTAINING COMPOSITIONS BASED ON COOLIGOMERS OF POLYOXYPROPYLENE GLYCOL AND 4,4'-DIPHENYL-METHANE DIISOCYANATE

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***A study was conducted in order to obtain a highly adhesive and highly elastic coating of the adhesive and sealing composition. Cooligomers of polyoxypropylene glycol and 4,4'-diphenyl-methane diisocyanate were obtained and their copolycondensation kinetics was studied. It has been established that the urethane formation reaction proceeds through a migration polymerization***

***based on the addition of mobile hydrogen atoms of the polyester to the nitrogen atom. The morphology of the synthesized cooligomers was studied by IR spectroscopy. It was shown that the combination of cooligomer with butyl rubber (a copolymer of isobutylene and isoprene) increases the elasticity of the system, relieves shrinkage and increases the thermal and chemical resistance. In order to obtain an adhesive and highly elastic composite material, we studied mixtures of polyurethane cooligomer with butyl rubber (BK), which has high rates of heat resistance, elasticity and chemical resistance in aggressive environments. It was shown that when polyurethane copolymer is mixed with butyl rubber, a polymer system is formed, the macromolecules of which mechanically interact with each other to form so-called interpenetrating grids. This composition is also the very stable in aggressive environments, which makes its application promising. Comparison of the data of differential thermal analysis of the cooligomer and the composition of cooligomer + butyl rubber shows that the resulting composition is thermally more stable than the cooligomer. An important scientific and technical problem of improving the quality of adhesive compositions has been solved. Because of the study, recommendations have been developed for improving the properties of adhesive compositions that are competitive with existing analogues, according to the formulation of the structure, technology for the preparation and use of adhesive compositions. The compositions obtained in this way can be used in the production of polymer products for the petroleum and engineering industries.***

**Key words:** chemical resistance, polyurethane cooligomer, butyl rubber, polymerization, heat resistance

**Для цитирования:**

Мустафаева Р.Э. Синтез и исследование свойств эластомерсодержащих композиций на основе соолигомеров полиоксипропиленгликоля и 4,4'-дифенил-метандиизоцианата. *Изв. вузов. Химия и хим. технология*. 2019. Т. 62. Вып. 2. С. 94–100

**For citation:**

Mustafayeva R.E. Synthesis and study of properties of elastomer-containing compositions based on cooligomers of polyoxypropylene glycol and 4,4'-diphenyl-methane diisocyanate. *Izv. Vyssh. Uchebn. Zaved. Khim. Khim. Tekhnol.* 2019. V. 62. N 2. P. 94–100

## INTRODUCTION

The chemical and physic-mechanical properties of polyurethane are largely determined by the variety of chemical bonds of various types in its structure, and in fact constitute a block of copolymers in which the chain alternates between sections of different chemical nature. This feature of the chemical structure of polyurethanes helps to obtain materials on their basis with a wide range of physical and mechanical properties [1-14].

However, narrow temperature ranges of use and relatively high cost characterize products based on polyurethane, along with high strength and excellent wear resistance. The elimination of these drawbacks can be achieved by combining polyurethane with other polymers and, thus, significantly expand their field of application [15-24].

The object of this study was the preparation of an adhesive polymer composition based on industrial polyether – polyoxidepropylene glycol, combined at room temperature with 4,4'-diphenyl-methane diisocyanates and 5% butyl rubber in toluene solution. It is known that the structure and properties of polyurethanes can vary widely by choosing suitable initial raw materials. They are one of the few polymers that can be purposefully regulated by the number of cross-links, the flexibility of polymer molecules, and the nature of intermolecular interactions. Polyurethanes can be obtained by the interaction of compounds containing isocyanate groups with polyfunctional hydroxyl-containing derivatives. As isocyanate, we used 4,4'-diphenylmethane diisocyanate, widely used in the production of flexible polyurethanes. Polyhydroxypropylene glycol was used as a hydroxyl-containing compound, which served as a good hydrophobic component. Thus, polyoxidepropylene glycol mainly determines the complex of physic-mechanical properties of obtained polyurethane materials.

The creation of these polyurethane materials was carried out in order to obtain promising polymers characterizing the complex of valuable operational properties, namely: a high rate of heat resistance, elasticity, chemical resistance in aggressive media and water resistance. To obtain coatings based on them, film materials are used practically in all industries.

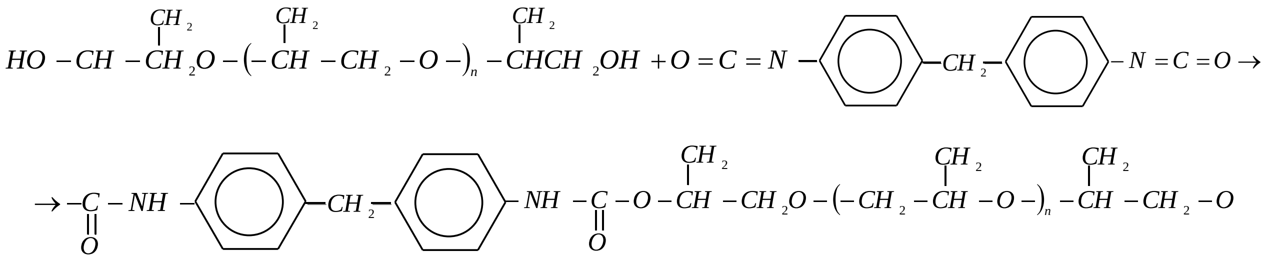
## EXPERIMENTAL METHOD

In work the following materials were used:

The industrial polyester of Laprol brand 2502 (TU-2226-026-10488-57-98) having the following physical and chemical characteristics: acid number, 0.1 mg KOH/g; molecular mass of 2500; the maintenance of OH-group, 6.7%; pH (a methanol: water –

70:30) – 6.1; the moisture content, 0.1%; viscosity kinematic at 30 °C, 540 $mm^2/s$. 4,4'-diphenyl-methanediisocyanate (MDI): melting temperature 40.5 °C; $d^{50}_4 = 1.850$; $n^{50}_d = 1.5906$. Butyl rubber (copolymer of isobutylene with isoprene) the BK-2045 brands (TU 38003169-74): viscosity according to Mooney at 100 °C, 8 min. – 45 ±4; not limitation – 2 ±0.2% mol.; $\rho = 865 kg/m^3$.

The combination of polyoxypropyleneglycol (PUR) with 4,4'-diphenyl-methanediisocyanate (MDI) was performed by mixing at room temperature. When polyglycolsare mixed with isocyanate type hardeners, the reaction of urethane formation proceeds according to the mechanism of migration polymerization, based on the ability to attach a polyether hydrogen atom to the nitrogen atom.



From the experimental results, it was revealed that the process of interaction of the components of the mixture is accompanied by an increase in viscosity with an increase in the concentration of 4,4'-diphenylmethanediisocyanate in Table 1.

*Table 1*

**The effect of the content of isocyanates groups on the viscosity of the reaction mixture**

***Таблица 1.* Влияние содержания изоцианатных групп на вязкость реакционной смеси**

| Indicator | Time of interaction, min | Molecular mass of mix, $\overline{M}_n$ | Dynamic viscosity, Pa·s |
|---|---|---|---|
| Maintenance of DMDIT, %: | | | |
| 30 | 60 | 800 | 20.0 |
| 20 | 60 | 500 | 15.0 |
| 15 | 60 | 300 | 13.0 |

It is shown that after 60 min the quantities of isocyanates groups' decreases to zero, which proves the course of chemical interaction due to the isocyanate groups. The copolycondensation process ends when they are fully consumed.

The viscosity of the reaction mass also increases with an increase in the copolycondensation time from zero to 60 minutes, and the time of complete consumption of the isocyanates groups coincides with the point of maximum viscosity of the cooligomer.

By extraction of the hinge plate of cooligomer with acetone in the Soxhlet apparatus we determined the degree of interaction, i.e. the output of polyurethane cooligomer (PUR), obtained with different ratios of the initial components.

*Table 2*

**Effects of the ratio of POPG: MDI on the output of polyurethane cooligomer**

***Таблица 2.* Влияния соотношения ПОПГ : ДМДИЦ на выход полиуретанового соолигомера**

| Name | The reaction mixture | | | | |
|---|---|---|---|---|---|
| | 1 | 2 | 3 | 4 | 5 |
| POPG: MDI, mass of the part | 100:10 | 100:15 | 100:20 | 100:30 | 100:40 |
| Output of PUR, % of mass | 90.5 | 92.5 | 95.6 | 95.0 | 94.8 |

As it can be seen from Table 2, the PURG:MDI ratio has a significant effect on the PUR output. The optimal ratio of POPG to MDI, which provides the highest yield of PUR (95.6% of possible), is 100:20.

The increase in the content of MDI in the reaction mixture to 30-40 parts by weight does not increases the yield of PUR. After depletion of OH groups, MDI remains an independent component in the system and, because of self-structuring, leads to a decrease in the elasticity of PUR.

PUR, along with high rates of physic-mechanical characteristics, possesses high chemical resistance. The thermal stability of PUR at elevated temperatures was studied using differential thermogravimetry (DTG) and differential thermal analysis (DTA) using a Paulik-Erdei system derivatograph with a temperature increase from 20 to 500 °C at a rate of 5 °C per minute.

$A1_2O_3$ was used as an inert substance; the sample weight was 200 mg (Fig. 1). Judging from the DTG curve, from a mark of 120 °C on the sample, unachieved POPG and MDI can diffuse with a weight loss of 5%. At around 340 °C, the destruction of the sample is fixed, and the mass loss in general is 84%.

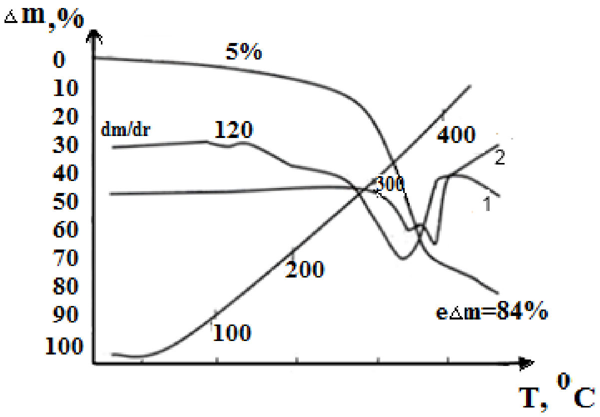


Fig. 1. DTA curves of a polyurethane cooligomer (1) and butyl rubber (2)

Рис. 1. Кривые ДТА полиуретанового соолигомера(1) и бутил каучука (2)

Judging from the DTG curve, from the 120 °C mark on the sample, unbound POPG and MDI can diffuse with a weight loss of 5%. At around 340 °C, the destruction of the sample is fixed, and the mass loss in general is 84%. To obtain a highly adhesive and highly elastic composite, we studied mixtures of polyurethane cooligomer with butyl rubber (VK), which have high heat resistance, elasticity, chemical resistance in aggressive media, and weather resistance and water resistance. It is shown that when PUR is mixed with BC, a polymer system is formed, the macromolecules of which mechanically interact, forming the so-called interpenetrating networks.

The crushed, suspended BC was placed in a laboratory reactor (V = 2.0 L) with toluene (or xylene) and stirred at 40-50 °C for 2-3 h until completely dissolved. After BK was completely dissolved in the solution, 4,4'-diphenyl-methane diisocyanate was added at a certain ratio of components and stirred for 1.5-2 h, after which the mixture was added to the mixture and stirred for another 50-60 min at room temperature. Compositions of prepared compositions with different component ratios are given in Table 3.

Of greatest interest was the compositional mixture 3. The IR-spectra of the POPG and MDI cooligomer (Fig. 2) was obtained by pressing it together with KBr (IR spectrophotometer Specord-75 JR). The components were taken in a ratio of 300 mg of salt and 2 mg of the test sample of cooligomer. The tablets were pressed at a pressure of 0.8 MPa. In the region of 1660 cm$^{-1}$, the absorption of C=O and C=N bonds is manifested. The frequency C=N band (1700 cm$^{-1}$) is shifted to the frequency range of the fluid due to conjugation of the C-NH group. In the spectrum of the mixture cooligomer and butyl rubber (Fig. 2, cf. 3), the absorption is 2900 cm$^{-1}$, 1400-1300 cm$^{-1}$. A 1710 cm$^{-1}$ band appeared, corresponding to a C=O group not related to the NH group. The absorption of 930 cm$^{-1}$ associated with $NH_2$ deformation vibrations increased. In all likelihood, during the formation of the composition, the CN bond is broken and the butyl rubber links are joined at the points where the bond is broken.

*Table 3*

**Compositions of prepared compositions based on mixtures of a solution**

***Таблица*. Составы композиций на основе смесей раствора**

| Name | Composition of mixtures | | |
|---|---|---|---|
| | 1 | 2 | 3 |
| Components, parts by weight:<br>5% solution of BK in: | | | |
| toluene | 100 | 100 | 100 |
| POPG | 100 | 100 | 100 |
| MDI | 10 | 15 | 20 |

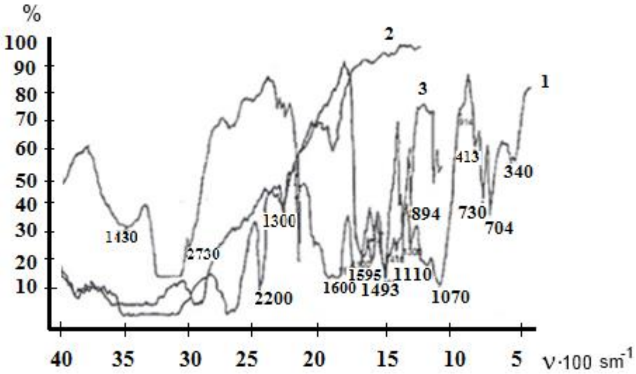


Fig. 2. IR spectra of polyurethane cooligomer (1), butyl rubber (2) and mixtures of PUR with BK (3)

Рис. 2. ИК спектры полиуретанового соолигомера (1), бутил-каучука (2) и смеси ПУСО с БК (3)

The thermal characteristics of the compositions are shown in a derivatogram (Fig. 3.). Two endothermic effects were recorded on the DTG curve: one at 150 °C, followed by a weight loss of 7.1%. Obviously, the solvent diffuses from the composition. The second peak of the DTG curve corresponds to a temperature of 362 °C, which probably indicates the beginning of the destruction of the composition, the mass loss is about 80%.

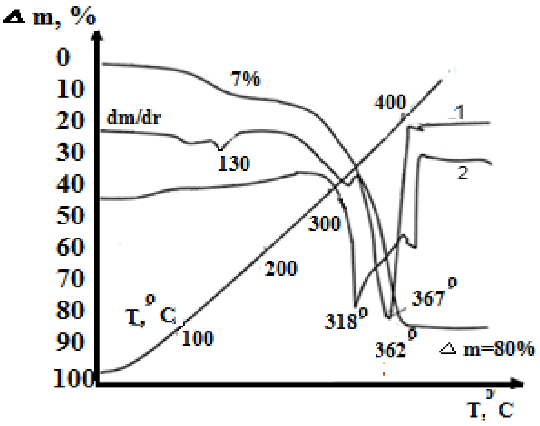


Fig. 3. Differential thermal analysis curves for thermooxidative degradation of the composition of polyurethane cooligomer (1) and a mixture of polyurethane cooligomer with butyl rubber (2)
Рис. 3. Кривые дифференциально-термического анализа при термоокислительной деструкции композиции полиуретоновой соолигомера(1) и смеси полиуретоновой соолигомера с бутилкаучуком(2)

***Table 4***

**Physical-mechanical properties of compositions of cooligomer + BK**

***Таблица 4*. Физико-механические свойства композиций полиуретанового соолигомера с бутилкаучуком**

| Indicators | Composition | | |
|---|---|---|---|
| | 1 | 2 | 3 |
| Tensile strength, MPa | 24.0 | 25.0 | 26.0 |
| Conditional voltage, MPa:<br>at 100% elongation<br>at 300% elongation | <br>11.0<br>17.0 | <br>11.7<br>18.1 | <br>12.5<br>22.5 |
| Relative elongation,% | 500 | 340 | 350 |
| Residual elongation,% | 40 | 45 | 20 |
| Tear resistance, N/mm | 41 | 54 | 60 |
| Adhesive strength, kN/m | 6.0 | 7.4 | 8.0 |
| Shorehardness, usl. units | 83 | 87 | 90 |
| Chemical resistance:<br>In $H_2SO_4$ (5%)<br>In $HNO_3$ (10%)<br>In HCl (30%) | <br>+<br>+<br>- | <br>+<br>+<br>- | <br>+<br>+<br>+ |

Comparison of the differential thermal analysis data of the co-oligomer and the cooligomer + BK composition shows that the resulting composition is thermally more stable than the initial cooligomer. The physic-mechanical properties of the obtained compositions have been studied, the results of which are presented in the Table 4.

Taking into account the values of IR, DTA, TGA and the results of the Table, it is clear that composition 3 based on a mixture of BK + POPG + MDI (100 + 100 + 20 parts by weight) is a highly adhesive polymer composition that has improved physical and mechanical properties.

## RESULTS AND DISCUSSION

Combining the cooligomer with butyl rubber (a copolymer of isobutylene and isoprene) increases the elasticity of the system, eliminates shrinkage and improves thermal and chemical resistance. A mixture of cooligomer with butyl rubber is recommended as a highly adhesive and highly elastic coating, adhesive and sealing composition. This composition is also the most stable in aggressive environments, which makes its application promising. The compositions thus obtained can be used in the manufacture of polymer products for the petroleum and engineering industries. The co-poly-condensation kinetics was studied, poly-oxide propylene glycol cooligomers and 4,4'-diphenyl-methanediisocyanate were obtained. It was found that the urethane formation reaction proceeds in accordance with migration polymerization, based on the addition of mobile hydrogen atoms of the polyester to the nitrogen atom. The morphology of the synthesized cooligomers was studied by IR spectroscopy. It has been shown that combining a cooligomer with butyl rubber (a copolymer of isobutylene and isoprene) increases the elasticity of the system, eliminates shrinkage and improves thermal and chemical resistance. A mixture of cooligomer with butyl rubber is recommended as a highly adhesive and highly elastic coating, adhesive and sealing composition.

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*Поступила в редакцию 28.02.2018*
*Принята к опубликованию 17.12.2018*

*Received 28.02.2018*
*Accepted 17.12.2018*