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ИССЛЕДОВАНИЕ СВОЙСТВ ОЛИГОМЕРОВ ИЗОПРЕНА, ПОЛУЧЕННЫХ ВОЗДЕЙСТВИЕМ МИКРОВОЛНОВОГО ИЗЛУЧЕНИЯ НА КАУЧУК СКИ-3

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Предложен способ получения олигомеров изопрена из каучука СКИ-3 воздействием микроволнового излучения на 10% раствор СКИ-3 в толуоле. Проведено исследование влияния мощности (144 Вт, 450 Вт, 675 Вт, 900 Вт) микроволнового излучения (МВИ) на степень деструкции СКИ-3. О глубине деструкции СКИ-3 судили по изменению значений средневязкостной молекулярной массы. Как показал анализ результатов экспериментов, процесс деструкции протекает по следующей закономерности: чем выше мощность микроволнового излучения, тем выше степень деструкции полиизопрена. При этом отмечено снижение молекулярной массы СКИ-3. Установлено, что фосфолипиды, используемые в качестве модификаторов СКИ-3, оказывают существенное влияние на процесс его деструкции. Увеличение количества фосфолипидов в системе толуол-СКИ-3 приводит к снижению степени деструкции полиизопрена независимо от мощности воздействия МВИ. Это проявляется в меньшем снижении молекулярной массы каучука в системе, где содержание фосфолипидов возрастает. Таким образом, фосфолипиды выполняют две функции: стабилизаторов, препятствующих процессу деструкции СКИ-3, и регуляторов степени деструкции каучука с получением олигомеров с определенной молекулярной массой. Полученные олигомеры использовали в качестве модификаторов изопренового каучука СКИ-3. Установлено, что введение олигомеров в резиновые смеси в количестве 7 и 10 мас.ч. на 100 мас.ч. каучука приводит к увеличению их когезионной прочности, а вулканизаты, содержащие олигомеры, характеризуются более высокими значениями физико-механических показателей.

Ключевые слова: олигомеры изопрена, каучук СКИ-3, микроволновое излучение, молекулярная масса, деструкция, фосфолипиды

STUDY OF PROPERTIES OF ISOPRENE OLIGOMERS OBTAINED BY MICROWAVE IRRADIATION OF SKI-3 RUBBER

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The present article proposes a method for production of low molecular weight rubber in microwave irradiation field. Isoprene rubber SKI-3 (10% solution in toluene) was used as the initial reactant. The effect of MWI power change (144 W, 450 W, 675 W, 900 W) on the degree of destruction was studied. The degree of destruction was measured based on the change in viscosity average molecular weight. The findings of the experiment data analysis indicate that the destruction process occurs in correspondence with the following dependency: the increase in the microwave irradiation power raises the destruction degree. At the same time, a decrease in molecular

weight was observed. Furthermore, it was found that the use of phospholipids as modifiers also affects the destruction process. The increase in phospholipids content in toluene-SKI-3 system reduces the degree of polyisoprene destruction regardless of the microwave irradiation power. It is demonstrated by the fact that rubber molecular weight is subject to a lower decrease in systems with higher phospholipids content. Phospholipids perform two functions. Firstly, they act as a stabilizer that inhibits the rubber destruction process. Secondly, varying their content allows controlling the destruction degree and obtaining oligomers with predetermined molecular weight. The obtained oligomers were used as SKI-3 isoprene rubber modifiers. It is observed that that the introduction of oligomers into rubber mixtures in the amount of 7 and 10 parts by weight per 100 parts by weight of rubber increases its cohesive strength. Furthermore, vulcanizates containing oligomers are characterized by improved physical and mechanical properties.

Key words: polyisoprene oligomers, SKI-3 rubber, microwave irradiation, molecular weight, destruction, phospholipids

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INTRODUCTION

The effect of microwave irradiation (MWI) on chemical reactions and physical processes is a relevant topic for scientific research. The research on this effect plays the leading role in intensification of these processes.

The effect of microwave irradiation on chemical reactions is usually measured by comparing the time spent to achieve the desired yield of the finished products with such observed when using conventional thermal heating. When carrying out processes in presence of MWI, it is necessary to account for molecular weight, polydispersity index and crystallinity of polymers, as well as for their mechanical (strength, elongation, modulus, viscosity) and thermal (glass transition point and melting point) properties. In some cases, products acquire properties that are not observed when conventional thermal heating is used. This feature can be considered an advantage as ability to change the properties of materials expands the field of opportunities for their application. The use of microwave irradiation for polymer synthesis and modification, as well as for polymer waste destruction was studied in many works of researchers in Russia and abroad [1-16].

Both monomeric and oligomeric products can be formed in a depolymerization reaction. Both are of great value as they can be used as initial reactant in polymer synthesis and modification processes. For example, oligomers are used in production of surfactants, coatings, rubbers, synthetic fibers, etc. Oligomers are obtained by polymerization and polycondensation with use of various techniques limiting the size of growing molecules [17-19].

Isoprene-based oligomers can be used as adhesion promoters to connect different layers of tire structure: tread, breaker, sidewall, carcass, etc.

This article is devoted to production of isoprene oligomers using the method of synthetic polyisoprene SKI-3 destruction by microwave irradiation.

EXPERIMENTAL PART

Isoprene rubber SKI-3. TU 20.17.10-141-05766801-2018 (PJSC «Nizhnekamskneftekhim»).

Phospholipids concentrate (PLC). TR 10-04-02-59-89. PLC contains (wt.-%): volatile – not more than 1.0; phospholipids – not less than 60; fatty acid triglycerides – not more than 40; acid number – not more than 10 mg KOH/g. Iodine value (I.v.) – 67.2 g I_2/100 g.

The rubber's viscosity average molecular weight is measured by timing the solvent (toluene) flowage and various concentrations of polymer solutions using an Ubbelohde viscometer.

A series of experiments on the destruction of SKI-3 (10% solution in toluene) with microwave irradiation was conducted (Table 1).

The SKI-3 destruction process was carried out in a toluene solution, since in this case its macromolecules are branched and demonstrate higher exposure to microwave irradiation than globular structures.

Table 1

Technological parameters of synthetic polyisoprene MV irradiation

***Таблица 1*. Технологические параметры процесса МВ-излучения синтетического полиизопрена**

Sample No.	MW radiation power, W	Process time, min	Modifier amount, % wt.
0	-	-	-
1	144	120	0
2	144	120	5
3	450	120	0
4	450	120	3
5	450	120	5
6	675	120	0
7	675	120	3
8	675	120	5
9	900	60	0
10	900	60	3
11	900	60	5

Phospholipids concentrate was used as a modifier due to its prominent antioxidant properties [20-21]. The degree of destruction was determined by measuring the viscosity average molecular weight of the obtained destruction products.

As an oligomeric modifier, the destructed SKI-3 was introduced directly into a Brabender rubber mixer together with other rubber mixture ingredients in proportion of 2-10 parts by weight of destructed SKI-3 to 100 parts by weight of initial SKI-3. Rubber mixtures not containing the destructed SKI-3 were being obtained at the same time (control samples). Model rubber mixtures were prepared according to proportions presented in Table 2 (per 100 parts by weight of rubber), parts by weight: stage 1 – initial SKI-3 (100.0), zinc oxide (5.0), IPPD (0.6), stearic acid (1.0), black carbon PM-100 (50.0), destructed SKI-3 (0 ÷ 10.0), Stage 2 - technical sulfur (1.0), diphenylguanidine (3.0), altax (0.6).

The rubber mixtures were prepared in two stages at the temperature of 70°C within 7 min. The modifier was introduced at the first stage of mixing together with initial SKI-3.

The vulcanization kinetics was studied using a Monsanto 100 S rheometer at 151 °C. Rheograms were analyzed according to the guidelines [22]. Vulcanization was carried out in a hydraulic press equipped with APVM-901 plates with electric heating. Vulcanizates tensile strength was measured according to GOST 270-75; tear strength – according to GOST 262-93; bond strength between rubber and a single cord thread – using the H-method; rubber elasticity – according to GOST 27110-86. Shore hardness number was measured according to GOST 263-75.

RESULTS AND DISCUSSION

The findings of this study show that an increase in MWI power leads to an increase in the degree of destruction (Table 2) both in presence of a modifier and its absence. Furthermore, the destruction of polyisoprene macromolecules proceeds more intensely in absence of a modifier (samples No. 1, 3, 6 and 9, Table 2) compared with the process in presence of phospholipids (samples No. 2, 4-5, 7-8 and 10-11, Table 2). With the increase in MWI power, viscosity average molecular weight of a polymer shows a fourfold (or higher) decrease in comparison to its initial molecular weight (sample 0, Table 2).

Table 2

Characterization of samples obtained

***Таблица 2*. Характеристики полученных образцов**

No	MWI power, W	Viscosity average molecular weight ($\bar{M}_\eta$)
0	-	550 000
1	144	95 300
2	144	101 300
3	450	39 800
4	450	73 000
5	450	75 900
6	675	36 700
7	675	78 900
8	675	81 400
9	900	33 550
10	900	41 960
11	900	49 400

When a phospholipids concentrate is used, the increase of its amount in the reaction mixture leads to a decrease in polymer molecular weight reduction. This finding may indicate either a certain stabilizing effect of the modifier or occurrence of parallel structuring processes in this system.

Thus, it was found that molecular weight characteristics of low molecular weight polyisoprene can be predetermined by varying destruction conditions.

Kinetic studies of viscosity average molecular weight ($\bar{M}_\eta$) change depending on MWI power (Fig. 1) show that an increase in electromagnetic radiation power raises the degree of polyisoprene destruction.

Then, various amounts of a phospholipid modifier were introduced into the toluene-SKI-3 system. It was found that an increase in the amount of phospholipid (in parts by weight per 100 parts by weight of SKI-3) reduces the degree of polyisoprene destruction regardless of the power of MWI exposure (Fig. 2). This finding indicates that in this process the phospholipid acts as a stabilizer and allows for controlling the degree of polymer destruction.

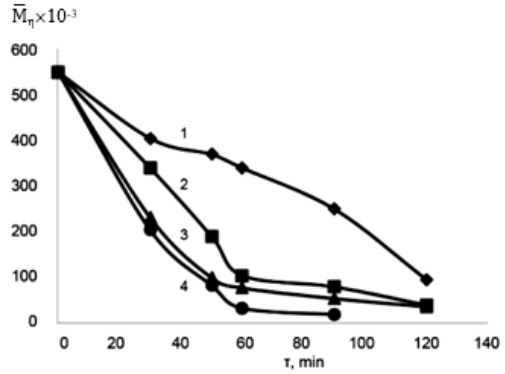

Fig. 1. SKI-3 viscosity average molecular weight ($\bar{M}_\eta$) change in the MWI field depending on the irradiation power and time (τ): 1 – 144 W, 2 – 450 W, 3 – 675 W, 4 – 900 W

Рис. 1. Изменение средневязкостной молекулярной массы ($\bar{M}_\eta$) СКИ-3 в токе МВИ в зависимости от мощности излучения и времени (τ): 1 – 144 Вт, 2 – 450 Вт, 3 – 675 Вт, 4 – 900 Вт

The control experiment was performed using the conventional method of heating the SKI-3 toluene solution in a retort at 90 °C. The findings of the experiment show that molecular weight of polyisoprene does not demonstrate any significant change.

Thus, it was found that the SKI-3 destruction process is influenced by electromagnetic radiation power and exposure time, as well as by the amount of phospholipid modifier. Furthermore, it is established that phospholipids demonstrate having a stabilizing effect and allow for controlling the molecular weight of forming oligomers.

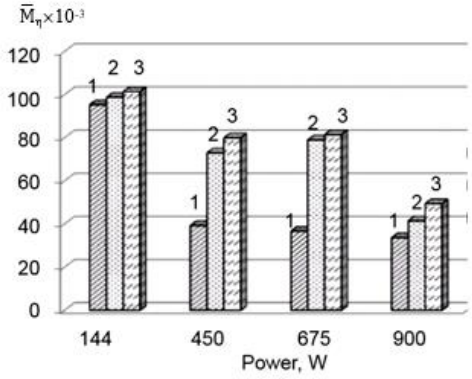

Fig. 2. Dependence of synthetic polyisoprene viscosity average molecular weight ($\bar{M}_\eta$) on the amount of phospholipids and the MWI-unit power: 1 – 0 parts by weight, 2 – 3 parts by weight, 3 – 5 parts by weight

Рис. 2. Зависимость средневязкостной молекулярной массы ($\bar{M}_\eta$) полиизопрена от количества фосфолипидов и мощности МВИ-установки: 1 – 0 мас.ч., 2 – 3 мас.ч., 3 – 5 мас.ч.

The continuing search for new SKI-3 modifiers is aimed at improving the properties of SKI-based products so that they best meet operation requirements. The oligomer obtained at 450 W power and in presence of a modifier in the amount of 3 phr. is proposed to be used as the modifier of rubber mixtures.

Experimental vulcanizates are characterized by the decrease in optimum vulcanization time of rubber mixtures (Table 3). It is possible that this effect is caused by the phospholipids contained in oligomers.

The results of physical and mechanical tests of rubber mixtures and vulcanizates (Table 3) were analyzed. The findings show that samples containing destructed SKI-3 in the amount of 7 and 10 parts by weight per 100 parts by weight of rubber demonstrate the optimal plasto-elastic properties (plasticity increase, elastic recovery reduction) and superior vulcanizates strength.

It was found that introducing an oligomeric component (destructed SKI-3) into rubber mixtures in the amount of 7 and 10 parts by weight of rubber leads to an increase in their cohesive strength (Table 3). Oligomer-containing vulcanizates are characterized by higher conditional tensile strengths. It may be attributed to the fact that oligomers (used as a modifier) have the same structural unit as isoprene rubber that results in composition compatibility being rather high. In turn, this provides an increase in strength characteristics.

The improvement of physical and mechanical properties can be attributed to the change of polymers structure entailed by the introduction of oligomers during mixing and vulcanization. The observed effects can be ascribed to the fact that the polymers mixture based on initial SKI-3 contains destructed SKI-3 low-molecular mixture. It imbeds between the macromolecules, thereby the intensity of heat transfer increases. Thus, the mobility of macromolecules escalates as a whole, and therefore the system compliance increases.

CONCLUSION

Oligomers were obtained by applying microwave irradiation to a solution of SKI-3 rubber. This is confirmed by the change in the viscosity average molecular weight. The potential for controlling the destruction degree of isoprene rubber by applying MWI at various powers was demonstrated. For instance, the impact of 144 W and 900 W power was observed to lead to more than double decrease in viscosity average molecular weight.

The use of a phospholipid concentrate in the toluene-SKI-3 system leads to a lesser decrease in the molecular weight. Thus, it allows for regulating the degree of destruction.

The obtained oligomers can be used as modifiers of SKI-3 rubber allowing for improving the technological, physical and mechanical properties of the composites.

Table 3

Physico-mechanical properties of model rubber mixtures and their vulcanizates

***Таблица 3*. Физико-механические свойства модельных резиновых смесей и их вулканизатов**

Indicator	Oligomers amount, parts by weight					
	0	2	3	5	7	10
Rubber mixture properties						
Conditional cohesive strength, MPa	0.23	0.25	0.27	0.28	0.30	0.31
Plasticity, conv. units	0.31	0.31	0.32	0.35	0.36	0.37
Elastic recovery, mm	1.4	1.4	1.3	1.2	1.1	1.0
Vulcanization characteristics						
Torque, dN × m						
min	38.0	38.0	35.2	32.7	31.0	30.0
max	64.0	63.0	56.5	55.0	41.6	29.4
Optimal vulcanization time, min.	17.0	16.0	15.5	15.3	15.0	14.0
Vulcanizates properties (151 °C)						
Conditional stress at 300% elongation, MPa	8.0	8.7	10.2	10.3	11.1	11.8
Conditional tensile strength, MPa	19.0	17.9	21.6	25.3	25.2	24.9
Elongation at break, %	520	510	510	540	550	560
Shore A hardness, conv. units	34	33	33	32	32	32
Bounce elasticity, %	37	39	40	38	39	42

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