Modification of synthetic polyisoprene by combination with high-density polyethylene

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Abstract:

Introduction/purpose: The development of elastomeric materials based on synthetic polyisoprene (IR) with high green strength is a rather urgent task, because it allows to replace, completely or partially, the main ingredient - natural rubber in responsible rubber products. The aim of the work was an additional increase in the green strength values of IR and rubber mixtures based on it by its modification with high-density polyethylene of PND 277-73.

Methods: The main methods of the research of the technological, physical-mechanical and operational properties were used. All tests conformed to ASTM or ISO standards. Rubber compounds were made in the Haake PolyLab rubber mixer. In parallel with the effects of modification, the *influence of the mixing temperature on the main properties of IR and NR based elastomeric materials was investigated.*

Results: It is shown that the increase in the mixing temperature of NR and IR for 20ºC leads to significant changes in the technological properties of rubber compounds (primarily green strength), as well as the physical-mechanical and operational properties of cured rubber. It has been established that a significant increase in the green strength of the rubber compound is achieved by combining IR with 7 mass. % HDPE.

Conclusion: The developed polymer composition based on IR and HDPE has a level of green strength of the rubber compound 2 times higher than that of natural rubber, while maintaining the physical-mechanical and operational characteristics of cured rubbers at the level of natural rubber.The polymer composition IR/HDPE 93/7 can be recommended for use in the manufacture of products whose manufacturing technology requires increased green strength of rubber compounds, including large-sized and all-metal tires.

Key words: synthetic polyisoprene, natural rubber, green strength, polymer mixtures, high-density polyethylene.

Introduction

The range of industrially synthesized rubber is quite limited, and the industry of synthetic rubber is unable to satisfy the ever-increasing needs of the rubber industry. The use of polymer mixtures in fact expands the range of elastomeric materials without requiring complex special equipment, which ultimately makes it possible to produce technical products with the required properties. Therefore, interest and attention to extraordinary mixtures of rubber with rubber or thermoplastics are constantly increasing. Previous experience and modern practice show that the use of polymer mixtures of different classes allows to improve the technological properties of rubber mixtures, the physical-mechanical properties of cured rubbers and the operational properties of finished products (Schwartz & Dinzburg, 1972; Kerber et al, 2024; Alekseenko et al, 2024).

Natural rubber is an indispensable elastomer for the manufacture of a wide range of rubber products, including tires, rubber and defense products. Now, one of the most popular materials in the tire industry is imported natural rubber (NR). This is especially true for all-metal, aviation and large-scale civil and defense tires, which are made of more than 85% NR. The only synthetic analogue for NR is synthetic isoprene rubber of the SKI-3 (IR) brand, which is inferior to natural rubber in several important indicators. Thus, one of the main disadvantages of IR compared to NR is the low green strength of rubber compounds based on IR, which is important in the prefabrication and assembly operations. Green strength is the most important technological characteristic of rubber compounds and is responsible for the confection adhesion and stable frame of the product elements until their complete assembly and vulcanization (Zolotarev et al, 2021; Zuev et al, 2024; Nasyrov et al, 2020; Lyusova & Chernyshov, 2022).

There are two ways to improve the green strength of IR: chemical modification (Aksenov, 2021; Akhmetov et al, 2023) and physical modification of rubber (Cruz-Morales et al, 2023; Chernyshov et al, 2023a, 2023b). Over the past more than 50 years, a lot of research work has been done on the first path, i.e. chemical modification of synthetic isoprene rubber (Aksenov, 2021). Many ways of chemical modification of IR gave an opportunity to increase cohesive strength, but did not receive further development for various reasons: carcinogenicity, toxicity or unsatisfactory processability. Physical modification, i.e. combining IR with other polymers, is the most promising way to increase green strength in the production of rubber products, as it does not require additional technological operations and is environmentally friendly.

As early as in the 1960s and 1970s, the possibility of increasing the green strength of rubber mixtures made of synthetic polyisoprene by introducing crystallizing polyolefins, including polyethylene, was considered (Schwartz & Dinzburg, 1972; Priklonskaya et al, 1969). Then high-density polyethylene (HDPE) was the most effective due to its high crystallinity. Presumably, the reinforcing effect of HDPE could be due to the presence of crystalline particles contributing to the orientation effect of the macromolecules of highly elastic polymer, accelerating the crystallization of rubber during deformation (Priklonskaya et al, 1969; Yanez Flores et al, 1997). But the number of the published works in this direction with specific test results is quite small, and those that exist are incomplete and the data in them are ambiguous. For example, there is no information on the used brands of polyethylene and on the process for producing a rubber compound containing polyethylene. There are no vulcanization characteristics and viscosity values for Mooney rubber compounds, no data on fatigue characteristics of vulcanizations, and no direct comparison with rubber mixtures and rubbers based on NR (Guseva et al, 2002). It should also be noted that since then the brand assortment of polyethylene has changed significantly, and there have been notable changes in the synthesis of SKI-3.

Materials and methods

The objects of the research in this work were elastomeric materials based on synthetic isoprene rubber (IR) brand SKI-3 (Mooney viscosity – 74 units) produced by LLC «Tolyattikauchuk». High-density polyethylene (HDPE) of LLC «Stavrolen» brand PND 277-73 was used to increase the green strength of the material. Based on the previous obtained data (Chernyshov et al, 2023a), the content of HPDE was 7 mass. %. The elastomeric material based on natural rubber brand RSS-1 was chosen as a standard.

The combination of the tested polymers and the production of the rubber compounds based on them were carried out in the Haake PolyLab rubber mixer (within 9 minutes) with the subsequent introduction of sulfur on rollers at a temperature of 50℃ for 1.5 minutes. The mixing mode is given in Table 1. The initial mixing temperature in the rubber mixer was 120-140°C for IR and NR based rubber compounds. For a mixture containing HDPE (melting point – 134°C), the initial mixing temperature was 140°C.

The formulations of the rubber compounds are presented in Table 2.

Table 1 – Mixing mode of the rubber compounds

Compound	Duration, min	General time, min.
1st stage		
Rubber (IR and/or NR)	1	1
HDPE (PND 277–73)	2	3
Stearic acid	6.0	9.0
Zinc oxide	6.0	9.0
Accelerator CBS	6.0	9.0
Carbon black N330	5.0	9.0
2nd stage		
Sulfur	1.5	10.5

The green strength of the rubber compounds and the physical-mechanical characteristics of cured rubber were determined on the Gotech AI-3000-U tensile testing machine according to ASTM D 6747-15 and ASTM D 412, respectively, and the vulcanization characteristics were determined on the MonTech MDR 3000 Professional rheometer according to ASTM D 5289. Using the MonTech MV 3000 Basic viscometer, the

Mooney viscosity was measured according to ASTM D 1646. The fatigue endurance at repeated elongation (ε = 125% at 3 Hz) and fatigue endurance under repeated bending with puncture (3 Hz) was determined on the MonTech FT 3000 CH according to ISO 6943 and ASTM D 813, respectively. Hardness and rebound resilience were determined according to ASTM D 2240 and ISO 4662.

Thus, the above methods and materials were used in the study of the influence of the mixing temperature on the technological properties of the rubber compounds based on NR and IR, and the physical-mechanical and operational properties of cured rubber based on them Their properties were also compared with the indicators of the polymer composition containing HDPE.

Table 2 – Formulations of the rubber compounds

Compound	Contents, parts per hundred rubber		
	NR	IR	IR/HDPE 93/7
NR (RSS-1)	100.0	-	-
IR (SKI-3)	-	100.0	93.0
HDPE (PND 277–73)	-	-	7.0
Stearic acid	1.0	2.0	2.0
Zinc oxide	5.0	5.0	5.0
Accelerator CBS	1.5	1.5	1.5
Carbon black N330	35.0	35.0	35.0
Sulfur	2.5	2.0	2.0

Results and discussion

As it is known, tire rubber mixtures are manufactured at temperatures above 120℃, so in the first stage of the work, the influence of the mixing temperature in the range of 130±10℃ on the main complex of the properties of elastomer materials was investigated.

As it can be seen in Figure 1, an increase in the mixing temperature leads to a decrease in the green strength of both NR and IR-based rubber compounds, and for natural rubber the decrease in green strength is much greater. It can be assumed that the mixing temperature increases, the rate of thermal-oxidative degradation increases, which leads to a decrease in the molecular weight of polymers. This assumption is supported by the Mooney viscosity data given in Table 3.

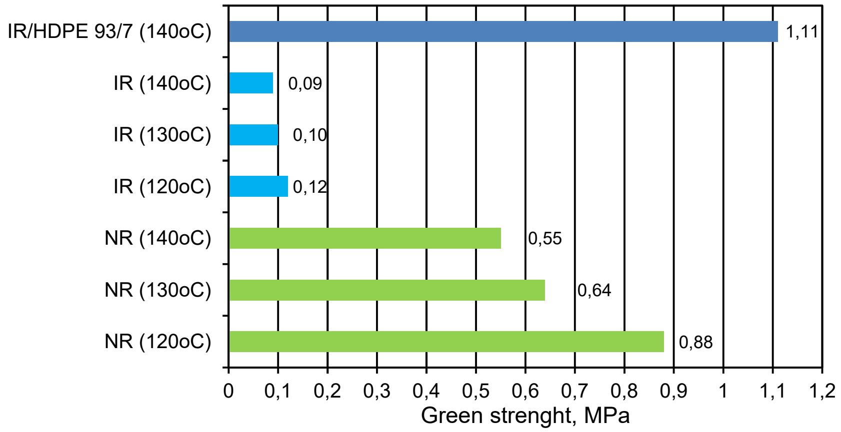

Figure 1 – Green strength of the rubber compounds

Table 3 – Technological properties of the rubber compounds and the physical and mechanical properties of the vulcanizates

Index	Polymer base						
	NR 120°C	NR 130°C	NR 140°C	IR 120°C	IR 130°C	IR 140°C	IR/HDPE 93/7 140°C
Technological properties of the rubber compounds							
Mooney Viscosity, units Mooney viscosity	40	39	37	46	39	39	49
ε_{CM}, %	725	612	550	887	1137	1350	762
Physical-mechanical properties of the vulcanizates							
f_{100}, MPa	3.2	2.7	2.4	2.5	1.7	1.5	2.9
f_{300}, MPa	18.6	14.3	14.6	14.7	10.2	9.4	14.2
f_p, MPa	30.7	27.7	26.9	31.4	27.4	25.3	27.5
ε_p, %	422	473	443	465	520	503	461
θ, %	32	23	21	31	20	22	36
P_r, kN/m	103.1	108.9	96	94	95.8	85.6	100.4

Note: ε_{CM} – elongation of a rubber compound in tension, f_{100}, f_{300} – stresses at an elongation of 100 and 300 %, f_p – tensile strength, ε_p –elongation at break, θ – residual elongation, and P_r –tear resistance.

As shown in Figure 1 and Table 3, the introduction of HDPE in the amount of 7 mass. % (IR/HDPE 93/7) can significantly increase the green strength of the rubber compound and increase the Mooney viscosity to some extent. When comparing rubber mixtures (Figure 1), it is shown that during mixing at 140℃, the green strength of the mixture is 2 times higher than that of NR and 12 times higher than that of IR.

The analysis of the curing curves (Figure 2) showed that an increase in the mixing temperature of the rubber compounds leads to a significant decrease in the torque increment (ΔM), as well as to a slight decrease in the induction period and an increase in the rate of the vulcanization process. The decrease in ΔM is probably due to thermo-oxidative destruction of rubber macromolecules during the mixing process. At the same time, the introduction of HDPE has practically no effect on the vulcanization process.

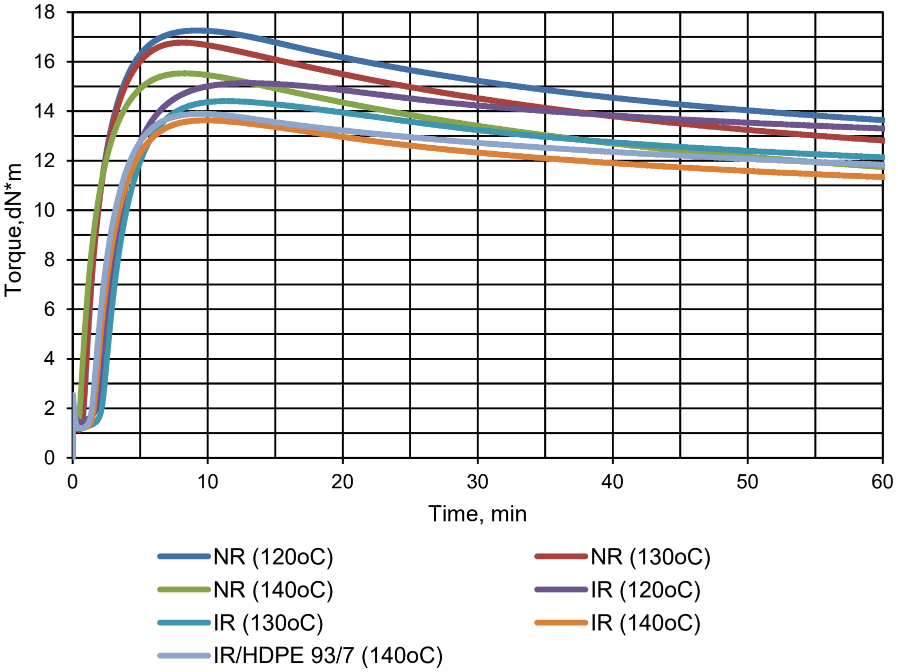

Figure 2 – Curing curves for the rubber compounds

The increase in the mixing temperature, which leads to an increase in the rate of thermo-oxidative destruction of rubber leads to a decrease in the physical-mechanical properties of rubber. Table 3 shows that there is a decrease in the tensile strength, the stresses at an elongation of 100 and 300%, and the tear resistance. The introduction of HDPE into the rubber composition leads, in comparison with IR based rubber, to a 93% increase in the stresses at an elongation of 100% and an increase in the tear resistance by 17%. As a result, the parameters of the vulcanizate are almost the same as for the NR-based rubber. The increase in these parameters is probably due to the same mechanism as the increase in green strength.

Table 4 shows that, as the temperature of mixing increases, there is a slight increase in rebound resilience and a decrease in hardness in rubbers regardless of the rubber base. The decrease in hardness is probably due to a decrease in the molecular weight of rubber macromolecule during destruction.

Table 4 – Hardness and rebound elasticity of the vulcanizates

Index	Polymer base						
	NR 120ºC	NR 130ºC	NR 140ºC	IR 120ºC	IR 130ºC	IR 140ºC	IR/HDPE 93/7 140ºC
Hardness (Shore A), units.	59	58	57	55	55	54	60
Rebound resilience, %	60	61	61	56	56	58	51

As shown in Table 4, the introduction of HDPE into rubber results in an increase in its hardness, since polyethylene is in a solid state at normal condition and its influence on this parameter is like that of fillers. When combining IR with polyethylene, there is a significant reduction in rebound resilience. This is probably due to the small number of physical and/or chemical bonds of the rubber matrix with polyethylene and the existence of the interface that prevents the distribution of fluctuations throughout the volume of the material.

One of the main operational properties of rubber and rubber products exposed to dynamic loads is fatigue, which characterizes the durability of the product. The fatigue characteristics for the tested compositions are shown in Figure 3.

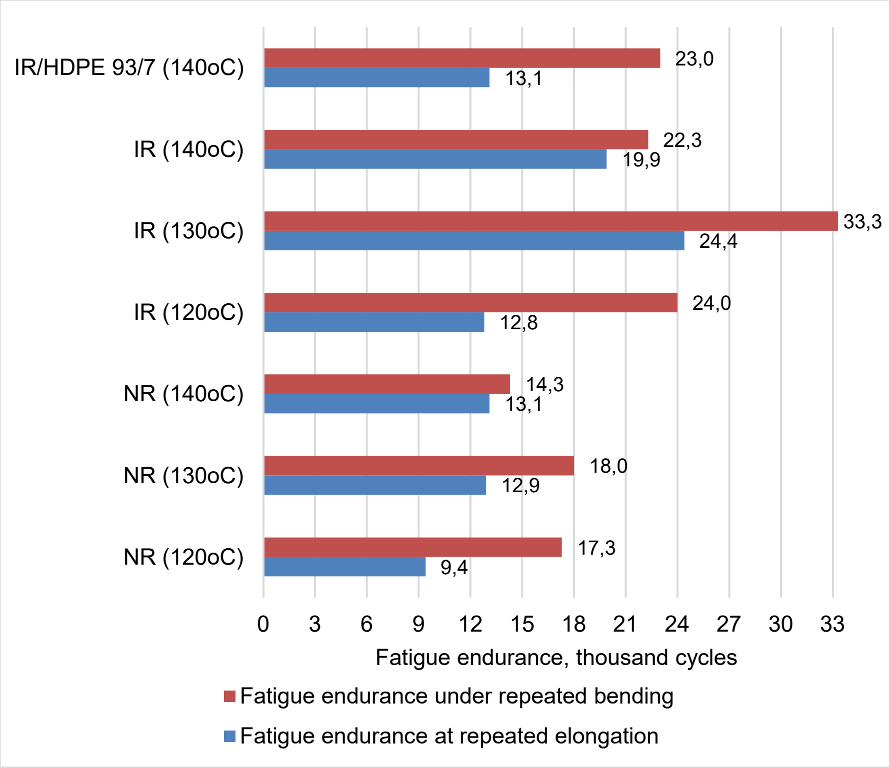

Figure 3 – Fatigue characteristics

It has been found that the increase in the mixing temperature of the rubber compounds has different effects on the fatigue endurance of NR and IR-based rubbers. As the mixing temperature rises, fatigue endurance increases in NR-based rubber and in IR-based rubber passes through the optimum with a maximum at 130ºC. At the same time, fatigue endurance under repeated bending changes similarly for both rubbers (passes through the optimum at a maximum temperature of 130℃). The increase in fatigue is due to the reduction of the rubber modules due to thermo-oxidative destruction.

The fatigue characteristics of the rubber presented in Figure 3 showed that the introduction of HDPE into IR-based rubber had little or no effect on fatigue endurance at repeated elongation and repeated bending when comparing equal modulus rubber. However, IR/HDPE’s fatigue endurance at repeated elongation is like NR-based cured rubber, and fatigue endurance under repeated bending is 28% higher.

Conclusions

Thus, it is shown that the increase in the mixing temperature of the rubber compounds based on NR and IR from 120℃ to 140℃ leads to significant changes in both the technological properties of the rubber compounds and the physical-mechanical properties of cured rubber and the operational properties of their vulcanizates. At the same time, it is worth noting separately that the increase in the temperature of the mixture negatively affects the green strength of the rubber compounds, especially based on natural rubber.

The work shows that combining IR with 7 mass. % HDPE leads to a significant increase in the green strength of the rubber compound. Moreover, at similar mixing temperatures, it exceeds the level of the green strength of a rubber compound based on natural rubber by 2 times.

The rubber based on the proposed polymer composition IR/HDPE 93/7 has the physical-mechanical and operational characteristics close to the NR-based rubber. The inventive polymer mixture is characterized in that it exceeds the NR-based rubber for fatigue endurance under repeated bending by 28%.

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Modificación del poliisopreno sintético mediante combinación con polietileno de alta densidad

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CAMPO: tecnología química, materiales
TIPO DE ARTÍCULO: artículo científico original

Resumen:

Introducción/objetivo: El desarrollo de materiales elastoméricos basados en poliisopreno (IR) sintético con alta resistencia en verde es una tarea bastante urgente, porque permite reemplazar, total o parcialmente, el ingrediente principal, el caucho natural, en productos de caucho responsable. El objetivo del trabajo era aumentar adicionalmente los valores de resistencia en verde del IR y de las mezclas de caucho basadas en él mediante su modificación con polietileno de alta densidad de PND 277-73.

Métodos: Se utilizaron los principales métodos de investigación de las propiedades tecnológicas, físico-mecánicas y operativas. Todas las pruebas se ajustaron a las normas ASTM o ISO. Los compuestos de caucho se fabricaron en el mezclador de caucho Haake PolyLab. Paralelamente a los efectos de la modificación, se investigó la influencia de la temperatura de mezcla sobre las principales propiedades de los materiales elastoméricos basados en IR y NR.

Resultados: Se demuestra que el aumento de la temperatura de mezcla de NR e IR por 20ºC conduce a cambios significativos en las propiedades tecnológicas de los compuestos de caucho (principalmente resistencia en verde), así como en las propiedades físico-mecánicas y operativas del caucho curado. Se ha establecido que se logra un aumento significativo en *la resistencia en crudo del compuesto de caucho combinando IR con 7 masa. %PEAD.*

Conclusión: La composición polimérica desarrollada a base de IR y HDPE tiene un nivel de resistencia en verde del compuesto de caucho 2 veces mayor que el del caucho natural, manteniendo las características físico-mecánicas y operativas de los cauchos curados al nivel del caucho natural. La composición polimérica IR/HDPE 93/7 se puede recomendar para su uso en la fabricación de productos cuya tecnología de fabricación requiere una mayor resistencia en verde de los compuestos de caucho, incluidos neumáticos de gran tamaño y totalmente metálicos.

Palabras claves: poliisopreno sintético, caucho natural, resistencia en verde, mezclas de polímeros, polietileno de alta densidad.

Модификация синтетического полиизопрена путем совмещения с полиэтиленом высокой плотности

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РУБРИКА ГРНТИ: 61.63.09 Полимерные материалы, используемые в производстве резин и изделий из них,
61.63.81 Методы испытаний и свойства резиновых изделий
ВИД СТАТЬИ: оригинальная научная статья

Резюме:

Введение/цель: Разработка эластомерных материалов на основе синтетического полиизопрена (IR), обладающего повышенной когезионной прочностью, является достаточно актуальной задачей, так как это позволяет полностью или частично заменить в ответственных резиновых изделиях основной ингредиент − натуральный каучук. Целью работы являлось дополнительное повышение значений когезионной прочности IR и резиновых смесей на его основе путем его модификации полиэтиленом высокой плотности марки ПНД 277–73.

Методы: Использованы основные методы исследования технологических, физико-механических и эксплуатационных свойств. Все испытания соответствовали стандартам ASTM или ISO. Резиновые смеси изготавливали в резиносмесителе

Haake PolyLab. Параллельно с эффектами от модификации, в работе было исследовано влияние температуры смешения на основные свойства эластомерных материалов на основе IR и NR.

Результаты: Показано, что увеличение температуры смешения резиновых смесей на основе NR и IR на 20°C приводит к существенным изменениям технологических свойств резиновых смесей (прежде всего когезионной прочности), а также физико-механических и эксплуатационных свойств резин на их основе. Установлено, что существенное увеличение когезионной прочности резиновой смеси достигается при совмещении IR с 7 мас. % HDPE.

Выводы: Разработанная полимерная композиция на основе IR и HDPE имеет уровень когезионной прочности резиновой смеси в 2 раза выше, чем у натурального каучука, при сохранении физико-механических и эксплуатационных характеристик вулканизатов на уровне резин на основе натурального каучука.

Ключевые слова: синтетический полиизопрен, натуральный каучук, когезионная прочность, смеси полимеров, полиэтилен высокой плотности.

Модификација синтетичког полиизопрена комбиновањем са полиетиленом високе густине

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ОБЛАСТ: хемијске технологије, материјали
КАТЕГОРИЈА (ТИП) ЧЛАНКА: оригинални научни рад

Сажетак:

Увод/циљ: Развој еластомерних материјала на бази синтетичког полиизопрена (IR) високе кохезионе чврстоће прилично је хитан задатак због потребе да се потпуно или делимично замени главни састојак критичних производа од гуме – природна гума. Циљ рада јесте да се додатно повећају вредности кохезионе чврстоће IR и гумених мешавина заснованих на њему путем његове модификације полиетиленом велике густине PND 277-73.

Методе: Коришћене су основне методе истраживања технолошких, физичко-механичких и експлоатационих својстава.

Сва испитивања била су у складу са стандардима АСТМ и ИСО. Мешавине гуме биле су припремљене у мешалици за гуме Haake PolyLab. Истовремено са ефектима модификације испитиван је утицај температуре мешања на главна својства еластомерних материјала на бази полиизопрена и каучука.

Резултати: Показано је да повећање температуре мешања полиизопрена и каучука за 20ºC доводи до значајних промена у технолошким својствима гумених смеша (пре свега кохезионе чврстоће), као и физичко-механичких и експлоатационих карактеристика вулканизата. Утврђено је да се постиже знатно повећање кохезионе чврстоће смеше гуме комбиновањем полиизопрена са 7 тежинских % HDPE.

Закључак: Изведено је полимерно једињење на бази IR и HDPE с нивоом кохезионе чврстоће гумене смеше два пута веће од природне гуме, а притом су задржане физичко-механичке и експлоатационе карактеристике вулканизата на нивоу природне гуме.

Кључне речи: синтетички полиизопрен, природна гума, кохезиона чврстоћа, полимерне мешавине, полиетилен велике густине.

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