Chemical Journal of Kazakhstan
Volume 1, Number 89(2025), 59-68 https://doi.org/10.51580/2025-1.2710-1185.06

УДК 678.029.46

TECHNOLOGICAL PROPERTIES OF RUBBER COMPOUNDS FOR HYDRAULIC SEALS (Part 1)

B.Sh. Khassankhodzhaeva[1], G.F. Sagitova[1]*, A.S. Sidikov[2], A.Sh. Kydyralieva[1], A.U. Sarsenbayeva[1], E.A. Tusupkaliyev [3], Zh.N. Kainarbayeva [3], M.N. Baiymbetov [3]

[1]M. Auezov South Kazakhstan University, Shymkent, Republic of Kazakhstan
[2] Branch of Gubkin Russian State University of Oil and Gas (NRU), Tashkent, Uzbekistan
[3]JSC "A.B. Bekturov Institute of Chemical Sciences", Almaty, Kazakhstan

**E-mail: t_ersin@mail.ru, guzalita.f1978@mail.ru*

Abstract. *Introduction.* This study investigates the technological properties of rubber compounds intended for the manufacture of water lock seals. The formulation is based on a rubber compound in which PN-6SH oil was replaced with an organic component extracted from oil sludge provided by Petro Kazakhstan Oil Products LLP. Additionally, the traditional filler (white carbon black) was replaced with zeolite from the Chankanai deposit, which resulted in satisfactory technological properties. *The aim* of the work was to study the properties of rubber compounds modified by the introduction of the organic part of oil sludge and zeolite, as well as to determine the optimal dosages of these components to improve the compound's characteristics. *Methodology.* The rubber compound was modified by adding the organic part of the oil sludge in an amount of 1.0–6.0 parts by weight. Zeolite was added in amounts of 3.0–20.0 parts by weight per 100 parts by weight of rubber. Five samples of rubber compounds were prepared. The technological properties of the mixtures were determined. *Results and Discussion.* It was established that increasing the content of the plasticizer (up to 6.0 parts by weight) and filler (up to 20.0 parts by weight) leads to an increase in the Mooney viscosity of the mixture. Optimal technological indicators were observed at a dosage of 6.0 parts by weight of the organic part of the oil sludge and 20.0 parts by weight of zeolite. The tests demonstrated that replacing traditional components with the proposed modifications ensures that the properties of the rubber compounds meet the control standards for water lock seals. *Conclusion.* The use of the organic part of oil sludge and zeolite in the formulation of rubber compounds allows the replacement of traditional plasticizers and fillers without compromising the properties of the mixtures intended for hydraulic seals, as confirmed by compliance with control standards.

Keywords: isoprene rubber, water lock seals, Mooney viscosity, plasticity, rubber.

Khassankhojayeva Bibi-Mariyam	*PhD doctoral student; E-mail: bibi-mariyam93@mail.ru*
Sagitova Guzaliya	*Candidate of technical sciences, professor; E-mail: guzalita.f1978@mail.ru*
Sidikov Abdujalol	*Doctor of Chemical Sciences, Professor; E-mail: sidikov_a@mail.ru*

Citation: Khassankhodzhaeva B.Sh., Sagitova G.F., Sidikov A.S., Kydyralieva A.Sh., Sarsenbayeva A.U., Tusupkaliyev E.A., Kainarbayeva Zh.N., Baiymbetov M.N. Technological properties of rubber compounds for hydraulic seals (part 1). *Chem. J. Kaz.*, **2025**, 1*(89)*, 59-68. DOI: https://doi.org/10.51580/2025-1.2710-1185.06

Kydyraliyeva Aigul Shazhalievna	*PhD doctoral student; E-mail: aigul.ukgu@mail.ru*
Sarsenbayeva Aizada	*Master; E-mail: aizada-uzakbaikyzy@mail.ru*
Tusupkaliyev Ersin Adietovich	*Candidate of Chemical Sciences; E-mail: t_ersin@mail.ru*
Kainarbayeva Zhaniya Nurbekovna	*Master; E-mail: zhaniya.kn@gmail.com*
Baiymbetov Mizambek Nurbayevich	*Candidate of Chemical Sciences; E-mail: bmizam@mail.ru*

1. Introduction

To explore the relationships between the components of rubber compounds and their technical characteristics, the authors [1] utilized a combination of the Plackett–Burman and Box–Behnken methods. Optimization of rubber compound formulations is performed using an integrated genetic algorithm based on support vector regression to minimize the mixture cost. Twelve components potentially affecting the technical properties of the rubber compound—namely, natural rubber, carbon black, white filler, stearic acid, zinc oxide, antiozonant, antioxidant, processing oil, curing retarder, curing agent, and accelerator - were selected using the Plackett–Burman system to determine significant variables.

One of the key technological parameters characterizing the properties of rubbers and rubber compounds is Mooney viscosity. Viscosity refers to the resistance of a material (e.g., rubber) to deformation or flow under external load. Mathematically, viscosity (η) is defined as the ratio of shear stress to shear rate. This parameter significantly depends on temperature: as temperature increases, material viscosity decreases [2].

The viscosity of the processed material plays a crucial role in the dynamics of the technological process, as it determines the forces required to achieve a given flow rate at various stages of processing. Exceeding permissible viscosity values can make rubber compound processing not only economically unfeasible but also technically impossible [3].

The technological assessment of Mooney viscosity provides insights into various aspects of the rubber mixing process. For example, high initial rubber viscosity increases energy consumption for preparing a quality rubber compound. At the same time, higher viscosity leads to greater shear stresses during rotor mixing, which improves the dispersion of powdered components and, consequently, enhances the quality of the final product. However, high initial rubber viscosity, all else being equal, results in greater heating of the rubber compound, necessitating additional measures to prevent premature vulcanization [4].

The Mooney viscosity parameter is invariably included in the technical specifications (TS) of all rubbers and serves as an important criterion for evaluating their properties. For instance, for isoprene rubber SKI-3 produced by Public Joint Stock Company Nizhnekamskneftekhim (PJSC NKNK), Mooney viscosity values vary depending on the group: for the first group, the range is 75–85 units, and for the second, 65–74 units [5].

This parameter significantly influences the technological parameters of semi-finished rubber product manufacturing processes, such as the temperature and speed of extrusion or calendering operations.

In this regard, an important scientific and practical task arises: to establish the relationship between the weight-average molecular weight (M_w) of SKI-3 rubber and the Mooney viscosity (ηm) of rubber compounds made from it. In previous studies by the authors [6–9], quantitative relationships were determined between the intrinsic viscosity and molecular weight of polymers, as well as between the initial viscosity (η_0) and weight-average molecular weight (M_w).

Considering environmental aspects associated with the use of white filler and PN-6SH oil, as well as their scarcity, the development of new rubber compound formulations becomes relevant. Of particular interest are mixtures containing the organic fraction of oil sludge (OFS) and zeolite from the Chankanay deposit, which can be used for sealants in hydraulic locks.

The purpose of this study is to investigate changes in the technological properties of rubber compounds incorporating OFS and Chankanay deposit zeolite and to develop new environmentally friendly formulations with improved characteristics.

2. Experimental part

The objects of the study are:

- Zeolite from the Chankanay deposit as a filler;
- Oil sludge from LOO "PKOP" as a plasticizer;
- Rubber compounds based on SKI-3 for manufacturing hydraulic seals.

In previous studies, the organic fraction of the oil sludge (OFS) was extracted.

The process of plasticizing isoprene rubber SKI-3 was carried out in a laboratory-scale closed rubber mixer. During the experiments, the rotors of the device rotated at a frequency of 30 revolutions per minute. The temperature and duration of the plasticizing process were varied within the ranges of 30 to 600 seconds and 30 to 130°C, respectively. After the plasticizing process, the resulting SKI-3 samples were used to prepare rubber compounds formulated for the production of hydraulic seals [10].

The rubber compound was prepared on rolling mills (in accordance with GOST 14333-79E Rubber Processing Mills).

The plastoelastic properties of the rubber were determined using a plastometer [11].

Mooney viscosity was measured using the rotational viscometry method, following the requirements of GOST R 54552–2011 [12].

3. Results and Discussion

The modern industry has a limited selection of plasticizers and fillers, necessitating the development and implementation of new components. Solving this problem is crucial for improving production efficiency.

The use of petroleum industry waste and natural minerals as ingredients in rubber compounds creates opportunities to expand the range of plasticizers and fillers. Furthermore, it allows the replacement of costly and scarce materials used in technical rubbers, significantly reducing production costs.

In line with the research objective, our study focused on examining the changes in the technological properties of rubber compounds when incorporating the organic fraction of oil sludge (OFS) and zeolite from the Chankanay deposit. A rubber compound formulation was developed based on a standard recipe for manufacturing hydraulic seals. In this formulation, PN-6SH oil was replaced with the organic fraction of oil sludge extracted from the petroleum waste of LOO "PKOP," and white carbon black was substituted with zeolite from the Chankanay deposit, while maintaining a satisfactory set of technological characteristics.

The developed mixtures included OFS in amounts ranging from 1.0 to 6.0 parts by weight and zeolite in amounts ranging from 3.0 to 20.0 parts by weight per 100 parts by weight of rubber. As a result, five rubber compound samples were prepared with varying component dosages. A comparative formulation of the reference and experimental rubber compounds for manufacturing hydraulic seals is presented in Table 1.

Table 1 – Rubber compound formulation for manufacturing hydraulic seals

Name of ingredients	Parts by weight per 100 parts of rubber					
	Прототип	1	2	3	4	5
SKI-3 (1st Grade)	100	100	100	100	100	100
Technical Sulfur	4	4	4	4	4	4
Sulfenamide M	0,8	0,8	0,8	0,8	0,8	0,8
Santoguard PVJ	0,2	0,2	0,2	0,2	0,2	0,2
Zinc White (Zinc Oxide)	5	5	5	5	5	5
Stearic Acid	2	2	2	2	2	2
Pine Rosin	2	2	2	2	2	2
ACMG Plasticizer	3	3	3	3	3	3
PN-6SH Oil	6	4	3	1	0	5
OFS	0	2	3	5	6	1
Protective Microcrystalline Wax 3B-1	1	1	1	1	1	1
Acetonanil R	2	2	2	2	2	2
Diafen FP	2	2	2	2	2	2
Technical Carbon P-220	60	60	60	60	60	60
White Silica	20	15	10	5	0	17
Zeolite	0	5	10	15	20	3
Total	208	208	208	208	208	208

A key technological parameter that characterizes the properties of rubbers and rubber compounds is Mooney viscosity. This parameter largely depends on the nature of the rubber used, as well as on the composition and quantity of the injected ingredients [13]. Mathematical models were used to analyze the Mooney viscosity data obtained experimentally at different temperatures of SKI-3 plasticization. At temperatures of plasticization up to 100 °C, the viscosity was described by equation (1), and for temperatures above 100 °C, equation (2) was applied. These models allowed us to determine the K_{st} constants with minimal confidence intervals, ensuring high accuracy in describing experimental data:

$$\eta_{м} = \eta_{м0} \cdot M_{w0}^{K\eta \cdot lgt}, \quad (1)$$

$$\eta_{м} = \eta_{м0} \cdot M_{w0}^{0,5K\eta \cdot (lgt) \cdot (lgt)}, \quad (2)$$

where η_{m0} is the viscosity of the rubber compound based on SKI-3 before plasticization, and K_{η} is the plasticization rate constant for the "Mooney viscosity" parameter.

Table 2 presents the obtained values of K_{η} and E_{eff} for the plasticization process of SKI-3 at different temperatures.

Table 2 – Values of K_{η} and E_{eff} for the plasticization process of SKI-3 at different temperatures

Indicator	**$K_{\eta} \cdot 10^3$, c^{-1}**	**E_{eff}, kJ/mol**	**$K_{\eta} \cdot 10^3$, c^{-1}**	**E_{eff}, kJ/mol**
At a temperature of, ^{0}C:				
30	-7,99 ± 0,33	-	-	
45	-6,14± 0,26	15,3	-	
55	-5,1± 0,23	16,4	-	
65	-4,57± 0,18	14,28	-	
80	-3,78± 0,17	10,3	-	
100	-7,99 ± 0,33	0,4	-3,36± 0,25	
115	-4,45± 0,57	-	-3,78± 0,23	-14,7
125	-4,95± 0,73	-	-4,18± 0,27	-18,5
130	-5,57± 0,91	-	-4,69± 0,33	-18,8

Note: The values of K_{η} and E_{eff} presented in the first and second columns of the table are calculated using Equation (1), while the values in the third and fourth columns are derived using Equation (2).

The confidence intervals for the determined values of the constants K_{η} do not exceed ±6–8%. Within the temperature range of 30 to 130°C, the activation energy values for the Mooney viscosity plasticization process of the rubber compound were calculated. Analysis of these data showed that within the temperature range of 30 to 100°C, the effective activation energy (E_{eff}) has a positive value, reaching its maximum at 55°C. However, as the temperature increases to 100°C, E_{eff} significantly decreases to 0.4 kJ/mol.

This phenomenon is attributed to the shifting balance between the mechanical and oxidative degradation processes of rubber macromolecules during plasticization at temperatures from 80 to 100°C [14–17]. At temperatures above 80°C, oxidative degradation processes begin to dominate, with mechanisms and kinetics distinct from those of mechanical degradation. This likely explains the form of equation (2), which describes the plasticization behavior of SKI-3 at higher temperatures.

Figure 1 presents the experimental data alongside the calculated results obtained using equations (1) and (2), illustrating the dependence of the viscosity (η_m) of the mixtures on the plasticization time of SKI-3 at various temperatures.

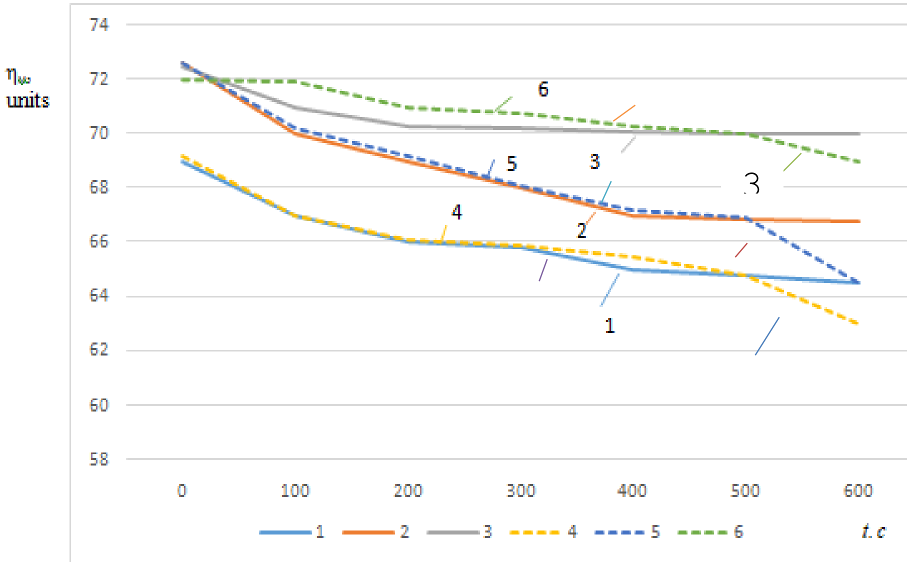

Figure 1 – Dependence of Mooney Viscosity (ηm\eta_mηm) of rubber compounds on SKI-3 plasticization time: calculated data (1, 2, 3) and experimental data (4, 5, 6) at various temperatures (°C): 30 – (1, 4), 80 – (3, 6), 130 – (2, 5).

There is excellent agreement between the experimental results and the calculated data. This allows the use of equations (1) and (2) to calculate the Mooney viscosity of the finished rubber compound with high accuracy for any temperature and pre-plasticization time of SKI-3 within the investigated ranges.

In the course of further research, the technological properties of raw rubber compounds were analyzed. It was established that the organic fraction of oil sludge and zeolite positively influence the characteristics of these compounds.

Figure 2 illustrates the dependence of the Mooney viscosity of the rubber compound on the content of the plasticizer and filler for the developed formulations.

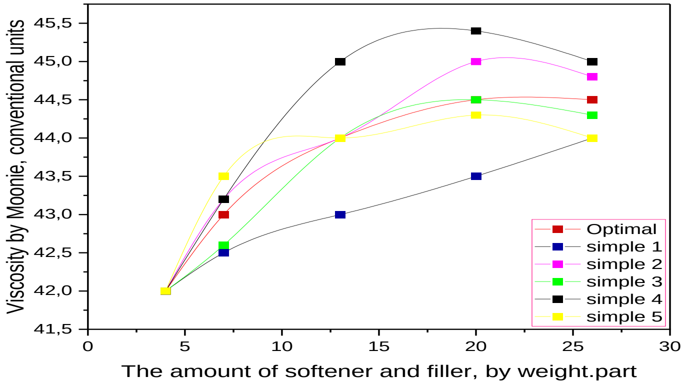

Figure 2 – Dependence of Mooney Viscosity on the Amount of Plasticizer and Filler Additives in Rubber Compounds for hydraulic seals.

An analysis of the viscous properties of rubber compounds shows that the effect of softeners and fillers on these characteristics is similar, which indicates the same mechanism of their action. According to the data shown in Figure 2, with an increase in the dosage of the softener (up to 6.0 wt.%) and filler (up to 20.0 wt.%) there is an increase in the Mooney viscosity. Optimal technological parameters for rubber compounds were achieved at a dosage of 6.0 wt.% of the organic part of the oil sludge and 20.0 wt.% of zeolite per 100 wt.% rubber, which meets the requirements for seals of hydraulic seals.

The test results of the technological properties of rubber compounds show that when replacing traditional softeners with the organic part of oil sludge and conventional fillers with zeolite, the properties of mixtures for sealing seals fully comply with regulatory requirements. In the future, the physico-mechanical properties of these rubbers will be investigated (Part 2).

4. Conclusions

Based on the study, the following was established:

1. Replacement of components: The use of PH and zeolite instead of traditional materials ensures environmental safety and reduces production costs.

2. Effect on viscosity: With an increase in the content of the softener to 6.0 wt.% and filler up to 20.0 wt.% increase in Mooney viscosity is observed, which improves the technological properties of the mixtures.

3. Optimal parameters: At a dosage of 6.0 wt.% PH and 20.0 wt.% zeolite achieves optimal performance for use in water locks.

The developed formulations of rubber compounds demonstrate competitive technological performance that meets regulatory requirements. These results highlight the promise of introducing new components into industrial production.

Acknowledgments: The work was carried out within the framework of the project of the Committee of Science of the Ministry of Science and Higher Education of the Republic of Kazakhstan under the project: № BR21882220.

Conflict of interests: The authors declare that there is no conflict of interest between the authors that requires disclosure in this article.

ГИДРАВЛИКАЛЫҚ ТЫҒЫЗДАҒЫШТАРҒА АРНАЛҒАН РЕЗИНА ҚОСПАЛАРЫНЫҢ ТЕХНОЛОГИЯЛЫҚ ҚАСИЕТТЕРІ (І БӨЛІМ)

Б.Ш. Хасанходжаева[1], Г.Ф.Сагитова[1*], С.С.Сидиков[2], А.Ш. Кыдыралиева[1], А.У.Сарсенбаева[1], Е.А. Тусупкалиев [3], Ж.Н. Кайнарбаева [3], М.Н. Баимбетов [3]

[1]*М. Әуезов атындағы Оңтүстік Қазақстан университеті, Шымкент, Қазақстан Республикасы*
[2]*И.М. Губкин атындағы Ресей мемлекеттік мұнай және газ университетінің филиалы, Ташкент, Өзбекстан Республикасы*
[3]*«А.Б. Бектұров атындағы Химия ғылымдары институты» АҚ, Алматы, Қазақстан*
[*]*E-mail: t_ersin@mail.ru, guzalita.f1978@mail.ru*

Резюме: *Кіріспе.* Бұл жұмыста гидрозатворлардың тығыздағыштарын дайындауға арналған резина қоспаларының технологиялық қасиеттері зерттелді. Рецепттің негізі ретінде «Petro Kazakhstan Oil Products» ЖШС мұнайшламынан бөлінген органикалық бөлігімен ПН-6Ш майы алмастырылған резина қоспасы пайдаланылды. Сонымен қатар, толықтырғыш ретінде дәстүрлі қолданылатын ақ күйе Шанқанай кен орнының цеолитімен алмастырылып, қанағаттанарлық технологиялық көрсеткіштерге қол жеткізілді. *Жұмыстың мақсаты* – мұнайшламның органикалық бөлігі мен цеолитті қосу арқылы модификацияланған резина қоспаларының қасиеттерін зерттеу, сондай-ақ осы компоненттердің қоспаларының сипаттамаларын жақсарту үшін оңтайлы мөлшерін анықтау. *Әдістер.* Резина қоспасына мұнайшламының органикалық бөлігі 1,0–6,0 массалық үлестегі мөлшері енгізілді. Цеолит каучуктің 100 массалық үлесіне 3,0–20,0 массалық үлесі мөлшерінде қосылды. Резина қоспаларының бес үлгісі дайындалды. Қоспалардың технологиялық қасиеттері анықталды. *Нәтижелер мен талқылау.* Жұмсартқыштың (6,0 масс. ү. дейін) және толықтырғыштың (20,0 масс. ү. дейін) мөлшері артқан сайын Муни бойынша қоспа тұтқырлығының өсуі анықталды. Технологиялық оңтайлы көрсеткіштер мұнайшламының органикалық бөлігін 6,0 масс. ү. және цеолитті 20,0 масс. ү. мөлшерінде қолданғанда байқалды. Сынақ нәтижелері көрсеткендей, дәстүрлі компоненттерді ұсынылған модификацияларға ауыстыру резина қоспаларының қасиеттерінің бақылау нормаларына сәйкес келуін қамтамасыз етеді. *Қорытындылар.* Мұнайшламының органикалық бөлігін және цеолитті резина қоспаларының рецептіне қосу жұмсартқыштар мен толықтырғыштарды дәстүрлі түрде алмастыруға мүмкіндік береді, сонымен қатар гидрозатворлардың тығыздағыштарына арналған қоспалардың қасиеттеріне зиян келтірмейді. Бұл бақылау нормаларына сәйкестігімен расталады.

Түйін сөздер: изопренді каучук, гидрозатворлардың резина тығыздағыштары, Муни бойынша тұтқырлық, иілімділігі, резина.

Хасанходжаева Биби-Мариям Шераликызы	*PhD докторант*
Сагитова Гузалия Фаритовна	*техника ғылымдарының докторы, профессор*
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Кайнарбаева Жания Нурбековна	*Магистр*
Баимбетов Мизамбек Нурбаевич	*Кандидат технических наук*

ТЕХНОЛОГИЧЕСКИЕ СВОЙСТВА РЕЗИНОВЫХ СМЕСЕЙ ДЛЯ УПЛОТНЕНИЙ ГИДРОЗАТВОРОВ (Часть 1)

Б.Ш. Хасанходжаева[1], Г.Ф. Сагитова[1*], С.С. Сидиков[2], А.Ш. Кыдыралиева[1], А.У. Сарсенбаева[1] , Тусупкалиев Е.А.[3], Кайнарбаева Ж.Н. [3], Баимбетов М.Н.[3]

[1]*Южно-Казахстанский университет им. М. Ауезова, Шымкент, Республика Казахстан*
[2]*Филиал* РГУ *нефти и газа (НИУ) имени И.М.* Губкина, Ташкент, Республика Узбекистан
[3]*«А.Б. Бектұров атындағы Химия ғылымдары институты» АҚ, Алматы, Қазақстан*
[*]*E-mail: t_ersin@mail.ru, guzalita.f1978@mail.ru*

Резюме. *Введение.* В данной работе исследованы технологические свойства резиновых смесей, предназначенных для изготовления уплотнений гидрозатворов. В качестве основы рецептуры использована резиновая смесь, в которое масло ПН-6Ш заменено органической частью, выделенной из нефтешлама ТОО «Петро Казахстан Ойл Продактс». Кроме того, белую сажу, традиционно используемую в качестве наполнителя, заменили на цеолит Чанканайского месторождения, что позволило достичь удовлетворительных технологических показателей. *Целью работы* было изучение свойств резиновых смесей, модифицированных введением органической части нефтешлама и цеолита, а также определение оптимальных дозировок данных компонентов для улучшения характеристик смесей. *Методология.* В резиновую смесь вводили органическую часть нефтешлама в количестве 1,0–6,0 масс.ч. Цеолит добавляли в количестве 3,0–20,0 масс.ч. на 100 масс.ч. каучука. Было приготовлено пять образцов резиновых смесей. Определялись технологические свойства смесей. *Результаты и обсуждение.* Установлено, что увеличение содержания пластификатора (до 6,0 масс.ч.) и наполнителя (до 20,0 масс.ч.) приводит к росту вязкости смеси по Муни. Оптимальные технологические показатели наблюдались при дозировке 6,0 масс.ч. органической части нефтешлама и 20,0 масс.ч. цеолита. Испытания показали, что замена традиционных компонентов на предложенные модификации обеспечивает соответствие свойств резиновых смесей требованиям норм контроля. *Выводы.* Применение органической части нефтешлама и цеолита в рецептуре резиновых смесей позволяет заменить традиционные мягчители и наполнители без ущерба для свойств смесей, предназначенных для уплотнений гидрозатворов, что подтверждается соответствием контрольным нормам.

Ключевые слова: изопреновый каучук, резиновые уплотнений гидрозатворов, вязкость по Муни, пластичность, резина.

Хасанходжаева Биби-Мариям Шераликызы	*PhD докторант*
Сагитова Гузалия Фаритовна	*кандидат технических наук, профессор*
Сидиков Абдужалол Сидикович	*доктор химических наук, профессор*
Кыдыралиева Айгуль Шажалиевна	*PhD докторант*
Сарсенбаева Айзада Узахбаевна	*магистр*
Тусупкалиев Ерсин Адиетович	*Кандидат технических наук*
Кайнарбаева Жания Нурбековна	*Магистр*
Баимбетов Мизамбек Нурбаевич	*Кандидат технических наук*

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