OPTIMIZING THE SWELLING DEGREE OF RUBBER AND ENHANCING ITS OPERATIONAL PROPERTIES USING CELLULOSE FIBERS DERIVED FROM WILD BARLEY STRAW

N.Kh. Karshiyev[5], K.S. Ibragimova[2], A. Khusenov[2], F.B. Eshkurbonov[1], N.J. Nurmatov[3], J.A. Bakirov[4], A.A. Ashurova[1], S.B. Eshkurbonov[4],R.I. Abdullaeva[1], A.Kh. Rakhimov[5],

[1]Termez State University of Engineering and Agrotechnology, Termiz, Uzbekistan
[2]Tashkent Institute of Chemical Technology, Tashkent, Uzbekistan
[3]Termiz State Pedagogical Institute, Termez, Uzbekistan
[4]Termez University of Economics and Service, Termez, Uzbekistan
[5]Denau Institute of Entrepreneurship and Pedagogy, Denau, Uzbekistan

Abstract:
This study investigates the optimization of the swelling degree of rubber compositions and the enhancement of their operational properties through the strategic incorporation of cellulose fibers derived from wild barley (Hordeumspontaneum) straw. In modern polymer science, replacing synthetic additives with environmentally sustainable fillers from local sources holds significant ecological and economic importance. This research evaluates the synergistic effect of cellulose obtained from wild barley when introduced into elastomer matrices along with technical-grade carboxymethylcellulose (CMC). The experimental methodology involved isolating cellulose through the chemical treatment of agricultural waste and systematically incorporating it into the rubber mixture at various concentrations. It has been conclusively demonstrated that these natural cellulose fibers function as both a reinforcing filler and a structural modifier. The primary focus of the research was on the material's performance in aggressive hydrocarbon environments. The results revealed a significant reduction in the degree of swelling when exposed to oil and gasoline, which in turn indicates a higher cross-link density between the fiber and the polymer matrix, as well as improved interfacial adhesion. Furthermore, mechanical tests demonstrated a considerable increase in tensile strength and hardness, resulting in a nearly twofold improvement in the operational durability of the resulting technical rubber products. The cellulose fibers reinforce the elastomer structure, reduce molecular mobility, and thereby prevent its premature degradation. This proposed technology not only paves the way for the creation of high-value-added industrial products but also promotes the principle of "green chemistry" by converting local plant waste into high-performance materials for the automotive and industrial sectors.

Keywords: Wild barley straw, cellulose, carboxymethylcellulose, elastomer, rubber, degree of swelling, mechanical strength, modifier, service life, filler.

1. Introduction.

Polymer materials with limited swelling capacity are used for pipe repair and joint sealing in aqueous environments. Additionally, such limited-swelling polymer materials, primarily rubbers, are in high demand for creating sealing agents used to isolate layers during oil extraction [Novakov et all., 2019; Rakhmanberdiev et all., 2016; Khamdamova et all.,2023].

To impart hydrosorptive properties to rubbers, hydrophilic fillers are incorporated into their composition, one of which is sodium carboxymethylcellulose (Na-CMC) [Egorov et all., 2019; Rakhmanov and Bozorov,2024]. However, many researchers have noted that using Na-CMC leads to a decline in the elastic and strength properties of the rubbers [Ibragimov et all., 2018]. For this reason, it is crucial to find new additives that can provide rubbers with a specific degree of swelling while maintaining the standard level of their physical and mechanical properties.

Many research studies [Eshqurbonov&Safarova, 2025; Safarova et all, 2025; Eshkurbonov& Safarova, 2024; Eshkurbonovet all., 2025;] have investigated the sorption properties of water-swellable polymers in relation to metal ions. In these studies, water-soluble polymers have been examined as sorbents, modifiers, ion exchangers, fillers, and polymer ligands. Experimental results indicate that the resulting polymers swell to a limited degree, and their constituent functional groups are capable of effectively sorbing cations from the solution.

This study investigated the potential of using cellulose derived from wild barley straw as a water-swellable polymer filler to produce butadiene-nitrile rubber-based composites with improved strength properties.

2. Research Methodology.

The methodology employed in this study is based on a systematic approach that encompasses raw material preparation, chemical extraction of cellulose, and the subsequent creation of rubber-based compositions. Initially, wild barley straw (Hordeumspontaneum) sourced from local agriculture was utilized as the primary precursor for cellulose extraction. To increase the effective surface area for chemical interaction, the straw was first meticulously cleaned of foreign impurities and then mechanically ground into uniform particles with a size of 0.2-0.5 mm. To isolate high-purity cellulose fibers, the ground raw material was subjected to alkaline treatment with a sodium hydroxide (NaOH) solution for 3 hours at a controlled temperature of 90-95°C. This delignification stage is crucial for separating hemicellulose and lignin, after which the resulting pulp was bleached with a 2% hydrogen peroxide (H_2O_2) solution to form a purified cellulose structure [Mulla, S., et al. (2020).]. The incorporation of the extracted cellulose into the elastomer matrix was performed using a laboratory-scale twin-roll mill, which ensured a high degree of dispersion. This degnification stage is important for the separation of hemicellulose and lignin, after which the resulting pulp was bleached with a 2% hydrogen peroxide (H_2O_2) solution to obtain a purified cellulose structure.

3. Experimental part.

Cellulose, prepared from wild barley straw, was introduced into the rubber composition as a water-swellable polymer using a planetary roller extruder (4 zones, central screw at 50 rpm). The cellulose sample from wild barley straw contained 7.0% lignin, 8.9% mineral components, and 1.8% resins and fats. For comparison, ASDACELL HV brand Na-CMC was used as the water-swellable polymer. This polymer is the most widely imported and used product in Uzbekistan. It is manufactured according to the ISO 13500:2008 standard and its composition, in terms of an absolutely dry technical product, includes at least 50% Na-CMC, approximately 35-40% of other water-soluble and insoluble cellulose types, and a water content of no less than 10%, with a degree of substitution of 0.8-0.9%.

Cellulose derived from wild barley straw, obtained using a planetary roller extruder, was introduced into the rubber compounds as a water-swellable polymer component. The cellulose sample was mixed with rubber in a 4-zone extruder, with the central screw rotating at 50 rpm. Chemical analysis of the obtained cellulose sample revealed a lignin content of 7.0%, mineral components of 8.9%, and resin and fat content of 1.8%. The absolute cellulose content was determined to be approximately 60%, while the polyanionic cellulose content was around 10-15%.

Wild barley cellulose and ASDACELL HV brand Na-CMC were initially separated into fractions using the sieve method. Fractions with a particle size of 1-0.5 mm were used in the study.

The main rubber mixture is composed of the following: BNKS-40AN rubber (TU 38.30313-2006), sulfur (GOST 127.4-93), 2-mercaptobenzothiazole (GOST 739-74), zinc oxide (GOST 202-84), stearic acid (GOST 6484-96), and P-324 technical carbon (GOST 7885-86). In some samples, BNK 90/30 bitumen was also used.

The base rubber compounds were prepared on laboratory roll mills. The process of incorporating the sorption polymers into the compound was performed in a laboratory mixer with a chamber volume of 40 cm^3.

Water-swellable polymers - wild barley straw cellulose, Na-CMC, or their combined mixture - were introduced into the base rubber composition at a 1:1 mass ratio relative to the rubber. Mixtures of water-swellable polymers, consisting of Na-CMC and wild barley straw cellulose, were prepared in 40:60 and 60:40 mass ratios, and their effect on the elastomer's properties was studied.

The rheometric properties of the rubber compounds were studied using a Monsanto-100S device in accordance with GOST 12535-84. Vulcanization of the samples was conducted in a hydraulic press equipped with electric heating plates. The tensile properties were evaluated according to GOST 270-75.

The impact resilience of the rubber was determined in accordance with the requirements of GOST 27110-86. The Shore A hardness of the samples was determined using a TSh-200 durometer (hardness tester) in compliance with the GOST 263-75 standard. The degree of swelling for the rubbers in an aqueous medium was determined by the gravimetric (weight) method (GOST R ISO 1817-2009). The relative change in the mass of the samples was calculated using the following formula:

$$Q = \frac{m_t - m_0}{m_0} \cdot 100\%$$

Here: m_t - mass of the sample after swelling for a specified period of time, g; m_0 - initial (dry) mass of the sample, g.

4. Results anddiscussion

In the initial stage of the research, the vulcanization kinetics of the prepared rubber mixtures were studied at a temperature of 170°C, and the optimal vulcanization time for the process was determined. An analysis of the results (Table 1) showed that the introduction of a water-swellable polymer into the rubber composition does not significantly affect the time required to reach the vulcanization optimum (t_{90}).

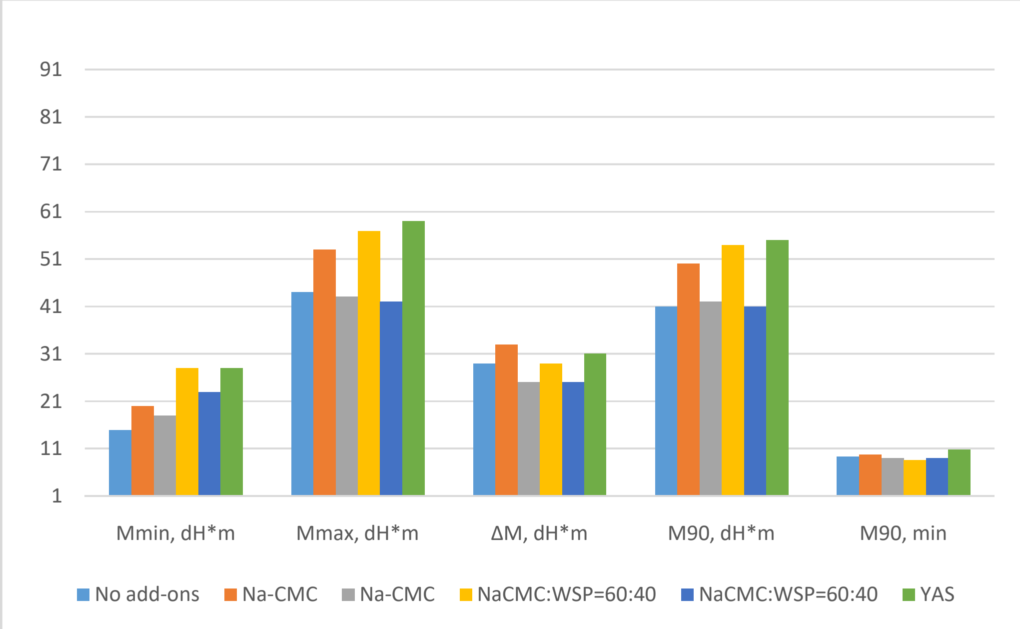

Figure 1. Rheometric properties of rubber compounds, %

(on a Monsanto 100 S instrument, Tvulc=170 °C, Ts,=0.8-1.0 min: Ratio of the base rubber compound to the water-swellable polymer is 1:1 by mass, particle size of the water-swellable polymer is 0.5-1 mm)

However, the introduction of a water-swelling polymer into the composition was observed to increase the internal friction resistance of the mixture (minimum (M_{min}) and maximum (M_{max})). Replacing a portion of the cellulose obtained from wild barley straw with Na-CMC resulted in a relatively smaller increase in the M_{min} and M_{max} indicators. To technologically adjust the viscosity of the mixture, bitumen was introduced into the composition. Experiments showed that adding bitumen in the amount of 20 parts by mass per 100 parts by mass of rubber makes it possible to reduce the Mmin and Mmax values to the level of the control sample, which does not contain the water-swelling polymer. It was also determined that the time to reach the vulcanization optimum remains close to that of the control sample.

The vulcanization of the studied rubber mixtures was carried out at a temperature of 170°C for 10 minutes, taking into account the vulcanization optimum (t_{90}) indicators. The analysis of the main physico-mechanical properties of the tested samples is presented in table 1.

Table 1
Influence of Hydrosorption Fillers and Plasticizer Content (in parts per 100 parts of rubber by mass) on Tensile Strength at Break

№	The amount of fillers and plasticizers	Tensile strength at break, MPa
1.	WSP-Free, non-bitumen Base Rubber	13,8
2.	WBP, non-bituminized	17,5
3.	NaCMC, non-bituminous	3,3
4.	60:40=NaCMC:WBP(bitumen 30 mas.c.)	2,4
5.	60:40=NaCMC:WBP (bitumen 20 mas.c.)	3,4
6.	60:40=NaCMC:WBP (bitumen 10 mas.c.)	4
7.	60:40=NaCMC:WBPnon-bituminous	6,2
8.	40:60=NaCM:WBP (bitumen 30 mas.c.)	3,1
9.	40:60=NaCM:WBP (bitumen 30 mas.c.)	3,9
10.	40:60=NaCMC:WBP (bitumen 30 mas.c.)	6,6
11.	40:60=NaCMC:WBPnon-bituminous	8,6

Analysis of the experimental data (Table 1) showed that using wild barley straw cellulose (WBP) as a water-swellable polymer has a positive effect on the physicomechanical properties of the vulcanizates. Specifically, the conditional tensile strength of the samples containing wild barley straw cellulose was found to have

increased by 26% (from 13.8 MPa to 17.5 MPa) compared to the control sample (without filler). This can be explained by the cellulose fibers forming a reinforcing framework within the elastomer matrix.
Conversely, the introduction of the traditional hydro-absorbent filler, Na-CMC, sharply reduces the tensile strength of the vulcanizates by a factor of four (from 13.8 MPa to 3.3 MPa). This indicates that the adhesive bonding between the Na-CMC particles and the rubber matrix is very weak.
Importantly, when WBP and Na-CMC were used in combination, significantly higher strength values (6.2-8.6 MPa) were obtained compared to samples containing only Na-CMC. This suggests that the cellulose fibers partially compensate for the structural defects caused by Na-CMC.
Due to the high hardness of highly filled rubber compositions, a bitumen plasticizer was introduced in an amount of 10-30 phr (parts per hundred rubber) to improve their processing properties.
As expected, the introduction of water-swellable polymers led to a 10-30% increase in the Shore A hardnes of the rubber compared to the control sample (Table 2). The addition of the plasticizer, in turn, made it possible to reduce this hardness and increase the material's elasticity.

Table 2
The effect of hydrosorption filler and plasticizer content on the Shore A hardness of rubber

№	Amount of fillers and plasticizers	Hardness, according to Schor A, etc.
1.	WSP-Free, non-bitumen Base Rubber	73
2.	WBP, non-bituminized	98
3.	NaCMC, non-bituminous	93
4.	60:40=NaCMC:WBP(bitumen 30 mas.c.)	82
5.	60:40=NaCMC:WBP (bitumen 20 mas.c.)	90
6.	60:40=NaCMC:WBP (bitumen 10 mas.c.)	92
7.	60:40=NaCMC:WBPnon-bituminous	96
8.	40:60=NaCM:WBP (bitumen 30 mas.c.)	90
9.	40:60=NaCM:WBP (bitumen 30 mas.c.)	89
10.	40:60=NaCMC:WBP (bitumen 30 mas.c.)	92
11.	40:60=NaCMC:WBPnon-bituminous	98

However, an increase in the amount of bitumen had a negative effect on the tensile strength properties of the vulcanizates, causing them to decrease to a certain extent. It should be noted that even when bitumen was introduced in quantities of 10-20 phr, the strength properties of the rubbers remained higher than those of the samples containing only Na-CMC. This indicates that the cellulose from wild barley straw retains its reinforcing role within the system.

Table 3.
The effect of hydrosorption filler and plasticizer content on rebound elasticity

№	Amount of fillers and plasticizers	Returnelasticity, %
1.	WSP-Free, non-bitumen Base Rubber	28
2.	WBP, non-bituminized	28
3.	NaCMC, non-bituminous	14
4.	60:40=NaCMC:WBP(bitumen 30 mas.c.)	10
5.	60:40=NaCMC:WBP (bitumen 20 mas.c.)	14
6.	60:40=NaCMC:WBP (bitumen 10 mas.c.)	17
7.	60:40=NaCMC:WBPnon-bituminous	18
8.	40:60=NaCM:WBP (bitumen 30 mas.c.)	15
9.	40:60=NaCM:WBP (bitumen 30 mas.c.)	16
10.	40:60=NaCMC:WBP (bitumen 30 mas.c.)	18
11.	40:60=NaCMC:WBPnon-bituminous	22

According to experimental data (Table 3), the introduction of wild barley cellulose does not significantly affect the rebound elasticity of the vulcanizates. However, the addition of Na-CMC reduces this indicator by a factor of two.

When wild barley cellulose and Na-CMC are used together, an increase in rebound elasticity was observed as the amount of wild barley cellulose in the system increased. The introduction of bitumen into the rubber compound leads to a decrease in rebound elasticity to the level of the sample containing Na-CMC.
At the next stage of the research, the influence of water-swellable polymers and the bitumen plasticizer on the degree of rubber swelling was studied in drinking water and formation water environments. It was determined that for all samples, the swelling process at room temperature is of a limited nature.
According to the results obtained (Table 2), it was noted that when wild barley straw cellulose is used alone, the degree of rubber swelling is lower compared to samples with Na-CMC. Bituminous and non-bituminous combinations of wild barley straw and Na-CMC demonstrated intermediate values for the degree of swelling.

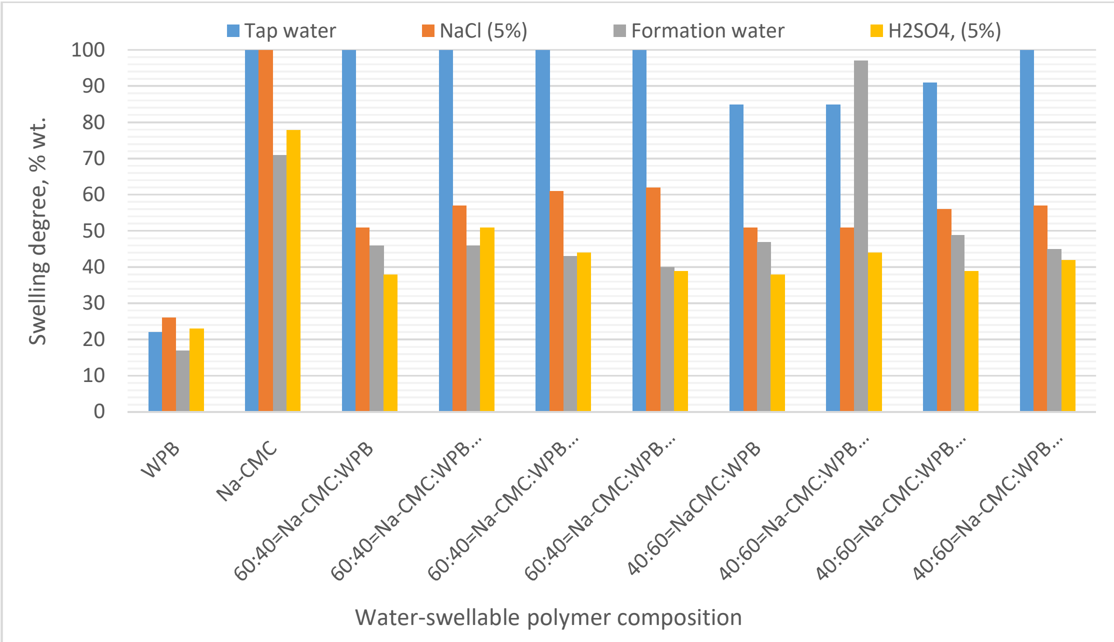

Figure 2. Effect of aqueous medium pH on the degree of rubber swelling
(Hydrochemical composition of formation water (mg-eq/l Cl^- - 139; SO_4^{2-} - 0,7; HCO_3^- - 0,2; Ca^{2+} - 11; Mg^{2+} - 3; Na^++K^+ - 70;

Plasticizer - grade 45/190 bitumen, in parts by mass per 100 parts of rubber)
An analysis of the experimental data revealed that, unlike Na-CMC, the pH of the medium has virtually no effect on the swelling degree of rubbers containing wild barley straw cellulose (Table 2). This demonstrates the versatility of rubbers based on wild barley straw cellulose for use in various mineralized aqueous environments.

5. Conclusion

Using wild barley straw cellulose as a hydrosorbent filler in rubber based on BNKS-40AN at a 1:1 ratio to the base rubber mass increases the vulcanizate's conditional tensile strength compared to a sample without a swellable polymer. The tensile strength of rubbers prepared with a mixture of wild barley straw cellulose and Na-CMC is significantly higher than that of the widely used Na-CMC sample, and it was determined that this indicator increases linearly as the cellulose content in the system rises. Although a high degree of filling with cellulose-containing polymers leads to increased material hardness, the elasticity of vulcanizates made with wild barley straw cellulose remains stable at the level of rubber without water-swellable polymers.
Furthermore, it was confirmed that the degree of swelling in aqueous solutions for rubber containing wild barley straw cellulose is 2-3 times lower than that of rubber using only Na-CMC as the hydrosorbent polymer, and the presence of a plasticizer has almost no negative effect on this process. In mixed compositions of water-swellable polymers made from wild barley straw cellulose and Na-CMC, the degree of swelling had intermediate values, and an increase in swelling indicators was observed as the Na-CMC content in the system increased.

References

Egorov, E. N., Ushmarin, N. F., &Efimov, K. V. (2019). Influence of functional ingredients on the technological properties of rubber water-swellable sealing elements. *Butlerov Communications*, 58(6), 146-151.

Eshkurbonov, F. B., & Safarova, E. R. (2024). Extraction of silver ions by complex-forming ionites based on epichlorohydrin and thiourea. *AIP Conference Proceedings*, 3244, 050018. https://doi.org/10.1063/5.0241890

Eshkurbonov, F. B., & Safarova, E. R. (2025). Application of diglycidylthiourea and melamine-based ionites in concentration and extraction of silver from Khanjizza ores. *AIP Conference Proceedings*, 3304, 040031. https://doi.org/10.1063/5.0269570

Eshkurbonov, F. B., Izatillayev, N. A., Rasulov, A. A., Ashurov, J. M., & Safarova, E. R. (2025). Physico-chemical studies of mono-aqua-coordinated copper-based bis(hydroxynaphthaldehyde) complex. *New Materials, Compounds and Applications*, 9(1), 134-141. https://doi.org/10.62476/nmca.91134

Ibragimov, M. A., Arkhireev, D. A., &Salikhov, A. Ya. (2018). Investigation of swellable packer damage. *PromyshlennoeProizvodstvoiIspolzovanieElastomerov* [Industrial Production and Use of Elastomers], (2), 35-40.

Khamdamova, D., Umarova, V., Primkulov, M., &Khusenov, A. (2023). Study on the microcrystalline cellulose from medicinal plants. *E3S Web of Conferences*, 434, 03038.https://doi.org/10.1051/e3sconf/202343403038.

Mulla, S., et al. (2020). Extraction and characterization of cellulose nanofibers from agricultural wastes for reinforcement in polymer composites. International Journal of Biological Macromolecules, 156, 1492-1502.

Novakov, I. A., Vaniev, M. A., &Lopatina, S. S. (2019). The state and development trends of the production and application of water- and oil-swellable elastomers for packer equipment. *KauchukiRezina* [Rubber and Rubber], 78(4), 228-237.

Rakhmanberdiev, G. R., Murodov, M. M., &Khusenov, A. Sh. (2016). Technologies for producing cellulose through the integrated processing of Central Asian poplar and Jerusalem artichoke. *KhimiyaiKhimicheskayaTekhnologiya* [Chemistry and Chemical Technology], Special Issue, 51-58.

Rakhmanov, J. J., &Bozorov, O. N. (2024). A Fundamental Scheme and Technology for Producing New Composite Types of Drilling Fluids for Reinforcement, Based on Low-Degree-of-Substitution Carboxymethylcellulose Derived from Fibrous Waste of Cotton Ginning and Textile Enterprises. *Scientific News of Kokand State Pedagogical Institute*, (4), 57-64.

Rakhmanov, J. J., Bozorov, O. N., &Normakhmatov, B. X. (2024). Technology for Obtaining Stabilizing Drilling Fluids of a Modified Composite Composition from Various Grades of Na-CMC Produced from Local Raw Materials. *Scientific News of Kokand State Pedagogical Institute*, (4), 65-76.

Safarova, E. R., Eshkurbonov, F. B., Daminova, Sh. Sh., Bobokulova, M. Sh., Esanov, R. R., &Saparova, S. B. (2025). Synthesis of complex-forming N, S-ionite photonic materials. *Proceedings of SPIE*, 14014, 140140W. https://doi.org/10.1117/12.3094070.