

Авторлар туралы мәліметтер

Дарья Юрьевна Островная – докторант, Манаш Қозыбаев атындағы Солтүстік Қазақстан университеті, Петропавл, Қазақстан; e-mail: darya_ostrovnoyaya@mail.ru. ORCID: <https://orcid.org/0000-0002-2843-472X>.

Антонина Николаевна Дюрягина – химия ғылымдарының кандидаты, профессор, Манаш Қозыбаев атындағы Солтүстік Қазақстан университеті, Петропавл, Қазақстан; e-mail: adyuryagina@inbox.ru. ORCID: <https://orcid.org/0000-0002-9109-8159>.

Юлия Сергеевна Бызова* – PhD, доцент, Манаш Қозыбаев атындағы Солтүстік Қазақстан университеті, Петропавл, Қазақстан; e-mail: yuliyabyzovva@gmail.com. ORCID: <https://orcid.org/0000-0002-2072-0238>.

Сведения об авторах

Дарья Юрьевна Островная – докторант, Северо-Казахстанский университет им. М. Козыбаева, Петропавловск, Казахстан; e-mail: darya_ostrovnoyaya@mail.ru. ORCID: <https://orcid.org/0000-0002-2843-472X>.

Антонина Николаевна Дюрягина – кандидат химических наук, профессор, Северо-Казахстанский университет им. М. Козыбаева, Петропавловск, Казахстан; e-mail: adyuryagina@inbox.ru. ORCID: <https://orcid.org/0000-0002-9109-8159>.

Юлия Сергеевна Бызова* – PhD, доцент, Северо-Казахстанский университет им. М. Козыбаева, Петропавловск, Казахстан; e-mail: yuliyabyzovva@gmail.com. ORCID: <https://orcid.org/0000-0002-2072-0238>.

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S.N. Sagatova*, Kh.S. Rafikova

Kazakh National Research Technical University named after K.I. Satbayev,
050013, Republic of Kazakhstan, Almaty, Satbayeva Street, 22

*e-mail: samira.sagatova9@gmail.com

PREPARATION AND ANALYSIS OF A BIODEGRADABLE POLYMER COMPOSITION BASED ON THERMOPLASTIC STARCH REINFORCED WITH STEARIC ACID-MODIFIED MICROCRYSTALLINE CELLULOSE

Abstract: Plastic waste pollution is a global environmental problem. Therefore, the transition to environmentally friendly, biodegradable polymer materials is an urgent challenge for humanity. Thermoplastic starch is one of the most promising alternatives to synthetic polymers due to its availability, biodegradability, and elasticity. However, its limited mechanical strength and poor barrier properties restrict its application in packaging. To address these limitations, cellulose – a rigid and renewable biopolymer – was used as a reinforcing agent and modified with stearic acid to improve hydrophobicity and barrier performance. A TPS/M-MCC composite was prepared via melt mixing and analyzed using tensile testing, water absorption, biodegradability, and dynamic mechanical analysis (DMA). Fourier-transform infrared (FTIR) spectroscopy confirmed the successful surface modification of cellulose. Compared with neat TPS, the composite demonstrated significantly enhanced mechanical strength, reduced water absorption and retained biodegradability. These findings suggest that TPS reinforced with stearic acid-modified cellulose is a promising environmentally friendly alternative to conventional synthetic polymers for packaging applications.

Key words: thermoplastic starch, cellulose, stearic acid, polymer composite, packaging materials.

Introduction

Thermoplastic starch is one of the most widely used alternatives among modern biopolymers. Thermoplastic starch is produced by intensive mixing and heating of starch in the presence of plasticizers. It is biodegradable, non-toxic, cost-effective, and exhibits excellent properties compared to many other natural biodegradable polymers. At present, it is applied in the production of packaging

materials, containers and tableware, as well as in pharmaceutical and the food industries, replacing many synthetic polymers [1].

The transition towards environmentally friendly, biodegradable and cost-effective natural materials has become a defining trend of the current generation. This shift is primarily driven by the growing concern over global plastic pollution and its associated toxicity. Every year, hundreds of thousands of tons of plastic waste enter and contaminate the environment worldwide [2]. For example, in Almaty, a major metropolis of Kazakhstan, the total amount of municipal household waste generated annually is estimated at approximately 1000-1200 thousand tons, of which about 23% consists of plastic waste. The morphological composition of household waste for the considered year is illustrated in Fig.1 [3].

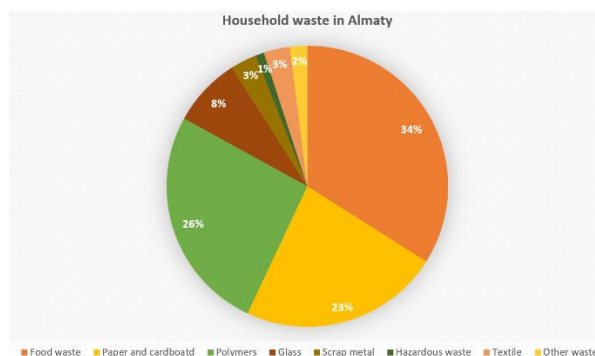


Figure 1 – Diagram of the morphological composition of household waste in Almaty for 2023 year

As evidenced by the data presented in the diagram, polymer waste ranks second after food waste. However, unlike food waste, polymer waste does not decompose or disappear naturally. On average, residents, including infants, generate approximately 42 kg/day of plastic waste capita per year. The major portion of plastic waste consists of packaging materials, primarily containers, bottles, and plastic bags, which are mainly produced from polymers such as polypropylene, polyethylene, polyethylene terephthalate, polystyrene, and polyurethane. These polymeric materials either do not degrade at all or require an extremely long period to decompose under natural conditions. Moreover, most of them are neither recycled nor reused, which makes them one of the largest contributions to environmental pollution [4].

Consider these factors; the transition to more environmentally friendly and biodegradable biopolymers has become an urgent necessity of present time. Among them, thermoplastic starch is regarded as one of the most promising materials for the production of eco-friendly packaging products [5].

The main drawback of thermoplastic starch lies in its relatively poor mechanical properties. To enhance its strength, various reinforcing agents are employed, including metal oxides, natural biomasses such as wood shavings, agricultural and food industry residues, clays, as well as biopolymers such as cellulose, chitosan and chitin. Below, studies conducted in this field and polymer composites developed based on thermoplastic starch are presented (Table 1).

Table 1 – Effect of different fillers on the properties of thermoplastic starch

Polymer composition	Preparation method	Influence of the filler on the matrix	Reference
TPS/ZnO	Solvent casting	The mechanical strength increased, and antimicrobial activity was also observed	[6]
TPS/eucalyptus nanofibrils	Extrusion	An increase in strength was observed with the incorporation of eucalyptus nanofibrils (ENF), with the optimal concentration determined to be 4%. Moreover, the composite demonstrated faster and more efficient biodegradation compared to neat thermoplastic starch (TPS)	[7]
TPS/Poly (butylene succinate) / citric acid	Thermal compression	The incorporation of fillers enhances the processability of cassava starch	[8]
TPS/nanocellulose	Solvent casting	Improvement in strength and resistance to mechanical stress	[9]

As can be seen, fillers used in thermoplastic starch generally improve its mechanical properties, thereby increasing the strength of the final product. However, most fillers primarily enhance the mechanical performance of the polymer composition, while the issue of barrier properties remains unresolved. Therefore, in the present work, both the barrier and mechanical properties of the resulting polymer composition were investigated [10].

The aim of this study was to develop a biodegradable polymer material based on thermoplastic starch with improved barrier and mechanical properties. For this purpose, cellulose was selected as the filler, as it possesses excellent mechanical properties and, according to literature data, significantly enhances the strength of thermoplastic starch. However, objectives of this study were not limited to improving mechanical performance; they also included enhancing the barrier properties of the material. In this context, barrier properties refer to the resistance of a material to water vapor and gases [11].

Since both cellulose and thermoplastic starch exhibit relatively low barrier properties, stearic acid was introduced to enhance moisture resistance. Stearic acid is a non-toxic and readily available hydrophobic component capable of improving the water resistance of the material [12]. To achieve better dispersion of stearic acid and to improve the homogeneity of the resulting polymer composite, a filler modification approach was employed, specifically the modification of cellulose.

In conclusion, this study resulted in the development of a cost-effective, environmentally friendly, and biodegradable polymer composite material suitable for use in the production of packaging products.

Materials and methods

Modification of cellulose with stearic acid

Microcrystalline cellulose (MCC) was mixed with 95% ethanol in a 1:3 ratio in a two-neck round-bottom flask. The mixture was thoroughly stirred for 30 minutes using a magnetic stirrer. Stearic acid, in an amount equivalent to that of cellulose, was then added to the mixture. The resulting mixture was continuously stirred with heating for 8 hours at 80°C. To prevent ethanol evaporation, a reflux condenser was attached to one neck of the flask, while a thermometer was placed in the other neck to monitor the temperature. The obtained modified MCC was filtered and dried in a drying oven at 50°C for 6 hours, followed by 12 hours at room temperature. The success of the modification was confirmed using FTIR spectroscopy (fig. 2).

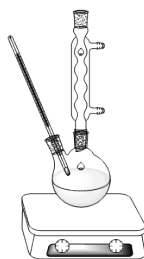


Figure 2 – Laboratory setup for cellulose modification

Preparation of TPS/M-MCC composite material

Corn starch was dissolved in distilled water at a 1:19 mass ratio and heated at 70°C for 10-15 minutes until the thermoplastic starch (TPS) reached a viscous consistency. Subsequently, modified MCC (M-MCC), pre-dissolved in ethanol at a 1:3 volume ratio, and 2 mL of glycerol were added. The resulting suspension contained 20 wt. % of M-MCC relative to TPS. The mixture was stirred for 20 minutes, after which it was poured into a Petri dishes to form films. The films were dried at room temperature for 24 hours.

In total, two types of films were prepared: one neat TPS film and one TPS/M-MCC composite film with 20 wt. % M-MCC. Initially, the films were cast as circular sheets. For further characterization and testing, specimens were cut from these films into standardized dumbbell-shaped samples for mechanical and barrier property measurements.

Methods for Sample Analysis

FTIR spectrometry

Fourier-transform infrared (FTIR) spectroscopy was performed to confirm the modification of microcrystalline cellulose with stearic acid. Samples of unmodified and modified microcrystalline

cellulose (0.3-0.5 mg) were thoroughly mixed with potassium bromide (0.5 g) and pressed into pellets with a diameter 13 mm. The FTIR spectra were collected in the range of 4000-400 cm^{-1} with a resolution of 4 cm^{-1} using an IR Fourier spectrometer.

Dynamic Mechanical Analysis (DMA)

Dynamic mechanical analysis (DMA) was performed to determine the glass transition temperature (T_g) of polymer samples. In this study, T_g and the loss tangent ($\tan \delta$) of thermoplastic starch (TPS) and TPS/M-MCC composite were measured using a DMA DX04 T analyzer (RM Electronic Measuring Instruments, Lázně Bělohrad, Czech Republic). Thermoplastic starch was used as a reference for comparison.

Rectangular specimens with dimensions of 40×8×2 mm, were subjected to sinusoidal three-point bending with a constant force amplitude of 500 mN at a frequency of 1 Hz. Measurements were conducted over a temperature range of -93 to 100 °C with a heating rate of 2 °C/min.

Mechanical properties

The mechanical properties of the samples were evaluated by tensile test. Specimens were prepared in a standardized «dumbbell» shape in accordance with the relevant ISO 527-3 standard. Tensile testing machine (Instron 3365, Instron, Norwood, MA, USA) at a constant crosshead speed of 100 mm/min at room temperature. For each component, five specimens were tested, and the average values were reported.

Barrier properties: Water Absorption

The barrier properties of the samples were evaluated by measuring their water absorption. Dumbbell-shaped specimens were conditioned in two controlled environments with relative humidities of 50% and 90%, respectively, following ASTM E 104. Samples were placed in sealed desiccators at room temperature for 7 days, after which they were weighed. Subsequently, the specimens were dried over silica gel in a desiccator at room temperature until a constant weight was achieved (approximately 14 days).

Water absorption (WA) was calculated using the following equation:

$$\text{WA}(\%) = ((W_2 - W_1)/W_1) \times 100 \quad (1)$$

where w_2 is the mass of the sample in the moistened state (g), and w_1 is the mass of the sample in the dried state (g).

Biodegradability

The biodegradability of TPS and TPS/M-MCC samples was evaluated in accordance with ASTM D5338. Specimens were placed in polyester mesh bags and buried in containers filled with pre-conditioned compost soil. The containers, equipped with perforated lids for ventilation, were kept in a drying oven at approximately 58° C for 7, 14 and 28 days. These compost moisture content was maintained at ~50% throughout the experiment by regular watering.

After one, two and three weeks, the first, second and third layers of samples was excavated, cleaned, dried to a constant weight, and weighed. The biodegradation (BD, %) was calculated according to the following equation:

$$\text{BD}(\%) = ((m_0 - m_1)/m_0) \times 100 \quad (2)$$

where m_0 is the mass of the sample before decomposition (g), m_1 is the mass of the sample after composting and drying (g).

Results and discussion

Modification of Microcrystalline Cellulose with Stearic Acid and Comparative FTIR analysis with Unmodified Microcrystalline cellulose

The modification of microcrystalline cellulose (MCC) with stearic acid (SA) is attributed to the formation of hydrogen bonds between the hydroxyl groups of cellulose and the carboxyl groups of stearic acid (fig. 3).

The resulting TPS/M-MCC composite exhibited a slightly different appearance compared to neat TPS, as the presence of small white M-MCC particles led to loss of transparency. Representative images of the TPS and TPS/M-MCC films are shown (fig. 4) below.

FTIR spectroscopy was employed to confirm the modification of MCC with stearic acid. The figure below (fig.5) presents the results of the comparative spectral analysis between unmodified MCC and modified MCC (M-MCC).

According to the FTIR spectroscopy data for MCC, a broad band in the range of 3300-3340 cm^{-1} was observed, corresponding to the stretching vibrations of hydroxyl (-OH) groups. In the spectrum of stearic acid-modified MCC (M-MCC) the intensity of this band decreased and its width

was partially reduced, indicating surface modification of MCC due to the formation of hydrogen bonds and partial interaction of carboxyl groups of stearic acid with the hydroxyl groups of cellulose.

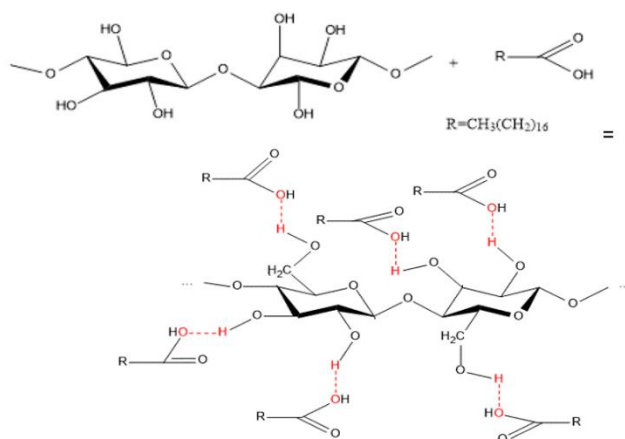


Figure 3 – Modification of MCC and formation of hydrogen bonds between MCC and SA



Figure 4 – Visual appearance of the obtained films: (a) TPS and (b) TPS/M-MCC

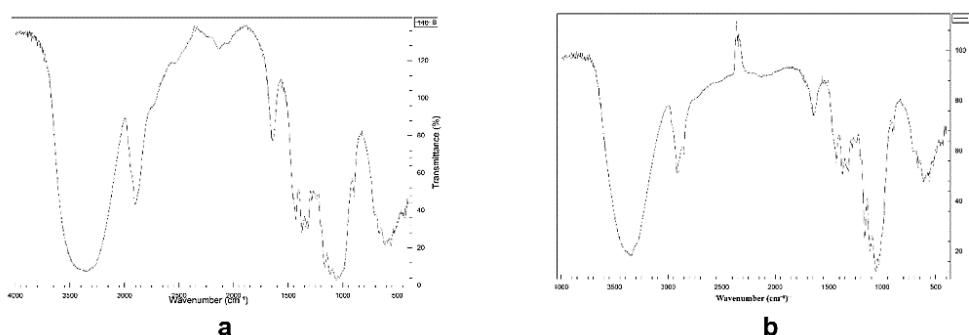


Figure 5 – Results of FTIR spectroscopy for: (a) MCC (b) M-MCC

Additionally, the peaks in the region of $1440\text{--}1390\text{ cm}^{-1}$ increased in intensity, which is attributed to the presence of CH stretching vibrations of the long alkyl chain of stearic acid. However, no new peaks were detected between $1745\text{ and }1715\text{ cm}^{-1}$, which excludes the formation of C=O bonds of esters and confirms that no covalent esterification occurred. This supports through hydrogen bonding rather than deep chemical modification.

At the same time, intense signals were observed in the range of $1050\text{--}1150\text{ cm}^{-1}$, typical for the vibrations of C–O–C glycoside bonds in the cellulose structure, confirming the preservation of the cellulose backbone.

Taken together, these spectral changes confirm the successful surface modification of MCC with stearic acid and the formation of a hydrophobic layer, which is consistent with the observed improvement in filler-matrix compatibility in the composite materials.

Dynamic mechanical analysis

Dynamic Mechanical Analysis (DMA) was conducted to investigate the influence of stearic acid-modified cellulose (M-MCC) on the thermomechanical behavior of thermoplastic starch (TPS). Figure 6 shows the temperature dependences of the storage modulus (E') and loss modulus (E'') for neat TPS and the TPS/M-MCC composite.

The glass transition temperature (T_g) of the samples was determined as the temperature at which the amorphous regions of the polymer transitions from a rigid, glassy state to a soft, rubbery state.

As shown in Figure 6a, neat TPS exhibited a characteristic decrease in E' with increasing temperature, indicating the softening of the starch-plasticizer matrix. The peak E'' and maximum $\tan \delta$ correspond to the glass transition region (T_g), which was observed at approximately 63°C.

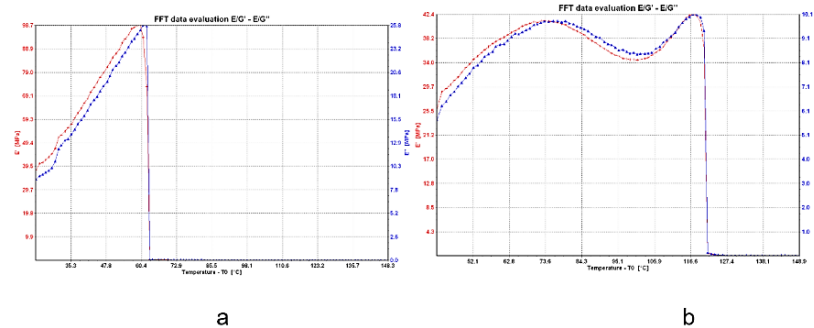


Figure 6 – Results of dynamic mechanical analysis (a) TPS, (b) TPS/M-MCC

In contrast, the TPS/M-MCC composite maintained a higher E' across the entire temperature range, indicating a pronounced reinforcement effect on the filler. The glass transition temperature, determined from the maximum of $\tan \delta$, shifted to approximately 120°C – about 57°C higher than that of neat TPS. This significant shift confirms the restricted mobility of the polymer chains, resulting from the interactions with the hydrophobically modified cellulose.

The $\tan \delta$ peak of the TPS/M-MCC composite was slightly lower and broader compared to that of neat TPS, indicating reduced viscous damping and a more elastic response. Additionally, the appearance of distinct «shoulders» on the E'' suggests the presence of heterogeneous relaxation processes, likely associated with the filler–matrix interface.

Overall, the incorporation of stearic acid-modified cellulose effectively enhanced the rigidity and thermal stability of TPS by improving filler-matrix compatibility and restricting the mobility of the starch chains.

Mechanical properties

The mechanical properties of neat TPS and the TPS/M-MCC composite were evaluated using uniaxial tensile testing. Table 2 summarizes the average values of Young's modulus, maximum tensile strength and elongation at break for the investigated samples.

Table 2 – Results of mechanical testing

Sample	Average value of Young's modulus (MPa)	Average value of maximum tensile strength (MPa)	Average value of elongation at break (%)
TPS	23±2	1,60±0,3	23±3
TPS/M-MCC	31±2	2,89±0,1	27±3

The stress-strain curves obtained from the tensile tests are presented in Figure 7.

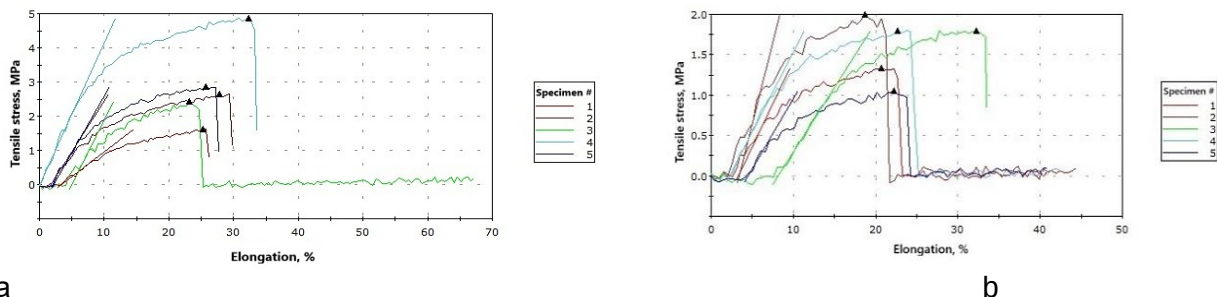


Figure 7 – Tensile strength results: (a) TPS (b) TPS/M-MCC

According to the results summarized in Table 2 and stress-strain curves in Figure 7, the incorporation of M-MCC into the TPS matrix significantly enhanced mechanical performance of the

material. The Young's modulus increased from 23 MPa to 31 MPa, indicating improved stiffness and more efficient stress transfer between the matrix and the filler. Furthermore, the maximum tensile strength nearly doubled, confirming the pronounced reinforcing effect of the M-MCC and its ability to improve the load-bearing capacity of the composite. Notably, the elongation at break also increased slightly, suggesting that the composite retained – and even slightly improved – its ductility. Overall, the TPS/M-MCC composite demonstrated excellent mechanical properties, combining increased stiffness with preserved elasticity.

Barrier properties

Barrier properties were evaluated by measuring the water absorption of the materials. Table 3 shows the water adsorption results for neat TPS and the TPS/M-MCC composite at relative humidity of 50% and 90%.

Table 3 – Results of water absorption tests

Samples	WA at 50%, %	WA at 90%, %
TPS	23±2	52±2
TPS/M-MCC	12±1	39±2

According to the data presented, the water absorption of the composite was nearly reduced by half compared to neat TPS. This indicates that the hydrophobicity of the material was significantly improved due to the modification with stearic acid. Enhanced moisture resistance is advantageous for applications in packaging, particularly for products requiring protection from humid environments or for extending the shelf life of dry goods under wet conditions.

Biodegradation

Biodegradation is a critical parameter for evaluating the environmental impact of bio composites, as it reflects their ability to decompose under natural conditions (Fig.8).



Figure 8 – Visual appearance of biodegradation tests

According to the data in the Table 4 and Figure 9, the incorporation of M-MCC slightly enhanced the biodegradability of the material. The degree of biodegradation increased from 54.35±3% for neat TPS to 57.38±3% for the TPS/M-MCC composite.

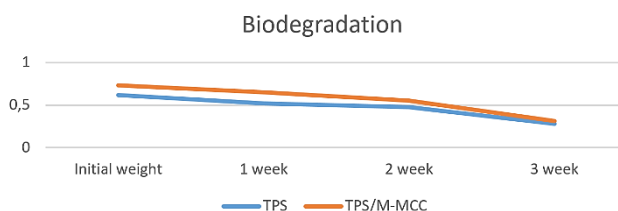


Figure 9 – Biodegradation of polymers as a function of time (weeks)

Table 4 – Results of biodegradation degree of the samples

Material	TPS	TPS/M-MCC
BD, %	54,35±3	57,38±3

In general, the incorporation of the filler did not substantially alter the overall biodegradation behavior of the composite. This is expected, as the filler itself is biodegradable and environmentally benign.

Consequently, the resulting material can be considered environmentally friendly, showing a high degree of biodegradation under composting conditions, and safe for disposal with minimal environmental impact.

Conclusion

In conclusion, the polymer composition based on thermoplastic starch showed excellent performance and fulfilled the intended objectives. Compared with neat thermoplastic starch, the TPS/M-MCC composition exhibited higher strength and toughness. The incorporation of stearic acid-modified cellulose reduced, water adsorption by nearly 50%, significantly improving the barrier properties of the material. The biodegradability of the composite remained comparable to that of neat TPS, confirming its environmental safety and sustainable applications. Overall, the reinforcement with modified cellulose led to a substantial improvement in the mechanical and barrier performance of TPS. Therefore, the TPS/M-MCC composite demonstrates strong potential as an environmental friendly, cost effective and fully biodegradable material for the production of packaging products, contributing to the reduction of plastic pollution.

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С.Н. Сагатова*, Х.С. Рафикова

Қ.И. Сатпаев атындағы Қазақ Ұлттық зерттеу техникалық университеті,
050013, Қазақстан Республикасы, Алматы қаласы, Сатпаев көшесі, 22

*e-mail: samira.sagatova9@gmail.com

СТЕАРИН ҚЫШҚЫЛЫМЕН МОДИФИЦИРЛЕНГЕН МИКРОКРИСТАЛДЫҚ ЦЕЛЛЮЛОЗАМЕН НЫҒАЙТЫЛҒАН ТЕРМОПЛАСТИКАЛЫҚ КРАХМАЛ НЕГІЗІНДЕГІ БИОЛОГИЯЛЫҚ ҮДІРАЙТЫН ПОЛИМЕРЛІ КОМПОЗИЦИЯНЫ АЛУ ЖӘНЕ ЗЕРТТЕУ

Қоршаған ортаның пластикалық қалдықтармен ластануы – жаһандық экологиялық проблемалардың бірі болып табылады. Сондықтан экологиялық таза, биологиялық ыдырайтын полимерлі материалдарға көшу бүкіл адамзат үшін өзекті мәселеу. Термопластикалық крахмал

өзінің қол жетімділігіне, экологиялық тазалығы мен зиянсыздығына және эластикалық қасиеттеріне байланысты синтетикалық полимерлерге ең перспективалы баламалардың бірі болып саналады. Дегенмен де, оның берітігің нашарлығы мен әлсіз тосқауылдық қасиеттері қаптама өнімдерін өндіруде қолдану мүмкіндігін шектейді. Осы кемшіліктерді жою үшін термопластикалық крахмал гидрофобтылығы мен тосқауылдық қасиеттерін арттыру мақсатында стеарин қышқылымен модифицирленген, беріктігі жоғары және қолжетімді биополимер – микрокристалды целлюлозамен нығайтылды. ТПК/М-МКЦ композиті балқымада араластыру әдісі арқылы алынып, созылуға беріктігі, су сіңіру қасиеті, биологиялық ыдырау дәрежесі және динамикалық механикалық талдау (DMA) әдістері арқылы зерттелді. Фурье түрлендірулі инфрақызыл спектроскопиясы (FTIR) целлюлозаның беттік модификациясын растады. Таза ТПК-мен салыстырғанда, алынған композит айтарлықтай жоғары механикалық беріктікті, төмен су сіңіруді және жақсы биологиялық ыдырау қабілетін көрсетті. Бұл нәтижелер модификацияланған целлюлозамен нығайтылған ТПК қаптама материалдарын өндіруге арналған дәстүрлі синтетикалық полимерлерге перспективалы экологиялық таза балама екенін көрсетеді.

Түйін сөздер: термопластикалық крахмал, целлюлоза, стеарин қышқылы, полимерлі композиция, қаптама материалдары.

С.Н. Сагатова*, Х.С. Рафикова

Казахский национальный исследовательский технический университет имени К.И. Сатпаева
050013, Республика Казахстан, Алматы, ул. Сатпаева, 22

*e-mail: samira.sagatova9@gmail.com

ПОЛУЧЕНИЕ И ИССЛЕДОВАНИЕ БИОРАЗЛАГАЕМОЙ ПОЛИМЕРНОЙ КОМПОЗИЦИИ НА ОСНОВЕ ТЕРМОПЛАСТИЧНОГО КРАХМАЛА, АРМИРОВАННОГО МИКРОКРИСТАЛЛИЧЕСКОЙ ЦЕЛЛЮЛОЗОЙ МОДИФИЦИРОВАННОЙ СТЕАРИНОВОЙ КИСЛОТОЙ

Загрязнение окружающей среды пластиковыми отходами является глобальной экологической проблемой. Поэтому переход на экологически чистые, биоразлагаемые полимерные материалы является актуальной задачей для всего человечества. Термопластичный крахмал, благодаря своей доступности, биоразлагаемости и эластичности, является одной из наиболее перспективных альтернатив синтетическим полимерам. Однако его низкая прочность и слабые барьерные свойства ограничивают возможности применения в производстве упаковочных изделий. Для устранения этих недостатков, термопластичный крахмал был армирован микрокристаллической целлюлозой – жестким и возобновляемым биополимером, модифицированным стеариновой кислотой для повышения гидрофобности и барьерных характеристик. Композит ТПК/М-МКЦ был получен методом смешивания расплава и исследован с помощью испытаний на растяжение, определения водопоглощения, оценки биоразлагаемости и динамического механического анализа (DMA). Инфракрасная спектроскопия с преобразованием Фурье (FTIR) подтвердила успешную модификацию поверхности целлюлозы. По сравнению с чистым ТПК, полученный композит показал значительно высокую механическую прочность, сниженное водопоглощение и сохраненную способность к биологическому разложению. Эти результаты свидетельствуют о том, что ТПК, армированный модифицированной целлюлозой, является перспективной экологически чистой альтернативой традиционным синтетическим полимерам для производства упаковочных материалов.

Ключевые слова: термопластичный крахмал, целлюлоза, стеариновая кислота, полимерная композиция, упаковочные материалы.

Information about the authors

Samira Nurbokzy Sagatova* – PhD student and teacher of Kazakh National Research Technical University named after K.I. Satbayev, Almaty, Kazakhstan; e-mail: samira.sagatova9@gmail.com. ORCID: <https://orcid.org/0000-0001-7078-8938>.

Khadichakhan Sabirzhanovna Rafikova – PhD, professor of Kazakh National Research Technical University named after K.I. Satbayev, Almaty, Kazakhstan; e-mail: kh.rafikova@satbayev.university. ORCID: <https://orcid.org/0000-0001-8028-2244>.

Авторлар туралы мәліметтер

Самира Нурболқызы Сагатова* – Қ.И. Сатпаев атындағы Қазақ Ұлттық Зерттеу Техникалық Университетінің оқытушысы және PhD студенті, Қазақстан Республикасы, Алматы қаласы; e-mail: samira.sagatova9@gmail.com. ORCID: <https://orcid.org/0000-0001-7078-8938>.

Хадичахан Сабыржановна Рафикова – PhD, Қ.И. Сатпаев атындағы Қазақ Ұлттық Зерттеу Техникалық Университетінің профессоры, Қазақстан Республикасы, Алматы қаласы; e-mail: kh.rafikova@satbayev.university. ORCID: <https://orcid.org/0000-0001-8028-2244>.

Сведения об авторах

Самира Нурболқызы Сагатова* – PhD студент и преподаватель Казахского национального исследовательского технического университета имени К.И. Сатпаева, Алматы, Казахстан; e-mail: samira.sagatova9@gmail.com. ORCID: <https://orcid.org/0000-0001-7078-8938>.

Хадичахан Сабыржановна Рафикова – PhD, профессор Казахского национального исследовательского технического университета имени К.И. Сатпаева, Алматы, Казахстан; e-mail: kh.rafikova@satbayev.university. ORCID: <https://orcid.org/0000-0001-8028-2244>.

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**B.Sh. Khassankhojayeva¹, G.F. Sagitova*, G.A. Takibayeva¹, S.Zh. Yegemberdiyeva²,
A.T. Kabylbekova³**

¹M. Auezov South Kazakhstan University,
160012, Republic of Kazakhstan, Shymkent, Tauke-khan avenue, 5,

²Regional Innovation University,
160000, Republic of Kazakhstan, Shymkent,
Ryskulova Street, 27/2,

³Miras University,
160000, Republic of Kazakhstan, Shymkent, Sapak Datka, 2
*e-mail: guzalita.f1978@mail.ru

OPTIMIZATION OF RECIPES OF DEVELOPED RUBBER MIXTURES TAKING INTO ACCOUNT THE EVALUATION OF PHYSICAL AND MECHANICAL PROPERTIES

Annotation: Optimization of the composition of rubber compounds is a complex and multifactorial process due to the variety of components used and the complex relationships between them. Conducting a series of experimental studies to select the optimal formulation requires a significant investment of time, raw materials and laboratory resources. In this paper, an approach based on the use of mathematical modeling methods is proposed, which makes it possible to predict the properties of rubber compounds and determine their optimal composition. Special attention is paid to the use of natural zeolite and oil refining waste as modifying additives, which helps to increase the efficiency and environmental friendliness of production. The additives used include the organic fraction of oil sludge and natural zeolite from the Chankanai deposit. Based on a priori information, an experimental design was developed to enable the construction of adequate mathematical models using the least squares method. These models describe the relationships between the component composition and both performance properties and economic criteria. An optimization problem is then formulated based on these models, followed by an analysis of its features and the selection of the most effective solution method. The effectiveness of the proposed approach is demonstrated through an example of optimizing the rubber compound formulation. Modification of rubber compounds makes it possible to obtain composite materials with increased oil resistance, stable elastic and strength properties. This approach helps to reduce production costs by using affordable raw materials and simplifying the technological process. The developed computational model for determining the optimal composition of a rubber compound can be used both in scientific research and in industrial practice in the development of new formulations of elastomeric compositions.

Key words: multicomponent mixtures, rubber mixtures for sealing the hydraulic seals, optimization problem, conditional tensile strength, composition – cost.

Introduction. Modern production of rubber, construction and other industrial products relies on the use of complex multicomponent systems, which include binders, fillers, resins, plasticizers and various chemical additives that provide the required performance characteristics of materials. These materials represent heterogeneous systems composed of particles and grains of varying