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EFFECT OF ULTRASOUND-TREATED CELLULOSE FILLER ON THE MORPHOLOGY AND INTERMOLECULAR INTERACTIONS OF TPS MATRIX

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Abstract. This study examines how cellulose fillers, including ultrasound-treated cellulose, affect the structure and interaction in thermoplastic starch (TPS). Scanning electron microscopy (SEM) has shown that pure TPS has a smooth and compact morphology, while TPS/cellulose composites have a coarser and heterogeneous structure with visible fibrillar formations and micro- and submicron scale features. The untreated cellulose forms a coarse layered structure with limited fibrillation, while the ultrasound-treated samples exhibit a more developed fibrous network, indicating improved dispersion and partial fibrillation of the filler. These observations suggest that cellulose functions as an effective hardening phase in the TPS matrix. Fourier Transform infrared analysis (FTIR) confirmed intermolecular interactions between starch and cellulose. Changes in the O–H stretching region and C–O/C–O–C bands indicate the formation of hydrogen

bonds and improved interfacial compatibility. The introduction of pretreated ultrasound cellulose enhances these interactions, resulting in a more integrated composite structure. The combination of SEM and IR analysis results makes it possible to understand how processing methods, such as ultrasound treatment, affect filler dispersion, adhesion, and overall morphology. These structural changes at the micro and submicron levels contribute to the creation of a more interconnected structure in the composite material. Overall, the results show that ultrasound treatment significantly enhances the structural role of cellulose in TPS, improving both morphology and intermolecular interactions. As a result, the developed starch–cellulose biocomposites demonstrate great potential for use in biodegradable packaging and other environmentally friendly applications.

Keywords: ultrasound treatment, cellulose, starch, SEM, FTIR

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УЛЬТРАДЫБЫСТЫҚ ӨҢДЕЛГЕН ЦЕЛЛЮЛОЗА ТОЛТЫРҒЫШЫНЫҢ ТПК МАТРИЦАСЫНЫҢ МОРФОЛОГИЯСЫ МЕН МОЛЕКУЛААРАЛЫҚ ӨЗАРА ӘРЕКЕТТЕСУІНЕ ӘСЕРІ

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Аннотация. Бұл жұмыс целлюлоза толтырғышының, соның ішінде ультрадыбыстық өңделген целлюлозаның термопластикалық крахмал матрицасындағы

(ТПК) морфологиясы мен молекулааралық өзара әрекеттесуіне әсері зерттелді. SEM талдауы негізінде, таза ТПК салыстырмалы түрде тегіс бетке ие екенін көрсетті, Ал ТПК /целлюлоза композиттерінде целлюлозаның көрінетін фибриллярлық құрылымдары, талшықтардың микро және субмикрондық масштабтағы гетерогенді морфологиясы бар екені анықталды. Өңделмеген сынамада өзіне тән талшықсыз құрылым сақталды, ал өңделген үлгілерде целлюлоза толтырғышының дисперсиясы мен ішінара фибрилляциясының жақсарғанын көрсететін жақсартылған талшықты тор пайда болды. Бұл морфологиялық өзгерістер целлюлозаның ТПК матрицасында тиімді орналасу фазасы ретінде әрекет еткенін көрсетеді. FTIR талдауы крахмал мен целлюлозаның өзара әрекеттесуін -OH созылу аймағындағы және C–O/C–O-C толқын жолақтарындағы өзгерістер арқылы растады, бұл сутектік байланысуды және фазааралық үйлесімділіктің жақсы екенін көрсетеді. Целлюлоза толтырғышы ТПК матрицасына енгізіліп қана қоймайды, сонымен қатар ультрадыбыспен алдын ала өзгертіледі, содан кейін микро және субмикрондық масштабтағы құрылымдық өзгерістерді ИҚ спектрін талдаумен бірге бағаланды. Бұл тәсіл толтырғышты өңдеу режимдерін сутектік байланыстарының сипатымен, композиттің морфологиясымен және фазааралық үйлесімділікпен тікелей байланыстыруға мүмкіндік береді, бұл крахмал-целлюлоза биокомпозиттеріндегі құрылымның пайда болу механизмдері туралы түсініктерді кеңейтеді.Тұтастай алғанда, зерттеу нәтижелері ультрадыбыстық өңдеудің целлюлозаның ТПК матрицасында орналасудағы құрылымдық рөлін күшейтетінін және өзара байланысты композициялық морфологияның қалыптасуына ықпал ететінін көрсетеді. Бұл зерттеулер болашақта биологиялық ыдырайтын қаптамаға және басқа да экологиялық таза қаптамаларда қолдану үшін перспективті болып келеді.

Түйін сөздер: ультрадыбыстық өңдеу, целлюлоза, крахмал, SEM, FTIR

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ВЛИЯНИЕ ОБРАБОТАННОГО УЛЬТРАЗВУКОМ ЦЕЛЛЮЛОЗНОГО НАПОЛНИТЕЛЯ НА МОРФОЛОГИЮ И МЕЖМОЛЕКУЛЯРНЫЕ ВЗАИМОДЕЙСТВИЯ В МАТРИЦЕ ТПК

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Аннотация: В работе исследовано влияние целлюлозного наполнителя, включая целлюлозу, подвергнутую ультразвуковой обработке, на морфологию и межмолекулярные взаимодействия в матрице термопластичного крахмала (ТПК). Результаты СЭМ-анализа показали, что чистый ТПК характеризуется относительно гладкой и компактной поверхностью, тогда как композиты ТПК–целлюлоза обладают более шероховатой и неоднородной морфологией с выраженными фибриллярными структурами микро- и субмикронного масштаба. Необработанная целлюлоза сохраняла крупную слоистую структуру с ограниченной фибриллизацией, тогда как ультразвуковая обработка способствовала формированию более развитой волокнистой сети, что свидетельствует об улучшении дисперсности наполнителя и его частичной фибриллизации. Полученные морфологические изменения указывают на эффективное армирующее действие целлюлозы в матрице ТПК. ИК-спектроскопический анализ подтвердил наличие взаимодействия между крахмалом и целлюлозой, проявляющегося изменением полос поглощения в области валентных колебаний O–H и C–O/C–O–C, что свидетельствует о формировании водородных связей и повышении межфазной совместимости компонентов. Научная новизна работы заключается в использовании предварительной ультразвуковой модификации целлюлозного наполнителя с последующей комплексной оценкой структурных изменений на микро- и субмикронном уровнях в сочетании с анализом ИК-спектров. Такой подход позволил установить взаимосвязь между режимами обработки наполнителя, характером межмолекулярных взаимодействий и морфологией композита. В целом результаты исследования показывают, что ультразвуковая обработка усиливает структурообразующую роль целлюлозы в матрице термопластичного крахмала и способствует формированию более однородной и взаимосвязанной структуры биокомпозита. Полученные материалы представляют интерес для создания биоразлагаемой упаковки и других экологически безопасных изделий.

Ключевые слова: термопластичный крахмал, целлюлоза, ультразвуковая обработка, биокомпозиты, СЭМ-анализ, ИК-спектроскопия, межфазная совместимость, биоразлагаемые материалы

Introduction. With the continuous growth of the world's population, energy demand and demand for traditional household materials are also increasing. These materials are often sourced from non-renewable resources, which entails a significant carbon footprint throughout the life cycle. This trend poses global environmental risks due to the release of pollutants during combustion and the accumulation of microplastics in ecosystems. In addition to pollution, an increase in the concentration of greenhouse atmosphere leads to a climate crisis, manifested in an increase in the global average temperature, a change in precipitation patterns and an increase in extreme weather events. One of the first approaches to solving the problems was to reduce dependence on non-renewable raw materials. This led to a breakthrough in the field of biopolymers, in other words, the use of renewable resources expanded its diversity by dividing it by the properties of decomposition in biological conditions, the method of production and origin. Classification by origin divides them into biobased and non-biobased. However, despite bio-occurrences, polymers such as Bio-PET, PE, PTT, PIT do not decompose in biological conditions and are capable of accumulating microplastics as well as synthetic polymers. On the contrary, biopolymers may be petroleum-based in origin but be capable of biodegradation, or may contain both types of raw materials.

These differences require separate sorting to maintain the efficiency and quality of recycling. Consequently, modern research focuses not only on developing an environmentally friendly composition but also on controlling the subsequent life cycle of biomaterials. For the production of bio-based polymers, raw materials of natural origin are used, such as edible sugars, starch, chitosan, and lactic acid. However, their targeted cultivation is expensive, especially for edible raw materials such as rice, corn, and potatoes. However, waste with a similar composition can reduce costs. Lignocellulosic biomass from agro-industrial and forest waste and even food waste (bread, pasta) can be substituted in the form of cheap raw materials (Mujtaba et al., 2023). In these circumstances, the transition to sustainable agriculture, which focuses on the rational use of resources, waste reduction, and the protection of ecosystem services, is particularly important. A distinctive feature of biogenic synthesis is the ability to select plant raw materials and process parameters to obtain nanomaterials with specific properties. Agricultural residues vary in lignin, cellulose, hemicellulose, polyphenols, organic acids, and other compounds, which affects their reducing ability, sorption properties, and role in stabilizing nanoparticles. Understanding how the size, morphology, chemical composition, and biocorona of nanoparticles affect their toxicity and bioavailability remains limited. Taken together, these factors create a scientific and practical contradiction: on the one hand, using agricultural waste for the "green" synthesis of nanomaterials has significant potential to solve urgent agroecological problems; on the other hand, existing knowledge about the properties, behavior, and safety of such materials remains fragmentary.

This necessitates comprehensive research aimed at analyzing the potential of agricultural waste as a raw material for nanomaterial production, systematizing existing approaches to their synthesis and application, and identifying key limitations and prospects for further development of this area. Therefore, technologies are required that simultaneously increase agricultural productivity and reduce its resource and environmental burden. Second, in many agricultural regions of the world, the problem of recycling plant residues and other types of agrobiomass persists, leading to additional greenhouse gas emissions and air and soil pollution during their combustion or uncontrolled decomposition. Transforming these wastes into highly functional materials not only reduces the negative impact on the environment but also creates new value chains in rural areas (Barik et al., 2026).

Literary review

Starch is a widely known polysaccharide of natural origin, consisting of amylose and amylopectin. It has hydrophilic properties due to the presence of OH groups and decomposes completely in the natural environment. In addition, it can be extracted both from primary resources such as rice, potatoes, corn, and from secondary resources in the form of plant waste. Despite its significant potential for application, native starch has disadvantages in mechanical strength and water resistance. As a result, the films are brittle and prone to swelling upon contact with water due to their hydrophilic nature, which limits their performance, especially when used as packaging films where condensation occurs during temperature changes. These shortcomings can be eliminated through modern modification methods. For example, ultrasonic treatment enables the creation of thinner, denser structures with reduced vapor permeability.; Crosslinking with citric acid significantly increases thermal stability at 164°C, transparency and hydrophobicity of the surface (Pooja et al. 2024). Due to this, starch finds application in various fields. One of the breakthroughs in modern science is the use of starch in the form of a coating as an indicator of external signals (pH, temperature, water, and enzymes) to control the release of fertilizers (Azeem, 2025). Starch-based materials are considered safe and non-toxic for food contact. It is for this reason that it is used as food packaging material for fruits, vegetables, snacks, and dry foods. Depending on the purpose of modification and functionalization, various materials can be obtained. For example, by filling the starch matrix with antimicrobial agents and essential oils, active packages can be obtained that can inhibit the growth of fungi and bacteria. To reduce gas permeability, nanoclay or titanium dioxide fillers are incorporated, and the resulting mixtures are also mixed with various biopolymers such as chitosan and/or PVA to obtain stronger films (Onyeaka et al., 2022). In addition, the ability to control biodegradation and biocompatibility expands starch applications in biomedicine and pharmacy, including wound-healing dressings, drug-delivery systems, and skeletons for bone and cartilage regeneration (Sobeih et al., 2025).

Common reed (Phragmites australis) is a promising source of lignocellulosic biomass for the production of high-strength fibers, packaging materials, and modern biocomposites. It consists of 35-45% cellulose and 18-25% lignin. In Kazakhstan, reed reserves are mainly concentrated in the southern part and distributed in deltas and along floodplains of large rivers, as well as on the shores of lakes located along the Syrdarya, Shu, Or Ili rivers, and the shores of Lake Balkhash. In the Syrdarya delta alone, its volume reaches 1.24 million tons on 58,935 hectares. Biomass is promising due to its high cellulose content, and its utilization is now limited not only to the paper industry but also to the production of innovative polymers, biofuels, and high-strength fibers. Despite its high potential, biomass in Kazakhstan is mainly used for animal husbandry as livestock feed.

Regarding chemical properties, the presence of hydroxyl groups (-OH) in cellulose allows it to interact with starch molecules, forming hydrogen bonds. High compatibility inhibits the mobility of starch chains, thereby increasing the rigidity and strength of the composite. Despite this, composites face several difficulties, including uneven dispersion, poor interfacial adhesion, and hydrophilicity, which hinder blending with hydrophobic polymers (Baibagyssov et al., 2020; Baibagyssov et al., 2025). To eliminate these disadvantages, modern research suggests various methods of physical and chemical modification: chemical modification of the cellulose surface is carried out by: - alkaline treatment to remove lignin/ hemicellulose and improve mechanical adhesion; - silanization by introducing hydrophobic groups, reducing hydrophilicity and improving adhesion; - esterification of nanocellulose fibers to increase the interfacial compatibility of composite films with hydrophobic polymers.

The use of compatibilizers and plasticizers. For example, maleic anhydride as a compatibilizer improves the dispersion of fibers in a polymer matrix (García-Castellanos et al., 2025). In the case of plasticizers, increasing their concentration to 40% by weight of starch also enabled more uniform dispersion of CNCs (cellulose nanocrystals) in the TPS polymer matrix (Csiszar et al., 2021).

Mechanical processing. According to the results of a study by Kong et al. (2023), ball milling of NFC (nanofibrillated cellulose) leads to improved adhesion between the matrix and the filler. In addition, it is worth noting that a more uniform distribution of cellulose can be achieved with a suspension than with a dry one. In this case, there are known methods for converting cellulose into a more homogeneous structure. For example, in the TEMPO-oxidation method, replacing hydroxyl groups on the cellulose surface with negatively charged carboxyl groups prevents the formation of agglomerates, ensuring the uniformity of the suspension. Acid hydrolysis (e.g., H3PO4) also helps to reduce particle size and improve compatibility with the matrix. Moreover, not only chemical but also physical effects, such as ultrasonic homogenization, make it possible to effectively disperse cellulose by breaking the hydrogen bonds between the fibers. Ultrasonic homogenization is a method that uses sound waves to induce acoustic cavitation in a liquid medium. Important process parameters include the amplitude, power, and duration of ultrasonic exposure, which directly affect the degree of dispersion, particle morphology, and the material's final properties. However, excessive ultrasonic treatment can degrade the structure, reducing the material's overall crystallinity and thermal stability. Based on this, when working with thermosensitive matrices, to avoid thermal destruction, it is necessary to maintain a low temperature using an ice bath (Hafid et al., 2023; Raeisi et al., 2025). This article is aimed at investigating the effect of reinforcing thermoplastic starch with modified cellulose from common reed.

Materials and methods. Materials: Lignocellulose biomass from common reed (Phragmites australis) was collected near the Talas River, Zhambyl region. Chemical reagents sodium hydroxide (NaOH) purity ≥ 98.3% (JSC "Base No. 1 of Chemical Reagents"), hydrogen peroxide (H_2O_2) 50% of PJSC "Khimprom", glycerol purity ≥ 99.5%, citric acid ≥ 99.9%, concentrated sulfuric acid (H_2SO_4) from which the solution was further prepared, were used for extraction with 1% H_2SO_4.

Methods.

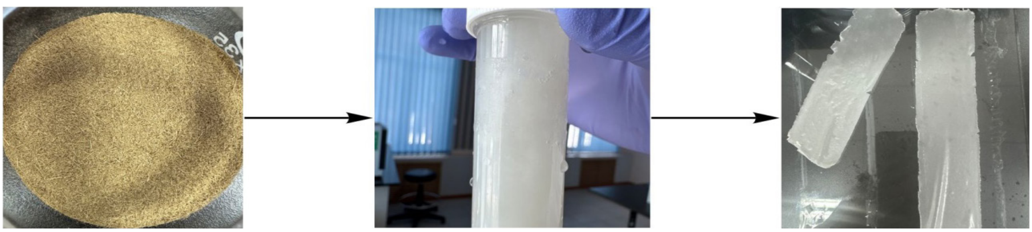
1. Cellulose extraction was carried out according to the method with minor modifications by Başaran Kankılıç and Metin (2020). The collected biomass was washed with distilled water and dried in an oven at 65°C for 48 hours. Next, the dried biomass was crushed with a blender and sieved through a sieve with a mesh size of 0.45 mm. The process of lignin removal (delignification) includes alkaline treatment and bleaching. For the first time, a 2-hour alkaline cooking of biomass was carried out in a 5% NaOH solution at 70°C. To bleach the pulp, a 4% NaOH and 7% H_2O_2 solution mixture was used at a 1:1 ratio, and the biomass was also boiled for 2 hours at 60°C. Each step is repeated 3 times and requires constant rinsing with distilled water between repetitions. Further processing was carried out according to the method of Asem et al. (2023), with minor modifications. First, 1 hour of mild acid hydrolysis was performed in 1% H_2SO_4 at 80°C, and the sample was then rinsed with distilled water 10 times. The resulting pulp was subjected to ultrasonic homogenization for 2 hours. An ice bath was used to maintain the temperature regime and prevent the loss of fiber properties. After that, the processed samples were stored in a refrigerator at 4°C. The pulp yield was calculated gravimetrically using the following formula (Başaran Kankılıç, G., & Metin, A. Ü., 2020):

$$\text{Cellulose efficiency (\%)} = \frac{\text{Weight of extracted dried cellulose}}{\text{Weight of untreated biomass used for extraction}} \cdot 100\%$$

2. Film preparation. Extraction of native starch from corn kernels using ultrasonic exposure was carried out as described by Liu et al. (2021). To identify differences between films made of pure TPS and TPS reinforced with cellulose, 2 samples were prepared. Citric acid was used instead of the co-plasticizer. The films were formed by casting using an Elcometer 3580 (casting knife). The ratio of starch and plasticizers for film (Table 1) formation was based on our previous study with minor changes (Yegissinova et al., 2025).

Table 1 — Ratio of components for biofilm preparation

The ratio of components	
TPS biofilm	TPS-cellulose biofilm
15 g Corn starch 125 ml Demineralized water 15 g Glycerol 15 ml Citric acid 6%	15 g Corn starch 100 ml Demineralized water 15 g Glycerol 15 ml Citric acid 6% 25 g Suspension of cellulose 0,5%



P. australis biomass   Cellulose suspension   TPS-cellulose biofilm

Figure 1 — Preparation process of TPS–cellulose biofilm from *Phragmites australis* biomass.

Results and discussions.

FTIR analysis

IR spectroscopy was used to study the molecular structure of TPS and evaluate the effect of an ultrasound-treated cellulose filler on intermolecular interactions in a composite system. The initial substances of the biocomposite were also studied: starch and suspension of fibrillated cellulose.

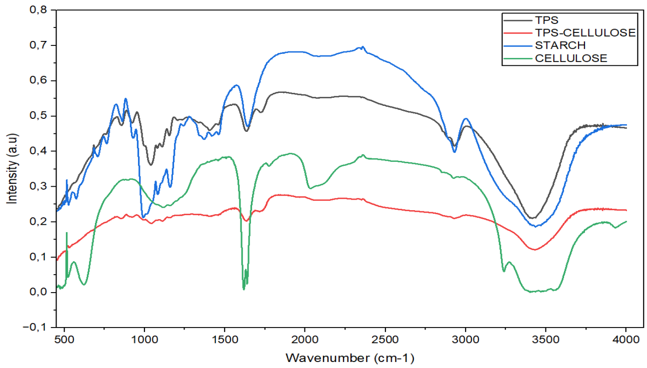
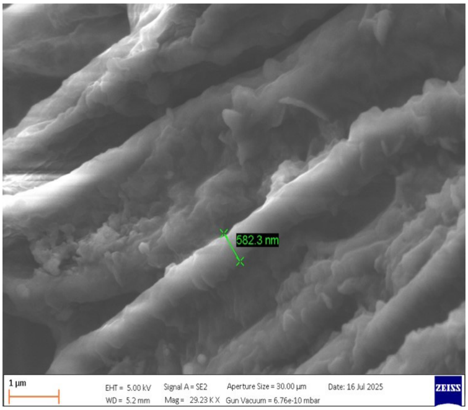


Figure 2 — FTIR spectra: 1 – thermoplastic starch (TPS); 2 – thermoplastic starch with cellulose filler (TPS-Cellulose); 3 – native corn starch (Starch); 4 – cellulose (suspension).

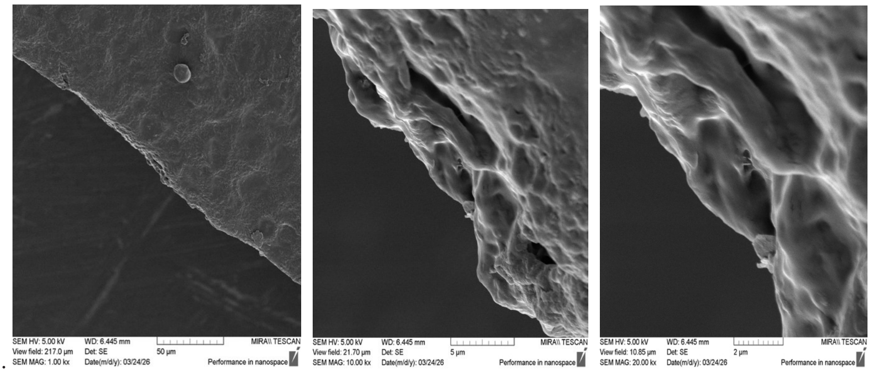
The spectrum of native starch is characterized by a wide absorption band in the range of 3200-3600 cm^{-1}, corresponding to O–H stretching fluctuations associated with hydroxyl groups and extensive intermolecular hydrogen bonds. The absorption peak near 2920 cm^{-1} is explained by the stretching fluctuations of the C–H aliphatic groups. Characteristic starch bands are observed in the fingerprint region between 1200 and 900 cm^{-1}, which corresponds to the stretching fluctuations of C–O, C–O–C and C–C glucopyranose units. The cellulose spectrum showed a similar wide band of O–H elongation of about 3330-3400 cm^{-1}, which reflects the structure of cellulose fibers with a high degree of hydrogen bonds. The band of about 2890 cm^{-1} corresponds to the C–H elongation fluctuations, while the absorption bands in the range of 1430-1160 cm^{-1} are associated with the bending of CH_3 and the asymmetric C–O–C elongation fluctuations characteristic of cellulose. Strong peaks in the range of 1050-1030 cm^{-1} are attributed to the stretching of the C–O polysaccharide chains of cellulose. After plasticization, the TPS spectrum retained its characteristic polysaccharide bands but showed noticeable changes in intensity. The O–H absorption band broadened and shifted slightly, indicating degradation of the native starch crystal structure and the formation of new hydrogen bonds between starch chains and the plasticizer. In addition, decreased intensities of several peaks in the fingerprint region indicate partial destruction of the starch structure during heat treatment. The spectrum of the TPS–Cellulose composite combines the characteristic absorption bands for both TPS and cellulose without the appearance of new absorption peaks. This observation indicates that no new covalent bonds were formed during the preparation of the composite. However, significant changes were observed in the intensity and shape of the O–H stretching region (3200-3600 cm^{-1}) compared with TPS without filler, which indicates an increase in intermolecular hydrogen bonds between the hydroxyl groups of starch and cellulose. A slight shift of the O–H band towards lower wavenumbers further supports stronger hydrogen-bond interactions in the composite matrix. In addition, the increased intensity of the absorption bands in the region of 1000-1150 cm^{-1} confirms the contribution of cellulose to the composite structure. These bands are associated with C–O–C and C–O-O tension fluctuations and indicate the successful incorporation of cellulose fibers into the TPS matrix.

SEM analysis



Figures 3 —TPS-hemp composite (without ultrasound and chemical treatment) (Yegissinova et al., 2025)

Figures 4 and 5, without filler, show a smoother, more solid, and more homogeneous surface, with fewer pronounced fiber elements. Images of cellulose-filled TPS samples show elongated fibrils, bridges, and fibrous inclusions, indicating more pronounced phase heterogeneity and the presence of a reinforcing cellulose mesh within the starch matrix. Samples show a denser, sometimes layered surface morphology and indicate the absence of a reinforcing fibrous phase. The structure remains compact, with relief, folds, and rare small defects; this confirms the relatively homogeneous matrix.



a b c

Figure 4 — SEM image of the surface (edge part) of thermoplastic starch without cellulose at magnification: a -1000; b – 10,000; c – 20,000

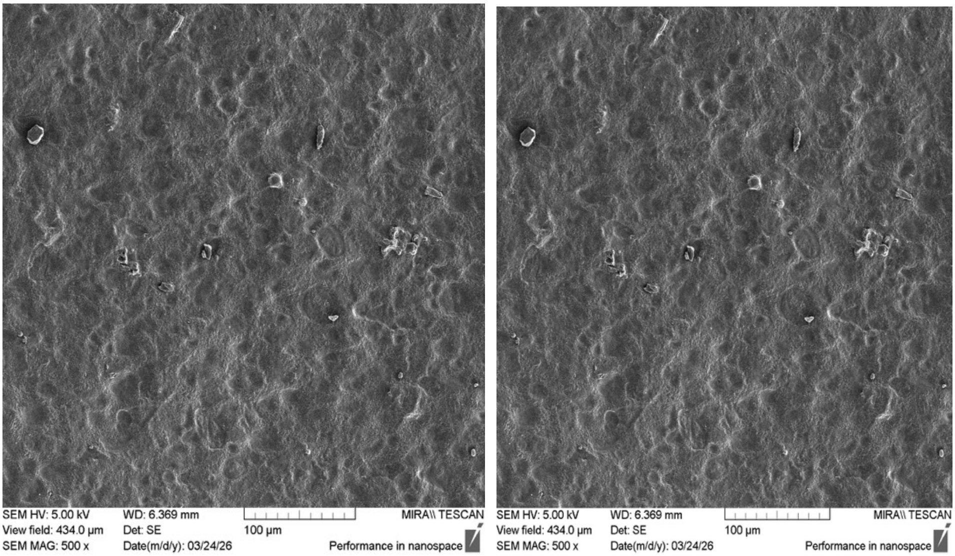


Figure 5 — SEM micrograph of the surface morphology of cellulose-free thermoplastic starch (TPS).

In Figure 6, in the TPS-cellulose biocomposite, thin and thicker fibrous elements appear, sometimes elongated relative to the matrix; this indicates good visibility of the cellulose phase and partial preservation of its fibrous nature. The surface here is not smooth: the cellulose protrudes from the starch matrix, stretches in places, and forms bridges. This indicates that the filler has not completely dissolved but has retained its fibrous nature and remains involved in reinforcing the material. D_1 = 3.54 μm is a larger fibrous element, probably a cellulose bundle or a thickened fibril. D_1 = 0.37 μm, D_2 = 0.33 μm; D_1 = 0.36 μm, D_2 = 0.32 μm are thinner fibrillar areas, indicating partial fibrillation of the filler after ultrasound treatment.

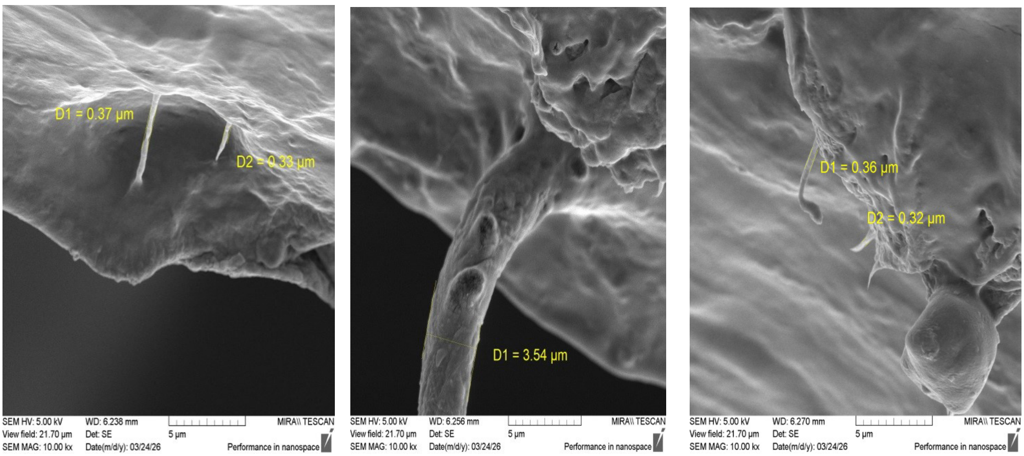


Figure 6 — SEM images of micro- and nanofibrillated cellulose structures in thermoplastic starch-cellulose composite (edge part)

In comparison with our previous researches paper result (Figure 3), where the effect on cellulose was only physical, using ball milling, larger fibers are observed than in this study, confirming the effective effect of ultrasound treatment on fibrillation. Also, visually, the chemically treated films, namely those that have undergone delignification and bleaching, differ significantly in uniformity compared with the previous study.

Conclusion. The results obtained confirm that the introduction of a cellulose filler significantly alters the structure and interfacial characteristics of thermoplastic starch (TPS). SEM analysis showed that pure TPS has a relatively smooth and compact morphology, while cellulose-containing samples have a rougher, heterogeneous, and fibrillar surface with visible fiber protrusions, bridges, and micro- and submicron-sized cellulose fragments. The untreated material retained a coarse-layered structure with limited fibrousness, while the processed samples showed a more developed fibrous network, indicating improved dispersion and structural modification of the filler. The morphology of TPS/cellulose composites suggests that ultrasound treatment and delignification contributed to partial cellulose fibrillation and increased its availability within the starch matrix. The presence of fibers of different sizes reflects a heterogeneous but effective reinforcement structure in which both larger bundles and smaller fibrils contribute to the composite architecture. This morphological change indicates that cellulose acted as an active reinforcement phase rather than a passive additive. IR analysis also confirmed the presence of intermolecular interactions between starch and cellulose. Changes in the O–H stretching region and in the C–O/C–O–C oscillation ranges indicate the development of hydrogen bonds and improved interfacial compatibility between the two biopolymer components. Spectral differences between TPS, starch, cellulose, and TPS-cellulose confirm that the composite is not a simple physical mixture but a system with a modified chemical environment and enhanced phase interactions. Overall, the study demonstrates that cellulose filler, especially after ultrasonic treatment, increases the morphological complexity and interphase organization of TPS. These structural changes are expected to increase the effectiveness of filler reinforcement and make the composite more suitable for further development as a biodegradable material for packaging and related applications.

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