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INVESTIGATION OF THE STRUCTURAL PROPERTIES OF NANOCELLULOSE MEMBRANES DERIVED FROM AGRICULTURAL WASTE

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Abstract. In this article, the structural properties of nanocellulose membranes obtained from agricultural waste were studied. Alfalfa and oat straw were used as raw materials for membrane production. The extraction process included the following stages: alkaline and acid treatment and a bleaching process to remove lignin and hemicellulose components from the plant biomass. The obtained cellulose was mechanically dispersed to form nanosized suspensions, and nanocellulose membranes were prepared by solvent casting. Various modifiers were introduced into the nanocellulose matrix to change the structural characteristics of the membranes. Copper nanoparticles were added to the alfalfa-based membranes, and graphene was added to the oat-based membranes. The structural features of the obtained composites were analyzed using Fourier transform infrared spectroscopy (FTIR) and X-ray diffraction (XRD). The IR analysis confirmed that the main functional groups characteristic of cellulose were preserved in all studied samples. After modification, significant changes in the intensity of the hydroxyl group absorption bands can be observed from the spectra, which indicate changes in intermolecular interactions and hydrogen bonds within the cellulose matrix. Changes in the regions corresponding to C–O–C and β -glycosidic bonds indicated structural changes caused by the incorporation of graphene and copper nanoparticles. X-ray diffraction analysis revealed diffraction peaks characteristic of the crystalline structure of cellulose in all membranes. However, nanomodified samples showed changes in peak intensity and crystallographic order compared to untreated cellulose membranes. The

decrease in diffraction peak intensity indicated partial disruption of ordered crystalline regions and changes in the supramolecular structure of cellulose after the incorporation of nanoparticles. The study revealed that the nanomodification process contributes to changes in the molecular and crystal structure of nanocellulose membranes obtained from agro-waste. The results of the study indicate that the obtained materials have high potential for use in the creation of environmentally safe functional biocomposites and membrane systems.

Keywords: agricultural waste, nanocellulose, nanocellulose membrane, graphene, copper nanoparticles, crystallinity

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АГРОҚАЛДЫҚТАРДАН АЛЫНҒАН НАНОЦЕЛЛЮЛОЗАЛЫ МЕМБРАНАЛАРДЫҢ ҚҰРЫЛЫМДЫҚ ҚАСИЕТТЕРІН ЗЕРТТЕУ

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Аннотация. Мақалада ауыл шаруашылығы қалдықтарынан алынған наноцеллюлозалы мембраналардың құрылымдық қасиеттері зерттелді. Мембраналарды алу үшін шикізат ретінде жоңышқа мен сұлы сабаны пайдаланылды. Целлюлозаны бөліп алу процесі өсімдік биомассасынан лигнин мен гемицеллюлозаны жоюға бағытталған сілтілік және қышқылдық өңдеу, сондай-ақ ағарту кезеңдерін қамтыды. Тазартылған целлюлоза наноөлшемді суспензиялар алу үшін механикалық дисперсияға ұшыратылып, наноцеллюлозалы мембраналар ерітіндіден құю әдісімен дайындалды. Мембраналардың құрылымдық сипаттамаларын өзгерту мақсатында наноцеллюлоза матрицасына әртүрлі наномодификаторлар енгізілді: жоңышқа негізіндегі мембраналарға мыс

нанобөлшектері, ал сұлы негізіндегі мембраналарға графен қосылды. Алынған композиттердің құрылымдық ерекшеліктері Фурье-түрлендірілген инфракызыл спектроскопия (FTIR) және рентгендік дифракция (XRD) әдістерімен зерттелді. FTIR талдауы барлық үлгілерде целлюлозаға тән негізгі функционалдық топтардың сақталғанын көрсетті. Модификациядан кейін гидроксил топтарының жұтылу жолақтары қарқындылығының өзгеруі целлюлоза матрицасындағы молекулааралық өзара әрекеттесулер мен сутектік байланыстардың өзгергенін көрсетті. C–O–C және β -гликозидтік байланыстарға сәйкес аймақтардағы өзгерістер графен мен мыс нанобөлшектерінің енгізілуіне байланысты құрылымдық қайта ұйымдасуды көрсетті. Рентгендік дифракциялық талдау барлық мембраналарда целлюлозаның кристалдық құрылымына тән дифракциялық максимумдардың бар екенін анықтады. Сонымен қатар, наномодификацияланған үлгілерде бастапқы мембраналармен салыстырғанда шыңдардың қарқындылығы мен кристаллографиялық реттелу дәрежесінің өзгеруі байқалды. Дифракциялық максимумдар қарқындылығының төмендеуі реттелген кристалдық аймақтардың ішінара бұзылғанын және нанобөлшектер енгізілгеннен кейін целлюлозаның супрамолекулалық құрылымының өзгергенін көрсетеді. Зерттеу нәтижелері наномодификация агроқалдықтардан алынған наноцеллюлозалы мембраналардың молекулалық және кристалдық құрылымын өзгертуге мүмкіндік беретінін көрсетті. Алынған материалдар экологиялық қауіпсіз функционалдық биокмозиттер мен мембраналық жүйелерді жасау үшін перспективалы болып табылады.

Түйін сөздер: ауылшаруашылық қалдықтары, наноцеллюлоза, наноцеллюлозалы мембрана, графен, мыс нанобөлшектері, кристалдылық

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ИССЛЕДОВАНИЕ СТРУКТУРНЫХ СВОЙСТВ НАНОЦЕЛЛЮЛОЗНЫХ МЕМБРАН, ПОЛУЧЕННЫХ ИЗ АГРООТХОДОВ

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Аннотация. В статье исследованы структурные свойства наноцеллюлозных мембран, полученных из сельскохозяйственных отходов. В качестве растительного сырья для получения мембран использовали люцерну и овсяную солому. Процесс выделения целлюлозы включал щелочную и кислотную обработку, а также отбеливание, направленное на удаление из растительной биомассы лигнина и гемицеллюлозы. Очищенную целлюлозу подвергали

механическому диспергированию с получением наноразмерных суспензий, после чего наноцеллюлозные мембраны формировали методом литья из раствора. Для изменения структурных характеристик мембран в наноцеллюлозную матрицу вводили различные наномодификаторы: в мембраны на основе люцерны добавляли наночастицы меди, а в мембраны на основе овсяной соломы — графен. Структурные особенности полученных композитов исследовали методами ИК-Фурье-спектроскопии (FTIR) и рентгеновской дифракции (XRD). ИК-спектроскопический анализ подтвердил сохранение во всех исследованных образцах основных функциональных групп, характерных для целлюлозы. После модификации отмечено изменение интенсивности полос поглощения гидроксильных групп, что свидетельствует об изменении межмолекулярных взаимодействий и системы водородных связей в целлюлозной матрице. Изменения в областях, соответствующих связям С–О–С и β-гликозидным связям, указывают на структурную реорганизацию, вызванную введением графена и наночастиц меди. Рентгенодифракционный анализ выявил во всех мембранах дифракционные максимумы, характерные для кристаллической структуры целлюлозы. Вместе с тем в наномодифицированных образцах наблюдались изменения интенсивности пиков и степени кристаллографической упорядоченности по сравнению с немодифицированными мембранами. Снижение интенсивности дифракционных максимумов свидетельствует о частичном нарушении упорядоченных кристаллических областей и изменении супрамолекулярной структуры целлюлозы после введения наночастиц. Установлено, что наномодификация способствует изменению молекулярной и кристаллической структуры наноцеллюлозных мембран, полученных из агроотходов. Полученные материалы обладают высоким потенциалом для создания экологически безопасных функциональных биокompозитов и мембранных систем.

Ключевые слова: сельскохозяйственные отходы, наноцеллюлоза, наноцеллюлозная мембрана, графен, наночастицы меди, кристалличность

Introduction. Currently, the issue of processing environmentally safe and renewable raw materials is of particular importance. It is known that the long-term non-degradation of synthetic polymers obtained from oil and oil products in the environment leads to aggravation of environmental problems. In this regard, research on the production of biodegradable materials based on natural polymers is currently being intensively developed. One of such promising biopolymers is cellulose (Ansari et al., 2024). It is a material that is widespread in nature, highly biocompatible, chemically stable and amenable to various types of modification (Wang et al., 2024).

In recent years, interest in technologies for obtaining nanocellulose through the processing of agricultural waste has increased significantly. This approach not only allows for the effective use of plant waste, but also creates conditions for obtaining materials with high functional properties. Oat straw, alfalfa and other agro-industrial waste are considered to be a suitable source of raw materials for obtaining nanocellulose due to the high cellulose content in their composition. Nanocellulose is characterized by

high specific surface area, mechanical strength, low density, and a developed hydrogen bond system. Due to these properties, it is widely used to produce membrane materials, sorbents, packaging materials, biomedical coatings, and functional biocomposites. The properties of nanocellulose membranes directly depend on their structural organization and degree of crystallinity. The ratio of crystalline and amorphous regions affects the mechanical stability, moisture resistance, and permeability properties of the material (Qureshi et al., 2024).

The nature of the plant-based raw material has a significant impact on the structure of nanocellulose. The difference in the amount of lignin, hemicellulose, and cellulose in the raw material causes changes in the morphological and structural features of the obtained materials. In addition, chemical processing methods also affect the crystalline structure of cellulose. In this regard, it is scientifically important to study the structural properties of nanocellulose materials obtained from various agro-wastes (Das et al., 2023).

Currently, one of the effective ways to improve the properties of nanocellulose is its modification with nanoparticles. Graphene-based carbon nanomaterials increase the mechanical strength and barrier properties of membranes, while copper nanoparticles give the materials antibacterial activity. The interaction of nanoparticles with the cellulose matrix leads to changes in hydrogen bonds and structural reorganization, which, as a result, changes in the degree of crystallinity of the material (Ma et al., 2025). Fourier transform infrared spectroscopy (FTIR) and X-ray diffraction (XRD) are widely used to characterize the structural features of nanocellulose materials. The IR method allows us to determine the changes in functional groups and intermolecular interactions, while XRD analysis is used to assess the crystal structure and degree of crystallinity of the material. The combined use of these methods allows us to comprehensively characterize the structural properties of nanocellulose membranes.

Although there are sufficient studies in the literature on nanocellulose obtained from plant residues, there is insufficient comparative study of the structural properties of nanocellulose membranes obtained from various agro-residues and modified with nanoparticles. In particular, there is limited information on the effect of graphene and copper nanoparticles on the structural organization and degree of crystallinity of the membranes. In this regard, the aim of this work is to study the structural properties of nanocellulose membranes obtained from agro-residues using IR spectroscopy and X-ray diffraction methods, as well as to determine the effect of graphene and copper nanoparticles on the structure and degree of crystallinity of the material.

Literature review. Currently, nanocellulose occupies a special place among biodegradable and environmentally friendly materials. Its high mechanical strength, large specific surface area, biocompatibility and susceptibility to chemical modification are of interest to many researchers. In recent years, research on the production of nanocellulose from agricultural waste has gained significant momentum, as this approach allows for the effective use of cheap and affordable raw materials. The literature indicates that nanocellulose based on agricultural waste is considered a functional material with low environmental impact (Ansari et al., 2024; Wang et al., 2024). Plant lignocellulosic

raw materials are often used to produce nanocellulose. Among them, wheat straw, rice husk, oat straw, sugarcane waste and various natural fibers are considered promising sources of raw materials. The amount of cellulose in plant waste, the ratio of lignin and hemicellulose directly affect the structure and properties of the obtained material. Therefore, comparative studies of the structural features of nanocellulose obtained from various agro-wastes are one of the important scientific directions (Qureshi et al., 2024).

Studies have shown that the properties of nanocellulose also depend on the methods of its production (Mamudu et al., 2025). Typically, alkaline treatment, bleaching and acid hydrolysis methods are widely used in the production of nanocellulose. As a result of such treatment, lignin and hemicellulose are separated, and the crystalline structure of cellulose is formed more clearly. In addition, the hydrolysis process contributes to the decomposition of amorphous regions, increasing the degree of crystallinity of the material. Some studies have shown that the crystallinity index of nanocellulose is significantly increased compared to the starting material (Rasheed et al., 2024). In order to improve the structural properties of nanocellulose materials, modification with various nanoparticles is being carried out (Ghosh et al., 2024). In recent years, graphene-based carbon nanomaterials and metal nanoparticles have been widely studied (Ren et al., 2024). The high mechanical strength and large specific surface area of graphene allow improving the barrier and strength properties of nanocellulose membranes. Copper nanoparticles are considered one of the important components for obtaining functional biocomposites due to their antibacterial activity.

The introduction of nanoparticles into the cellulose matrix leads to a change in the structure of the material. It has been shown in the literature that such changes are associated with the reorganization of hydrogen bonds, a change in the ratio of crystalline and amorphous regions. As a result, the mechanical stability, thermal properties and sorption activity of the membranes change (Makhado et al., 2025). Therefore, the study of the structural properties of nanomodified nanocellulose is also important from a practical point of view.

IR and X-ray diffraction methods are widely used in the study of the structure of nanocellulose materials. IR spectroscopy allows us to identify functional groups, hydrogen bonds and chemical changes in cellulose. In particular, the vibrations of O-H groups in the 3200-3400 cm^{-1} region and β -glycosidic bonds in the 890 cm^{-1} region are considered the main signals characterizing the structure of cellulose. X-ray diffraction analysis is an important method for determining the crystal structure, degree of crystallinity, and structural order of cellulose (Rao et al., 2023; Mamudu et al., 2025).

Although the number of works on the structural properties of nanocellulose obtained from various agro-wastes has increased in recent years, the comparative study of the structure and crystallinity of nanocellulose membranes modified with graphene and copper nanoparticles is still insufficiently studied. In particular, information on the combined effect of the nature of the plant raw material and nanomodification is limited. In this regard, the study of the structural properties of nanocellulose membranes obtained from agro-wastes is currently one of the most relevant scientific directions.

Materials and methods of research. *Preparation of nanocellulose-based membranes.* Alfalfa straw and oat straw grown on the Zhumabai farm, Zhualy district,

Zhambyl region, were used as plant agro-residues in the research work. The initial plant raw materials were dried in natural conditions, mechanically crushed, and cleaned of foreign impurities. Nanocellulose extraction consisted of several stages: alkaline treatment, acid treatment, and bleaching (Reshmy et al., 2015). At the first stage, the plant raw materials were treated with a 2% sodium hydroxide (NaOH) solution to remove hemicellulose and lignin. The treatment was carried out in an autoclave at a temperature of 120°C and a pressure of 20 bar for 40 minutes. After alkaline treatment, the samples were washed several times with distilled water and cleaned to a neutral environment. Then, a mixture of nitric acid (HNO_3), acetic acid (CH_3COOH), and distilled water was used for additional purification of the cellulose. As a result of acid treatment, the amount of residual lignin and hemicellulose was reduced and the purity of the cellulose structure was increased. During the bleaching stage, the material obtained was treated with a 1% sodium hypochlorite (NaOCl) solution, resulting in a high-purity white cellulose.

The purified cellulose was processed to a nanoscale structure by mechanical dispersion, and a stable nanocellulose suspension was prepared. Nanocellulose membranes were formed by solvent casting (Norrahim et al., 2021; Ruan et al., 2023). The prepared suspension was poured into special molds and dried at room temperature. In order to improve the functional properties of the membranes, various nanomodifiers were introduced into the nanocellulose matrix. 1% copper nanoparticles were added to the alfalfa-based membranes, and 1% graphene was added to the oat-based membranes. The nanoparticles were pre-dispersed and then mixed with the nanocellulose suspension by mechanical mixing until a homogeneous system was formed. The resulting composite mixtures were poured into molds and dried to prepare functionalized nanocellulose membranes. Four different samples were considered in the study: raw cellulose from alfalfa (AC), alfalfa-based membrane modified with copper nanoparticles (AC2), raw cellulose from oats (OC), and graphene-modified oat-based membrane (OC2). The technological scheme for obtaining nanocellulose-based membranes based on agroresidues is shown in Figure 1.

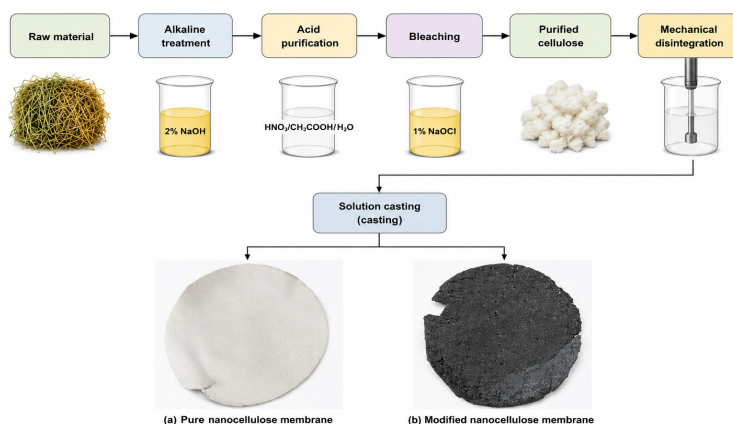


Figure 1 – Scheme for the production of nanocellulose membranes based on agro-waste

X-ray diffraction (XRD). X-ray diffraction (XRD) was performed to determine the crystal structure and degree of crystallinity of the samples. The crystalline properties of cellulose materials significantly affect their mechanical strength, thermal stability, and moisture absorption capacity. In addition, the occurrence of structural changes as a result of chemical treatment and nanomodification is accompanied by a change in the degree of crystallinity. In this regard, the crystalline features of the original and modified nanocellulose membranes were studied using the XRD method. X-ray diffraction patterns were taken on a Bruker D8 Advance (Germany) X-ray diffractometer using CuK α radiation ($\lambda = 1.5405 \text{ \AA}$). Measurements were carried out in the range of $2\theta = 10\text{--}90^\circ$. Based on the obtained diffractograms, the main diffraction maxima, structural arrangement, and changes in the degree of crystallinity of cellulose were analyzed.

Fourier transform infrared spectroscopy (IR). The Fourier transform infrared spectroscopy (FTIR) method was used to determine the chemical structure and functional groups of nanocellulose membranes. FTIR analysis allows us to assess the changes in functional groups in the material structure, intermolecular interactions, and structural features resulting from nanomodification. FTIR spectra of the samples were recorded on a Shimadzu infrared spectrometer. Measurements were carried out in the range of $4000\text{--}400 \text{ cm}^{-1}$. The obtained spectra were analyzed for changes in hydroxyl groups, β -glycosidic bonds, and the main functional groups of cellulose. All experiments were repeated at least three times. The results obtained were statistically processed and expressed as the arithmetic mean and standard deviation. Experimental data processing was carried out using the OriginPro program.

Research results

Analysis of IR spectroscopy results. In order to determine the chemical structure and functional groups of the obtained nanocellulose membranes, IR spectra were studied (Fig. 2). IR results showed that the main functional groups characteristic of cellulose were preserved in all samples. In addition, certain changes were observed in the spectra as a result of nanomodification.

IR spectra of alfalfa-based AC and AC2 samples are shown in Fig. 2a. A broad intense absorption band is observed in the spectra in the region of $3200\text{--}3400 \text{ cm}^{-1}$, which corresponds to the valence vibrations of hydroxyl groups (O–H). A decrease in the intensity of this band in the AC2 sample indicates a change in the hydrogen bond system due to the introduction of copper nanoparticles. In addition, signals in the region of approximately 2900 cm^{-1} are characteristic of the valence vibrations of C–H bonds. The weak signals observed in the range of $1400\text{--}1600 \text{ cm}^{-1}$ are associated with the deformation vibrations of adsorbed water molecules and cellulose structure. The intense absorption bands in the region of $1000\text{--}1160 \text{ cm}^{-1}$ correspond to the vibrations of C–O–C and C–O bonds. The clear signal in the region of $1030\text{--}1050 \text{ cm}^{-1}$ proves the preservation of β -1,4-glycosidic bonds. In the AC2 sample, the intensity of the bands in this region decreases and the spectral shape changes, which may be due to the interaction of copper nanoparticles with the cellulose matrix (Fig. 2a).

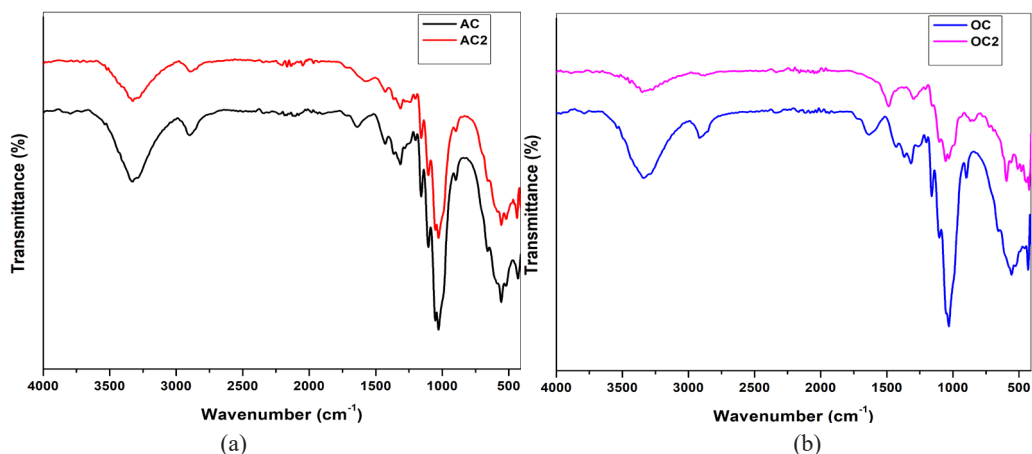


Figure 2 – IR spectra of nanocellulose membranes: (a) based on alfalfa; (b) based on oat straw.

The FTIR spectra of oat-based OC and OC2 samples are shown in Fig. 2b. In the OC sample, intense signals of the main functional groups characteristic of cellulose are observed. In the OC2 sample, the intensity of the absorption band characteristic of the O-H group in the 3200-3400 cm^{-1} region decreased after the introduction of graphene. This indicates that the graphene particles affected the intermolecular interactions between the cellulose chains. In addition, the change in the signals in the 1000-1100 cm^{-1} region indicates that structural reorganization occurs due to the binding of graphene to the cellulose structure.

Overall, the IR results showed that the hydrogen bonding system in the cellulose matrix changes upon the introduction of nanoparticles, and these changes lead to the reorganization of the material structure.

X-ray diffraction analysis of nanocellulose membranes. In order to assess the crystal structure and degree of crystallinity of nanocellulose membranes, X-ray diffraction analysis was performed. The X-ray diffraction results showed that there are differences in the structural features of the original and modified samples.

The X-ray diffraction patterns of the alfalfa-based AC and AC2 samples are shown in Figure 3a. In the AC sample, a clear intense peak is observed in the region of $2\theta \approx 22-23^\circ$, which corresponds to the (200) crystallographic plane characteristic of the cellulose I structure. In addition, a weak maximum is observed in the region of $2\theta \approx 15-16^\circ$, which characterizes the semi-crystalline structure of cellulose. In the AC2 sample, the intensity of the main diffraction peak is observed to decrease. This indicates a decrease in the structural order in the cellulose matrix as a result of the introduction of copper nanoparticles. In addition, the appearance of additional weak maxima in the range of $30-45^\circ$ proves that the copper nanoparticles contributed to the structural changes.

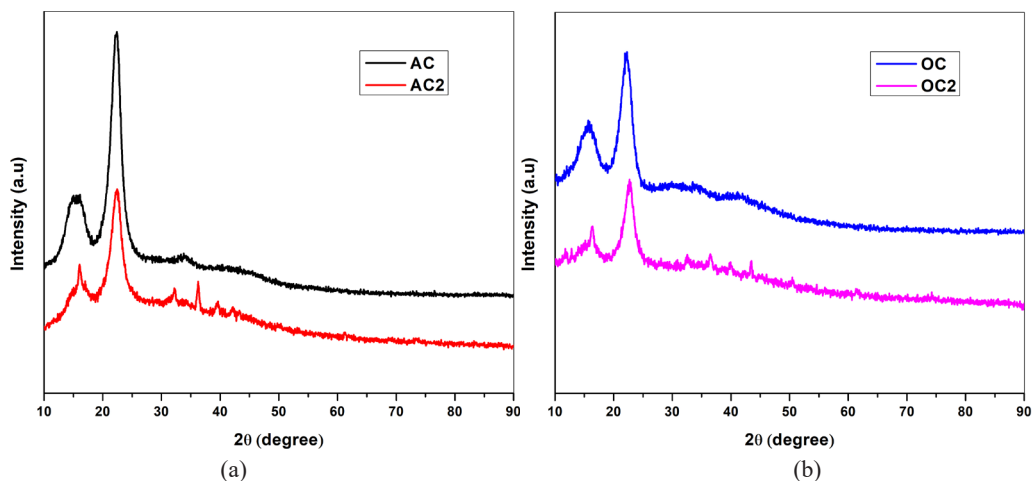


Figure 3 – X-ray diffraction patterns of nanocellulose membranes: (a) based on alfalfa; (b) based on oat straw.

The X-ray diffraction patterns of the OC and OC2 samples are shown in Fig. 3b. In the OC sample, an intense peak in the region of $2\theta \approx 22^\circ$ was clearly observed, indicating that the crystalline structure of cellulose was preserved. In the OC2 sample modified with graphene, the intensity of the main peak decreased, and some signs of amorphization of the structure were observed. In addition, the appearance of weak diffraction maxima in the range of $35\text{--}45^\circ$ may be due to the interaction of graphene with the cellulose matrix.

The results of the general X-ray phase analysis showed that graphene and copper nanoparticles affect the crystalline structure of nanocellulose membranes. As a result of nanomodification, a decrease in the degree of crystallinity and structural reorganization phenomena were observed. These changes are explained by the influence of nanoparticles on the intermolecular bonds between cellulose chains.

Discussion of the results. The conducted IR and X-ray diffraction studies showed that the structural organization of nanocellulose membranes obtained from agroresidues depends on the nature of the plant raw material and the type of nanomodifier introduced. The spectral and X-ray characteristics of the samples based on alfalfa and oats differed from each other, which may be due to their chemical composition and supramolecular organization. In addition, a comparative analysis of the obtained spectra and diffractograms revealed that the original cellulose structure was preserved, but the introduction of graphene and copper nanoparticles led to a change in intermolecular interactions and reorganization of crystalline regions.

In the IR spectra of all samples, a broad absorption band was observed in the range of $3200\text{--}3400\text{ cm}^{-1}$ (Fig. 2). This region is characteristic of the valence vibrations of hydroxyl groups and indicates the formation of hydrogen bonds in cellulose. However, the intensity of this band decreased in the AC2 and OC2 samples. This change indicates a reorganization of the interactions between cellulose chains after the introduction of nanoparticles. Similar phenomena were described in the work of Mokhena et al.,

where it was shown that the hydrogen bonds in the nanocellulose structure weaken and the internal organization of the matrix changes when graphene oxide is introduced (Mokhena et al, 2024).

In all samples, the signals observed around 2900 cm^{-1} correspond to the vibrations of C–H bonds. This indicates that the main polysaccharide backbone of cellulose was preserved during chemical treatment. In addition, the intense absorption bands in the range of $1000\text{--}1160\text{ cm}^{-1}$ were characteristic of C–O–C and C–O bonds. In particular, the maximum in the region of $1030\text{--}1050\text{ cm}^{-1}$ confirms the preservation of β -1,4-glycosidic bonds. However, in the AC2 and OC2 samples, the change in signals in this region indicates that structural reorganization is taking place due to the interaction of nanomodifiers with the cellulose matrix.

The clearer observation of signals characteristic of some functional groups in the oat-based OC sample compared to the alfalfa-based AC sample may be due to the higher degree of structural organization of oat fibers. The literature shows that the lignocellulosic composition of different agro-industrial wastes is not the same, and these differences affect the morphology and crystallinity of the obtained nanocellulose. Qureshi et al. noted that the properties of cellulose nanocrystals obtained from different plant residues depend on the amount of cellulose, hemicellulose and lignin in the raw material (Qureshi et al., 2024).

The X-ray diffraction results complemented the structural changes detected by IR analysis. A clear intense maximum was observed in the $2\theta \approx 22\text{--}23^\circ$ region in the diffractograms of AC and OC samples (Fig. 3). This peak is associated with the main crystallographic plane characteristic of the cellulose I structure. In addition, weak signals in the $15\text{--}16^\circ$ region indicated the semi-crystalline nature of the materials. Such results are consistent with the data given in the literature for natural cellulose. Das et al. showed that the XRD maxima of nanocellulose obtained from agro-residues are located in this range (Das et al., 2023).

In the nanomodified AC2 and OC2 samples, the intensity of the main diffraction peaks was observed to decrease. This phenomenon indicates that the crystalline regions were disrupted to some extent as a result of the introduction of graphene and copper nanoparticles. In other words, the nanoparticles affected the initial ordered arrangement of cellulose chains and increased the proportion of amorphous regions. A review article by Yusuf and colleagues in 2024 stated that similar structural changes were observed in nanocellulose-graphene hybrid composites and that graphene affects the internal organization of the matrix (Yusuf et al., 2024).

The observation of weak additional peaks in the $35\text{--}45^\circ$ range in the OC2 sample may be associated with the arrangement of graphene particles in the cellulose structure. Such results have also been described in studies of graphene-based nanocellulose composites. Yang and colleagues showed that additional weak peaks may appear in the X-ray diffraction spectrum as a result of the formation of new interfacial bonds of graphene in the cellulose matrix (Yang et al., 2023).

The structural changes in the AC2 sample are explained by the influence of copper nanoparticles. Copper particles can interact with hydroxyl groups and change the

intermolecular bonds between polymer chains. As a result, the degree of order of the crystal structure decreases and the intensity of diffraction maxima weakens. However, since only X-ray diffraction and IR methods were used in this work, it was not possible to fully determine the exact phase state of copper particles. In this regard, in the future, there is a need to use additional methods such as SEM, TEM and X-ray diffraction.

Analysis of the obtained results showed that nanomodification significantly affects the structural organization of nanocellulose membranes. Based on the X-ray diffraction and IR data, it was determined that the introduction of graphene and copper nanoparticles changes the intermolecular interactions in the cellulose matrix and the reorganization of the crystal structure. A decrease in the intensity of the main diffraction maxima in nanomodified samples indicates a change in the degree of structural order. In addition, the change in the bands characteristic of functional groups in the IR spectra confirms the interaction of nanoparticles with cellulose chains. The obtained results showed that the structural properties of nanocellulose membranes depend on the nature of the starting plant raw materials and the type of nanomodifier used. Overall, the obtained results prove that the structural properties of nanocellulose membranes obtained from agro-waste depend on the nature of the starting raw materials and the type of nanoparticles introduced. While the IR results indicate changes in the level of intermolecular bonds, the X-ray diffraction analysis confirmed that these changes affected the crystal structure. It was found that the introduction of graphene and copper nanoparticles allows to reorganize the structure of nanocellulose membranes and change their functional properties. The results obtained indicate that such materials have high prospects for use in sorption systems, filter membranes, and antibacterial coatings.

Conclusion. Thus, the structural features of nanocellulose membranes obtained from agro-waste were studied using X-ray diffraction and IR methods. As a result of the analyses, it was found that the main functional groups and crystal structure characteristic of cellulose were preserved in the obtained materials. It was also observed that the introduction of graphene and copper nanoparticles affected the structural organization of the membranes and led to a change in intermolecular interactions. The preservation of bands characteristic of hydroxyl groups and β -glycosidic bonds in the IR spectra confirmed the cellulose nature of the obtained membranes. The change in the intensity and shape of some absorption bands in the nanomodified samples indicated the interaction of nanoparticles with the cellulose matrix. The X-ray diffraction results revealed that the initial and modified samples have a semi-crystalline structure. The change in diffraction maxima after nanomodification was associated with the reorganization of the structure and a change in the degree of crystal ordering. As a result of the comparative analysis, it was found that the structural properties of nanocellulose membranes based on alfalfa and oats are not the same. This is explained by the chemical composition and fiber structure of the original plant raw materials. In addition, the effect of graphene and copper nanoparticles was observed to be of different nature, which indicated that it depends on the nature of the modifiers used. The results obtained prove that it is possible to obtain functional nanocellulose membranes based on agro-waste and that their structural properties can be changed through nanomodification.

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