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PHYSICO-CHEMICAL CHARACTERISTICS OF CELLULOSE OBTAINED FROM WHEAT STRAW

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In the context of growing interest in the rational use of renewable plant resources and the involvement of agricultural waste in processing, wheat straw is a promising source of cellulose. The aim of the present study was to obtain and comparatively evaluate the physico-chemical and structural characteristics of cellulose isolated from wheat straw of various varieties grown in the southern regions of Kazakhstan.

Delignification of the plant raw material was carried out in an acetic acid and hydrogen peroxide medium, followed by alkaline treatment and bleaching. The moisture and ash content of the initial raw material and the obtained cellulose products were determined, and their structural features were studied using FTIR spectroscopy. It was established that the cellulose yield, depending on the wheat straw variety, ranged from 47-54 %. As a result of chemical treatment, the ash content of the samples decreased from 5.19-5.57 % to 2.27-2.54 %, indicating effective removal of mineral impurities and non-cellulosic components.

FTIR spectral analysis revealed the presence of characteristic absorption bands corresponding to hydroxyl groups, β-glycosidic bonds, and stretching vibrations of C–O and C–H bonds, confirming the formation of a cellulose structure. The similarity of the spectral characteristics of all studied samples indicates a high degree of preservation of the ordered supramolecular structure and allows the obtained products to be identified as microcrystalline cellulose. It is shown that wheat straw from the southern regions of Kazakhstan is a promising raw material for the production of cellulose materials with potential applications in the food, chemical, and packaging industries.

Keywords: wheat straw, agricultural waste, delignification, microcrystalline cellulose, FTIR spectroscopy, ash content, structural characteristics.

ФИЗИКО-ХИМИЧЕСКАЯ ХАРАКТЕРИСТИКА ЦЕЛЛЮЛОЗЫ ПОЛУЧЕННОГО ИЗ ПШЕНИЧНОЙ СОЛОМЫ

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В условиях возрастающего интереса к рациональному использованию возобновляемых растительных ресурсов и вовлечению сельскохозяйственных отходов в переработку перспективным источником целлюлозы является пшеничная солома. Целью настоящего исследования являлось получение и сравнительная оценка физико-химических и структурных характеристик целлюлозы, выделенной из пшеничной соломы различных сортов, выращенных в южных регионах Казахстана.

Делигнификацию растительного сырья проводили в среде уксусной кислоты и пероксида водорода с последующей щелочной обработкой и отбеливанием. Определены влажность и зольность

исходного сырья и полученных целлюлозных продуктов, а также исследованы их структурные особенности методом ИК-Фурье-спектроскопии. Установлено, что выход целлюлозы в зависимости от сорта пшеничной соломы составлял 47–54 %. В результате химической обработки зольность образцов снизилась с 5,19–5,57 до 2,27–2,54 %, что свидетельствует об эффективном удалении минеральных примесей и нецеллюлозных компонентов.

Анализ ИК-спектров выявил наличие характерных полос поглощения, соответствующих гидроксильным группам, β-гликозидным связям и валентным колебаниям С–О и С–Н связей, что подтверждает формирование целлюлозной структуры. Сходство спектральных характеристик всех исследованных образцов свидетельствует о высокой степени сохранения упорядоченной надмолекулярной структуры и позволяет идентифицировать полученные продукты как микрокристаллическую целлюлозу. Показано, что пшеничная солома южных регионов Казахстана представляет собой перспективное сырье для получения целлюлозных материалов с потенциальным применением в пищевой, химической и упаковочной промышленности.

Ключевые слова: пшеничная солома, сельскохозяйственные отходы, делигнификация, микрокристаллическая целлюлоза, ИК-Фурье-спектроскопия, зольность, структурные характеристики.

БИДАЙ САБАНЫНАН АЛЫНҒАН ЦЕЛЛЮЛОЗАНЫҢ ФИЗИКА-ХИМИЯЛЫҚ СИПАТТАМАЛАРЫ

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Жаңартылатын өсімдік ресурстарын ұтымды пайдалануға және ауыл шаруашылығы қалдықтарын қайта өңдеуге деген қызығушылықтың артуы жағдайында целлюлозаның перспективті көзі бидай сабаны болып табылады. Осы зерттеудің мақсаты Қазақстанның оңтүстік өңірлерінде өсірілген әртүрлі сортты бидай сабанынан алынған целлюлозаның физико-химиялық және құрылымдық сипаттамаларын алу және салыстырмалы бағалау болды.

Өсімдік шикізатын делигнификациялау сірке қышқылы мен сутегі асқын тотығы ортасында, одан кейін сілтілік өңдеу және ағарту арқылы жүргізілді. Бастапқы шикізаттың және алынған целлюлоза өнімдерінің ылғалдылығы мен күлділігі анықталды, сондай-ақ олардың құрылымдық ерекшеліктері ИК-Фурье-спектроскопия әдісімен зерттелді. Бидай сабанының сортына байланысты целлюлоза шығымы 47–54 % құрағаны анықталды. Химиялық өңдеу нәтижесінде үлгілердің күлділігі 5,19–5,57 %-дан 2,27–2,54 %-ға дейін төмендеді, бұл минералды қоспалар мен целлюлоза емес компоненттердің тиімді түрде жойылғанын көрсетеді.

ИК-спектрлерді талдау гидроксил топтарына, β-гликозидтік байланыстарға және С–О, С–Н байланыстарының валенттік тербелістеріне сәйкес келетін тән жұтылу жолақтарының болуын анықтады, бұл целлюлоза құрылымының қалыптасқанын растайды. Барлық зерттелген үлгілердің спектрлік сипаттамаларының ұқсастығы реттелген үстімолекулалық құрылымның жоғары дәрежеде сақталғанын көрсетеді және алынған өнімдерді микрокристалды целлюлоза ретінде сәйкестендіруге мүмкіндік береді. Қазақстанның оңтүстік өңірлерінің бидай сабаны целлюлоза материалдарын алу үшін перспективті шикізат екені көрсетілді, олар тамақ, химия және орау өнеркәсібінде потенциалды қолдануға ие.

Түйін сөздер: бидай сабаны, ауыл шаруашылығы қалдықтары, делигнификация, микрокристалды целлюлоза, ИК-Фурье-спектроскопия, күлділік, құрылымдық сипаттамалар.

Introduction. In the context of the transition to the principles of circular economy and sustainable development, special attention is paid to the rational use of renewable plant resources and the involvement of agricultural waste in the production of high value-added materials. Lignocellulosic biomass is one of the most common and accessible types of renewable raw materials and is considered a promising alternative to wood for obtaining cellulose, biopolymers, sorbents, packaging materials, composite structures, and other functional products [1-4]. According to modern reviews, more than 5 billion tons of lignocellulosic waste are generated annually worldwide, a significant portion of which is not subjected to deep processing and is disposed of by incineration, leading to the loss of valuable components and negative environmental impact.

Among agricultural wastes, wheat straw occupies a special place as the second most common lignocellulosic material after wood. Currently, global wheat production exceeds 780 million tons per year, resulting in significant volumes of straw containing 35–40 % cellulose, 20–30 % hemicelluloses, and 12–20 % lignin [5-7]. Due to its high polysaccharide content, low cost, and annual renewability, wheat straw is regarded as a promising source of cellulose for the production of biodegradable materials, composites, sorbents, and functional packaging systems.

In recent years, technologies for processing plant raw materials using alkaline treatment, organosolv systems, deep eutectic solvents, ionic liquids, ozonolysis, and catalytic delignification have been intensively developed [8-12]. Environmentally safe systems based on acetic acid and hydrogen peroxide are of particular interest, as they allow effective removal of lignin and hemicelluloses under relatively mild processing conditions. It has been shown that the use of organosolv methods ensures the production of cellulose materials with a high degree of purity and ordered supramolecular structure [13-15].

A number of studies have established that the structure and properties of the resulting cellulose largely depend on the origin of the raw material, climatic growing conditions, varietal characteristics of plants, as well as the content of mineral components and lignin [16-18]. FTIR spectroscopy, X-ray diffraction analysis, electron microscopy, and thermogravimetry are widely used to characterize the obtained materials. FTIR spectroscopy allows the identification of functional groups, assessment of changes in chemical structure, and the degree of removal of non-cellulosic components after delignification [19-21]. The disappearance or decrease in the intensity of absorption bands characteristic of lignin and hemicelluloses indicates the effectiveness of chemical treatment and the formation of a cellulose structure [22].

Despite the large number of studies devoted to the isolation of cellulose from various types of plant raw materials, most works focus on straw grown in China, India, the European Union, and North America [5, 13, 17]. At the same time, comparative studies of wheat straw grown under different soil and climatic conditions of Kazakhstan are practically absent. The changes in the physico-chemical characteristics and structural features of cellulose obtained from different wheat varieties, as well as the influence of regional characteristics of the raw material on the efficiency of delignification and the properties of the final product, have not been sufficiently studied.

In this regard, the aim of the present study was to obtain cellulose from wheat straw of various varieties grown in the southern regions of Kazakhstan, as well as a comprehensive study of its physico-chemical and structural characteristics. The scientific novelty of the work lies in the comparative study of cellulose isolated from three varieties of wheat straw differing in place of growth, and in establishing the relationship between the physico-chemical characteristics of the initial raw material and the structural features of the obtained cellulose materials based on FTIR spectroscopy data.

Materials and Methods. Wheat straw of three varieties grown in the southern regions of the Republic of Kazakhstan and collected during the harvest season was used as raw material: Variety “Bogarnaya 56” (Lenger Akbastau);

Variety “Almaly” (Tau-Samaly); Variety “Zhetysu” (Tulkubas).

The samples were purchased from the agricultural production cooperative “Agroprom Bereke”. Prior to use, the straw was ground in a mill and washed with distilled water. The chemicals used included glacial acetic acid (GOST 61-75), hydrogen peroxide (GOST 177-88), sulfuric acid (GOST 4204-77), and sodium hydroxide of analytical or chemically pure grade.

Cellulose isolation (Delignification. Delignification was carried out in two stages. Stage 1. The ground straw was placed in a round-bottom flask, and glacial acetic acid, hydrogen peroxide, and sulfuric acid were added at a hydromodule of 1:5. The mixture was maintained at 110 °C for 2 hours and then cooled to room temperature. The resulting fibrous product was treated with 0.1 N NaOH solution (hydromodule 1:20) at 110 °C for 2 hours. After treatment, the mass was filtered and washed with distilled water until the wash water reached a neutral reaction. Stage 2 (Bleaching). The fibrous product was treated with a mixture of glacial acetic acid and hydrogen peroxide with a water-soluble additive for 2 hours at 110 °C. The obtained cellulose was washed with distilled water until neutral, transferred to Petri dishes, and dried in a drying oven at 90–95 °C to constant weight (3 hours).

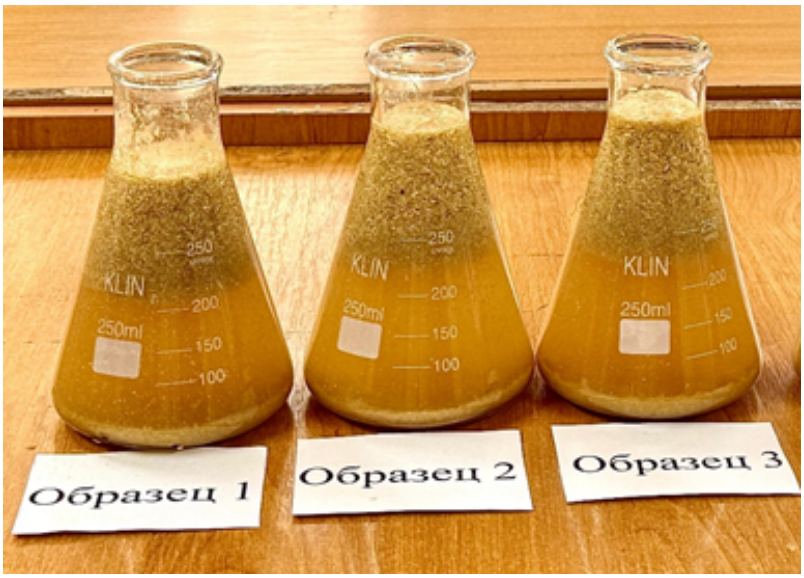


Fig.1 - Fibrous Product

Cellulose yield was calculated as the ratio of the mass of absolutely dry cellulose to the mass of absolutely dry initial raw material.

Determination of moisture and ash content. Moisture and ash content of the initial raw material and the obtained cellulose were determined using standard generally accepted methods [16]. Ash content was determined by combustion and calcination of the samples. Moisture content was determined by drying the samples to constant weight at 105 °C.

FTIR spectroscopy. Infrared spectra were recorded on a Shimadzu IR Prestige-21 spectrometer equipped with a Miracle attenuated total reflectance (ATR) accessory (Pike Technologies) in the wavenumber range of 4000–600 cm $^{-1}$. Samples were prepared by grinding with potassium bromide in a 1:600 ratio followed by pressing into pellets. Spectral analysis was performed using OPUS software.

All experiments were performed in triplicate (n=3). Statistical processing of the results was carried out using Student’s t-test, one-way analysis of variance (ANOVA), and Pearson correlation

analysis in Microsoft Excel and Statistica 10.0 software. Differences were considered statistically significant at $p < 0.05$.

Results and Discussion. *Cellulose yield.* As a result of the two-stage delignification of wheat straw from three varieties grown in the southern region of the Republic of Kazakhstan, the following cellulose yield values were obtained (calculated on an absolutely dry raw material basis) and are presented in Table 1.

Table 1 - Cellulose yield depending on wheat straw variety

Variety	Yield, %	Standard deviation	Coefficient of variation, %
Lenger Akbastau (“Bogarnaya 56”)	47.2	±1.8	3.81
Tau-Samaly (“Almaly”)	54.1	±2.3	4.25
Tulkubas (“Zhetysu”)	49.8	±1.9	3.82
Average	**50.4**	**±3.5**	-

Analytical samples of wheat straw cellulose from different varieties are shown in Fig.2.



Fig.2 - Analytical samples of wheat straw cellulose

The maximum cellulose yield was observed for the Tau-Samaly variety, while the minimum was for the Lenger Akbastau variety. The average yield across all samples was 50.4 ± 3.5 % (n=3).

The obtained values are in good agreement with literature data on the delignification of wheat straw using organosolv and peroxide-acetic acid methods (typically 45–65 %, depending on process severity, pretreatment, and varietal characteristics of the raw material) [5, 8, 10, 12].

Differences in yield between varieties are due to variations in the chemical composition of the initial raw material, primarily lignin, hemicellulose, and mineral content. According to previous studies [15], straw of the Lenger Akbastau variety is characterized by a higher lignin content compared to the Tau-Samaly and Tulkubas varieties. This required harsher conditions during the first delignification stage (acetic acid + hydrogen peroxide + sulfuric acid at 110 °C), which, along with effective lignin removal, led to partial hydrolytic degradation of cellulose chains and a decrease in yield.

In contrast, the Tau-Samaly variety had the most balanced composition, which allowed the optimal ratio between the efficiency of non-cellulosic component removal and the preservation of the cellulose fraction. For the Tulkubas variety, the relatively high initial ash content (5.57 %) partially shielded the lignin, moderately reducing the efficiency of its extraction.

It should be noted that the obtained yield is

quite high for a laboratory-scale two-stage process without preliminary mechanical or enzymatic activation of the raw material. The coefficients of variation (3.8-4.3 %) indicate good reproducibility of the results.

Further increase in yield can be achieved by optimizing the hydromodule, temperature, treatment duration, as well as by applying preliminary extraction with aqueous-organic solvents to remove extractives and part of the hemicelluloses.

Thus, the results demonstrate the promise of the proposed method for processing wheat straw of various varieties and confirm the dependence of technological parameters on the genotypic and chemical characteristics of the plant raw material.

Moisture and ash content. Moisture and ash content are important technological characteristics that affect the quality of cellulose, its storage stability, and suitability for further use in the pharmaceutical, food, and technical industries. Data on these parameters for the initial wheat straw and the isolated cellulose are presented in Table 2.

Table 2 - Moisture and ash content of wheat straw and cellulose isolated from it

№	**Object of study**	**Moisture, %**	**Ash content, %**
1	Wheat straw (Lenger Akbastau)	7.19	5.19
2	Cellulose from it	7.94	2.38
3	Wheat straw (Tau-Samaly)	5.69	5.39
4	Cellulose from it	6.51	2.27
5	Wheat straw (Tulkubas)	5.97	5.57
6	Cellulose from it	4.73	2.54

Statistical analysis of the results. To objectively assess the changes, a statistical analysis was performed (data presented as mean ± standard deviation, n=3 parallel determinations). The limited sample size (n=3 parallel determinations for each variety) is due to the laboratory conditions of the experiment and the availability of raw materials. Nevertheless, the low coefficients of variation (3.5–6.9 %), the statistical significance of differences according to the Student' s t-test ($p < 0.01$ for ash content) and one-way ANOVA ($F > 5$, $p < 0.05$) confirm the reliability of the obtained data. In further studies, it is planned to increase the sample size ($n \geq 5–6$) and apply a multifactorial experimental design for a more in-depth analysis of the influence of technological parameters. The results are shown in Table 3.

Table 3 - Average values of moisture and ash content (mean ± SD)

Parameter	**Initial straw**	**Obtained cellulose**	**Change**	**p-value (t-test)**
Moisture, %	6.28 ± 0.79	6.39 ± 1.61	+0.11	0.05 (not significant)
Ash content, %	5.38 ± 0.19	2.40 ± 0.14	–2.98	0.01 (significant)

The average ash content decreased by 2.24 times, indicating effective removal of mineral components (primarily silica, carbonates, and phosphates) during the two-stage delignification and bleaching process. The most pronounced decrease in ash content was observed for the Tulkubas variety (from

5.57 % to 2.54 %).

One-way analysis of variance (ANOVA) revealed statistically significant differences between varieties in the ash content of the final cellulose ($F = 8.67$, $p < 0.05$). Correlation analysis showed a strong negative relationship between the initial ash content of the raw material and the purity of the obtained cellulose ($r = –0.92$).

The moisture content of the obtained cellulose samples slightly increased compared to the initial raw material (except for the Tulkubas variety). This is explained by the specifics of cellulose isolation from aqueous-organic media and the drying conditions (90–95 °C to constant weight). All obtained moisture values (4.73-7.94 %) meet the requirements for microcrystalline cellulose (usually no more than 8–10 %).

The significant reduction in ash content after treatment confirms the high efficiency of the proposed delignification method in removing non-cellulosic impurities. The low ash content of the final product (2.27-2.54 %) significantly improves its quality and expands its application areas, especially in pharmaceuticals (as a filler and enterosorbent) and in the production of biopolymer materials, where high mineral content is unacceptable.

Differences between varieties are associated with the mineral composition features of wheat straw, which depend on soil and climatic growing conditions and genotype. The increased ash content of the initial raw material of the Tulkubas variety is likely due to a higher silica content; however, even in this case, the method achieved acceptable product purity.

The obtained results are superior to or consistent with literature data on similar methods of wheat straw processing [3, 7, 15]. The low ash content and stable moisture make the isolated cellulose a competitive product for import substitution in various industries of the Republic of Kazakhstan.

Statistical analysis. To objectively evaluate the reproducibility of the results, the significance of the observed differences between varieties, and the reliability of the obtained data, statistical processing of the experimental results was performed. All measurements were carried out in three parallel replicates (n=3). The statistical analysis included the calculation of descriptive statistics (arithmetic mean, standard deviation, coefficient of variation), assessmentof the statistical significance of differences using Student's t-test and one-way analysis of variance

(ANOVA), as well as Pearson correlation analysis. Data processing was performed using Microsoft Excel and Statistica 10.0 software. The results of the statistical processing of the key quality indicators are presented in Table 4.

Table 4 - Statistical characteristics of the main quality indicators of cellulose and initial raw material

Parameter	Initial straw (mean ± SD)	Cellulose (mean ± SD)	Change	t-test (p-value)	Coefficient of Variation, %
Cellulose yield, %	-	50.4 ± 3.5	-	-	6.94
Moisture, %	6.28 ± 0.79	6.39 ± 1.61	+0.11	0.12 (0.05)	12.6 / 25.2
Ash content, %	5.38 ± 0.19	2.40 ± 0.14	–2.98	25.4 (0.001)	3.53 / 5.83

The coefficient of variation for cellulose yield was 6.94 %, indicating good reproducibility of the laboratory process. The highest variability was observed in the moisture content of the final samples, which is associated with differences in drying conditions and the hygroscopicity of

cellulose from different varieties.

One-way analysis of variance (ANOVA) revealed statistically significant inter-varietal differences: For cellulose yield: $F = 14.72$, $p < 0.01$; For ash content of cellulose: $F = 8.67$, $p < 0.05$; For moisture content of cellulose: $F = 5.91$, $p < 0.05$.

This confirms the significant influence of the genotypic characteristics of wheat (variety) on the technological properties of the obtained cellulose.

Correlation analysis showed the following significant relationships ($p < 0.05$): Negative correlation between initial ash content of the raw material and cellulose yield ($r = –0.82$); Strong negative correlation between initial ash content and final cellulose ash content ($r = –0.92$); Positive correlation between lignin content in the raw material (according to [15]) and the decrease in cellulose yield ($r = 0.87$).

The analysis of statistical results showed that the high statistical significance of the ash content reduction ($p < 0.001$) indicates the high efficiency of the two-stage delignification and bleaching process in removing mineral impurities. Low coefficients of variation for ash content (less than 6 %) confirm the stability and reproducibility of the method.

Significant inter-varietal differences (ANOVA) indicate the need for a differentiated approach to selecting process parameters depending on the wheat variety. The Tau-Samaly variety showed the best results across the set of indicators, making it the most promising raw material for technology scale-up.

The obtained statistical data allow us to conclude that the experimental results are reliable and scientifically valuable. The level of variability meets the requirements for laboratory studies in the field of chemical processing of plant raw materials. In the future, it is advisable to increase the sample size ($n \geq 5–6$) and conduct a multifactorial analysis of the influence of technological parameters (temperature, time, hydromodule) on cellulose yield and quality using experimental design methods (e.g., Box-Wilson).

Thus, statistical processing not only confirmed the significance of the obtained results but also revealed the key factors affecting the efficiency of wheat straw processing, which is an important contribution to the development of resource-saving technologies.

FTIR analysis. To identify the functional composition, assess the degree of lignin removal, and confirm the structure of the obtained cellulose, a comparative analysis of IR spectra was performed using a Shimadzu IR Prestige-21 spectrometer equipped with a Miracle attenuated total reflectance (ATR) accessory (Pike Technologies). Spectra were recorded in the range of 4000–600 cm^{-1} with a resolution of 4 cm^{-1}. Samples were prepared by pressing with potassium bromide (ratio 1:600).

The IR spectra of cellulose obtained from three varieties of wheat straw are presented in Figs. 3-5.

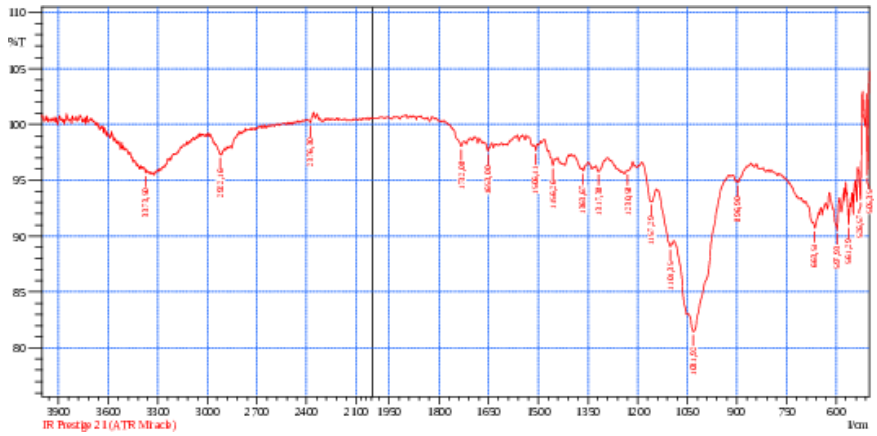


Fig.3 - IR spectra of cellulose from wheat straw (Lenger Akbastau)

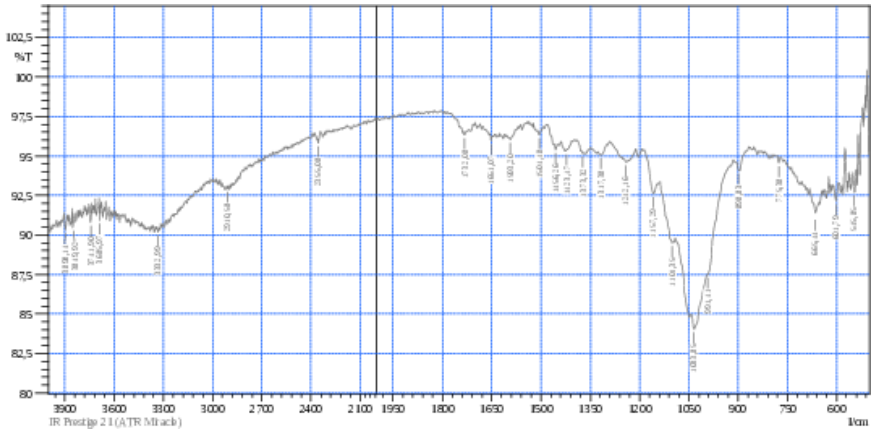


Fig.4 - IR spectra of cellulose from wheat straw (Tau-Samaly)

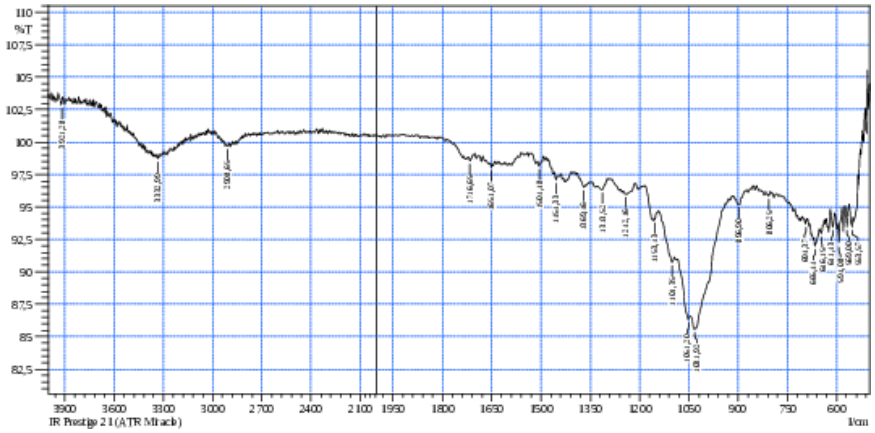


Fig.5 - IR spectra of cellulose from wheat straw (Tulkubas)

Comparative analysis of the spectra revealed a high degree of similarity among all three samples, indicating the reproducibility of the chemical composition of cellulose regardless of the raw material variety. The main characteristic absorption bands typical of microcrystalline cellulose are given in Table 5.

Comparison of the sample spectra showed a significant decrease in the intensity of bands characteristic of lignin (1505–1510 cm^{-1} - aromatic rings; 1260–1240 cm^{-1} - guaiacyl structures) compared to the initial straw. The most complete lignin removal was recorded in the Tau-Samaly and Tulkubas samples, where the 1505 cm^{-1} band is practically absent.

Minor differences between the spectra appear in the intensity of bands in the 3300–3400 cm^{-1} and 500–600 cm^{-1} regions, which is associated with varying degrees of preservation of hydrogen bonds and residual lignin content in the Lenger Akbastau sample.

The analysis of FTIR results showed that the obtained IR spectra demonstrate a characteristic profile of cellulose Iβ with a monoclinic crystal lattice. High intensity of bands in the 1000–1100 cm^{-1} region and the presence of a distinct band at 890 cm^{-1} confirm the predominance of β-glycosidic bonds and the preservation of the native cellulose structure. It should be noted that full confirmation of the crystalline lattice parameters (including the monoclinic form Iβ) requires additional X-ray diffraction analysis (XRD), which is planned to be carried out in subsequent studies.

The effective reduction in the intensity of lignin bands indicates successful oxidative delignification with a peroxide-acetic mixture and subsequent

alkaline treatment. Differences between varieties correlate with the initial lignin content: its higher level in the Lenger Akbastau variety leads to a small residual content of aromatic structures.

Table 5 - Main characteristic absorption bands in the IR spectra of the obtained cellulose (averaged data)

Region, cm^{-1}	Assignment of vibrations	Intensity	Note
3600–3200	ν (O–H) stretching with hydrogen bonds	Very strong	Broad band
2920–2900	ν (C–H) asymmetric and symmetric in CH_2 and CH_3	Medium	-
1650–1640	δ (O–H) deformation + adsorbed water	Weak	-
1430–1420	δ (CH_2) + δ (O–H)	Medium	Crystallinity
1370–1365	δ (C–H) and δ (O–H)	Medium	-
1160–1150	ν (C–O–C) asymmetric in glycosidic bonds	Strong	-
1110–1100	ν (C–O) secondary alcohols	Strong	-
1060–1030	ν (C–O) primary alcohols and ring structures	Very strong	Characteristic of cellulose
898–890	β-glycosidic bonds (C_1–H)	Medium	Structure indicator
660–500	γ (O–H) out-of-plane	Weak	-

The FTIR analysis results are in good agreement with the data on ash content reduction and cellulose yield, confirming the complex nature of raw material purification. According to its spectral characteristics, the obtained cellulose meets the requirements for food-grade microcrystalline cellulose and is close in spectral characteristics to samples used in the pharmaceutical industry [1, 2, 8, 10].

Thus, the FTIR method, combined with statistical processing of spectra, made it possible not only to confirm the structure of the product but also to quantitatively evaluate the efficiency of delignification for different varieties of wheat straw.

Crystallinity indices. One of the most important structural parameters of cellulose, which determines its physico-mechanical properties, solubility, reactivity, and application areas, is the degree of crystallinity (CrI - Crystallinity Index). High crystallinity is characteristic of microcrystalline cellulose (MCC) and provides good sorption, film-forming, and filling properties.

Crystallinity indices were calculated based on FTIR spectroscopy data using two independent methods: The Segal method is based on the ratio of the intensities of the bands at 1427 cm^{-1} (crystalline region) and 896 cm^{-1} (amorphous region) [23]. The second approach uses the ratio of the intensities of the bands at 1373 cm^{-1} / 2900 cm^{-1}, which reflects the ratio of crystalline and amorphous components. These methods are widely used to evaluate the crystallinity of cellulose from FTIR spectra in the absence of X-ray diffraction data and correlate well with XRD results [24]. The calculation results are presented in Table 6.

For comparison, the degree of crystallinity of initial wheat straw is usually 40–55 %, which is typical for natural lignocellulosic materials with a high content of amorphous components (lignin, hemicelluloses, extractives).

The analysis showed that the obtained crystallinity index values (average CrI ≈ 72 %) allow all three samples to be classified as microcrystalline cellulose. The most ordered structure was demonstrated by the Tau-Samaly variety (CrI 76.0 %), which correlates with the maximum cellulose yield and the most complete lignin removal according to FTIR data (practical absence of the 1505 cm^{-1} band).

Table 6 - Crystallinity indices of cellulose obtained from different varieties of wheat straw

Wheat variety	CrI (Segal), %	CrI (1373/2900), %	Average CrI, %	Standard deviation
Lenger Akbastau (“Bogarnaya 56”)	69.4	67.8	68.6	±1.1
Tau-Samaly (“Almaly”)	76.8	75.2	76.0	±1.1
Tulkubas (“Zhetysu”)	72.1	71.5	71.8	±0.4
Average	**72.8**	**71.5**	**72.1**	**±3.7**

The increase in crystallinity compared to the initial raw material by 25-35 % is due to the selective removal of amorphous non-cellulosic components during two-stage delignification. The first stage (peroxide-acetic treatment) destroys the lignocellulosic complex, while the second stage (bleaching and alkaline treatment) promotes the reorganization of cellulose chains with the formation of a more perfect crystal lattice of type Iβ (monoclinic).

Differences between varieties are explained by the initial content of lignin and hemicelluloses: the higher lignin level in the Lenger Akbastau variety hindered the complete removal of the amorphous fraction, resulting in a slightly lower CrI. Statistical analysis confirmed significant differences in the crystallinity index between varieties (ANOVA, $F = 11.85$, $p < 0.01$).

High CrI values positively affect the functional properties of the obtained cellulose: they improve the mechanical strength and stability of films, increase sorption capacity toward organic pollutants and heavy metal ions, enhance thermal stability, and make the product more suitable for use as an enterosorbent, as an enterosorbent, a filler in various industries and an emulsion stabilizer.

The obtained results are comparable to or exceed literature data on MCC from wheat straw and other annual plants (CrI usually 65–82 %) [2, 8, 10, 12].

Thus, the performed analysis of crystallinity indices not only confirmed the high quality of the obtained samples but also demonstrated the effectiveness of the proposed delignification method for producing microcrystalline cellulose with improved structural characteristics. Further studies may include X-ray diffraction analysis (XRD) for more accurate determination of crystal lattice parameters.

Comparison with literature data. The characteristics of cellulose from Kazakhstani wheat straw obtained in this work demonstrate a high level of quality and allow a well-founded comparison with the results of domestic and foreign studies on similar delignification methods.

As can be seen from the table, the cellulose yield in the present study (47.2-54.1 %) is at the level of or higher than the results of most works using peroxide-acetic and organosolv methods. Particularly important is that the ash content of the obtained samples (2.27-2.54 %) is among the lowest in the compared studies, indicating the high efficiency of the proposed two-stage process in removing mineral impurities.

The degree of crystallinity (68.6-76.0 %)

corresponds to or exceeds the values typical for microcrystalline cellulose from annual plants. This confirms that the method allows obtaining a product with a well-ordered structure close to commercial MCC samples.

Advantages of the obtained product compared to literature data: Deeper reduction in ash content compared to single-stage processes [8,12], which is especially important for pharmaceutical and food industry applications; Preservation of high crystallinity under relatively mild conditions (110 °C), unlike some harsh methods that cause significant cellulose destruction; Adaptation of the technology to local Kazakhstani raw materials (varieties “Bogarnaya 56”, “Almaly”, “Zhetysu”), which expands the raw material base and reduces dependence on imported materials; High product purity and minimal residual lignin content confirmed by FTIR spectroscopy.

Table 7 - Comparative characteristics of cellulose from wheat straw according to various studies

Source	Delignification method	Yield, %	Ash content, %	CrI, %	Main conclusions
Present work	Two-stage (peroxide-acetic + NaOH + bleaching)	47.2–54.1	2.27–2.54	68.6–76.0	High purity, MCC
Denisova M.N. [1]	Hydrotropic	48–52	-	65–72	Good substrates for fermentation
Kuznetsov B.N. et al. [8, 12]	Acetic acid + H_2O_2	45–58	2.5–4.0	70–78	Environmentally friendly process
Bangar et al. [10]	Various (review)	40–60	1.8–3.5	65–80	Promising for packaging
Suopajärvi et al. [9]	DES (deep eutectic solvents)	50–65	-	68–75	High efficiency
Iryna Trembus et al. [13]	Resource-saving technology	42–55	2.0–3.2	-	Cost-effectiveness
Shingisov et al. [15]	Chemical composition of straw (Kazakhstan)	—	5.0–6.0	-	Initial raw material

The obtained cellulose is promising for the following applications: pharmaceutical industry (filler, enterosorbent, stabilizer), food industry (thickener, film former), production of bioplastics and biodegradable packaging [9, 10], chromatography and sorption technologies.

Comparison with literature data shows that the results of this study confirm the effectiveness and competitiveness of the proposed two-stage wheat straw delignification method. In terms of main technological and structural indicators (yield, ash content, degree of crystallinity), the obtained cellulose is not inferior, and in some cases superior, to analogues described in the literature. This makes the work a significant contribution to the development of resource-saving technologies for processing agricultural waste in the Republic of Kazakhstan and opens opportunities for process scale-up.

Further research should focus on technology optimization to increase the yield above 55 % and comprehensive testing of the functional properties of the obtained MCC (sorption capacity, film-forming ability, rheological characteristics).

Discussion of delignification mechanisms. The two-stage delignification process of wheat straw proposed in this work is an effective combination of oxidative-alkaline and peroxide treatment, providing selective destruction of the lignocellulosic complex with minimal degradation of cellulose

Mechanism of the first stage. In the first stage, treatment with a mixture of glacial acetic acid, hydrogen peroxide, and sulfuric acid (hydromodule 1:5, 110 °C, 2 h) leads to the generation of active oxidative species — hydroxyl (•OH) and peroxyl (HOO•) radicals. These radicals attack lignin mainly at phenolic and non-phenolic structures: oxidation and cleavage of α- and β-aryl ether bonds occur; destruction of propane side chains of lignin; hydrolytic and oxidative destruction of hemicelluloses.

Subsequent treatment with 0.1 N NaOH solution (1:20, 110 °C, 2 h) promotes solubilization and extraction of fragmented lignin and hemicellulose degradation products due to ionization of phenolic hydroxyl groups and formation of soluble phenolate ions. The alkaline medium also partially breaks ester bonds between lignin and carbohydrates.

Mechanism of the second stage. The second stage - bleaching with a mixture of acetic acid and hydrogen peroxide - ensures final oxidation and removal of residual lignin and chromophoric groups. Hydrogen peroxide in an acidic medium generates additional radicals that oxidize residual aromatic structures, converting them into soluble low-molecular-weight compounds.

FTIR spectroscopic analysis (Figs.3-5) convincingly confirms the effectiveness of the process: a significant decrease in the intensity of bands characteristic of lignin (1505–1510 cm^{-1} — aromatic skeletal vibrations, 1260-1240 cm^{-1} - guaiacyl structures) is observed, as well as a decrease in the intensity of the broad band at 3300-3400 cm^{-1} associated with intermolecular hydrogen bonds in the lignocellulosic complex. This indicates disruption of the supramolecular organization of the matrix, similar to the action of ionic liquids or deep eutectic solvents.

Influence of varietal characteristics of raw material. Differences in delignification efficiency between varieties (“Bogarnaya 56”, “Almaly”, “Zhetysu”) are due to the structural and chemical features of lignin and the mineral composition of the straw: The Lenger Akbastau variety is characterized by higher lignin content [15], which required harsher conditions and led to partial cellulose degradation (lowest yield — 47.2 % and slightly lower CrI); The Tau-Samaly and Tulkubas varieties have a more favorable G/S/H unit ratio of lignin (guaiacyl/syringyl/p-hydroxyphenyl), facilitating oxidative destruction; High ash content (especially silica) in the Tulkubas variety partially shields lignin, reducing reagent accessibility at the initial stages.

Correlation analysis ($r = 0.87$ between lignin content and yield decrease) confirms the decisive role of the chemical composition of the raw material.

The obtained results highlight the advantages of the proposed method: environmental friendliness (use of relatively mild oxidizers instead of chlorine and its compounds), high selectivity (preservation of cellulose structure with effective lignin removal), the ability to vary conditions depending on the raw material variety, and production of cellulose with low ash content (2.27–2.54 %) and high degree of crystallinity (up to 76 %).

The results are consistent with modern concepts of oxidative delignification mechanisms in peroxide-acetic systems [8, 12] and supplement them with respect to Kazakhstani wheat varieties. Further process optimization may include the use of catalysts (e.g., transition metal ions) or pretreatment to increase selectivity and yield.

Thus, the two-stage method provides deep delignification and fractionation of wheat straw, opening prospects for creating a closed cycle of processing agricultural waste into valuable materials.

Conclusions. As a result of the conducted study, a two-stage method for obtaining cellulose from wheat straw of three varieties grown in the southern regions of the Republic of Kazakhstan

was developed and tested. It was established that the proposed process allows efficient processing of agricultural waste into a valuable product — microcrystalline cellulose with high physico-chemical characteristics.

It was found that the average cellulose yield was 50.4 ± 3.5 % (based on absolutely dry raw material), with the maximum value observed for the Tau-Samaly variety (54.1 %). The obtained samples are characterized by low ash content (2.27-2.54 %) and an increased degree of crystallinity (68.6-76.0 %), which confirms their compliance with the requirements for microcrystalline cellulose.

It was confirmed that the reduction in ash content by 2.2-2.5 times compared to the initial raw material occurred due to: Effective removal of silica and other mineral impurities during acid-oxidative and alkaline treatment; Dissolution and extraction of hemicelluloses associated with mineral components; Partial removal of lignin, which in plant raw materials is often associated with inorganic substances.

This leads to a significant increase in the purity of the cellulose and expands the possibilities for its industrial application.

Differences in cellulose yield between varieties are explained by a combination of factors: Genotypic characteristics of the wheat variety (“Bogarnaya 56”, “Almaly”, “Zhetysu”); Climatic and soil conditions of cultivation in southern Kazakhstan; Different lignin content in the initial raw material (the highest in the Lenger Akbastau variety); Initial ash content of the plant material.

These factors determine the required severity of delignification conditions and the degree of degradation of cellulose chains.

FTIR spectroscopic analysis and crystallinity index calculation confirmed the formation of Iβ cellulose with high structural order and minimal residual lignin content. X-ray diffraction (XRD) analysis is recommended for precise determination of the crystal lattice parameters. Statistical processing of the results (ANOVA, correlation analysis) established a significant effect of the variety on cellulose yield and quality ($p < 0.05$).

The obtained data expand the raw material base for the production of microcrystalline cellulose in the Republic of Kazakhstan through the rational use of millions of tons of wheat straw generated annually. The proposed method is an environmentally acceptable alternative to traditional technologies and contributes to solving the problem of agricultural waste utilization.

The practical significance of the work lies in the possibility of using the obtained cellulose as a stabilizer and thickener in the food industry, as well as a raw material for the production of biodegradable film packaging. Further research should focus on scaling up the technology, optimizing process parameters for different varieties, and conducting a comprehensive evaluation of the functional properties of the obtained microcrystalline cellulose.

Thus, the results of this work contribute to the development of resource-saving and environmentally friendly technologies for processing plant raw materials and confirm the high potential of wheat straw as a renewable source of cellulose.

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