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NANOCELLULOSE FROM REED STALKS TO IMPROVE THE PROPERTIES OF PAPER FOR PACKAGING FOOD PRODUCTS

Background. The development of technologies for obtaining materials from plant raw materials, the use of which improves the consumer properties of cardboard and paper products and does not pollute the environment with harmful substances from synthetic polymers, is an urgent problem of our time.

Objective. The purpose of the paper is to obtain pulp and nanocellulose from reed stalks by environmentally friendly methods and apply nanocellulose to improve the quality parameters of paper for packaging food products on automatic machines.

Methods. To obtain pulp from reed stalks with a minimum residual content of lignin and minerals, two processing stages were used: alkaline extraction and organosolv cooking at a temperature of 97 ± 2 °C. Nanocellulose was obtained by the oxidation of organosolv reed pulp with 2,2,6,6-tetramethylpiperidine-1-oxyl (TEMPO) in the TEMPO / NaBr / NaClO system, which is more environmentally friendly than acid hydrolysis. The resulting nanocellulose was applied to paper samples from waste paper and sulphate unbleached pulp at a consumption from 1 to 3 g/m².

Results. Organosolv pulp was obtained from reed stalks with a residual lignin content of 0.53 % and an ash content of 0.045 %, which was used to obtain nanocellulose. The resulting nanocellulose particles have a transverse size in the range of 5–20 nm, a length of up to several micrometers, and the tensile strength of nanocellulose films is up to 60 MPa. It is shown that the application of nanocellulose to the surface of the samples increases the breaking strength and breaking length, and reduces the surface absorbency of paper. It was determined that with a nanocellulose consumption of up to 3 g/m², paper samples have indicators that meet the requirements of the standard for paper for packaging food products on automatic machines.

Conclusions. The use of nanocellulose from reed stalks as a hardening substance for paper production will allow replacing environmentally harmful polymer additives and up to 50 % of more expensive softwood pulp with waste paper, while maintaining paper quality indicators at the level of standard requirements.

Keywords: reed stalk; organosolv pulp; TEMPO-mediated oxidation; nanocellulose; paper.

Introduction

In connection with the deteriorating environmental situation in recent years, there is a growing interest in the production of materials from biodegradable raw materials, which are renewed annually and are not inferior to products from oil, gas and coal. Polymers from these fossils take hundreds of years to decompose, causing irreparable damage to the environment. Plastic accounts for 85 % of all waste in the world's oceans, half of which are disposable plastic products [1]. The European Parliament Directive of 5 June 2019 on the reduction of the environmental impact of certain plastic products prohibits single-use plastic products and provides for their substitution with natural polymers [2]. The use of natural polymers from cellulosic plant materials is being seen as an alternative to plastics and could be a viable approach to reducing deforestation, increasing the use of agricultural surplus and developing biodegradable materials [3]. The processing products of such renewable plant

materials are widely used in the pulp, paper, chemical, textile, pharmaceutical, medicine and electronic industries [4, 5]. New technologies for processing plant raw materials into consumer goods contribute to the sustainable development of society, solving environmental and economic problems.

The main component of plant materials is cellulose, which is the most abundant renewable biopolymer in nature. Cellulose has attracted considerable interest as a raw material for the production of nanocellulose. Nanocellulose (NC) belongs to a group of nanomaterials consisting of the nanosized cellulose particles. The NC exhibits unique properties, such as high elastic modulus, high specific surface area, optical transparency, low thermal expansion coefficient, and chemical reactivity. NC has high transparency, biodegradability and biocompatibility, a low lightweight and production cost in comparison with synthetic polymers [5–7].

Nanocellulose is produced using the following methods: mechanical processing, chemicals and enzymatic hydrolysis, or a combination of both. Among

chemical methods, in addition to the production of nanocellulose with the use of mineral acids, ecologically safe substances such as phthalimide-N-oxyl (PINO) and 2,2,6,6-tetramethylpiperidine-1-oxyl (TEMPO) have recently been actively developed [8, 9]. The literature provides results on the production of nanocellulose with these reagents, mainly from wood cellulose [10, 11].

The main raw material for cellulose production in the world pulp and paper industry is wood. For countries that do not have large reserves of free wood, alternative sources of fibrous raw materials may be non-wood plant raw materials – annual and perennial plants and fibrous waste from agricultural production. Common reed (*Phragmites australis*) also belongs to such raw materials. Common reed is a cheap and common plant that grows on the banks of rivers and lakes. Common reed has the ability to form monospecific stands occupying large areas. Only in Ukraine, common reed harvesting averages 3200 tons per year and tends to grow. Reed stalks can be used for the production of pulp, paper and board, but there is no data on the production of nanocellulose by TEMPO-mediated oxidation and the use of this reed nanocellulose in papermaking.

Problem statement

The aim of the study was to obtain and use nanocellulose from reed stalks to improve the quality parameters of one of the mass types of paper – paper for packaging food products.

Methods for obtaining pulp, nanocellulose and paper

We used the biomass of common reed from the Cherkasy region of Ukraine after the harvest in 2019. Before research, the raw material was crushed to 2–5 mm and stored in a desiccator to maintain a constant moisture content and chemical composition. The chemical composition of reed stalks was determined according to TAPPI standards [12]. The analyses for the chemical characterization were done in triplicate and the mean and standard deviation were calculated. Sodium hydroxide, glacial acetic acid, hydrogen peroxide, ethanol, 2,2,6,6-tetramethylpiperidine-1-oxyl (TEMPO), sodium bromide, and sodium hypochlorite were chemical grade.

The pulp was obtained from reed stalks in two stages. At the first stage, an alkali solution was used as a preliminary treatment of plant raw materials to remove the main part of hemicelluloses and minerals and partially remove lignin. For this, the

reed stalks were extracted with a NaOH solution with a concentration of 50 g/l at a temperature of 97 ± 2 °C for 180 min, the ratio of liquid-to-solid is 10 : 1. At the second stage, to remove residual lignin and extractives, organosolv cooking was carried out using a solution of glacial acetic acid and 35 % hydrogen peroxide in a volume ratio of 7 : 3 with a liquid-to-solid ratio of 10 : 1 at a temperature of 97 ± 2 °C for 120 min. These values of technological parameters were previously determined for the extraction of different representatives of non-wood plant raw materials [13].

The TEMPO-oxidized nanocellulose (TONC) has been prepared using the TEMPO/NaBr/NaOCl system followed by ultrasonic treatment. This treatment leads to the selective removal of the amorphous regions of cellulose while maintaining the crystalline regions of cellulose macromolecules [14]. The method of preparing of the TONC was described in [15]. The optimal conditions of preparing of the reed TONC were consumption of TEMPO was of 1.6 % by weight of ORP, the duration of the oxidation was 24 hours. Ultrasonic treatment of a suspension of nanocellulose oxidized with TEMPO was carried out for 10–30 min until a transparent nanocellulose gel was formed. The resulting nanocellulose suspension was used for papermaking.

For the production of samples of packaging paper used waste paper brand MS-4A and sulfate unbleached pulp from softwood. Paper samples were made weighing 83 ± 1 g/m² and 110 ± 2 g/m² with the addition to the mass and without the addition of starch glue “Callpress” (SG) and alkyl ketene dimer (ACD) with a consumption of 1.5 % by weight of pulp. Waste paper and pulp were ground to a size of 2–5 cm, soaked for 1 hour in cold water, dissolved for 20 min in a water breaker, and then another 10–22 min in a mill to ensure the homogeneity of the suspension from the degree of grinding 30 °SR (Shopper-Riegler). Paper sheets were made on a Rapid-Köthen sheet-forming machine according to the standard technique. After drying, the suspension of reed nanocellulose was evenly applied to the manufactured castings with a consumption of 1, 2, and 3 g/m². In the obtained samples were determined quality indicators in accordance with GOST 7247-90 “Paper for food packaging on machines” [16].

Results and discussion

Chemical analysis of plant materials showed that reed stalks had the following content of components: 49.3 % of cellulose, 22.9 % of lignin; hot water solubility – 10.6 %; 1 % NaOH solubility – 19.4 %;

2.3% of resin, fats, and waxes; 3.5% of mineral substances (ash) relative to the mass of dry raw material. Thus, the studied non-wood plant material contains less lignin than wood of coniferous and deciduous species, which a priori suggests a lower consumption of reagents and a shorter duration of the process of its delignification in comparison with obtaining pulp from wood. The reed pulp after pre-treatment with alkali for 180 min contains 12.2% of lignin and ash content of 0.87% by weight of dry raw materials. After the first stage of thermochemical processing of plant raw materials, the resulting reed pulp contains a significant content of lignin and mineral substances (ash), which do not allow obtaining nanocellulose from it. Therefore, the second stage was carried out – organosolv cooking – as a result of which reed pulp was obtained, suitable for chemical processing, in particular for obtaining nanocellulose from it. The pulp after peracetic cooking for 120 minutes has a lignin of 0.53% and an ash content of 0.045% by weight of the pulp. This organosolv reed pulp (ORP) has been used to produce nanocellulose.

A suspension of TEMPO-oxidized nanocellulose (TONC) was extracted from ORP using the TEMPO / NaBr / NaClO oxidation system followed by sonication. It is shown that an increase in the duration of the oxidation process promotes the production of TONC films with a higher density and a high content of carboxyl groups, as well as an increase in their transparency and mechanical strength [15]. An increase in the mechanical parameters of TONC is associated with a decrease in the size of cellulose macromolecules in the process of TEMPO-mediated oxidation with their transformation into nanoparticles, which form denser structures with stronger bonds between them. The nanosize of cellulose particles is confirmed by AFM and TEM data. The transverse particle size of nanocellulose is in the range of 5–20 nm, but individual nanofibers have a width of up to 28 nm and a length of several micrometres. The tensile strength of nanocellulose films is up to 60 MPa [15].

To study the effect of reed nanocellulose on the physical and mechanical properties of paper for food packaging, a suspension of reed nanocellulose was applied to laboratory paper samples. The composition of the laboratory samples and the values of the paper weight 83 g/m² and 110 g/m² are given in Tables 1 and 2, respectively, and in Figs. 1 and 2.

Table 1. Physico-mechanical properties of paper weight 83 g/m² of different composition

№	Composition	Breaking force, N	Degree of sizing, mm	Surface absorbency, Cobb ₃₀ , g
1	Waste Paper (WP)	29.5	1.0	176.2
2	WP + ACD*	30.2	2.0	48.2
3	WP + SG**	31.3	2.0	52.3
4	WP + ACD + SG	31.8	2.0	41.5
5	WP + NC 1 g/m ²	33.4	2.0	75.9
6	WP + NC 2 g/m ²	38.7	2.0	71.8
7	WP + NC 3 g/m ²	49.2	2.0	46.3
8	Requirements of the standard [16] for brand E-II	No less 30	No less 1.0	No more 50

*ACD – alkyl ketene dimer; **SG – starch glue

As can be seen from Table 1, the values of the breaking force for paper samples weight 83 g/m² without the addition of sizing agents to the composition and the surface coating are within the requirements of the standard [16]. The addition of sizing agents to the fibrous composition slightly increases the value of this indicator, but the addition of suspension of reed nanocellulose on the paper surface leads to an excess of the breaking force by 11–64%. The application of nanocellulose to the surface of the paper also leads to a natural increase in the breaking length of the paper samples (Fig. 1) due to the formation of additional hydrogen bonds between the cellulose fibers. The same dependence of an increase in physical and mechanical parameters with an increase in the consumption of nanocellulose is observed for laboratory samples of paper weight 110 g/m² (Table 2 and Fig. 2).

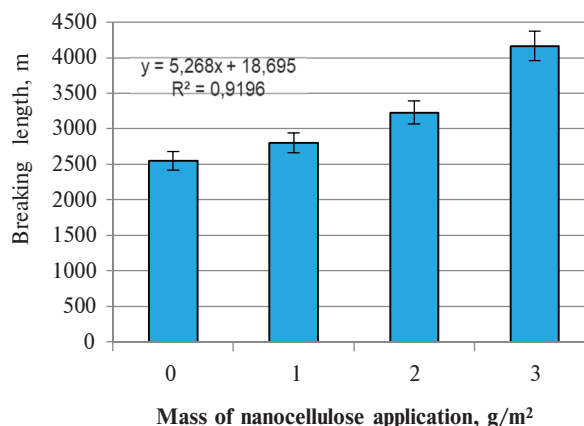


Fig. 1. The effect of reed nanocellulose consumption on the breaking length of paper samples weight 83 g/m²

Table 2. Physico-mechanical properties of paper weight 110 g/m² of different composition

№	Composition	Breaking force, N	Degree of sizing, mm	Surface absorbency, Cobb ₆₀ , g
1	Waste Paper (WP)	76.4	1.0	202.7
2	Sulfate pulp (SP)	80.4	1.0	144.3
3	50WP:50SP	78.4	1.0	169.5
4	50WP:50SP + ACD	71.5	2.0	31.8
5	50WP:50SP + SG	76.4	2.0	21.7
6	50WP:50SP + SG + ACD	77.4	2.0	18.3
7	50WP:50SP + SG + NC 1 g/m ²	91.1	2.0	20.2
8	50WP:50SP + SG + NC 2 g/m ²	92.1	2.0	18.4
9	50WP:50SP + SG + NC 3g/m ²	99.9	2.0	14.8
10	Requirements of the standard [16] for brand D	No less 39	No less 2.0	No more 40

From the data in Tables 1 and 2 it is seen that the values of the degree of sizing, which is determined by the dashed method, for paper meet the requirements of the standard for samples weight 83 g/m² with the addition and without addition to the composition of the paper modified starch glue or ACD. And for samples of paper weight 110 g/m² the values of this indicator meet the requirements of the standard only after the introduction into the fibrous composition of the above sizing agents (Table 2).

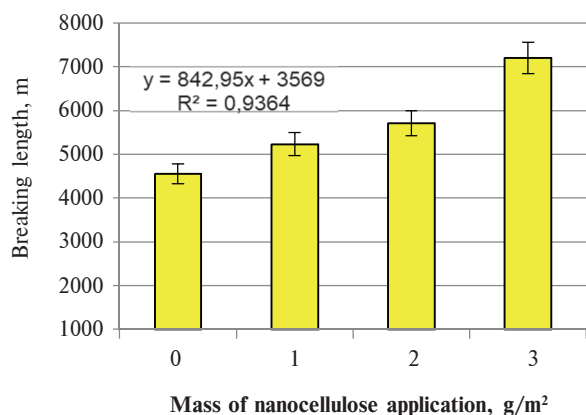


Fig. 2. The effect of reed nanocellulose consumption on the breaking length of paper samples weight 110 g/m²

One of the important indicators of paper for packaging food products is the surface absorbency of paper with a single-sided wetting area of 1 m² (Cobb₃₀ or Cobb₆₀ depending on the weight of the paper). As can be seen from the data in Table 1, the addition to the fibrous composition of modified starch paper and ACD at consumption of 1.5 % by weight of pulp or on its surface 3 g/m² of reed nanocellulose allows obtaining paper samples with the value of surface absorbency of paper that meet the requirements of the standard [16] for paper for packaging food products for brand E-II. For paper samples with the mass of 110 g/m², the value of this indicator required by the standard is fulfilled after adding modified starch glue or ACD to the pulp, and also significantly improves when a suspension of nanocellulose is applied to its surface.

The improvement in the strength and surface absorbency of the paper with increasing nanocellulose consumption is due to the formation of additional hydrogen bonds between the fibers of the paper and the nanocellulose during its surface application. The formation of the film on the surface of the paper and the partial impregnation of the nanoparticles into the inner layers of the paper was published previously [17].

These conclusions are confirmed by SEM images of paper with and without the application of nanocellulose on its surface (Fig. 3). The data in Fig. 3, *a* show that the surface of the paper without applying of nanocellulose has a porous structure (Fig. 3, *a*), the fibers are long and clearly expressed.

The surface coating by nanocellulose suspension (Fig. 3, *b*) contributes to a reduction of porosity of paper. In this case, the surface becomes more uniform and smooth. There is a decrease in the number of pores between layers and an increase in the density of paper. As seen in the cross-sectional view of paper (Fig. 3, *c*), spun fibers are placed in paper layers with a loose structure and cavities between them. When a nanocellulose suspension is applied to the surface (Fig. 3, *d*), the density of the surface layer of paper increases.

Thus, it is shown that the application of reed nanocellulose on the surface of laboratory paper samples improves the physical and mechanical properties of paper for packing of food products on automatic machines that meet the requirements for paper brand E-II (for the manufacture of packages for packaging groceries weight up to 3 kg) and brand D (for the manufacture of packages for packaging groceries weight up to 5 kg) in accordance with GOST 7247-90 [16].

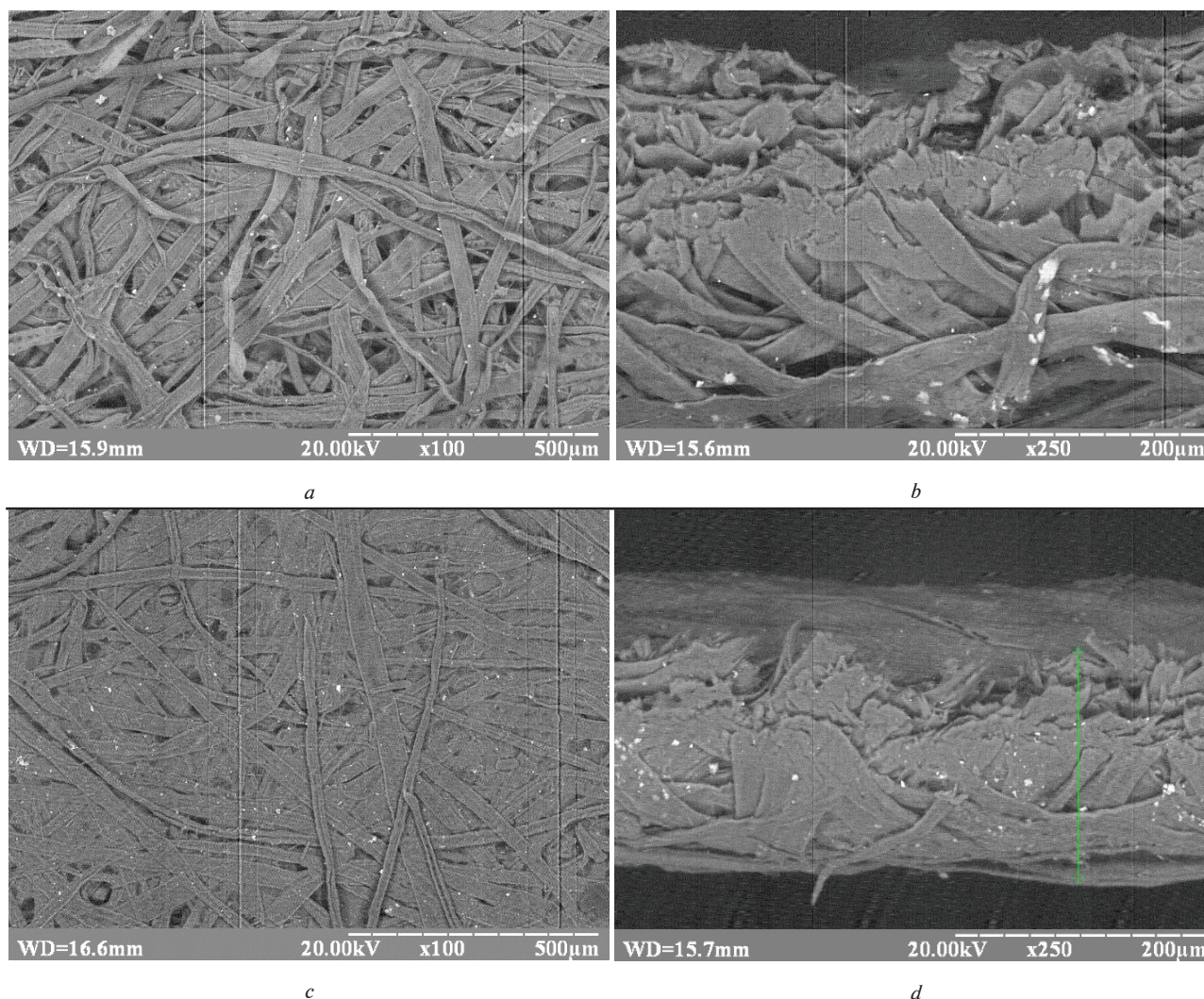


Fig. 3. SEM images of paper for packing of food products without coating (a); with coating of nanocellulose (b); c and b – cross-sectional view of a and b, respectively

Conclusions

1. Thermochemical treatment of reed stalks in two stages – alkaline extraction and organosolv cooking – make it possible to obtain cellulose with a minimum residual content of lignin and minerals. Such cellulose is suitable for subsequent chemical treatment, including the production of nanocellulose.

2. To obtain nanocellulose, we used 2,2,6,6-tetramethylpiperidine-1-oxyl (TEMPO) in the TEMPO/NaBr/NaClO system, which is more environmentally friendly than acid hydrolysis. Subsequent ultrasonic treatment makes it possible to obtain a stable nanocellulose gel with high mechanical properties.

3. It is shown that the deposition of up to 3 g/m² of nanocellulose on the surface of the paper obtained samples that meet the requirements for paper for packaging food products on automatic machines. It was found that the use of nanocellulose from reed stalks as a hardening agent for paper production makes it possible to replace up to 50 % of the more expensive pulp with waste paper, which will reduce the cost of paper production while maintaining the requirements of the standard.

4. A prospect for further research may be the use of nanocellulose from reed stalks for the production of other mass types of paper and cardboard.

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НАНОЦЕЛЮЛОЗА ЗІ СТЕБЕЛ ОЧЕРЕТУ ДЛЯ ПОКРАЩЕННЯ ВЛАСТИВОСТЕЙ ПАПЕРУ ДЛЯ ПАКУВАННЯ ХАРЧОВИХ ПРОДУКТІВ

Проблематика. Розроблення технологій отримання з рослинної сировини матеріалів, застосування яких покращує споживчі властивості картонно-паперової продукції та не забруднює довкілля шкідливими речовинами з синтетичних полімерів, є актуальною проблемою.

Мета дослідження. Отримати екологічно безпечними способами целюлозу і наноцелюлозу зі стебел очерету та застосувати наноцелюлозу для покращення якісних параметрів паперу для пакування харчових продуктів на автоматах.

Методика реалізації. Для отримання зі стебел очерету целюлози з мінімальним залишковим вмістом лігніну та мінеральних речовин використовували дві стадії обробки: лужну екстракцію й органосольвентне варіння за температури 97 ± 2 °C. Наноцелюлозу отримували окисненням органосольвентної очеретяної целюлози 2,2,6,6-тетраметилпиперидин-1-оксидом (ТЕМПО) в системі ТЕМПО / NaBr / NaClO, що є більш екологічним, ніж гідроліз кислотами. Отриману наноцелюлозу наносили на зразки паперу з макулатури та сульфатної невивіленої целюлози з витратою від 1 до 3 г/м².

Результати дослідження. Отримано органосольвентну целюлозу зі стебел очерету з залишковим вмістом лігніну 0,53 % і зольністю 0,045 %, яку використали для отримання наноцелюлози. Одержані внаслідок окиснення частинки целюлози мали поперечний розмір 5–20 нм, довжину до декількох мікрометрів. Наноцелюлозні плівки мали високі механічні показники: щільність до 1,5 г/см³ і міцність на розрив до 60 МПа. Показано, що нанесення наноцелюлози на поверхню зразків збільшує розривну силу та розривну довжину, зменшує поверхневу вбирність паперу. Визначено, що з витратою наноцелюлози до 3 г/м² зразки паперу мають показники, що відповідають вимогам стандарту до паперу для пакування харчових продуктів на автоматах.

Висновки. Використання наноцелюлози зі стебел очерету як зміцнювальної речовини для виробництва паперу дасть змогу замінити екологічно шкідливі полімерні добавки та до 50 % більш вартісної целюлози на макулатуру зі збереженням показників якості паперу на рівні вимог стандарту.

Ключові слова: стебло очерету; органосольвентна целюлоза; ТЕМПО-окиснення; наноцелюлоза; папір.

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НАНОЦЕЛЛЮЛОЗА ИЗ СТЕБЛЕЙ КАМЫША ДЛЯ УЛУЧШЕНИЯ СВОЙСТВ БУМАГИ ДЛЯ УПАКОВКИ ПИЩЕВЫХ ПРОДУКТОВ

Проблематика. Разработка технологий получения из растительного сырья материалов, применение которых улучшает потребительские свойства картонно-бумажной продукции и не загрязняет окружающую среду вредными веществами из синтетических полимеров, является актуальной проблемой.

Цель исследования. Получить экологически безопасными способами целлюлозу и наноцеллюлозу из стеблей камыша и применить наноцеллюлозу для улучшения параметров качества бумаги для упаковки пищевых продуктов на автоматах.

Методика реализации. Для получения из стеблей камыша целлюлозы с минимальным остаточным содержанием лигнина и минеральных веществ использовали две стадии обработки: щелочную экстракцию и органосольвентную варку при температуре 97 ± 2 °С. Наноцеллюлозу получали окислением органосольвентной камышовой целлюлозы 2,2,6,6-тетраметилпиперидин-1-окислом (ТЕМПО) в системе ТЕМПО / NaBr / NaClO, что является более экологичным, чем гидролиз кислотами. Полученную наноцеллюлозу наносили на образцы бумаги из макулатуры и сульфатной небеленой целлюлозы с расходом от 1 до 3 г/м².

Результаты исследования. Получена органосольвентная целлюлоза из стеблей камыша с остаточным содержанием лигнина 0,53 % и зольностью 0,045 %, которую использовали для получения наноцеллюлозы. Полученные в результате окисления частицы целлюлозы имели поперечный размер 5–20 нм, длину до нескольких микрометров. Наноцеллюлозные пленки имели высокие механические показатели: плотность до 1,5 г/см³ и прочность на разрыв до 60 МПа. Показано, что нанесение наноцеллюлозы на поверхность образцов увеличивает разрывную силу и разрывную длину, уменьшает поверхностную впитываемость бумаги. Определено, что с расходом наноцеллюлозы до 3 г/м² образцы бумаги имеют показатели, соответствующие требованиям стандарта к бумаге для упаковки пищевых продуктов на автоматах.

Выводы. Использование наноцеллюлозы из стеблей камыша как упрочняющего вещества для производства бумаги позволит заменить экологически вредные полимерные добавки и до 50 % более дорогостоящей целлюлозы на макулатуру при сохранении показателей качества бумаги на уровне требований стандарта.

Ключевые слова: стебель камыша; органосольвентная целлюлоза; ТЕМПО-окисление; наноцеллюлоза; бумага.

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