

## ХІМІЧНІ ТЕХНОЛОГІЇ

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T.V. Zelenchuk, I.M. Deikun, V.A. Barbash\*  
Igor Sikorsky Kyiv Polytechnic Institute, Kyiv, Ukraine

## OBTAINING OF PERACETIC CELLULOSE FROM OAT STRAW FOR PAPER MANUFACTURING

**Background.** Development of technology for obtaining peracetic pulp from oat straw and its use in the production of one of the paper mass types.

**Objective.** Determination of peracetic cooking technological parameters' optimal values for oat straw peracetic cellulose quality indicators.

**Methods.** The oat straw cooking was carried out with peracetic acid at  $95 \pm 1$  °C from 90 to 180 min for hydromodulus 8:1 and 7:1, using a sodium tungstate catalyst. To determine the oat straw peracetic cellulose mechanical indexes, laboratory samples of paper weighing 70 g/m<sup>2</sup> were made.

**Results.** Technological parameters' optimum values (temperature, cooking duration, hydromodulus, hydrogen peroxide and acetic acid concentration) for the oat straw delignification process were established. It is shown that the sodium tungstate catalyst addition to the cooking solution at a rate of up to 1 % of the plant raw material weight helps to reduce the lignin content in cellulose to 15 %. A diagram of the cellulose yield dependence on its residual lignin content for various methods of non-wood plant material species delignification is constructed. The high efficiency of the peracetic method for obtaining cellulose from non-wood plant raw materials, in particular from oat straw, has been confirmed. It is determined that the obtained peracetic cellulose from oat straw has high mechanical indexes.

**Conclusions.** Oat straw peracetic cellulose can be used for the production of paper and cardboard mass types, in particular wrapping paper.

**Keywords:** oat straw; cellulose; delignification; catalyst; paper; mechanical indexes.

## Introduction

The studies in the field of plant macromolecular materials create a scientific basis for the rational use of renewable raw materials and have important signification for obtaining consumer goods. One of the indicators of the development of a society in each country is the level of consumption of paper and cardboard per capita. This indicator reflects the achieved Gross Domestic Product and indicates the satisfaction of the basic needs of people. Its average world value in 2014 was 56 kg per person, in Europe – 157 kg, in North America – 224 kg, in China – 75 kg per capita [1]. In Ukraine, this indicator by the results of 2016 was only 26.2 kg, of which only 19.5 kg was produced by domestic enterprises, and the rest was imported [2]. Taking into account the long-term global economic growth, the demand for cardboard and paper products will grow annually by 1.1 % to 2030 [3], which requires an increase in the volumes of cellulose production.

The main raw material for the production of pulp in the world is wood. For countries that do not have large stocks of free wood, including Ukraine, an urgent problem is searching for alternative sources of raw materials, in particular non-wood plant raw materials, for obtaining pulp [4, 5].

Among the representatives of non-wood plant raw materials for obtaining cellulose, special atten-

tion researchers attracted to the of cereal stalks and technical plants, for which no rational application has been found until now, and most of them remain in the fields or burned. The main advantage of such raw materials is its annual renewal and lower price than wood [6]. Obtained pulp from non-wood plant raw materials has satisfactory quality indicators and is used for the production of various types of paper and cardboard [7]. Annual world potential resources of non-wood plant raw materials exceed 1.0 billion tons, and the most promising ones are of cereal crop stalks with the volume of million tons: wheat – 550, rice – 180, rye – 60, oats – 50, barley – 40 [8].

In the pulp and paper industry, traditional cellulose production methods are sulfate, sulfite, and sodium, which remain the main pollutants in the industry. From an environmental point of view, cooking in different organic solutions is safer. This is so-called organosolvent methods of delignification. Organic solvents used for delignification of plant material may belong to different classes of organic compounds – monoatomic and polyhydric alcohols, ethers and esters, ketones and amines, phenols and carboxylic acids [9]. In particular, the peracetic acid is widely used as the main delignification reagent of the cooking solution. It is formed in the process of interaction of hydrogen peroxide and acetic acid [10, 11]. Hydrogen peroxide is widely used in various indus-

\* corresponding author: v.barbash@kpi.ua

tries, in particular in the process of pulp bleaching, in cooking solution [12], and more than 65% of the world's production of acetic acid using in production of polymers [13].

The results of obtaining cellulose by organo-solvent methods of delignification from wheat and rice straw are sufficiently highlighted in the literature [14–16]. At the same time, processes of cellulose production from oats straw require additional research.

Oat (*Avéna sativa*) – is a genus of annual herbaceous plants, widely grown on an industrial scale as a food and forage plant. The oat occupies the 7th place in the world agriculture. Oat straw stalks, like wheat and rye, have a hollow straw height of 140 cm and a thickness of 4–4.5 mm [17]. Therefore, the development of resource-saving technologies for obtaining cellulose from oats straw is an actual scientific and practical task.

### Problem statement

The purpose of this study is to determine the optimum values of technological parameters for obtaining cellulose from oat straw by the peracetic method for the production of paper and cardboard.

To achieve this purpose there are the following tasks:

- to determine the chemical composition of oat straw and compare it with other representatives of plant raw materials;
- to investigate the influence of main technological parameters – the duration and the hydromodulus of cooking and the concentration of delignification reagents on the quality indices of peracetic cellulose;
- to establish the dependence of the quality indicators of the obtained cellulose from the catalyst consumption;
- to evaluate the effectiveness of the peracetic pulping for different representatives of non-wood plant raw materials;
- to determine the mechanical indexes of peracetic cellulose from oat straw and to investigate the possibility of its using in the production of one of the paper mass types.

### Research methods

The oat straw from the Poltava region harvested in 2015 was used in this study. Oat straw stalks were carefully sorted from the impurities of herbs and ears, crushed to a size from 15–20 mm and stored in a desiccator to maintain constant moisture.

The chemical composition of oat straw and other plant raw materials for comparison by standard methods [18] is determined (Table 1). As can be seen from the data in Table 1, oat straw on the content of the main components – cellulose, lignin, pentosans, is close to hardwood, but contains 15–35 times more mineral substances than wood. The lower content of lignin in oat straw than in wood, a priori suggests the need for lower consumption of cooking reagents to achieve the same residual content of lignin in pulp. The oat straw by chemical composition is slightly inferior to the most widespread representative of cereal crops – wheat straw because it has a higher content of lignin and minerals and less content of pentosans. But compared to the chemical composition of corn stems, oat straw has a higher content of cellulose and 2 times less amount of substances that are extracted with hot water, which will promote a greater yield of cellulose from the mass of abs. dry raw materials (a.d.r.m).

**Table 1.** Chemical composition of plant materials, %

Plant material	Cellulose	Lignin	Pentosans	Ash content	Water-soluble fraction
Oat straw	44.3	22.6	18.7	6.8	5.7
Wheat straw	46.2	18.6	26.4	4.2	6.0
Corn stalks	42.6	17.9	25.7	4.7	11.8
Birch-tree	46.8	24.3	19.2	0.4	7.2
Pine-tree	47.0	27.5	10.4	0.2	6.7

The cellulose cooking from oat straw was carried out in heat-resistant flasks connected to the refrigerators to provide a constant concentration of the cooking solution. In this study, two variants of pulp obtaining from oat straw were studied: the pre-prepared solution of peracetic acid (PAA) without a catalyst and with using a catalyst (sodium tungstate). The consumption of the sodium tungstate was varied from 0.25 to 1% by mass of a.d.r.m. The cooking solution of PAA was prepared in advance by mixing glacial acetic acid and hydrogen peroxide at a concentration of 30% in the ratio of 70:30. The cooking solution, depending on the duration of its saturation, had a concentration of hydrogen peroxide from 2.8 to 6.3 % and a PAA from 5.1 to 9.7 %. The cooking

was carried out at  $95 \pm 1$  °C duration from 90 to 180 min for hydromodulus 8:1 and 7:1. In the spent solution, the residual concentration of PAA and hydrogen peroxide were determined. In the obtained peracetic cellulose from oat straw, the following indexes were determined: pulp yields the residual lignin content and ash quality from the mass of a.d.r.m. To determine the mechanical indexes of peracetic cellulose from oat straw, obtained cellulose was pre-grounded in a centrifugal-grinding apparatus to a milling degree of 60° Shopper–Riegler, and laboratory sheet was made weighing 70 g/m<sup>2</sup>. For the production of wrapping paper, one of the industry product mass types, the obtained peracetic cellulose from oat straw was ground in a centrifugal-grinding apparatus and in the prepared cellulose mass was added rosin glue at a concentration of 20 g/dm<sup>3</sup> with a consumption of 2 %, and alum sulphate at a concentration of 50 g/dm<sup>3</sup> with consumption of 3 % from the weight of the finished paper. The production of pulp and paper laboratory sheet and its testing was carried out in accordance with standard methods [18].

### Analysis of the results

In order to determine the influence of cooking time, concentration of H<sub>2</sub>O<sub>2</sub> and PAA on the quality indexes of peracetic cellulose from oat straw, a series

of studies have been carried out, the results of which are given in Table 2.

As can be seen from data in Table 2, the obtained peracetic cellulose from oat straw has a low content of residual lignin from 1.86 to 3.93 % from a.d.r.m. It shows the effective passage of the delignification process of plant raw material by a PAA solution. The low residual concentration of PAA and H<sub>2</sub>O<sub>2</sub> in the spent solution indicates a high degree of cooking reagents use. It should be noted that with the PAA concentration increase in the cooking solution and cooking time, the process of plant material delignification is improved. The relatively high ash content in the obtained peracetic cellulose indicates weak interaction of PAA with mineral substances, in particular with silicic acid salts, which are part of plant raw material.

The changing of the structural characteristics of the plant raw material under the action of the cooking solution is shown in the photographs of the samples obtained by scanning electron microscopy (Fig. 1).

As can be seen from Fig. 1, *a*, oat straw has thin and short fibers and open structure that provides rapid diffusion of the cooking solution to the fiber of the raw material. This relatively low lignin content contributes to a better pulping even at a relatively low cooking temperature. In the process of chemical-thermal processing of plant raw material, extraction of lignin and extractives was carried out, which had a positive effect on the separation of fibers (Fig. 1, *b*).

**Table 2.** Dependences of quality indexes of peracetic cellulose from oat straw versus the concentration of H<sub>2</sub>O<sub>2</sub> and PAA for a hydromodulus 8:1

Reagent concentration, %		Cooking time, min	Pulp yield, %	Lignin content, %	Ash content, %	Residual concentration, %	
H <sub>2</sub> O <sub>2</sub>	PAA					H <sub>2</sub> O <sub>2</sub>	PAA
6.3	5.1	90	61.9	3.93	5.80	1.65	1.16
		120	59.7	2.91	5.10	1.48	1.04
		150	56.8	2.58	3.87	1.12	0.79
5.1	6.2	90	57.6	3.80	5.50	1.59	1.26
		120	54.9	2.33	4.90	1.39	1.14
		150	49.7	2.06	3.41	1.01	0.89
3.9	7.4	90	56.1	3.39	5.22	0.62	0.53
		120	53.5	2.05	4.83	0.55	0.51
		150	48.1	1.95	4.51	0.45	0.50
2.8	9.4	120	43.6	2.52	5.20	0.90	1.0
3.2	9.7	120	38.6	1.86	5.08	1.03	1.22

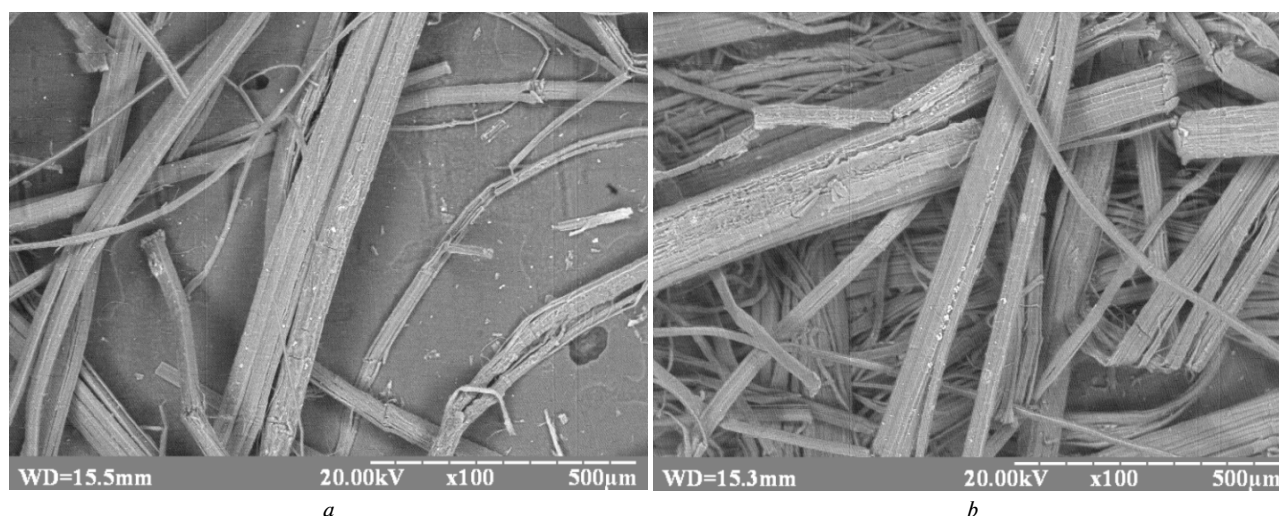


Fig. 1. SEM photographs of oat straw samples: *a* – raw material; *b* – peracetic cellulose

On the basis of the conducted researches investigation it is possible to recommend to carry out the process of obtaining organosolvent cellulose from oat straw at the following values of technological parameters: temperature  $95 \pm 1$  °C, the hydromodulus 8:1, duration 150 min, concentrations of  $H_2O_2$  – 3,9 % and PAA – 7,4 %. Under these cooking conditions, the maximum use (minimum residual concentration) of cooking reagents, insignificant residual content of lignin and minerals for a relatively high yield of cellulose from oat straw is observed.

To determine the influence of the cooking hydromodulus on the quality indices of peracetic oat cellulose, a series of cooking was carried out at the concentration of  $H_2O_2$  2.38 % and PAA 8.61 %. These results are shown in Table 3.

As can be seen from the data in Table 3, the cooking hydromodulus increase contributed to peracetic acid quantity increase that participates in the

process of delignification. This leads to the production of cellulose with a lower content of residual lignin for different cooking times, but the content of ash is slightly different. The low residual concentration of PAA in the spent solution indicates the high degree of acid use during cooking. It should also be noted that the peracetic oat cellulose at hydromodulus 8:1 has a lighter color and is more readily to disintegrate than cellulose, which obtained for a hydromodulus 7:1.

From the literature data, it is known that the effective catalysts of the peracetic cooking are transition metal salts, in particular tungsten and sodium molybdate [19]. In this case, sodium tungsten has a greater effect of lignin oxidation than sodium molybdate. Therefore, the effect of the  $Na_2WO_4$  charge on the quality indices of peracetic oat cellulose was investigated in the study. The cooking was made at PAA concentration of 9.8 % and at hydromodu-

**Table 3.** The quality indexes of peracetic oat cellulose for different cooking time and hydromodulus

Hydro-modulus	Cooking time, min	Pulp yield, %	Lignin content, %	Ash content, %	Residual concentration of	
					$H_2O_2$ , %	PAA, %
7:1	90	59.3	3.11	5.11	1.30	1.50
	120	56.4	2.73	4.83	0.97	1.01
	150	55.6	2.50	4.40	0.84	0.90
	180	54.2	2.25	4.32	0.75	0.77
8:1	90	58.5	2.91	5.03	1.43	1.45
	120	55.2	2.55	4.95	1.02	1.12
	150	53.1	2.23	4.67	0.93	0.98
	180	52.8	2.11	4.72	0.86	0.87

lus 8:1, during 90 and 150 min. The obtained results are shown in Fig. 2. Data in Fig. 2 show that at catalyst consumption from 0.25 to 1 % by mass a.d.r.m. both cellulose yield and the residual lignin content decreases. Fig. 2, *b* also shows that at a lower consumption of the catalyst, but for longer cooking time, it produces the peracetic cellulose with the same indicator of lignin content (2.4 %), as well as with higher catalyst consumption at a shorter cooking time.

The obtained results also show that the catalyst consumption and cooking time did not significantly affect the ash content in peracetic cellulose, and the low residual PAA content in the spent solution (0.22–0.80 %) indicates a high degree of acid use during cooking.

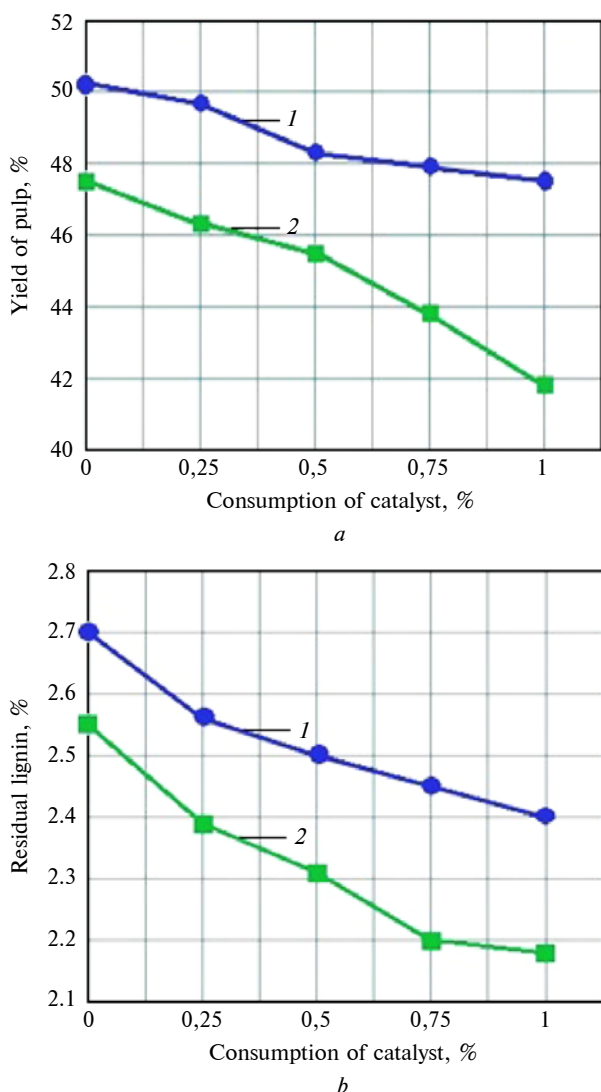


Fig. 2. Dependences of yield (*a*) and residual lignin content in peracetic cellulose (*b*) from oat straw versus catalyst consumption at different cooking time: 1 – 90 min; 2 – 150 min

Peracetic cellulose, obtained with  $\text{Na}_2\text{WO}_4$  in comparative to cellulose without catalyst has better milling ability even with shorter cooking time. That helps to reduce energy consumption in its preparations for paper and paperboard production.

To evaluate the delignification efficiency of non-wood plant raw material by the peracetic method, a diagram of the cellulosic material yield dependence on the residual lignin content in it was constructed (Fig. 3). The proposed diagram differs from the known diagrams of Girz, Ross, and Schmidt [20] by the methodology of its construction. Figure structured as follows: the axis of ordinates deposited pulp yield of 40 % (for clarity on a few percent lower cellulose content in plant material) to 100 %. On the vertical axis also delayed the point corresponding to the content in plant material holocellulose (the amount of cellulose, pentosans, and hexosans). On the abscissa from left to right, lignin content, as a pulp percentage, is postponed from zero to the maximum value of the content of lignin in plant raw materials. The intersection of the horizontal line of 100 % pulp yield lines and vertical lignin content in plant material provides a point corresponding to the initial content of all plant material components (cellulose, hemicellulose, lignin, resins, fats, waxes, minerals, and extractives).

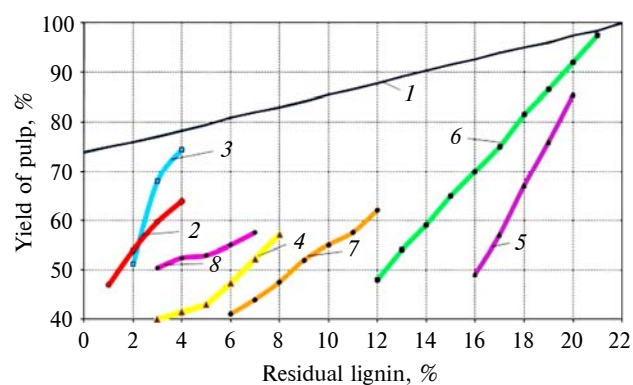


Fig. 3. The line of “ideal” delignification (1) and pulp yield dependence of different raw plant materials on the residual lignin content for the oat straw peracetic method delignification (2), wheat straw (3), corn (4), and wheat straw delignification by next methods: acetic (5); ester (6); soda (7); neutral-sulfite (8) [21–22]

The line joining this point with the holocellulose content can be considered as the line of “ideal” delignification. It describes the maximum polysaccharide content plant for certain residual lignin content in the pulp. Therefore, as the closer the particular process line delignification line of “ideal” delignification for a residual lignin value, the more pulp polysaccharide yield obtained by storing carbohy-

drates (cellulose and hemicelluloses) and is a more effective method of producing cellulose from plant raw material. As can be seen from Fig. 3, the peracetic method delignification of oat and wheat straw and corn stalks is the closest to the line of "ideal" delignification compared to traditional methods of cooking – soda and neutral-sulfite (lines 7 and 8 in Fig. 3). At the same time Fig. 3 shows, that peracetic method of producing cellulose from oat straw is better than from corn stalks but less effective than obtaining cellulose from wheat straw. This finding confirms a similar assumption, mentioned above in the analysis of the chemical composition of plant raw materials (see Table 1).

From building diagram (Fig. 3) we can conclude that the above delignification methods of examined plant raw materials by the affectivity of obtaining cellulose are approaching the line of "ideal" delignification in the following sequence: acetic – ester – soda – neutral-sulfite – peracetic.

Such order of investigated delignification methods indicates that peracetic method of producing cellulose can more selectively remove the lignin from the plant raw material and to obtain pulp with greater polysaccharides content than traditional methods of cooking methods and organosolvent methods such as acetic and ester.

Peracetic oat cellulose obtained from cooking duration of 150 min and hydromodulus of 8:1, depending on the concentration of cooking reagents has the following mechanical indexes (Table 4).

As can be seen from the data in Table 4, with increasing concentrations of peracetic acid in the cooking, all the values of peracetic cellulose mechanical indexes from oat straw also increase. This is due to the fact that the increase of the peracetic acid concentration in the cooking liquid solution helps to better remove lignin from the plant raw material, and improves the paper-making properties of the cellulose due to the better formation of hydrogen bonds between the individual cellulose fibers.

To study the possibility of using the obtained peracetic cellulose from oat straw in a composition of one of the paper mass types, laboratory samples

of wrapping paper weighing 70 g/m<sup>2</sup> were made. For this purpose, cellulose, which was obtained by cooking with a solution with a PAA concentration of 9.6 % without a catalyst (Sample I), and with a PAA concentration of 6.2 % and with a catalyst in the amount of 1 % by mass a.d.r.m. was used (Sample II). Mechanical indices of Samples I and II of peracetic wrapping paper are shown in Table 5.

**Table 5.** The indexes of wrapping paper from peracetic oat cellulose

Index	Sample I	Sample II	Basic requirement
Bursting strength, kPa	195	186	Not less than 180
Breaking strength, m	5350	4850	Not less than 3000
Sizing degree, mm	1.0	1.0	Not less than 0.6

As can be seen from the data in Table 5, laboratory paper samples from peracetic oat cellulose exceed all indexes for the wrapping paper grade G and may be recommended for the manufacture of pulp and paper industry enterprises.

## Conclusions

It was established that oat straw the content of the main components is close to hardwood, but contains 15–35 times more mineral substances than wood. The lower content of lignin in oat straw than in wood indicates the need for lower cooking reagents to achieve the same residual lignin content in the resulting cellulose.

It was shown that increasing the PAA concentration in the cooking solution contributes to the better oat straw delignification process operation. The optimal values of the technological parameters of the cellulose peracetic cooking from oat straw are: H<sub>2</sub>O<sub>2</sub> concentration – 3.9 %, PAA – 7.4 %, hydromodulus 8:1, at a temperature of 95 ± 1 °C for 150 min.

It was established that adding of sodium tungstate to a cooking solution at 1 % by mass a. d. r. m. contributes to lignin content decrease in cellulose to 15 %.

The high efficiency of the peracetic method for the cellulose production from non-wood plant raw materials, in particular from oat straw, has been confirmed. By the efficiency of cellulose production, different methods of delignification of the

**Table 4.** Mechanical indexes of peracetic oat cellulose

H <sub>2</sub> O <sub>2</sub> concentration, %	PAA concentration, %	Density, g/cm <sup>3</sup>	Bursting strength, kPa	Tearing strength, mH	Breaking strength, m
6.3	5.1	0.526	225	20	5050
5.1	6.2	0.536	231	40	5800
2.8	9.4	0.577	244	70	6200
3.2	9.7	0.593	251	80	6850

plant straw under consideration are approaching the line of “ideal” delignification in the following sequence: acetic – ester – soda – neutral-sulfite – peracetic.

It is determined that the obtained peracetic cellulose from oat straw has high values of mechanical indexes and can be used for the mass type production of paper and cardboard, wrapping paper, in particular.

In the future, it is planned to explore peracetic oat cellulose in a composition of other types of paper, to carry out alkaline treatment of cellulose for the further removal of minerals and to consider the possibility of chemical processing of peracetic oat cellulose in its derivative.

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Т.В. Зеленчук, І.М. Дейкун, В.А. Барбаш

### ОДЕРЖАННЯ ПЕРОЦТОВОЇ ЦЕЛЮЛОЗИ ІЗ СОЛОМИ ВІВСА ДЛЯ ВИРОБНИЦТВА ПАПЕРУ

**Проблематика.** Розробка технології одержання пероцтової целюлози із соломи вівса та використання її у виробництві одного із масових видів паперу.

**Мета дослідження.** Визначення оптимальних значень технологічних параметрів пероцтового варіння на показники якості пероцтової целюлози із соломи вівса.

**Методика реалізації.** Варіння січки соломи вівса проводили пероцтовою кислотою за температури  $95 \pm 1$  °C упродовж 90, 120, 150 і 180 хв за гідромодуля 8:1 і 7:1, а також із використанням каталізатора – вольфрамату натрію. Для визначення фізико-механічних показників пероцтової целюлози із соломи вівса виготовляли лабораторні зразки паперу масою 70 г/м<sup>2</sup>.

**Результати дослідження.** Встановлено оптимальні значення технологічних параметрів (температури, тривалості, гідромодуля, концентрації пероксиду водню і пероцтової кислоти) процесу делігніфікації соломи вівса. Показано, що додавання до варильного розчину каталізатора вольфрамату натрію з витратою до 1 % від маси рослинної сировини сприяє зменшенню вмісту лігніну в целюлозі до 15 %. Побудовано діаграму залежності виходу целюлози від вмісту в ній залишкового лігніну для різних способів делігніфікації представників недеревної рослинної сировини. Підтверджено високу ефективність пероцтового методу одержання целюлози із недеревної рослинної сировини, зокрема із соломи вівса. Визначено, що одержана пероцтова целюлоза із соломи вівса має високі значення фізико-механічних показників.

**Висновки.** Пероцтова целюлоза із соломи вівса може використовуватися для виробництва масових видів паперу і карто-ну, зокрема обгорткового паперу марки Г.

**Ключові слова:** солома вівса; целюлоза; делігніфікація; каталізатор; папір; фізико-механічні показники.



Т.В. Зеленчук, И.М. Дейкун, В.А. Барбаш

#### ПОЛУЧЕНИЕ ПЕРУКСУСНОЙ ЦЕЛЛЮЛОЗЫ ИЗ СОЛОМЫ ОВСА ДЛЯ ПРОИЗВОДСТВА БУМАГИ

**Проблематика.** Разработка технологии получения перуксусной целлюлозы из соломы овса и использование ее в производстве одного из массовых видов бумаги.

**Цель исследования.** Определение оптимальных значений технологических параметров перуксусной варки на показатели качества перуксусной целлюлозы из соломы овса.

**Методика реализации.** Варку сечки соломы овса проводили перуксусной кислотой при температуре  $95 \pm 1$  °С в течение 90, 120, 150 и 180 мин при гидромодуле 8:1 и 7:1, а также с использованием катализатора – вольфрамата натрия. Для определения физико-механических показателей перуксусной целлюлозы из соломы овса изготавливали лабораторные образцы бумаги массой  $70 \text{ г/м}^2$ .

**Результаты исследования.** Установлены оптимальные значения технологических параметров (температуры, продолжительности, гидромодуля, концентрации пероксида водорода и перуксусной кислоты) процесса делигнификации соломы овса. Показано, что добавление к варочному раствору катализатора вольфрамата натрия с расходом до 1 % от массы растительного сырья способствует уменьшению содержания лигнина в целлюлозе до 15 %. Построена диаграмма зависимости выхода целлюлозы от содержания в ней остаточного лигнина для различных способов делигнификации представителей недревесного растительного сырья. Подтверждена высокая эффективность перуксусного метода получения целлюлозы из недревесного растительного сырья, в частности из соломы овса. Определено, что полученная перуксусная целлюлоза из соломы овса имеет высокие значения физико-механических показателей.

**Выводы.** Перуксусная целлюлоза из соломы овса может использоваться для производства массовых видов бумаги и картона, в том числе оберточной бумаги марки Г.

**Ключевые слова:** солома овса; целлюлоза; делигнификация; катализатор; бумага; физико-механические показатели.

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