USE (UTILIZATION) AND WASTE RECYCLING (ORGANIC AND INORGANIC) IN THE CHEMICAL INDUSTRY

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RESOURCE-SAVING TECHNOLOGY IN CHEMICAL PROCESSING OF LIGNOCELLULOSIC RAW MATERIALS

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INTRODUCTION

Reducing the consumption of natural resources through the development of the latest efficient resource-saving processes is anrgent task of chemical technology. A necessary condition for the further development of the pulp industry is the reduction of the negative impact of mills on the environment. One of the ways to solve such task is the development of new methods for cellulose production from non-woods, in particular organosolv methods of pulping[1]. They are environmentally friendly and allow to obtain high-yields pulps at relatively low energy costs[2]. As anlternative to traditional methods of delignification, which are a significant source of air and water pollution with sulfur and chlorine compounds, catalytic oxidative organosolv methods of delignification of plant biomass with hydrogen peroxide in ancidic medium are proposed[3]. Hydrogen peroxide is aild oxidant and is considered one of the most environmentally friendly reagents for the delignification process[4]. It forms with organic acids peracids, which are characterized by high delignificating activity and are generated during

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[3] Deykun I., Halysh V., Barbash V. Rapeseed straw as anlternative for pulping and papermaking. *Cellulose Chemistry and Technology*. 2018. № 52(9–100). P. 833–839.

[4] Trembus I. V., Sokolovska N. V., Halysh V. V., Nosachova J. V., Overchenko T. A. Low-temperature method for manufacturing of cellulose from wheat straw. *Voprosy Khimii i Khimicheskoi Tekhnologii*. 2019. № 1. P. 116–122.

delignification[5]. The use of organic peroxyacids at their concentration of 4–10 % allows the delignification of biomass at atmospheric pressure and temperature up to 100 ºC and reduce the consumption of fresh water[6]. The use of peroxyacids does not lead to the destruction of the high molecular weight component of polysaccharide nature, final cellulosic product has a high value of whiteness[7], which allows it toe used in the composition of different types of paper and cardboard without the use of additional bleaching stage with the application of chemicals[8]. Another important positive point is the possibility of precipitation of reactive lignin from spent pulping solution[9].

Technologies of organosolv pulping of hardwoods are widely studied and described in the literature, in contrast to the obtaining cellulose from non-woods, in particular agricultural residues.

1. The problem's prerequisites emergence and the problem's formulation

The growing needs in the cellulosic goods and the shortage of wood for its production makes an important problem for the pulp and paper industry to expand the plant raw material base through the application of agricultural residues[10]. Ukraine is aeader in world export of sunflower seeds and oil and share in world trade in sunflower processing is estimated at 56 %[11]. Due to this, a huge quantity of solid residues, such as stems, is formed and left in the fields, which has a negative impact on the environment[12]. The use

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of only 25 % of this amount will allow to obtain 1–3 million tons of cellulosic products for the production of various goods.

The type of the process for non-woods processing into cellulosic products, for example, for the production of cardboard and paper products, connected with the anatomical structure and chemical composition of biomass, quality requirements, as well as with the technical and economic parameters of the delignification method[13].

Properties of cellulosic products from annual plants and agricultural residues are largely determined on morphological structure of elementary fibers of biomass, its chemical composition, type of delignification process; yield of pulp and its degree of delignification, the degree of grinding, their ability to hydrate and to fibrillate. Cellulose from non-woods contains a large number of small parenchymal cells, short vessels, nodes, which causes high fat content of the fibrous mass. Small cells in biomass impact the yield of the final product greatly as they can be destructed during the chemical treatment.

The most common in the world practice sulfate and sulfite methods of obtaining cellulose allow to obtain cellulose products with a yield of upo 48 % by weight of wood, and more than 50 % of wood biomass dissolves in the cooking solution and are subject to various methods of regeneration, which leads to additional energy losses and environmental pollution.

Therefore, the aim of the work was to characterize the process of obtaining cellulose from sunflower stalks by oxidative-organosol treatment with peracids.

2. Formulating a task for the optimal pulping of sunflower stalks

The analysis of existing research data shows that the use of organosolv methods of delignification of biomass, allows to obtain cellulosic products suitable for the further chemical processing or for the production of cardboard and paper products and to solve the environmental problems.

High-quality cellulose from annual plants and agricultural residues can be obtained and can be used in compositions with wood pulp for the production of many types of paper: writing, printing, drawing, typewriting, and bleached high-grade cardboard[14]. The application of straw cellulose in the composition improves the structure of the paper, the smoothness of the

[13] Akgul M., Tozluoglu A. Alkaline-ethanol pulping of cotton stalks. *Scientific Research and Essays*. 2010. № 5(10). P. 1068–1074.

[14] Barbash V., Trembus I., Nagorna J. Obtaining pulp from corn stalks. *Chemistry and Chemical Technology*. 2012. № 1. P. 83–87.

surface, reduces air permeability, and improves the density of the paper[15]. Cellulose from non-woods materials can be used for the production of fat-resistant types of paper[16] and cardboard[17].

Therefore, the characterization of organosolv method of obtaining cellulose from sunflower stalks is anrgent scientific and technical problem. So, the following tasks for the creation of resource-saving technology for pulp production should be solved:

1. The effect of the main technological parameters (type of peracids, temperature, cooking time, catalyst consumption) on the quality of cellulosic products from sunflower stalks should be evaluated;
2. The optimal technological parameters of obtaining organosolv cellulosic products from sunflower stalks using the methods of mathematical planning of the experiment and optimization methods should be applied.
3. The selectivity of lignin dissolution should be defined.
4. The paper-forming properties of the obtained cellulosic products from sunflower stalks should be evaluated.

3. Evaluation the efficiency of resource-saving technology for pulping of sunflower stalks

Sunflower stalks (Helianthus L.) were used to obtain cellulosic products. Stalks were sorted, cut to aize of 10–20 mm and were stored in aesiccator to maintain constant humidity. The chemical composition of sunflower stalks was determined in accordance with standard methods[18], the results are given in Table 1 in comparison with wheat straw[19], coniferous and deciduous wood[20].

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Table 1

Chemical composition of non-wood and wood biomass, %

Biomass	Cellulose	Lignin	Extractives		Resins, fats and waxes	Pentosans	Ash
			In hot water	In 1 % NaOH			
Coniferous wood	40–50	28–30	2.1–7.3	11–18.3	0.9–7.5	10.8	1–0.18
Deciduous wood	31–49	18–25	1.8–2.4	10.9–11.3	0.4–3	28.0	0.14–0.5
Wheat straw	44–45	16–17	10.1–10.6	37.2–38.4	5.2	26.7	6.6–6.9
Sunflower stalks	40.6	21.8	4.8	35.8	2.1	19.8	2.9

As can be seen from the Table 1, the content of lignin and cellulose in the stalks is close to the content of lignin and cellulose in hardwoods. The content of resins, fats and waxes and pentosans also slightly different. It should be noted that the sunflower stalks contain several times more minerals (ash content) and soluble in NaOH components (starch, pectin, inorganic salts, cyclic alcohols, dyes, tannins, hemicelluloses and low molecular weight cellulose fractions). Obtained results show that for organosolv delignification of the stalks will requires significantly lower costs of cooking reagents and shorter duration of heat treatment compared to similar wood cooking conditions to achieve the same degree of delignification[21].

Performic pulping of sunflower stalks was performed with a cooking solution containing 60 % formic acid and 30 % hydrogen peroxide in aatio of 50: 50 % by volume, the ratio slid to liquor was 10:1. The duration of treatment ranged from 30 to 120 min and the temperature of the process was from 70 to 90 °C.

Pulping of sunflower stalks with peracetic acid was performed with a cooking solution of glacial acetic acid and 35 % hydrogen peroxide at aatio of chemical reagents of 70: 30 % by volume. The temperature of the process

[21] Трембус І. В. Пакувальний папір із стебел соняшнику. *Молодий вчений*. 2016. № 3(30). С. 280–283.

was from 70 to 90 °C, duration was from 30 to 120 minutes, the ratio slid to liquor was 10:1.

The quality of sunflower cellulose is surely influenced by aumber of technological parameters, such as the duration of the delignification process and the cooking temperature. The research results are shown in Fig. 1a and 1b.

As can be seen, the yield of cellulosic pulp and the content of residual lignin in iteduced with increasing temperature and time of treatment. This is happened due to the intensification of the processes of cleavage of α- and β-ether alkylaryl bonds in lignin macromolecules, its solubilization, as well as extractive and mineral substances of biomass transfer into pulping solution.

It should mention that the yield of cellulosic pulp in case of performic acid is higher in comparison with the use of peracetic acid in the range from 30 to 120 min of treatment.

It is obvious that cellulosic products obtained by the peracetic method of delignification have a low content of residual lignin (Fig. 1 b). This is due to the fact that in the peracetic acidic medium, the structure of lignin is destroyed as aesult of acid cleavage of α-ether bonds with the formation of intermediate carbocations. In this case, the organic solvent, as aeak nucleophile, blocks the active centers of lignin and prevents its condensation. When using performic acid, the processes of acidolysis and acid fragmentation of lignin, the splitting of lignocellulosic complex and the destruction and dissolution of hemicelluloses is observed. It should be noted that the whiteness of cellulose obtained using peracetic acid is inhe range 68–72 %, and in the case of using performic acid only 40–44 %.

Results show that peracetic acid has a more selective effect on lignin and dissolves and converts it better than performic acid, which dissolves mainly hemicelluloses, which significantly reduces the yield of pulp.

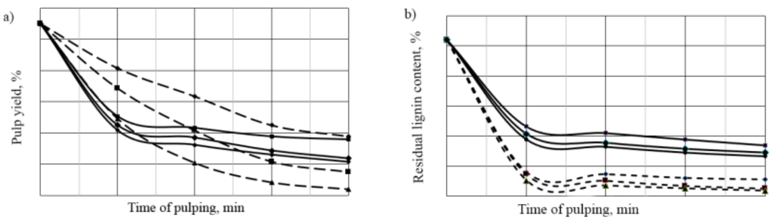

Fig. 1. Dependence of yield of pulp (a) and residual lignin content in pulp (b) obtained from sunflower stalks by peracid delignification:
--♦-- peracetic pulping at 70 °C; --■-- peracetic pulping at 80 °C;
--▲-- peracetic pulping at 90 °C; –■– performic pulping at 70 °C;
–♦– performic pulping at 80 °C; –●– performic pulping at 90 °C

To evaluate the possibility of cellulosic pulp application in the composition of paper laboratory sheets of 75 g/m^2 were papered and physical and mechanical properties were studied. The results of the rare given in Table 2.

Table 2

Properties of cellulosic products obtained by pulping of sunflower stalks with peracids

Time of pulping, min	Temperature of pulping, °C	Breaking length, m		Tear resistance, mN	
		Performic	Peracetic	Performic	Peracetic
30	70	2860	3050	102.0	140.1
60		4260	5240	142.1	205.5
90		5020	5750	176.4	262.3
120		5520	7070	215.8	287.8
30	80	4090	4350	128.1	157.2
60		5230	5850	137.3	216.5
90		5730	6700	235.4	275.3
120		6160	8650	274.6	314.4
30	90	4390	5100	137.3	272.4
60		5390	5900	296.2	325.3
90		5960	8050	313.9	379.4
120		6870	9050	402.0	478.9

As can be seen from the data in Table 2, the physical and mechanical characteristics of the obtained pulps increase with increasing the temperature and time of pulping, due to the formation of additional hydrogen bonds between polysaccharides and high content of hemicellulose especially in the case of peracetic pulping, which helps to improve mechanical strength.

Strength characteristics of the organosolv cellulosic products from sunflower stalks are similar to sulfite coniferous cellulose[22], which indicates the prospects for their use in the pulp and paper industry.

To evaluate the selectivity of cellulose obtaining (the ratio of dissolved lignin and carbohydrates dissolved during treatment), researchers have proposed carbohydrate diagrams of Ross[23], Schmidt[24], Goertz[25]. To compare

[22] ГОСТ 6501-82. Целлюлоза сульфитная небеленая их хвойной древесины. Технические условия. М. : Государственный комитет СССР по управлению качеством продукции и стандартам. 1982. 8 с.

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the efficiency of different processes of lignin removal during organosolv pulping of sunflower stalks, Fig. 2 shows a diagram, which characterized the dependence of the yield of the organosolv cellulosic product on the content of residual lignin.

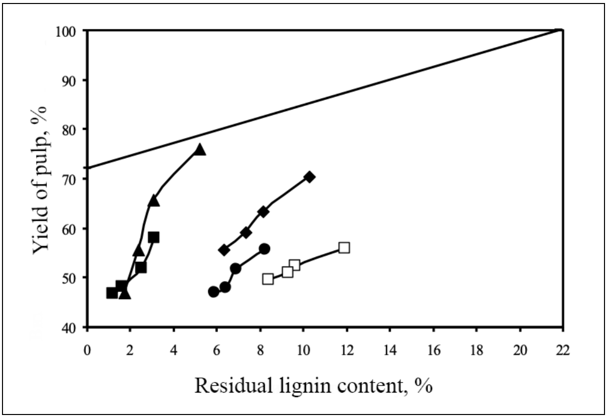

Fig. 2. Dependence of cellulosic pulp yield from sunflower stalks obtained by different delignification methods on residual lignin content:
-●- neutral-sulfite pulping; -□- soda pulping; -■- alkaline-sulfite-alcohol pulping; -♦- performic pulping; -▲- peracetic pulping.

The sloping line of “ideal delignification” (Fig. 2) characterizes the maximum content of polysaccharides in biomass for a certain content of residual lignin in the cellulosic product[26]. According to theory, the closer the line of aarticular delignification process to the line of “ideal delignification” for a certain residual lignin, the greater the yield of cellulosic pulp due to the preservation, of components of polysaccharide nature (cellulose and hemicellulose). It can be seen that studied processes can be arranged by the increase in efficiency of delignification in the following order: soda pulping – neutral-sulfite pulping – alkaline-sulfite-alcohol pulping – performic pulping – peracetic pulping. The results of research indicate that the highest selectivity corresponds to peracetic

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[26] Barbash V., Trembus I., Alushkin S., Yashchenko O. Comparative pulping of sunflower stalks. *Scientific Journal “ScienceRise”*. 2016. № 3/2(20). P. 71–78.

method of delignification of sunflower stalks, for which the optimal cooking conditions were also determined.

Factorial design of type 2^n was used as aathematical method for obtaining mathematical dependences of the quality of cellulosic products from sunflower stalks Y_i onheir main technological parameters.

Cooking temperature (x_1) and cooking time (x_2) were selected as the main technological parameters influencing the quality of cellulosic products from sunflower stalks. For the Y_i the cellulosic products were selected: Y_1 – yield of cellulosic product; Y_2 – residual lignin content; Y_3 – the breaking length; Y_4 – the tear resistance.

As a result of mathematical processing of the obtained experimental data, regression equations that adequately describe the dependences of quality indicators on the main technological parameters and can be used as mathematical models of peracetic method of delignification of sunflower stems were calculated. The defined adequate regression equations are the following:

Yield of cellulosic product, %

$$Y_1 = +\,59{,}509 – 11{,}214x_1 – 9{,}0637x_2 + 0{,}146x_1x_2 + 6{,}1222x_1^2 + 1{,}0688x_2^2$$

Content of residual lignin, %

$$Y_2 = +\,2{,}6925 – 0{,}67467x_1 – 0{,}68625x_2 – 0{,}065x_1x_2 + 0{,}22x_1^2 + 0{,}15875x_2^2$$

Breaking length, m

$$Y_3 = +\,5840{,}3 + 3183{,}3x_1 + 998{,}75x_2 + 150x_1x_2 – 1004{,}4x_1^2 + 63{,}75x_2^2$$

Tear resistance, mN

$$Y_4 = +\,251{,}89 + 70{,}267x_1 + 61{,}5x_2 – 2{,}1x_1x_2 – 18{,}222x_1^2 + 44{,}5x_2^2$$

Multicriteria optimization using the Harrington desirability function defines the compromise area of peracetic delignification of sunflower stalks depending on the main technological parameters (Fig. 3). The calculated value of the generalized desirability function D is equal to 0.67 than 0.6, which indicates a good consistency of quality indicators with the values of technological parameters.

The following parameter values were determined as the optimum point: x_1 (pulping temperature) = 80 °C, x_2 (pulping time) = 120 min. The quality indicators of the cellulose at the optimum point have the following parameter values: yield – 55.1 %; residual lignin content – 2.2 %; breaking length – 7930 m, tear resistance – 299.7 mN.

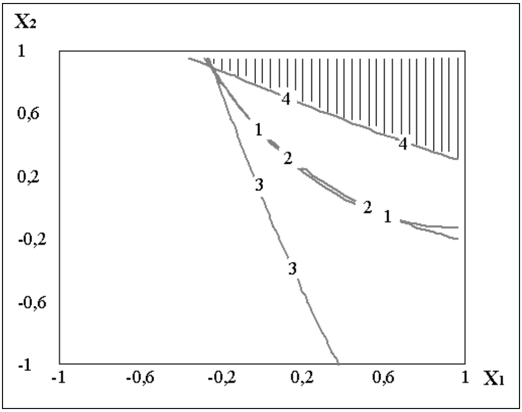

Fig. 3. Compromise area of peracetic delignification of sunflower stalks: yield of cellulosic product, % (1); residual lignin content, % (2); breaking length, m (3); tear resistance, mN (4).

4. Catalytic organosolv pulping of sunflower stalks

For the optimal conditions of delignification of biomass with peracetic acid it was decided to investigate the catalytic peracetic treatment of sunflower stalks with the application $Na_2WO_4{\cdot}2H_2O$, $Na_2MoO_4{\cdot}2H_2O$, TiO_2 as catalysts. Catalysts consumption was in the range from 1 to 6 %. As aesult, cellulosic products were obtained with wide range of yield and residual lignin content (Fig. 4). As can be seen, the effect of catalysts consumption on the pulp characteristics is quite different. For example, titanium oxide has a very little effect among the studied catalysts. The increase in consumption of this catalyst does not significantly affect the yield and residual lignin content, compared with cellulosic products obtained by non-catalytic peracetic delignification.

The cellulosic product obtained with the application of sodium molybdate has a better delignifying effect. It should be noted that the residual lignin content is reduced by 1 % and the yield is reduced by 11 % at aatalyst consumption of 5 %, which is also inefficient. There was also a significant change in the color of final pulps, which makes certain difficulties in bleaching stage.

Sodium tungstate showed the best effect on lignin removal. In cellulose obtained with the application of this catalyst, the yield decreased by 4 %, while the residual lignin content is reduced by 0.8 % at aatalyst consumption of 5 %.

The results of the calculation of the selectivity of delignification are presented in Table 3.

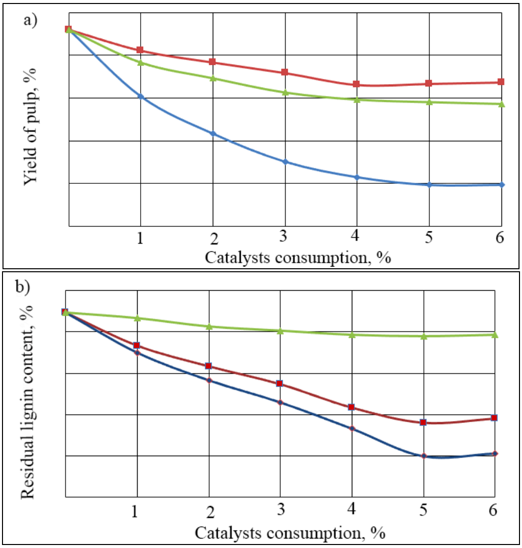

Fig. 4. Dependence of yield of pulp (a) and residual lignin content in pulp (b) obtained from sunflower stalks by peracid delignification at different catalysts consumption:
–♦– $Na_2MoO_4 \times 2H_2O$; –■– $Na_2WO_4 \times 2H_2O$; –▲–TiO_2

Table 3

Indicators of selectivity for dissolving lignin during peracetic delignification of sunflower stalks using catalysts: $Na_2MoO_4 \times 2H_2O$; $Na_2WO_4 \times 2H_2O$; TiO_2

Indicators of selectivity of lignin dissolution	Catalyst	Catalyst consumption, %						
		0	1	2	3	4	5	6
Degree of delignification%	$Na_2MoO_4 \times 2H_2O$	94.12	95.17	95.75	96.20	96.64	96.90	96.84
	$Na_2WO_4 \times 2H_2O$	94.12	94.99	95.54	95.99	96.50	96.96	96.91
	TiO_2	94.12	94.46	94.72	94.89	95.01	95.05	95.04
Selectivity, %	$Na_2MoO_4 \times 2H_2O$	68.96	63.23	60.03	57.61	56.31	55.66	55.66
	$Na_2WO_4 \times 2H_2O$	68.96	67.26	66.31	65.46	64.49	64.67	64.79
	TiO_2	68.96	66.11	64.78	63.55	62.94	62.72	62.56
The degree of removal of carbo-hydrates, %	$Na_2MoO_4 \times 2H_2O$	31.55	37.27	40.44	42.84	44.10	44.73	44.73
	$Na_2WO_4 \times 2H_2O$	31.55	33.20	34.11	34.93	35.86	35.63	35.52
	TiO_2	31.55	34.41	35.74	36.97	37.58	37.79	37.96
Optimality for lignin removal, %	$Na_2MoO_4 \times 2H_2O$	64.90	60.18	57.48	55.42	54.41	53.93	53.90
	$Na_2WO_4 \times 2H_2O$	64.90	63.88	63.36	62.83	62.23	62.78	62.70
	TiO_2	64.90	62.45	61.35	60.30	59.79	59.62	59.46

As can be seen from, the optimum point of peracetic delignification of sunflower stalks using catalysts is observed for the consumption of sodium tungstate in the amount of 5 %.

The obtained results indicate that the highest selectivity for lignin removal has sodium tungstate. The residual lignin content is reduced by almost 1.5 times compared to the pulping of sunflower stalks by non-catalytic peracetic delignification. It isational to use sodium tungstate with a consumption of 5 %.

The results of the study of microscopic structure of cellulose sample obtained through the peracetic pulping of sunflower stalks using sodium tungstate show testify about deep delignification as separate fibers are observed (Fig. 5).

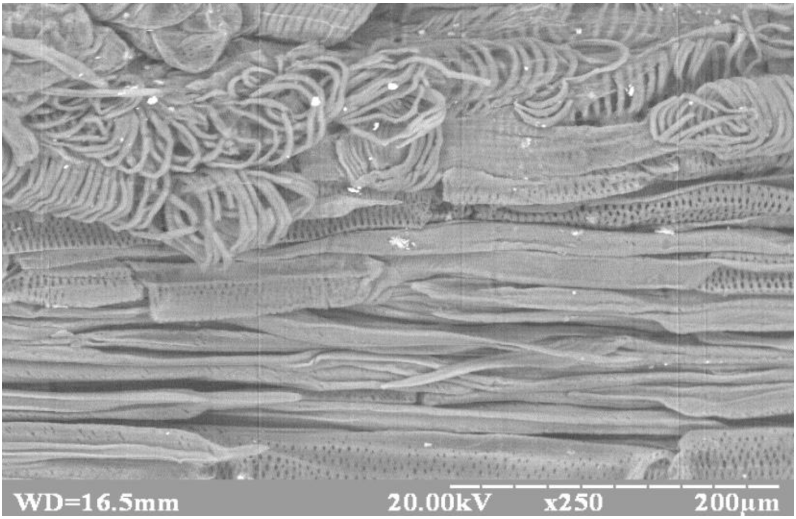

Fig. 5. SEM image of cellulosic product

For the cellulosic pulp obtained from sunflower stalks during organosolv delignification at optimal conditions and with the application of sodium tungstate with consumption of 5 % were obtained and its properties were compared with calculated values. Results are given in Table 4.

As can be seen from the data the use of the catalyst has a positive effect on the properties cellulosic pulp. The physical and mechanical characteristics of the obtained cellulosic product using the catalyst $Na_2WO_4 \times 2H_2O$ increase, due to the better paper-forming properties of pulp due to the formation of additional hydrogen bonds between polysaccharides resulting in high strength.

Table 4

Properties of the cellulose from sunflower stalks

Properties	Estimated values (optimized)	Experi-mental values	Values obtained for catalytic pulping $Na_2WO_4 \times 2H_2O$
Yield, %	55.1	54.81	51.0
Content of residual lignin, %	2.2	2.34	1.54
Breaking length, m	8650	8050	8680
Tear resistance, mN	314.4	302.7	315.92

It should be noted that the application of the catalyst during organosolv pulping improves the physical and mechanical properties of cellulose from sunflower stalks.

CONCLUSIONS

Based on the results of own research, the expediency of sunflower stalks application as anlternative raw material for the pulp industry and peracids as pulping solutions of biomass is evaluated.

The possibility of processing sunflower stalks with solutions of peracetic and performic acids into cellulosic products, which are not inferior to wood technical cellulose in terms of quality and can be used for the production of mass types of cardboard and paper products, is shown.

Regression equations were calculated. The method of multicriteria optimization were used to determin the compromise area of the peracetic delignification process of sunflower stalks.

SUMMARY

The strategy of using plant waste from agriculture to obtain useful products can be successfully implemented in many existing enterprises for the production of cardboard and paper products, because it isimple. This will allow domestic enterprises to abandon the use of expensive imported pulp, which will have a positive impact on the cost of finished paper products. The proposed technology is resource efficient because it allows efficiently process all components of biomass.

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