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V.A. Barbash, O.V. Yaschenko

National Technical University of Ukraine "KPI", Kyiv, Ukraine

OBTAINING OF STRAW PULP IN ISOBUTANOL MEDIUM

Background. Decreasing of wood consumption and hazardous wastes in environment due to ecologically safe methods of plant raw material delignification.

Objective. Identification of technological parameters influence on obtaining of organosolvent straw pulp and impact of catalysts (anthraquinone and hydrazine) on the delignification process, main quality indices of pulp, kinetic characteristics.

Methods. Pulping of organosolvent straw pulp with using of catalysts was performed in 0.25 dm³ steel autoclaves placed in glycerol bath heated at temperature 140–180 °C and 30–150 minutes duration.

Results. Obtained organosolvent straw pulp with anthraquinone and hydrazine catalysts are characterized by such physical and mechanical properties: breaking length – 7900–6900 m; burst index – 4.33–4.52 kN/g; folding strength – 128–600 double folds. It was established that using of hydrazine catalyst allows obtaining pulp with lesser lignin, ash and pentosans content then with anthraquinone. It was determined that delignification in isobutanol–KOH–water–catalyst system is described by second-order kinetic equation. Kinetic characteristics (reaction rate and energy of activation) of wheat straw delignification and characteristics of lignin removal from plant raw material were calculated.

Conclusions. Obtained organosolvent straw pulp is suitable for production of paper and cardboard products and after bleaching and alkali treatment – for further chemical treatment into different cellulose derivatives.

Keywords: wheat straw; cellulose; isobutanol; anthraquinone; hydrazine; kinetic characteristics; selectivity indices.

Introduction

Cellulose, as the most abundant natural polymer [1], has found application in the numerous branches of industry (pulp and paper, chemical, food, pharmaceutical, medical, cosmetic), in the processes of obtaining new nanocellulose materials and for household purposes [2].

The main raw material for obtaining pulp is hardwood and softwood. Stocks of wood are limited in the numerous countries and Ukraine in particular. Countries with developed agriculture annually produce millions of fibrous wastes during the treatment of technical and grain crops. Some of them can be considered as the alternative to wood for obtaining pulp. Wheat stalks belong to perspective representatives of non-wood plant raw material for obtaining pulp. According to Ukraine's Ministry of agricultural politics [3] annual potential of unused wheat straw is up to 20 million tones depending on crop yield.

In worldwide practice of pulp and paper industry sulfate and sulphite processes have become the most common pulping methods of obtaining pulp plant raw material. However, they pollute environment with hazardous wastes. Organosolvent methods are ecologically safer [4]. They apply organic solvents, which serve as chemical reagent and medium where reaction occurs. Introduction of organic solvent into pulping liquor changes its dielectric penetrability and viscosity, impacts on

solvation of delignification products, decreases energy of activation and increases rate of delignification. As the most common organic solvents aliphatic and aromatic alcohols, amines and organic acids, which are relatively easy to regenerate, are used. Some organic solvents, as isobutanol, are characterized by their ability to separate into two layers in mixtures with water. In such case upper layer of organic solvent can be used in the next pulping without regeneration, and bottom water layer containing dissolved mineral and organic compounds (lignin, extractives and hemicellulose) can be used as mineral fertilizer [5]. For such purpose it is recommended to add components, which facilitate improvement of fertilizer properties, particularly, compounds of potassium and nitrogen. And, as it is known, lignin improves agrochemical efficiency of mineral fertilizers [6]. Unfortunately, there is limited data considering application of isobutanol as component in pulping liquor in literature [5, 7, 8]. In order to intensify delignification, different catalysts are added into pulping liquor. Efficiency of anthraquinone was shown in literature multiple times [9] as well, as results of application of other compounds, particularly, hydrazine [7, 8]. That is why in this work wheat straw delignification by the isobutanol-water-KOH-anthraquinone and isobutanol-water-KOH-hydrazine pulping liquors for evaluation of obtained straw pulp characteristics was investigated.

Objective

The aim of this work is to investigate the influence of main technological parameters on the process of obtaining wheat straw pulp in isobutanol medium. Also, there is purpose to investigate the influence of different catalysts (anthraquinone and hydrazine) on delignification process and cellulose quality indices, kinetic characteristics and lignin removal characteristics.

Methods of research

In order to obtain pulp stalks of wheat straw from Kyiv region harvested in 2014 were used. Averaged chemical composition related to absolutely dry raw material was: 46.2 % cellulose, 18.6 % lignin, 25.2 % pentosans, 6.6 % ash, 5.2 % resins, fats and waxes, 74.1 % holocellulose. Values of chemical composition in wheat straw stalks were identified according to standard methods [10]. Before researches raw material was reduced in size to 2-5 mm and stored in desiccator for maintaining constant humidity and chemical composition.

Pulping was performed in isobutanol medium with potassium hydroxide and different catalysts inside steel autoclaves with volume 0.25 dm³. Autoclaves were placed in glycerol bath heated to set temperature. Pulping liquor with 10 % KOH, 15 % hydrazine, 0.1 % anthraquinone charges related to a.d.r.m. in medium of isobutanol – water 50:50 volume % mixture with liquid-to-solid ratio 6:1 was used. Temperature of pulping was 140–160 °C and from 60 to 150 minutes, pulping with anthraquinone was at 150–180 °C and from 30 to 90 minutes. Such values of technological parameters were considered as the best after series of previous experiments [9]. Autoclaves were cooled under streaming water when the pulping had been finished. Waste liquor was used for further regeneration. Obtained pulp was transferred in flask and 5 % KOH solution was poured into it. After 30 minutes pulp was filtered on Buchner's funnel and rinsed with hot distilled water for reaching neutral pH. Yield of pulp was identified by gravimetrical method after drying it to air-dry condition during 24 hours and yield of pentosans, residual lignin and ash – by the standard methods [10].

In order to obtain additional characteristics of organosolvent delignification method in isobutanol medium, calculations of following indices: selectivity (*SI*), carbohydrates removal degree (*CRD*) and delignification degree (*DD*) were performed using formulas:

$$SI = \frac{B}{100 - \frac{A \cdot DD}{100}} \cdot 100, \quad (1)$$

$$CRD = 100 - \frac{B \cdot (100 - C)}{100 - A}, \quad (2)$$

$$DD = 100 - \frac{B \cdot C}{A}, \quad (3)$$

where: *A* – original lignin content, %; *B* – plant residue yield, %; *C* – residual lignin content in pulp, %.

In order to compare influence of technological parameters on delignification process in isobutanol – water – KOH – catalyst system, kinetic characteristics, namely, specific reaction rate and energy of activation were additionally calculated. First order (4) and second order (5) kinetic equations were used for specific reaction rate calculation:

$$k = \frac{1}{t} \ln \frac{[A_0]}{[A]}, \quad (4)$$

$$k = \frac{1}{t} \left(\frac{1}{[A]} - \frac{1}{[A_0]} \right), \quad (5)$$

where: *k* – reaction rate; *t* – duration of delignification, min; *[A₀]* – lignin content in plant material, %; *[A]* – lignin content in pulp, %.

Arrhenius equation was used to calculate energy of activation:

$$k = k_0 e^{E_a/RT},$$

where: *k₀* – multiplier; *E_a* – energy of activation; *R* – molar gas constant; *T* – temperature of delignification process, K.

Aggregative pulping of wheat straw in isobutanol medium, anthraquinone and hydrazine catalysts at temperature 150 °C during 90 minutes was performed for evaluation of physical and mechanical properties of organosolvent pulps. Obtained straw pulps were beaten in centrifugal apparatus for reaching 60 ± 2 °SR (Schopper–Riegler) freeness. Samples of hand-sheets were made on sheet-making apparatus and tested according to standard methods [10].

Solution of objective

Results of pulping straw pulp in isobutanol medium with potassium hydroxide at different tem-

peratures and catalysts are presented in Table 1. As it is seen from Table 1, increasing of temperature and duration of delignification in the investigated system leads to decreasing of all indices of obtained straw pulp. It is related to intensification of lignin macromolecules destruction due to breakage of ether bond between separate chains of lignin and to transferring lignin, hemicelluloses, minerals and other extractive compounds from plant into pulping liquor. In addition, it can be concluded from table 1 data that pulping of wheat straw in isobutanol medium with hydrazine catalyst leads to significant decrease of lignin, ash and pentosans contents related to original values at the same tem-

Table 1. Quality indices of straw pulp obtained under different conditions in isobutanol–water–KOH–catalyst system, % related to a.d.r.m.

Duration of pulping, min	Yield of pulp	Residual lignin content	Pentosans content	Ash content
Anthraquinone catalyst				
Temperature of pulping 150 °C				
30	58.2	15.4	3.22	4.84
60	57.3	14.8	3.15	3.60
90	55.3	12.3	3.03	3.42
Temperature of pulping 160 °C				
30	55.0	10.5	2.90	3.83
60	54.1	9.5	2.85	2.79
90	53.5	8.6	2.81	1.93
Temperature of pulping 180 °C				
30	53.0	7.3	2.78	2.36
60	52.1	5.6	2.67	1.84
90	50.7	3.2	2.57	1.47
Hydrazine catalyst				
Temperature of pulping 140 °C				
60	58.2	4.03	3.75	2.68
90	57.3	3.75	3.27	2.60
120	56.3	3.25	2.42	2.35
150	55.2	2.6	2.07	1.96
Temperature of pulping 150 °C				
60	56.5	3.2	3.05	2.59
90	55.1	2.9	2.8	2.51
120	53.5	2.7	2.9	2.19
150	51.6	2.1	1.3	1.76
Temperature of pulping 160 °C				
60	53.0	1.9	2.3	2.45
90	51.1	1.5	2.2	2.29
120	49.7	1.3	1.5	1.92
150	49.0	1.1	0.93	1.63

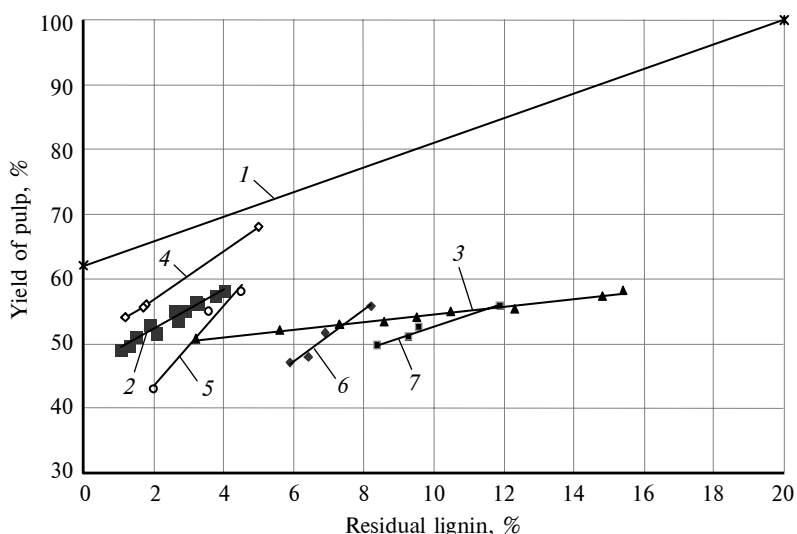
peratures and durations compared to pulping with anthraquinone catalyst. It is related to significant influence of hydrazine on delignification of plant raw material due to 150 times greater consumption of hydrazine than anthraquinone consumption in investigated systems.

Obtained in isobutanol medium organosolvent straw pulp with anthraquinone and hydrazine catalysts at 150 °C temperature and 90 minutes duration are characterized by such physical and mechanical indices correspondently: breaking length – 7900 and 6900 m; burst index – 4.33 and 4.52 kN/g; folding strength – 128 and 600 double folds. Thus, at the same pulping conditions straw pulp obtained in isobutanol–water–potassium hydroxide–hydrazine has less lignin, pentosans and ash content, higher burst index and folding strength compared to pulp obtained with anthraquinone. Such values of indices show the possibility of organosolvent pulp application in production of paper and cardboard. After bleaching and alkali treatment pulp can be used in further chemical treatment for production of different cellulose derivatives.

To compare efficiency of wheat straw delignification in isobutanol medium with other investigated methods of obtaining pulp, diagram of yield dependency on residual lignin content was constructed (Figure).

This diagram differs from known diagrams of Hirtz, Ross and Schmidt by the methodology of its construction. Yield of obtained pulp is placed from 40 % (few percent lower than original cellulose content) to 100 % on the *y*-axis. Point corresponding to holocellulose content (sum of cellulose, pentosans and hexosans) is also placed on the *y*-axis. Lignin content in pulp is placed from left to right on the *x*-axis starting with zero percent up to maximum lignin content in plant raw material (for wheat straw – 20 %). Cross of horizontal line of 100 % pulp yield with vertical line of lignin content in plant raw material gives point corresponding to original content of all plant components (cellulose, hemicellulose, lignin, resins, fats, waxes, mineral and extractive compounds). Line, which links this point with holocellulose content, can be considered as the line of “ideal delignification”. It characterizes maximum polysaccharide content in plant raw material for certain residual lignin content in pulp.

Then points corresponding to values of yield and residual lignin content in each delignification methods are placed on the diagram. Points are linked with line. The closer line to the line of “ideal delignification”, the more polysaccharide yield in pulp can be obtained at the same values of residual



Dependency of pulp yield on residual lignin content for different wheat straw delignification methods: 1 – line of “ideal delignification”; 2 – IBKH; 3 – IBKA; 4 – ASAE; 5 – PAA; 6 – neutral-sulphite; 7 – soda

lignin content due to preserving of carbohydrates (cellulose and hemicellulose), i.e. this method is more effective. As it is seen from Figure, among the observed wheat straw delignification methods the closer to the line of “ideal delignification” are following organosolvent methods: alkali–sulfite–alcohol (ASAE), pulping in isobutanol–water–KOH–hydrazine system (IBKH) and peracetic acid (PAA). Other provided pulping methods in isobutanol–water–KOH–anthraquinone (IBKA), neutral-sulfite and soda are significantly inferior by their efficiency of lignin removal. It also can be concluded from Figure, that above-mentioned methods can be placed in the following sequence by increasing their efficiency and approach to line of “ideal delignification”:

$$\text{Soda} - \text{neutral-sulfite} - \text{IBKH} - \text{PAA} - \text{IBKA} - \text{ASAE.} \quad (6)$$

Such arrangement shows, that organosolvent delignification methods allows more selective lignin removal and obtaining pulp with more polysaccharide content than, for example, traditional soda and neutral-sulfite methods.

Calculated by the formulas (1)–(3) values of lignin removal indices can validate delignification methods arrangement in obtained sequence (6). These values are presented in Table 2.

As it’s seen from Table 2, with the increase of pulping duration such indices as CRD and DD are expectedly increasing and SI – decreasing. Decreasing of SI with the increase of technological parameters of pulping is explained by the equation (1)

due to the decreasing of residual lignin content and yield. Data from Table 2 shows that pulping of wheat straw in isobutanol medium with hydrazine allows obtaining pulp with 97 % DD, which corresponds to the values of DD in ASAE method. By the values of CRD organosolvent method of delignification in isobutanol medium with hydrazine is even more effective than ASAE. That’s why analysis of provided data in table 2 allows concluding that more effective methods of delignification are IBKH system, ASAE, PAA, IBKA system, while soda and neutral-sulphite methods have the lowest values of lignin removal indices among the investigated methods. Obtained dependency of presented delignification

methods efficiency confirms abovementioned sequence (6) of approaching to the line of “ideal delignification” on the diagram of yield dependency on residual lignin content.

Table 2. Indices of lignin removal for different delignification methods

Delignification methods	Duration of pulping, min	DD, %	SI, %	CRD, %
IBKA	30	71.41	64.27	38.31
	60	74.64	63.70	38.62
	90	77.17	63.38	38.73
IBKH	60	94.59	64.31	36.13
	90	95.93	62.20	38.15
	120	96.53	60.58	39.74
	150	97.10	59.80	40.47
Neutral-sulphite	30	79.29	67.67	34.24
	60	83.86	63.46	38.21
	90	86.13	59.16	42.45
	120	87.43	58.34	43.105
Soda	90	69.92	66.11	36.78
	120	77.19	63.29	39.08
	150	78.58	61.59	40.74
	180	81.15	60.44	41.68
PAA	30	79.96	69.85	31.86
	60	86.11	59.22	42.38
	90	91.08	53.62	47.54
	120	93.81	50.77	50.08
ASAE	30	91.85	72.89	27.73
	60	94.47	61.8	38.79
	90	96.66	58.62	41.77
	120	97.56	57.24	43.05

Table 3. Kinetic characteristics of wheat straw delignification process in isobutanol–water–KOH–catalyst system

Pulping temperature, °C	Graphic method		Analytic method	
	Specific reaction rate k , $\text{min}^{-1} \cdot (10^{-3})$	Energy of activation E , kJ/mole	Specific reaction rate k , $\text{min}^{-1} \cdot (10^{-3})$	Energy of activation E , kJ/mole
Hydrazine catalyst				
140	3.5	76.6	2.4	75.1
150	4.2		3.2	
160	9.8		6.5	
Anthraquinone catalyst				
150	0.6	125.9	0.3	128.8
160	0.8		1.0	
180	6.7		2.6	

Additional characteristics of delignification are such kinetic characteristics as specific reaction rate and energy of activation. In order to determine specific reaction rate and energy of activation of delignification in isobutanol-water-KOH-catalyst system, kinetic curves described by first-order (4) and second-order (5) equations were constructed. Analysis of these curves has shown that delignification in isobutanol-water-KOH-catalyst is described by second-order equation, as kinetic curves for second-order equation do not have inflections and described by the linear dependencies with correlation coefficient close to 1. Second-order reaction is specific for wood and non-wood delignification by different methods [11]. Results of kinetic characteristic calculation for wheat straw delignification in isobutanol medium by graphic and analytic methods are presented in Table 3.

As it is seen from Table 3, data reaction rate of wheat straw delignification expectedly increase with the increase of pulping temperature. Average energy of activation value is 75.8 kJ/mole for hydrazine and 127.3 kJ/mole for anthraquinone. Calculated kinetic characteristics of obtaining wheat straw pulp in isobutanol medium with different catalysts testify about the necessity of higher energy consumption compared to sooner investigated organosolvent wheat straw delignification [11]. As the molecule of isobutanol has a bigger size than methanol or ethanol, its penetrability to lignin molecules is lower and characterized by lesser locking of hydroxyl groups in α -carbon in benzyl alcohol lignin molecule. That is why additional energy consumption for wheat straw delignification is required. Also, investigated pulping liquors in isobutanol medium has low consumption of delignification agent – potassium hydroxide (10 % related

to a.d.r.m.), which is insufficient for complete lignin removal.

Conclusions

Performing wheat straw delignification in isobutanol-water-KOH-catalyst (anthraquinone or hydrazine) allows obtaining pulp, which is not inferior by its physical and mechanical characteristics to sulfate and sulphite wood pulps and can be considered as an alternative resource for paper and cardboard production and further chemical treatment. Pulping in isobutanol medium is ecologically safe and does not require complicated regeneration system, as the upper layer of black liquor contains organic solvent, which is possible to reuse in next pulping, and bottom water layer contains dissolved mineral and organic compounds (lignin extractives, hemicellulose), which can be used as mineral fertilizer. Presence of potassium compounds due to KOH and nitrogen from hydrazine increases agrochemical efficiency of such mineral fertilizers.

Efficiency of different wheat straw delignification methods is analyzed on the proposed diagram of dependency of yield on residual lignin content. It was shown, that with the approach to the line of “ideal delignification”, which characterizes efficiency of delignification, investigated methods are arranged in following sequence: soda – neutral-sulphite – IBKA – PAA – IBKH – ASAE.

Calculated values of lignin removal indices for different delignification methods can validate the obtained sequence of investigated methods by their efficiency.

It was identified, that delignification in isobutanol-water-KOH-catalyst system is described by the second-order kinetic equation. It was shown, that obtaining wheat straw pulp in isobutanol medium with

anthraquinone requires higher energy consumption, than with hydrazine and corresponds to the sooner investigated wheat straw delignification methods.

In further research it is planned to develop the technological scheme of production straw pulp in the medium isobutanol.

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ОДЕРЖАННЯ СОЛОМ'ЯНОЇ ЦЕЛЮЛОЗИ В СЕРЕДОВИЩІ ІЗОБУТАНОЛУ

Проблематика. Зменшення витрат деревини та викидів шкідливих речовин у довкілля за рахунок екологічно безпечних способів делігніфікації рослинної сировини.

Мета дослідження. Визначення впливу технологічних параметрів процесу одержання органосольвентної солом'яної пшеничної целюлози та дії каталізаторів (антрахінону і гідразину) на процес делігніфікації та основні показники якості целюлози, кінетичні показники.

Методика реалізації. Варіння органосольвентної солом'яної целюлози з використанням каталізаторів проводили в сталевих автоклавах об'ємом $0,25 \text{ дм}^3$, занурених у гліцеринову баню за температури $140\text{--}180 \text{ }^\circ\text{C}$ упродовж $30\text{--}150$ хв.

Результати дослідження. Отримана органосольвентна солом'яна целюлоза з використанням як каталізатора антрахінону і гідразину характеризуються відповідно такими фізико-механічними показниками: розривна довжина – $7900\text{--}6900$ м; індекс продавлювання – $4,33\text{--}4,52 \text{ кН/г}$; опір згину – $128\text{--}600$ подвійних перегинів. Встановлено, що використання гідразину порівняно з антрахіноном дає змогу отримати целюлозу з меншим вмістом лігніну, золи та пентозанів. Визначено, що процес делігніфікації в системі ізобутанол–KOH–вода–каталізатор описується кінетичним рівнянням другого порядку. Розраховано кінетичні характеристики (константи швидкості та енергії активації) процесу делігніфікації пшеничної соломи і показники вилучення лігніну із рослинної сировини.

Висновки. Одержана органосольвентна солом'яна целюлоза придатна для виготовлення масових видів картонно-паперової продукції, а після проведення процесів вибілювання та облагороджування – для подальшої хімічної переробки на різні похідні целюлози.

Ключові слова: пшенична солома; целюлоза; ізобутанол; антрахінон; гідразин; кінетичні характеристики; показники вибіркості.

В.А. Барбаш, О.В. Яценко

ПОЛУЧЕНИЕ СОЛОМЕННОЙ ЦЕЛЛЮЛОЗЫ В СРЕДЕ ИЗОБУТАНОЛА

Проблематика. Уменьшение расходов древесины и выбросов вредных веществ в окружающую среду экологически безопасными способами делигнификации растительного сырья.

Цель исследования. Определение влияния основных технологических параметров процесса получения органосольвентной соломенной пшеничной целлюлозы и действия различных катализаторов (антрахинона и гидразина) на процесс делигнификации и основные показатели качества целлюлозы, кинетические показатели.

Методика реализации. Варку органосольвентной соломенной целлюлозы с использованием катализаторов проводили в стальных автоклавах объемом $0,25 \text{ дм}^3$, погруженных в глицериновую баню, нагретую до температуры $140\text{--}180 \text{ }^\circ\text{C}$, на протяжении $30\text{--}150$ мин.

Результаты исследования. Полученная органосольвентная соломенная целлюлоза с использованием в качестве катализатора антрахинона и гидразина характеризуются соответственно такими физико-механическими показателями: разрывная длина – $7900\text{--}6900$ м; индекс продавливания – $4,33\text{--}4,52 \text{ кН/г}$; сопротивление сгибу – $128\text{--}600$ двойных перегибов. Установлено, что использование гидразина в сравнении с антрахиноном позволяет получить целлюлозу с меньшим содержанием лигнина, зола и пентозанов. Определено, что процесс делигнификации в системе изобутанол–KOH–вода–катализатор описывается кинетическим уравнением второго порядка. Рассчитаны кинетические характеристики (константы скорости и энергии активации) процесса делигнификации пшеничной соломы и показатели удаления лигнина из растительного сырья.

Выводы. Полученная органосольвентная соломенная целлюлоза пригодна для изготовления массовых видов картонно-бумажной продукции, а после проведения процессов отбеливания и облагораживания – для дальнейшей химической переработки в производные целлюлозы.

Ключевые слова: пшеничная солома; целлюлоза; изобутанол; антрахинон; гидразин; кинетические характеристики; показатели избирательности.

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