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CHARACTERISTICS OF BLACK LIQUOR AFTER ALKALINE DELIGNIFICATION OF PAULOWNIA WOOD

The object of research is the black liquor of the alkaline pulping sample Paulownia Clone in Vitro 112® with active alkali consumption of 14, 18 and 22 % in Na_2O units from the mass of completely dry raw materials.

The problem solved in the work is related to the formation of waste in the form of black liquor during pulp production. It is noted that as a result of the production of 1 ton of cellulose, up to 7 tons of black liquor is formed.

In the course of the work, an analysis of the total titrated and active alkalinity in the white and black liquor after cooking paulownia wood was carried out, and the pH value was determined. The dependence of the content of dry substances in black liquor on the consumption of active alkali, the duration of cooking, the presence of a catalyst, and the final temperature of the delignification process is given. The amount of organic and mineral components of dry substances of black liquor per 1 ton of air-dry fibrous semi-finished products was calculated.

It was established that with an increase in temperature by 20 °C (from 150 to 170 °C) and a duration of approximately 1.5 times (from 60 to 90 min and from 90 to 150 min), with the same consumption of active alkali and the use of a catalyst in the conditions after sodium pulping, the content of dry substances in the black liquor increases by 1.5–3 %. This regularity is explained by the transition into the solution of a greater number of dissolution products of lignin, hemicelluloses, and mineral substances.

Estimated values for the content of mineral and organic substances in black liquor after cooking paulownia wood for a duration of 150 min and a final temperature of 170 °C at different consumptions of active alkali and the use of catalysts in the form of anthraquinone and ethyl alcohol are within the range of 1024–1518 kg per 1 ton dry pulps of normal yield and can be used in practice. The obtained results can be used to form an assessment of the black liquor as an additional resource potential for the economy of production.

Keywords: *paulownia, alkaline pulping, black liquor, pH, total and active alkalinity, dry solids, organic and mineral compounds.*

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1. Introduction

The process of cellulose production by any cooking method is reduced to the liberation of cellulose from other accompanying components of plant tissue, such as lignin, hemicelluloses, resins, fats, waxes, and mineral substances. In the course of chemical pro cessing of plant raw materials, hemicellulose, which are easily hydrolyzed, first pass into the solution, which weakens the cell membrane and makes it more accessible to cooking reagents. Further dissolution of hemicelluloses is associated with the destruction and splitting of bonds with lignin, which is the main substance from which raw materials are released in the process of delignification [1]. As a result, the cooking solution is transformed into spent, which is called «black liquor» – a by-product of pulp production at factories and an available renewable energy source with a dry matter content of 15–17 % [2, 3]. According to its chemical composition, black liquor is a complex mixture of organic and inorganic substances [4].

Depending on the initial conditions of the delignification process of plant raw materials, black liquors are traditionally divided into sulfate and sulfite. The composition of the organic part of sulfate liquor, which is 60–70 %, includes soap in the amount of 40–45 %, lignin – 30–35 %, breakdown products of carbohydrates (hemicellulose, cellulose) – 30–35 % and low molecular weight acids – aliphatic carboxylic acids (acetate, formate), which are formed as a result of various chemical transformations during cooking in a boiler. Such components as lignosulfonic acids of various degrees of sulfonation, monosaccharides, furfural, methyl alcohol, resin, starch, and low molecular weight compounds are characteristic of sulfite liquor [1, 5–7].

Black liquor also contains approximately 30 % to 40 % inorganic substances [1]. The inorganic part of the composition of black liquor depends on the type of plant material, consumption of lye and cooking conditions. Approximately 18–60 % of the sodium present in black liquor may be chemically bound to organic components in the form of sodium salts, which include carboxylate and phenolate salts.

The average fractional composition of individual inorganic compounds (% of the sum of compounds) in a typical black liquor is: sodium carbonate (Na_2CO_3) – 10–40 %, free alkali (NaOH) – 2–10 %, sodium sulfide (Na_2S) – 15–20 %, Na_2SO_3 – 5–10 %, $Na_2S_2O_3$ – 15–20 %, Na_2SO_4 from 10 to 15 % and from 0.2 to 0.7 % of silicon dioxide (SiO_2) and about 10 % of other inorganic non-technological components [8].

The ratio between the constituent components of black liquor depends on the type of wood and the technological conditions of delignification. Black liquors of alkaline cooking are very similar to each other in terms of chemical composition. It is known that delignification under the conditions of sulfate cooking only accelerates in comparison with sodium cooking, due to the presence in the solution of both hydroxyl ions and hydrogen sulfide ions, which have greater nucleophilicity in contrast to only hydroxyl ions, which are the main and only ones during sodium cooking. However, in both cases of alkaline cooking, the degradation and dissolution of lignin is due to the breaking of certain types of chemical bonds in it and the release of phenolic hydroxyl groups, which dissociate into sodium phenolates with a simultaneous increase in the hydrophilicity of lignin fragments. Thus, the lignin of sodium and sulfate cooking is very similar in chemical composition.

During delignification of cellulose, lignin undergoes fragmentation and is in solution with different molecular weights. Generally, hardwood black liquor contains less lignin and more aliphatic carboxylic acids, especially acetic acid, and other organic matter, including xylan residues, compared to softwood black liquor.

In modern conditions, pulp mills use the waste of delignification of vegetable raw materials in the form of black liquor as a source of energy. Traditionally, at pulp mills, black liquor is burned in recovery boilers such as, for example, Tomlinson to generate steam and recover chemicals for reuse [9].

An alternative and new commercial technology is gasification of black liquor. Chemrec's technology can provide double the amount of electricity for a pulp mill. Its purpose is the production of a combustible mixture of raw gases, as well as the separation of inorganic chemicals from the production of cellulose for their further regeneration. The gasification process can occur at temperatures of 600–1000 °C [10, 11].

It is generally accepted that black liquor is the fifth most important source of fuel on Earth after coal, oil, natural gas and gasoline. According to the data of the International Energy Agency (IEA), the future use of lignin compounds of black liquor as biofuel is promising, although expensive and not very common at present [2]. According to the IEA report «World Investments in Energy 2023», it is expected that annual investments in clean energy will grow by 9 % due to renewable energy sources, which directly includes the by-product of cellulose production – lignin [12].

Based on IEA data, the global pulp and paper industry currently processes about 170 million tons of black liquor (as solids) per year with a total energy content of approximately $2 \cdot 10^{18}$ J, making black liquor a very important source of biomass. Compared to other potential sources of biom ass for chemical production, black liquor has the great advantage that it is already partially processed and exists in a liquid form that can be pumped [3, 10].

In the previously presented works, it was shown that most of the black liquor in the world is subject to concentration by evaporation and subsequent combustion for energy production [7]. Nevertheless, this strategy involves the destruction of many valuable compounds of great industrial interest (for example, lignin, hemicelluloses, etc.) and does not always justify itself. This is partly due to the fact that the equipment used is quite complex and expensive to maintain, which significantly limits the increase in pulp production capacity and, accordingly, the decrease in the competitiveness of production [13].

In 2020, German scientists stated that the properties of black liquor were underestimated and showed that lignin as a promising biopolymer, being the second most common polymer on earth, can play an important role in the development of substances based on oil, as well as substances based on biochemical components. New approaches have been developed to help transform existing waste streams into solid board materials for furniture manufacturing and as flexible alternatives to leather in the fashion industry [14].

Among the scientists, there are opinions about the more valuable use of lignin of black liquor, for example, to obtain carbon fibers, phenolic resins, activated carbon, as a portable or biodiesel fuel. Thus, partial extraction of lignin can be an alternative technology for the recovery of inorganic components from black liquor [8].

In scientific circles, there are thoughts of rethinking the general process of obtaining cellulose as a biorefinery with the possibility of fractionation of numerous chemical compounds, including the breakdown products of polysaccharides, making them available to replace petroleum-based chemicals.

Based on the above researches, a pressing issue in the process of pulp production is the solution of the problem of waste in the form of black liquor and the formation of additional resource potential for the economy of production.

Therefore, *the aim of research* is to analyze the spent solutions after the sodium process of delignification of *Paulownia Clone in Vitro 112*® wood, which will make it possible to evaluate them for further processing. This goal can be achieved by solving the following tasks:

– establish the dependence of the content of dry substances in black liquor on the consumption of active alkali, the duration of cooking, the presence of a catalyst and the temperature of the delignification process;

– calculate the amount of organic and mineral components of dry substances of black liquor per 1 ton of air-dry FSFP.

2. Materials and Methods

The object of the work research is the black liquor of sodium boiling of the *Paulownia Clone in Vitro 112*® sample at the consumption of active alkali of 14, 18 and 22 % in Na_2O units from the mass of absolutely dry (abs. dry) raw materials.

Pre-prepared air-dry wood chips were loaded into steel autoclaves with a volume of 0.5 l in the amount of 40 g (based on abs. dry raw material). The compacted chips in the middle of the autoclave were filled with a cooking solution with a hydromodulus of 5:1, and a catalyst was added if necessary. Anthraquinone (AQ) was used as a process catalyst for some samples at a rate of 0.05 and 0.1 % and ethyl alcohol (C_2H_5OH) in an amount of 20 % by volume (vol.). Cooking was carried out with a gradual rise in temperature from 100 °C to 150 or 170 °C, followed by cooking at the final temperature for 60, 90 or 150 minutes.

At the end of cooking, the autoclaves were cooled, the black liquor was separated from the fibrous semi-finished product (FSFP), which was washed with running water on sieves (Fig. 1).

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Fig. 1. The general scheme for obtaining fibrous semi-finished products and black liquor:
1 – wood chips; 2 – sodium chloride; 3 – washed fibrous semi-finished product after cooking; 4 – black liquor

The yield of the semi-finished product [15] and the mass fraction of lignin [16] were determined in the solid residue. The content of dry substances [17] and their ash content [18] were determined in black liquor. The total titrated and active alkalinity was determined in the original and used solution according to the standard method [18]. The pH of the solution was also measured using a pH-150MI pH meter and litmus paper. The result was taken as the average arithmetic value of three parallel measurements.

The amount of mineral and organic substances in the liquor after cooking (per 1 ton of air-dry FSFP) was calculated according to the following formulas:

$$G_{\min} = \frac{800 \cdot 100}{B} \cdot \frac{a}{100} \cdot 1.5 \ \frac{\text{kg}}{\text{t}}, \qquad (1)$$

$$G_{org} = 880 \cdot \frac{100 - B}{B} \ \frac{\text{kg}}{\text{t}}, \qquad (2)$$

where B – yield of FSFP, % of the mass of abs. dry wood; a – consumption of active alkali for cooking, % in Na_2O units to abs. dry wood; 1.5 – conversion factor from Na_2O units to proper units.

The calculated value of the total amount of dry substances was obtained by the formula:

$$G_{dry\ matter} = G_{\min} + G_{org} = \frac{800}{B}(100 - B + 1.5a) \ \frac{\text{kg}}{\text{t}}. \qquad (3)$$

The research was conducted at the Department of Ecology and Technology of Plant Polymers of the National Technical University of Ukraine «Igor Sikorsky Kyiv Polytechnic Institute».

3. Results and Discussion

The results of the analysis of the white and black liquors after cooking *Paulownia Clone in Vitro 112*® wood are shown in Table 1.

It should be noted that the pH value of the spent solutions stably remains in the alkaline region – 11.3–11.6, which is explained by a sufficiently high buffer alkalinity. The main part of the active alkali binds to the acidic products of the breakdown of wood polysaccharides. The concentration of hydroxyl ions in the cooking solution remains at a high and almost constant level throughout the cooking process.

According to the residual value of active alkali, it can be seen that after the end of cooking, 5.7–8 % of alkali remains, which ensures the normal passage of wood delignification processes.

An important characteristic of the black liquor after cooking hardwood and softwood is the content of dry substances in it. This indicator makes it possible to evaluate the rationality of using black liquor with the aim of its further regeneration.

Table 1

The results of the analysis of the white and black liquors after cooking Paulownia Clone in Vitro 112® wood

Consumption of active alkali, % in Na_2O units	White liquor			τ, min	Black liquor	
	pH	C_{TA}, g/l in Na_2O units	C_{AA}, g/l in Na_2O units		C_{TA}, g/l in Na_2O units	C_{AA}, g/l in Na_2O units
14	11.97	64.5	55.0	90	17.8	3.2
				150	18.3	3.8
18	11.87	91.4	80.7	90	23.0	4.6
				150	26.4	5.6
22	11.79	142.9	130.5	90	26.2	8.4
				150	28.2	10.4

The results of the influence of the consumption of active alkali and the duration of the wood delignification process at a final temperature of 150 °C on the content of dry substances in black liquor are shown in Fig. 2, as well as the quality of the obtained semi-finished products are shown in Table 2.

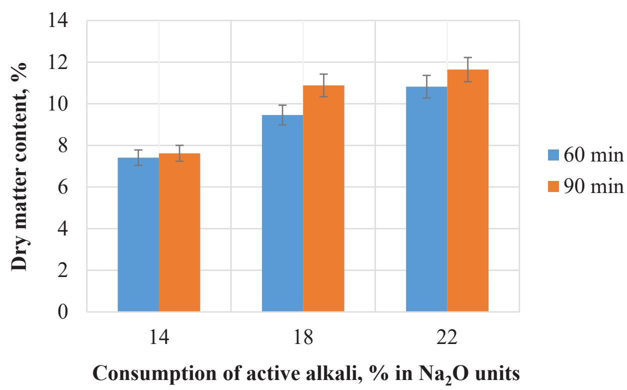

Fig. 2. Dependence of the content of dry matter in black liquor from Paulownia Clone in Vitro 112® wood on the duration of cooking and consumption of active alkali during sodium cooking at a temperature of 150 °C

As can be seen from the diagram (Fig. 2), with an increase in the duration of the delignification process and consumption of active alkali, the increased content of dry matter up to 11.34 % is contained in the black liquor obtained in 90 min and the consumption of active alkali is 22 % in Na_2O units.

This is about 0.5 % more than during 60 min with the same consumption of active alkali, where the dry residue is at the level of 10.82 %. That is, with an increase in the duration of cooking from 60 to 90 min at a temperature of 150 °C, the hydrolytic effect on carbohydrates and lignin increases, which is reflected in the increase in the amount of organic and mineral substances that have become part of the spent solution. However, at lower costs of active alkali, for example, within 14 % in Na_2O units by mass abs. dry raw materials, the influence of the duration of cooking is not significant. The content of dry substances in the black liquor under these conditions is in the range of 7.41–7.62 %. This indicates the insufficient effect of the active reagents of sodium chlorite on plant raw materials.

The obtained experimental data on the content of dry matter in black liquor are confirmed by the yield of FSFP and the content of lignin in it (Table 2).

Table 2

Quality indicators of fibrous semi-finished products obtained at different consumptions of active alkali and duration of cooking at a temperature of 150 °C

Consumption of active alkali, % in Na_2O units	τ, min	Yield FSFP, % of mass abs. dry raw materials	Lignin content, %
14	60	88.9	19.7
	90	88.0	18.5
18	60	81.4	17.6
	90	69.2	15.6
22	60	79.4	15.2
	90	63.8	12.6

As can be seen from the data in the Table 2, with higher consumption of active alkali and longer duration of cooking, there is a deepening of the degree of delignification and, accordingly, a decrease in the FSFP yield. This regularity is accompanied by the dissolution of wood components under the action of active hydroxide ions and their accumulation in the solution in the form of a dry residue.

In order to understand the chemical processes that occur during the cooking of wood, it is important to determine the amount of organic and mineral substances in black liquor.

The calculated values of the content of mineral and organic substances in the black liquor, obtained at different rates of consumption of active alkali and duration of cooking at a final temperature of 150 °C, are shown in Table 3.

Table 3

The results of the calculation of the content of mineral and organic substances in black liquor after cooking Paulownia Clone in Vitro 112® wood at a temperature of 150 °C

Consumption of active alkali, % in Na_2O units	τ, min	G_{min}, kg/t	G_{org}, kg/t	$G_{dry\ matter}$, kg/t
14	60	208	110	318
	90	210	120	330
18	60	292	201	493
	90	344	393	736
22	60	366	228	594
	90	456	500	956

It should be taken into account that the dry substances of the black liquor can be lost during cooking, washing and evaporation. Accordingly, a smaller number of them will be used for regeneration. It is accepted to consider the loss of dry substances at the level of 10 %. It should be noted that the total content of dry substances in the amount of 956 kg/t is typical for high yield cellulose.

The study of the influence of the use of catalysts in the form of AQ and C_2H_5OH in the conditions of sodium delignification of paulownia wood at different consumptions of active alkali on the content of dry matter in black liquor is shown in Fig. 3. The duration of the process was 150 min and the final temperature was 170 °C.

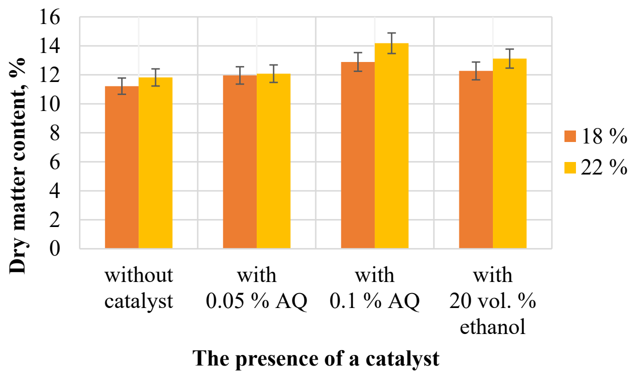

Fig. 3. The dependence of the content of dry matter in the black liquor on the consumption of active alkali and the use of catalysts during sodium boiling of Paulownia Clone in Vitro 112® wood for a duration of 150 min and a temperature of 170 °C

From those shown in Fig. 3 of the data shows that with an increase in the consumption of active alkali for the process of cooking wood by 4 % (from 18 to 22 % in Na_2O units), the number of its components, which are transferred to the composition of black liquor, increases within 1.5 %. This dependence is followed during the non-catalytic and catalytic delignification process, namely the use of 0.1 % AQ and 20 vol. % C_2H_5OH.

The obtained content of dry substances in the spent liquor can be confirmed by experimental data on the quality indicators of the FSFP (Table 4).

Table 4

Quality indicators of fibrous semi-finished products obtained at different consumptions of active alkali and the use of catalysts under the conditions of sodium cooking for a duration of 150 min and a temperature of 170 °C

The presence of a catalyst	Consumption of active alkali, % in Na_2O units	Yield FSFP, % of mass abs. dry raw materials	Lignin content, %
–	18	51.1	13.4
	22	48.8	7.5
0.05 % AQ	18	54.2	8.4
	22	50.1	6.3
0.1 % AQ	18	56.1	5.5
	22	53.2	4.3
20 vol. % C_2H_5OH	18	58.7	7.1
	22	57.5	6.2

Based on the given data, it can be seen that the increased content of dry matter up to 14.18 % was obtained with a minimum lignin content of 4.3 % and with an active alkali consumption of 22 % in Na_2O units and the addition of a delignification process catalyst of 0.1 % AQ by mass abs. dry raw materials. The obtained data are explained by the regularity of the influence of the AQ catalyst on deepening the delignification of wood at the same time as the consumption of active alkali is higher, which is consistent with theoretical information.

Data on the content of mineral and organic substances in black liquor, obtained with different consumptions of active alkali and the use of catalysts in the form of AQ and C_2H_5OH under the conditions of sodium boiling at a final temperature of 170 °C, calculated according to formulas (1) and (2) are given in Table 5.

Table 5

The results of the calculation of mineral and organic substances in the black liquor after cooking Paulownia Clone in Vitro 112® wood for a duration of 150 min and a temperature of 170 °C

The presence of a catalyst	Consumption of active alkali, % in Na_2O units	G_{min}, kg/t	G_{org}, kg/t	$G_{dry\ matter}$, kg/t
–	18	465	841	1306
	22	595	923	1518
0.05 % AQ	18	438	744	1182
	22	580	876	1456
0.1 % AQ	18	424	689	1112
	22	546	775	1321
20 vol. % C_2H_5OH	18	405	619	1024
	22	505	652	1157

As can be seen from the Table 5, the total amount of dry matter in black liquor is calculated to be in the range of 1024–1518 kg per 1 ton dry pulp of normal yield, which is generally confirmed by literature data (from 1250 to about 1800 kg/t).

In the dry residue of black liquor, its ash content was determined. It should be noted that the value of ash depends on both the temperature and the duration of cooking. Thus, at a cooking temperature of 150 °C, the ash content of the dry residue is within 25–35 %. As the temperature rises to 170 °C, the content of the mineral part increases to 31–40 %, which is consistent with literature data. In part, this regularity is explained by the fact that the deepening of the degree of delignification and the decrease in the yield of semi-finished products shift the ratio between the organic and mineral parts in the direction of the mineral part.

The obtained research results can be useful for scientists and technological engineers in the pulp and paper industry. Data on the content of the organic and mineral components of dry matter of the black liquor after sodium delignification of paulownia wood per 1 ton dry FSFP will be useful to theoretical scientists and practical researchers in the conditions of solving the tasks of regeneration of black liquor to improve the competitiveness of the entire process of pulp production. Determination of the pH value, total titrated and active alkalinity of black liquor should be carried out within one day after cooking the plant material.

In connection with the state of war in Ukraine, scheduled power outages occurred, which led to interruptions in the conduct of research.

The prospect of further research consists in the study of the chemical composition of the organic and mineral components of the dry substances of the black liquor.

4. Conclusions

Studies have shown that during sodium boiling of paulownia wood, spent solutions are formed, which contain wood degradation products and their amount depends on the technological factors of the process.

It was experimentally established that the content of dry matter after sodium cooking depends to a greater extent on the consumption of active alkali compared to the increase in the duration of cooking.

However, if the temperature is increased by 20 °C (from 150 to 170 °C) and the duration is approximately 1.5 times (from 60 to 90 min and from 90 to 150 min), with the same consumption of active alkali and the use of a catalyst in the conditions of sodium during cooking, the content of dry substances in the pulp naturally increases by 1.5–3 %, which is associated with the transition into the solution of a greater number of dissolution products of lignin, hemicelluloses, and mineral substances.

The obtained results allow to proceed to the study of the chemical composition of the organic and mineral part of the dry substances of the black liquor from the processing of paulownia wood for the assessment of their future use, which will be covered in the following publications.

Conflict of interest

The authors declare that they have no conflict of interest in relation to this study, including financial, personal, authorship, or any other, that could affect the study and its results presented in this article.

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The study was conducted without financial support.

Data availability

The manuscript has no associated data.

Use of artificial intelligence

The authors confirm that they did not use artificial intelligence technologies during the creation of the given work.

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