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# INFLUENCE OF KEY PARAMETERS OF ALKALINE PRETREATMENT OF BIOMASS IN A ROTARY-PULSATION APPARATUS FOR LIGNIN PRODUCTION

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***Abstract.*** *In bioethanol production technology from plant raw materials, pretreatment with subsequent hydrolysis is the most energy-consuming stage. Pretreatment technology is improved by combining chemical methods with physical methods. Hydrodynamic cavitation-based technology appears as a promising biomass pretreatment at industrial scales, which may be tailored to continuous or semi-continuous operation, saving time, energy, and operational costs. This study aimed to assess how the treatment parameters of an alkaline suspension of wheat straw in an RPA affect lignin extraction efficiency during pretreatment for hydrolysis. The wheat straw's alkaline suspension was processed in the RPA in recirculation mode. The variables examined were process temperature and alkali concentration. The solid/liquid ratio and rotor rotation speed remained constant throughout the study series. Lignin content was measured colorimetrically using acetyl bromide.*

*It was determined that the medium heating during processing due to energy dissipation in the working zone of the RPA contributes to an increase in lignin extraction.*

*It was established that an increase in the initial temperature of the alkaline suspension of wheat straw from 40 to 90 ºC within one hour leads to an increase in the lignin yield to 69.7%. An increase in the alkali concentration from 1 wt.% to 4 wt.% leads to an increase in the lignin yield from 69.7 to 81.5% of its initial content in the raw material. Comparing the obtained results of lignin extraction with similar results for other types of processing, it is possible to identify certain advantages, namely, the extraction process occurs at a temperature below 100 °C and atmospheric pressure. As a result of the research, it was found that the impact of a complex of physical processes occurring in the RPA working area allows for an increase in the yield of lignin in comparison with traditional processing with the same parameters of alkaline pretreatment of plant raw materials for fermentation.*

***Keywords:*** *bioethanol, alkaline pretreatment, lignin extraction, cavitation, rotary-pulsation apparatus.*

# ВПЛИВ КЛЮЧОВИХ ПАРАМЕТРІВ ПОПЕРЕДНЬОЇ ЛУЖНОЇ ПІДГОТОВКИ БІОМАСИ В РОТОРНО-ПУЛЬСАЦІЙНОМУ АПАРАТІ НА ОТРИМАННЯ ЛІГНІНУ

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*Анотація. У технології отримання біоетанолу з рослинної сировини попередня обробка з подальшим гідролізом є найбільш енерговитратним етапом. З метою удосконалення технології попередньої обробки застосовують не тільки хімічні методи, а і їх комбінацію з* *методами фізичного впливу. Технологія на основі гідродинамічної кавітації видається перспективною попередньою обробкою біомаси в промислових масштабах, яка може бути адаптована для безперервної або напівбезперервної роботи, що дасть змогу заощадити час, енергію та експлуатаційні витрати. Метою цього дослідження було визначити вплив параметрів обробки лужної суспензії соломи пшеничної в роторно-пульсаційному апараті на ефективність екстракції лігніну протягом попередньої підготовки до гідролізу. Лужну суспензію пшеничної соломи обробляли в кавітаційному реакторі роторного типу в режимі рециркуляції. Параметрами варіювання були температура процесу та концентрація лугу. Співвідношення тверда речовина / рідина та швидкість обертання ротора залишались незмінними протягом всієї серії досліджень. Вміст лігніну визначали колориметрично з використанням ацетилброміду.*

*Було виявлено, що нагрівання середовища протягом обробки внаслідок дисипації енергії в робочій зоні роторно-пульсаційного апарата сприяє збільшенню екстракції лігніну.*

*Встановлено, що підвищення початкової температури лужної суспензії соломи пшеничної від 40 до 90 °C протягом однієї години приводить до збільшення виходу лігніну до 69,7 %. Підвищення концентрації лугу від 1 до 4 мас.% призводить до збільшення виходу лігніну з 69,7 до 81,5 % його початкового вмісту в сировині.*

*Порівнюючи отримані результати екстракції лігніну з аналогічними результатами для інших видів обробки, наведених вище, можна відмітити певні переваги, а саме, процес екстракції відбувається за температури нижче 100 оС і атмосферного тиску. Внаслідок досліджень встановлено, що під впливом комплексу фізичних процесів, які відбуваються в робочій зоні роторно-пульсаційного апарата, вихід лігніну підвищується в порівнянні з традиційною обробкою з такими самими параметрами лужної попередньої обробки рослинної сировини для ферментації.*

***Ключові слова:*** *біоетанол, лужна попередня підготовка, екстракція лігніну, кавітація, роторно-пульсаційний апарат.*

**Introduction**

The production of second-generation ethanol is more expensive than from sugar- or starch-containing raw materials because the conversion of lignocellulose into reducing sugars is more complex than the conversion of starch [1, 2].

Successful competition of second-generation ethanol with first-generation ethanol, as well as with fossil fuels, can be achieved, in particular, through the implementation of technologies and equipment that can increase the degree of conversion of raw materials into ethanol without a significant increase in costs, and through the extraction of by-products processing and their subsequent processing into valuable products [3].

Lignin, as a product of plant biomass treatment, is used as dispersants, emulsifiers, binders and adhesives, sorbents for medical and technological purposes, and sorbents for wastewater treatment [4, 5].

One of the stages of the plant biomass treatment is the extraction of lignin and hemicellulose from the lignocellulose complex. This stage is a necessary part of biomass pretreatment for hydrolysis, but it is the most energy-consuming in the production process [6]. The purpose of pretreatment is to disrupt the structure of cellulosic biomass, making cellulose more accessible to enzymes that convert carbohydrate polymers into fermenting sugars [7]. The reason for the high energy consumption of biomass pretreatment for hydrolysis is that the process takes place at elevated temperatures and pressures [8].

The result of the hydrolysis stage, which includes pretreatment and hydrolysis itself, is the production of a hydrolysate with the required concentration of reducing sugars, which is then fed to the fermentation stage. At the pretreatment stage, cellulose, lignin, furfural, and hemicelluloses are isolated.

Traditional pretreatment methods include physical pretreatment, chemical pretreatment, and biological pretreatment, each with its limitations [9]. Among the most well-known methods for pretreatment of lignocellulosic feedstock for hydrolysis are dilute acid pretreatment and alkali pretreatment.

A comparison of the effects of different pretreatment methods, including acidic and alkaline, on the improvement of enzymatic hydrolysis of wheat straw and ethanol production was reviewed by Torquero [10]. The treatment methods were as follows: thermal autoclaving (without the addition of chemical reagents), autoclaving with dilute HCl solution (1.5%, w/w), autoclaving with dilute NaOH solution (1% w/w), and alkaline peroxide $H_2O_2$ (5% w/w).

The treatment by the first three methods was carried out for one hour at a temperature of 121ºC and a solid/liquid ratio of 1/10.

The unwashed or washed solid was then used as a substrate in the next enzymatic hydrolysis and subsequent fermentation. According to the authors, autoclaving with dilute alkali is advisable as a pretreatment for biomass, followed by washing, while thermal autoclaving and dilute acid pretreatment give worse results. The advantages of using alkali were noted both at the stage of obtaining cellulose during pretreatment and at the stage of obtaining glucose and xylose during subsequent enzymatic hydrolysis.

In particular, the solid fraction of alkaline pretreatment showed a significant increase in cellulose content by 44.0% due to a sharp decrease of 51.0% in the percentage of acid-insoluble lignin and a less noticeable decrease in hemicellulose content by 11.1% compared to the thermal and acid treatments. Autoclaving in dilute alkaline solution favored the separation of some fibers more than others, thereby increasing the external surface area and porosity, as confirmed by electron microscopy.

The study [11] compared the pretreatment of wheat straw with dilute sulfuric acid, hydrochloric acid, nitric acid, and potassium hydroxide solution (1%) at a temperature of 130 ± 3 °C, a pressure of 0.3 MPa, for one hour, followed by enzymatic hydrolysis for 24 hours and fermentation for a week.

The authors claim that the highest yield of ethanol is achieved by alkaline treatment with pre-washing of raw materials with distilled water, and is 57.7% (hydrolysis) and 76.3% (fermentation), which is the most effective among all methods.

Research on the impact of key parameters of alkaline pretreatment of sugarcane bagasse for hydrolysis on lignin removal was conducted by Wunna et al. [12]. The effect of solid phase concentration and alkali concentration on the yield, particularly lignin, was assessed when alkaline dispersions with bagasse particles smaller than 0.5 mm were autoclaved at 121 °C for 120 min. It was determined that the percentage of lignin removed increased with increasing alkali concentration.

The maximum lignin removal for a solid phase concentration of 20% was 87.7% for 1.5% (wt.) with sodium hydroxide after 60 min autoclaving. For a solid phase concentration of 10%, lignin removal of 87.3% was achieved at a sodium hydroxide concentration of 1.5% (wt.) in 30 min.

It was found that the percentage of lignin and glucan removal did not increase significantly with longer autoclaving time at a solid phase concentration of 10%.

In an alkaline medium, the processes of destruction and extraction of lignin proceed more efficiently, and in an acidic medium, the processes of depolymerization of polysaccharides [13]. Alkaline pretreatment is more effective for agricultural waste and grass crops than for wood materials [14]. In alkaline pretreatment methods, in comparison with dilute acidic ones, hydrolysis proceeds under milder conditions, sugar decomposition is minimal, and, therefore, enzyme inhibitors (furfural and hydroxymethylfurfural) are formed in smaller quantities.

Improving pretreatment technology involves not only using chemical methods, but also combining them with physical methods.

To overcome the limitations of single-pretreatment methods, combining multiple pretreatment approaches allows for the complementary advantages of different methods, thereby improving the degradation efficiency and utilization of lignocelluloses [15].

The effect of ultrasonic cavitation on lignin yield from wheat straw and its effect on enzymatic hydrolysis has been studied in several studies [16, 17].

Calcio Gaudino reports the dominance of physical effects of ultrasonic pretreatment in improving wheat straw delignification at a solid/liquid (biomass/water) ratio of 1:20, showing low frequency/high power (25 kHz, 2 or 3 kW) as the preferred conditions.

It was demonstrated that the positive effect of ultrasound alkali pretreatment of wheat straw on sugar yield was due to a combination of factors, including an easier accessibility of the biomass carbohydrate network to enzymes due to the increased wheat straw porosity, the partial removal of lignin, which increases the relative proportion of carbohydrates in the treated biomass, and the disordering (amorphization) of the cellulose structure, which promotes carbohydrate hydrolysis as well as saponification of the acetyl groups in the hemicellulose.

Vandenbossche et al. assessed the impact of thermo-mechanical and thermo-mechano-chemical pretreatments in a twin-screw extruder on the digestibility of wheat straw. The study selected thermo-mechanical and thermo-mechano-chemical pretreatments using acidic, alkaline, oxidizing, and organic solvents [18]. The extruders had seven modular barrels, each 200 mm long for the BC 45 extruder and 100 mm for the BC21 extruder, as well as various twin-screw extruders with segmented screw elements of 50 and 100 mm length for the BC 45 and 12.5, 25, and 50 mm.

Twin-screw extrusion was performed for 30 min. Certain processing parameters were provided in each module. Acidic thermo-mechano-chemical pretreatment of wheat straw led to improved cellulose digestibility.

As a result of extrusion under alkaline treatment conditions, almost 50% of the hemicelluloses were removed at a low NaOH/straw ratio (7.3%), and nearly all of them were removed at a longer extruder residence time and a NaOH/straw ratio of 35%. At the same time, 36% of the lignin was dissolved.

Recently, there has been an increase in research on using hydrodynamic cavitation for the pretreatment of lignocellulosic feedstock before hydrolysis. Hydrodynamic cavitation is more effective than acoustic cavitation in many applications due to its ability to oxidize organic matter combined with low operating cost, easy scalability, high energy efficiency, and less pollution without the formation of by-products [19]. Devices used to generate cavitation include a Venturi nozzle [20, 21], an orifice plate [22-25], and cavitation generators based on centrifugal pumps [26, 27].

Specifically, the experimental setup for hydrodynamic cavitation in the study by Terán et al. consisted of a tank and a cylindrical stainless steel cavitation reactor, which were connected to a 1.5 kW centrifugal pump. The radially shaped orifice plate had 27 holes with a diameter of 1 mm. The upstream and downstream pressures were maintained at 0.3 and 0.03 MPa, respectively. The solid/liquid ratio was calculated based on a cavitation reactor volume of 100 ml. Bagasse particles were used in the range of 1.18 to 1.70 mm. The composition of the feedstock was as follows: 40.6% glucan, 26.3% xylan, and 24.9% lignin on a dry weight basis. The pretreatment conditions were set as follows: NaOH concentrations 0.1–0.5 M, solid/liquid ratio 3–10%, reaction time 15–45 min. The temperature in this study increased from 23 ºC to 64 ºC in 45 min. Thus, lignin removal and structural changes can be attributed to the cavitation effect below 50 °C and the alkaline impact above. 53% lignin removal can be achieved under 5% NaOH, 5% solid/liquid ratio, and 40 minutes in hydrodynamic cavitation treatment mode.

Rotational reactors, or rotating generators of hydrodynamic cavitation, or rotor-pulsation apparatus (mainly disc and cylindrical), are based on centrifugal pumps, to the housing of which a modified rotor and stator are added.

In this type of reactor, the cavitation phenomenon is generated by numerous cavitation generation units located on the rotor and stator. A geometric structure of the units is fundamental for the generation of cavitation as well as the effectiveness and economic efficiency [28]. Furthermore, the frequency of energy release is significantly higher compared to non-rotational generators of hydrodynamic cavitation [29]. Rotational reactors have exhibited excellent performance compared to non-rotational reactors in technologies related to mixing, homogenization, dispersion, etc.

An example of equipment that significantly intensifies heat and mass transfer in liquid multicomponent media is the rotor-pulsation apparatus, which is an effective device in technologies related to mixing, homogenization, dispersion, etc.

The principle of such a device`s operation involves accumulating energy at locally disconnected discrete points within the system with subsequent impulsed implementation to achieve the necessary physical effects, namely, pressurizing and depressurizing, adiabatic boiling, water hammer, shock waves, shear stresses, local turbulence, and cavitation [30]. An advantageous feature of such devices is the combination of hydrodynamic cavitation created in cavitation zones located in the working area of the device and highly gradient shear stresses arising between the rotor and stator.

There are several studies on the use of devices with a rotor (rotor-stator pair) for cavitation treatment of lignocellulosic raw materials. In particular, Kosel et al. [26] investigated the effect of hydrodynamic cavitation generated by a rotational generator on the refining process of pulp that was a mixture of coniferous species and eucalyptus in the ratio of water/solids 1, 2, 3% in the paper production. The refining causes the breaking of hydrogen bonds between microfibrils, which subsequently increases the surface area of microfibrils and the ability of fibers to establish bonds between them and, thus, fiber fibrillation.

During the work, the drainage rate was evaluated as an indicator of the efficiency of fibrillation, a visual assessment of the operating conditions of the setup for creating developed cavitation in the cavitation zone, as well as the effect of hydrodynamic cavitation on the physical properties of the resulting paper. The model system is assembled from a 2 L reservoir, piping, a heat exchanger, pressure and flow meters and a rotation generator. The piping and connections are made of standard household water system materials. Both the rotor and the stator are 50mm in diameter and have a specially designed geometry with 12 radial teeth, 3mm deep and 4mm wide. The rotor speed of the generator was set to 7000 rpm, which set the flow rate to 11.3 L/min.

Among the conclusions made by the authors, it should be noted that increasing the rotation frequency and shear rate in the working zone of the rotary hydrodynamic cavitator, in the interval of intensive cavitation development, resulted in obtaining pulp with a higher drainage rate, which indicates the ability to break hydrogen bonds between cellulose microfibrils and subsequently cause fibrillation [26].

The aim of the study by Lauberte et al. [27] was to define the chemical composition of liquid fractions and solid biomass residues resulting from ultrasonic and/or hydrodynamic pretreatment of wheat straw for hydrolysis [27]. In addition to the chemical profile, the antioxidant power and biological activity of the liquid fractions obtained after pretreatment were evaluated.

High-intensity ultrasonic pretreatment (30, 60, 90, and 120 min) was performed in a multi-frequency reactor at a frequency range (25 and 80 kHz and their combinations). The raw material was wheat straw, initially crushed to a particle size of 0.2 mm.

During ultrasonic treatment, the temperature inside the reactor was stabilized at 25, 30, or 35 ºC. Pretreatment was performed at 29°C in water or aqueous NaOH solution (10% by weight, based on oven-dried biomass). The solid/liquid ratio was 1/20.

Pretreatment by hydrodynamic cavitation was carried out in a rotor-stator reactor operating at 3,000 rpm at a temperature of 50°C in aqueous NaOH solutions (10% by weight, based on oven-dried wheat straw biomass) for 30 min. The solid/liquid ratio was 1/50. The authors did not provide the design features of the working unit of the rotor-stator reactor. The author's data demonstrate that in the presence of alkali, both hydrodynamic and ultrasonic treatment significantly reduce the lignin content in the solid fraction after pretreatment of wheat straw. The lignin content was 21.2 ± 0.2% to 16.1 ± 0.2% for hydrodynamic treatment. Furthermore, these pretreatments significantly reduced the ash and extractives content of the wheat straw solid biomass. The analytical pyrolysis (Py-GC/MS analyses) indicated that hemicelluloses were partially removed from the wheat straw biomass and that cellulose was more susceptible to depolymerization.

The literature analysis that was conducted allows us to draw the following conclusion. Adaptation of hydrodynamic cavitation pretreatment to an industrial level requires a lignocellulosic biomass sustainable conversion with cost minimization and process efficiency maximization.

Hydrodynamic cavitation-based technology appears as a promising biomass pretreatment method at industrial scales, which may be tailored to continuous or semi-continuous operation, saving time, energy, and operational costs. Unfortunately, to date, there is a lack of studies on the use of devices of various designs for these purposes, especially industrial or semi-industrial ones. This is important given the fact that there are still no universal criteria for the development of such processing devices and corresponding processing modes [28].

The goal of the study was to determine the effect of temperature, sodium hydroxide concentration, and processing duration of an alkali suspension of wheat straw in a cylindrical type rotary-pulsation apparatus (RPA) on the efficiency of the lignin extraction during pretreatment of wheat straw for hydrolysis.

To achieve the goal, it was necessary to accomplish the following tasks:

– to determine the effect of the initial temperature of the dispersed medium on the degree of extraction of lignin when processing an alkali suspension of wheat straw in the RPA;

– to determine the effect of the initial sodium hydroxide concentration on the degree of lignin extraction when processing an alkali suspension of wheat straw in the RPA.

**Experimental**

*Biomass feedstock*

Wheat straw harvesting was carried out in August 2020 in the Kyiv region of Ukraine. The straw contained 45.6% cellulose, 25.8 % hemicelluloses,17,1% lignin, 5.4% extractives, and 4/2% ash.

The straw was coarsely crushed to a fraction of 1 cm on the Rozumnitsa electric chopper. The resulting chaff was ground once on a DZ 300-2 pin mill. Figure 1 shows the obtained distribution of wheat straw particles after sieving on sieves. The main fraction of wheat straw particles after sieving was 100-400 microns.

Since the grinding of plant raw materials takes place at a separate stage, the impact of particle size on the extraction of the target component during alkaline pretreatment, as well as the effect of the complex of physical processes occurring in the working zone of the RPA on the contact surface area of the phases, was not considered in this study.

*Experimental setup*

The studies were conducted on the setup, the scheme of which is presented in Fig. 1.

The working elements of the cylindrical type RPA are two concentric cylinders (Fig. 1c).

Each cylindrical working element has 24 holes arranged in a staggered order in two rows with a hole diameter of 14.3 mm. The inner cylinder (rotor) rotates at a given speed relative to the outer, stationary cylinder (stator). The rotor and stator are arranged in series. The rotor has three blades, which perform the functions of a centrifugal pump. The rotor rotation speed throughout the entire series of studies was 47.75 rps. The outer diameter of the rotor is 170.5 mm. The outer diameter of the stator is 195.7 mm. The width of the rotor and stator is 44 mm. The inter-cylinder gap is 0.3 mm. The flow rate in nominal operation mode is 7.7 $m^3$/h.

The extraction of lignin from wheat straw was carried out as follows. The required amount of wheat straw was soaked in water and heated to the temperature determined by the conditions of the experiment until a homogeneous suspension was formed.

The receiving tank and reactor-mixer of the setup were filled with the rest of the water, and mixed with sodium hydroxide in the ratio determined by the experimental conditions. The solid/liquid ratio of suspension remained unchanged at 10% wt.

After starting the engine, the frequency converter set the rotor rotation frequency, which was equal to 47.75 rps for the entire series of studies. During the process, samples of suspension were taken at regular intervals.

*Determination of lignin concentration*

Lignin content was determined using the method described by [31]. Briefly, 5 mg of the sample was suspended in 2.5 mL of acetyl bromide (25% AcBr in acetic acid). 0.1 ml of perchloric acid was added and mixed. The mixture was dried in an oven three times for 10 minutes at 70 °C and cooled with ice water for 30 minutes, then transferred to a volumetric flask containing 10 mL of 2 M NaOH and 12 mL of acetic acid. Lignin content was determined by the UV-VIS method at 280 nm using 20.09 $g^{-1}\cdot L\cdot cm^{-1}$.

For each series of studies, the measurement was carried out three times. The error did not exceed 5%.

**Results and Discussion**

Most published studies on lignocellulosic biomass pretreatment for hydrolysis identify temperature, process duration, and alkali concentration as the key factors affecting lignin extraction.

Vancov and colleagues, having studied the process of alkaline pretreatment using sorghum and wheat straw, particularly concluded that temperature had the greatest effect on lignin extraction, followed by alkalinity, then processing duration [32].

It should be emphasized that the processing of liquid media in rotary generators of hydrodynamic cavitation is accompanied by significant heating of the processing medium due to energy dissipation. Heating occurs both due to shear stresses in the radial gaps between the rotor and stator, and due to cavitation in the channels of the stator or rotor (depending on the design) [33]. The increase in temperature due to energy dissipation allows reducing the supply of heat from the outside to achieve the operating temperature of the process, and reducing the viscosity of the raw material being processed allows reducing the energy consumption for its pumping.

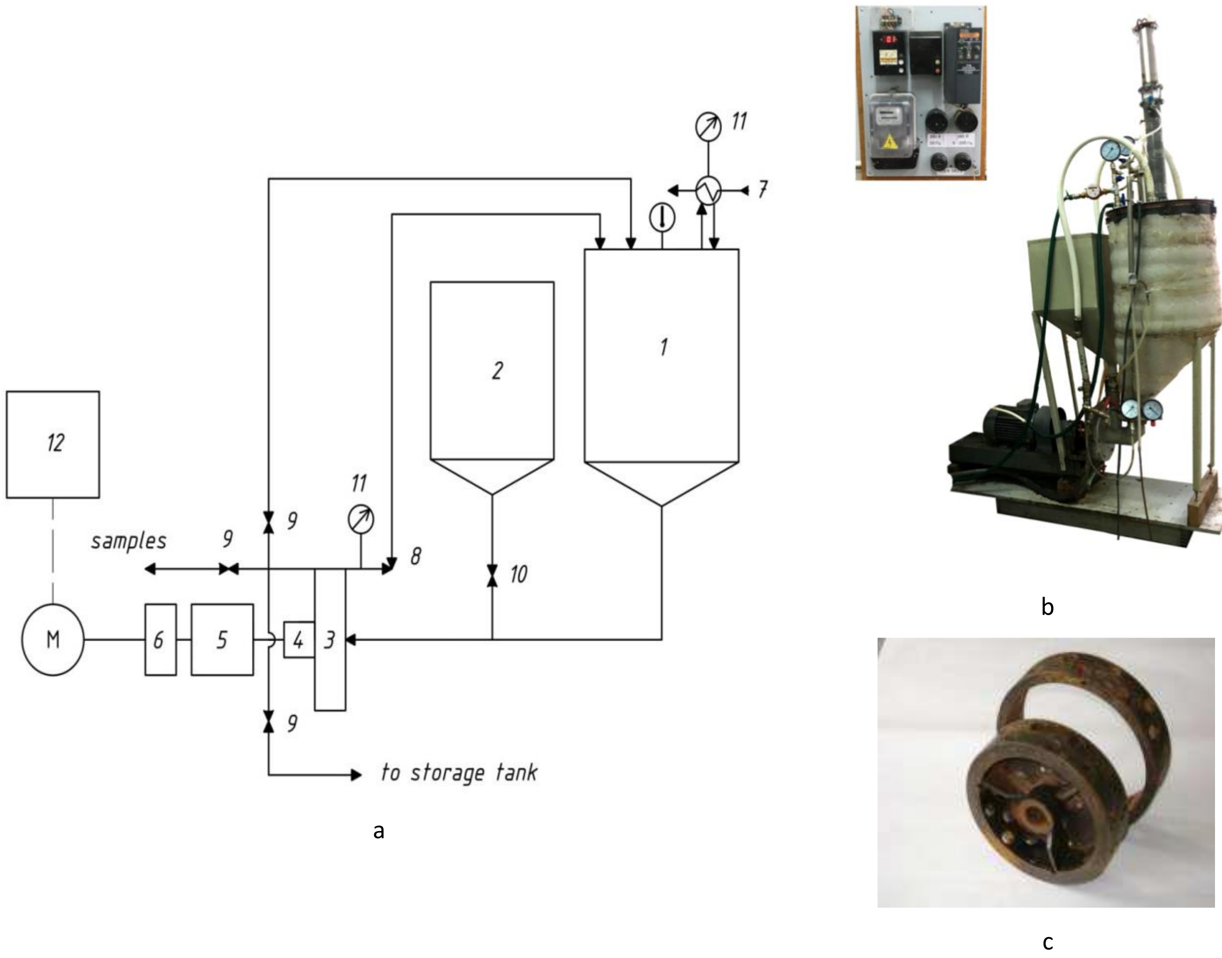


*Fig. 1. a) Schematic hydraulic diagram of the experimental setup: 1 – receiving tank; 2 – raw materials tank; 3 – reactor-mixer; 4 – stuffing unit; 5 – bearing unit; 6 – coupling; 7 – reflux condenser; 8 – corner ball valve; 9 – ball valve ½ ″; 10 – ball valve 1″; 11 – gauge; 12 – control unit; b) appearance of the setup; c) appearance of the rotor-pulsation unit*

The effect of temperature increase due to energy dissipation on the lignin extraction process is considered in Fig. 2. Pretreatment of wheat straw suspension in 1% (wt.) sodium hydroxide solution occurred within an hour.

In the first case, the process temperature was maintained constant by immersing the cooling coil into the receiving tank and setting the appropriate refrigerant flow rate. In the second case, heat removal from the processed raw material did not occur. The data shown in the figure indicate that over the entire temperature range, lignin extraction from plant raw materials increases when the temperature of the processing medium is not maintained constant. It should be noted that with increasing initial temperature, the difference between the amount of lignin extracted in both methods decreases from 38.5% at 40°C to 1.5% at 90°C.

Fig. 3 shows the dynamics of lignin extraction within an hour for an alkali concentration of 1% (w/w) in the temperature range of 40...90 °C.

It can be seen from the presented diagram that the degree of lignin extraction increases with time in the entire temperature range. The maximum lignin yield for 40 °C is 20.34%, for 90 °C is 9.71%. The extraction is most intense in the first 10 minutes, which is obviously due to the effect of alkali on the surface layers of lignocellulosic biomass.

Data obtained as a result of studies are comparable with the results of pretreatment of wheat straw in an autoclave, presented by Chen [34]. In particular, at a temperature of 60°C, 28.33% of lignin was extracted during treatment in an autoclave for 30 minutes, 34.01% during pretreatment in the RPA, and 31.1 and 41.9% in 60 minutes, respectively.

In contrast to Vancov et al., the authors of the work [34], after statistical analysis of data on lignin removal from corn stover during alkali pretreatment, concluded that the most influential parameter, compared to temperature and duration of treatment, is the alkali concentration.

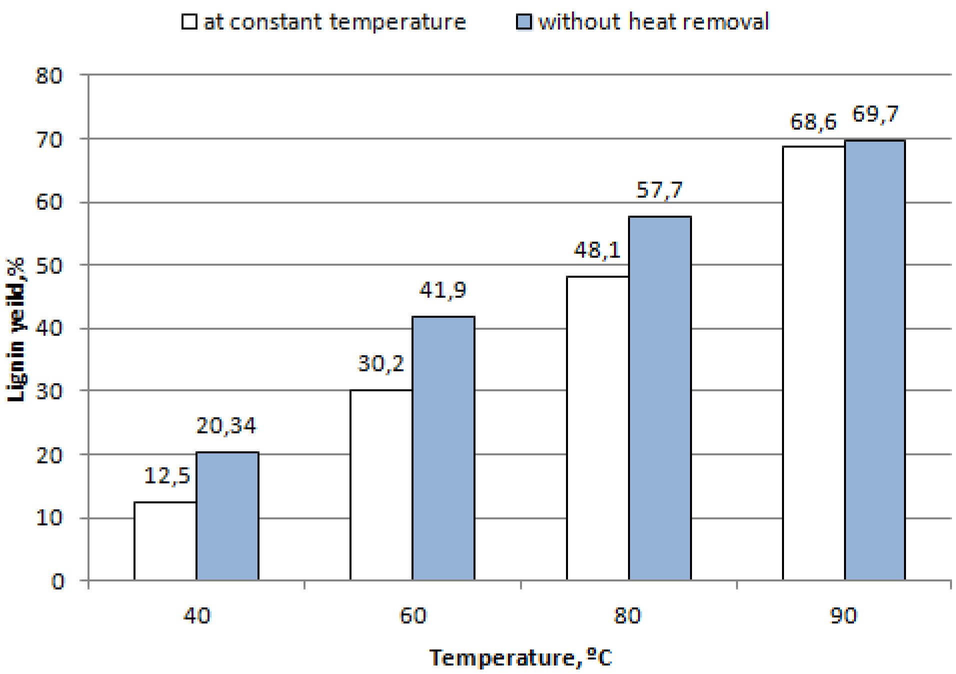


*Fig. 2. Lignin yield depending on the processing temperature mode*

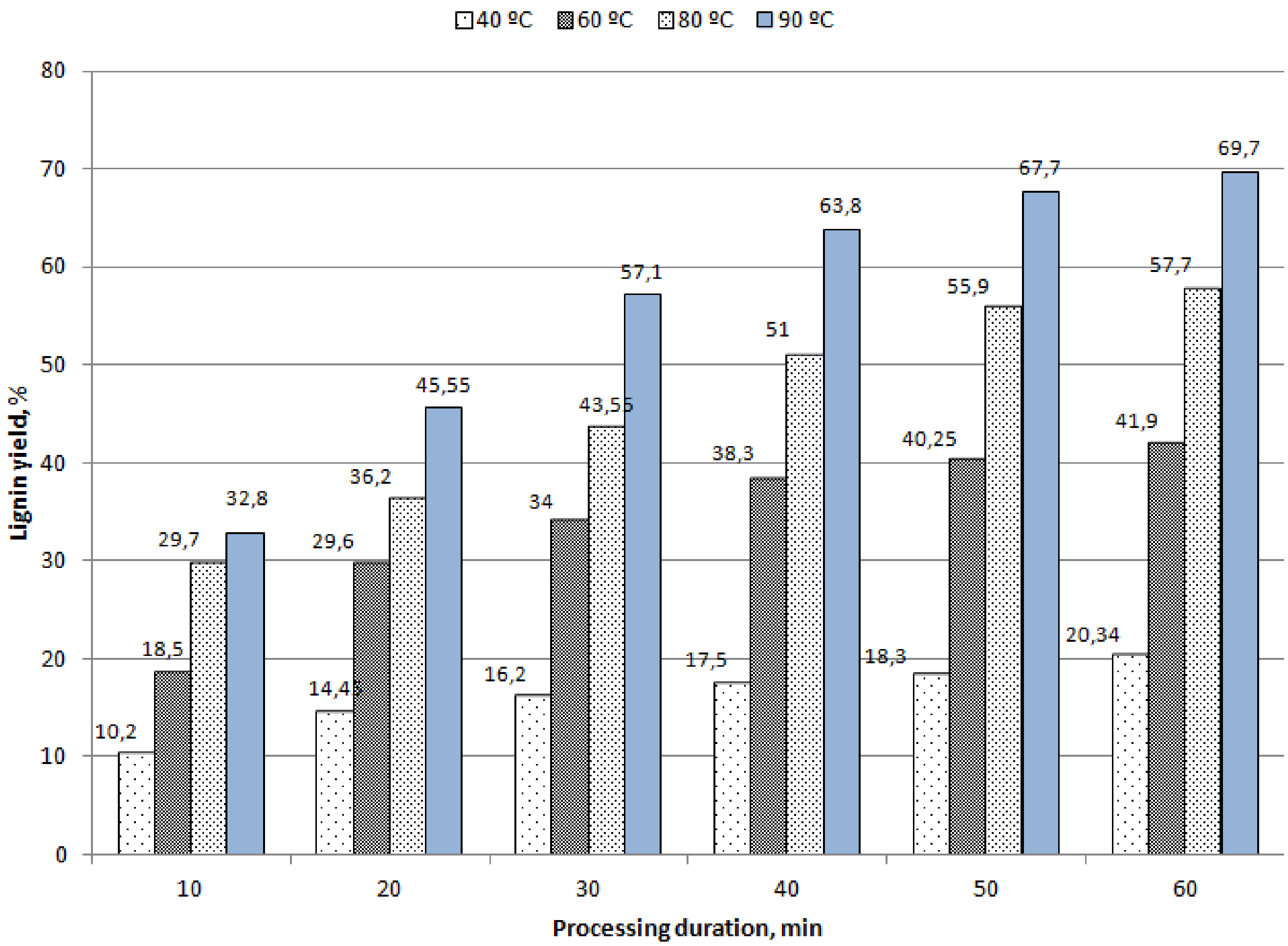


*Fig. 3. Dependence of lignin yield on the initial temperature of alkaline wheat straw suspension*

The effect of alkali concentration on the dynamics of lignin extraction from the alkali suspension of wheat straw is presented in Fig. 4. Lignin extraction took place for one hour at a temperature of 90 °C. The alkali concentration was varied from 1 to 4%wt.

Extraction of lignin from lignocellulosic biomass, in particular, wheat straw, increases with increasing alkali concentration, which is confirmed by a number of authors [7, 32, 35]. During pretreatment in the RPA, 69.7% of 1% wt alkali and 81.5% of 4% wt alkali were extracted per hour. The obtained values are comparable to the values obtained by autoclaving at a temperature of 121°C.  As in the case of increasing temperature, the fastest increase in the degree of lignin extraction with increasing alkali concentration occurs within the first 10 min.

The obtained results of alkaline pretreatment of plant raw materials for hydrolysis in a cylindrical-type RPA and their comparison with the results of similar studies using hydrodynamic cavitation generators of other types allow us to conclude that in addition to the physical parameters of impact, in particular the liquid/solid ratio, processing duration, temperature and alkali concentration, on the extraction of lignin from plant raw materials, additionally complex thermodynamic and hydrodynamic processes occurring in the working zone of the RPA, in particular high shear stresses, high turbulence, the formation of vortices in the inter-cylinder gap, as well as cavitation in the rotor-stator channels, have an impact on the extraction of lignin from plant raw materials.

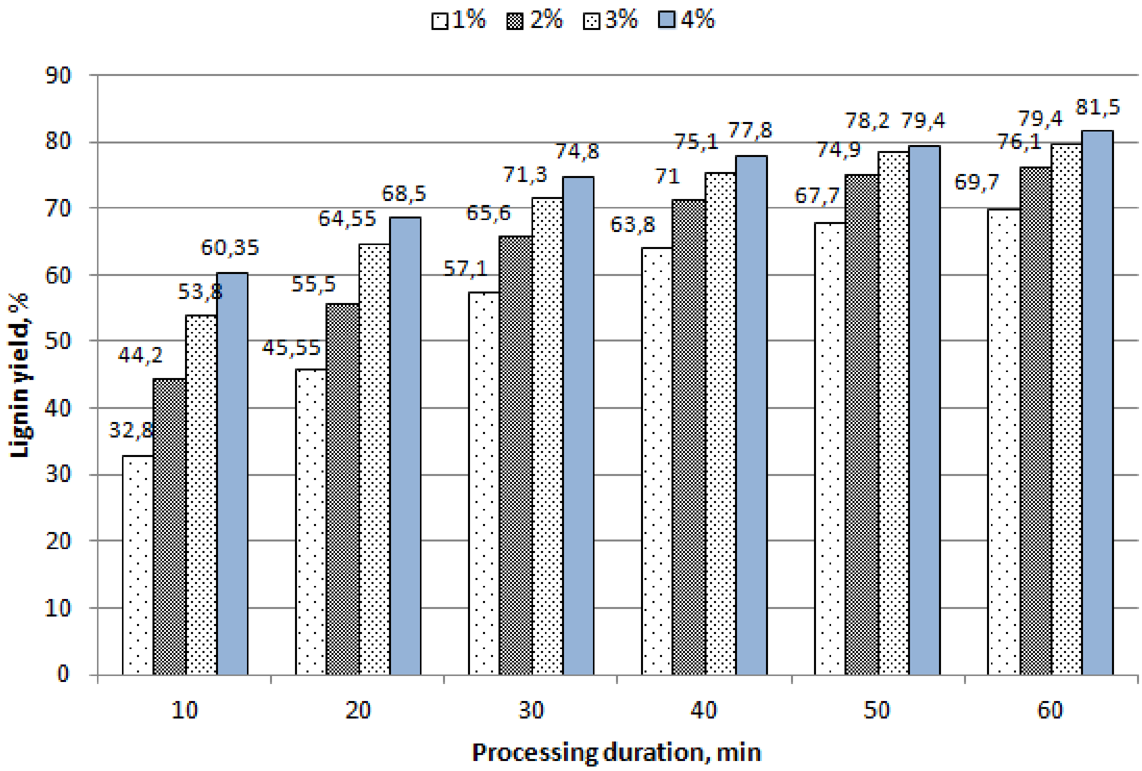


*Fig. 4. Dependence of lignin yield on alkali concentration*

The presented design of the experimental setup with the RPA enables high-performance processing of highly viscous liquid media, which include a suspension of wheat straw with particles of 0.1...0.4 mm with a solid/liquid ratio of 10% (w/w). The passage of the suspension through the rotor and stator channels is ensured by round holes in the rotor and stator, as well as a sufficiently wide (0.3 mm) inter-cylinder gap.

**Conclusion**

In a study on the use of an RPA during alkaline pretreatment of plant raw materials (wheat straw) with variable key parameters, namely temperature, process duration and alkali concentration for lignin extraction, the following results were obtained for the first time:

– increasing the initial temperature of the process from 40°C to 90°C with a further increase in temperature due to energy dissipation in the working area of the RPA for the lignin extraction at an alkali concentration of 1% within an hour leads to the extraction of 20.34 to 69.7% of lignin, respectively. Lignin extraction in the corresponding range of constant temperatures ranged from 12.5% to 68.6%;

– it was determined that increasing the duration of the pretreatment of wheat straw suspension for hydrolysis in an RPA from 10 min to an hour at an alkali concentration of 1% leads to an increase in the yield of lignin in the entire range of initial temperatures from 40°C to 90°C. The highest yield of lignin was 69.7% at a temperature of 90°C in one hour;

– increasing the alkali concentration from 1% to 4% also leads to an increase in the yield of lignin. The highest lignin yield was 81.5% per hour at an alkali concentration of 4%.

Thus, it was found that the impact of a complex of physical processes occurring in the RPA working area increases the yield of lignin compared to traditional processing at the same technological parameters of alkaline pretreatment of plant raw materials for hydrolysis. Comparing the obtained results of lignin extraction with similar results for other types of processing, it is possible to identify certain advantages, namely, the extraction process occurs at a temperature below 100°C and atmospheric pressure.

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