

## Discrete-pulse energy input for preretreatment of corn stalk in bioethanol production

Valerii Myronchuk<sup>1</sup>, Oleksandr Obodovych<sup>2</sup>, Olesya Stepanova<sup>2</sup>,  
Oleksandr Gavva<sup>1</sup>, Serhii Volodin<sup>1</sup>, Bogdan Tselen<sup>2</sup>

1 – National University of Food Technologies, Kyiv, Ukraine

2 – Institute of Engineering Thermophysics of NAS of Ukraine, Kyiv, Ukraine

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### Abstract

#### Keywords:

Bioethanol  
Corn  
Stalk  
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Cellulose  
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Rotor-pulsating  
unit

**Introduction.** The aim of the work is to determine the effect of discrete-pulse energy input on the reactivity of lignocellulosic biomass, in particular corn stalks, during their preliminary processing for the production of bioethanol fuel.

**Materials and methods.** The corn stalks were crushed in a disintegrator to particles no larger than 500 micrometers, poured with a 1% NaOH solution in a ratio of 1:10 and sent for processing in a rotary-pulsating unit. The treatment in the rotary-pulsating unit was carried out in recirculation mode for 1–70 minutes at a temperature of 50 °C.

**Results and discussion.** To increase the reactivity of lignocellulosic biomass during enzymatic conversion, preliminary treatment is necessary, the main task of which is to remove lignin and disrupt the structure of cellulose fibers. With a processing time of 70 minutes and a flow shear rate from  $20 \cdot 10^3$  to  $60 \cdot 10^3$  s<sup>-1</sup> at different pulsation frequencies, the reactivity (degree of conversion) of corn stalks increases to 61%. The main increase in reactivity from 10 to 61% occurs within 50 minutes at a shear rate of  $60 \cdot 10^3$  s<sup>-1</sup>. Further increase in treatment duration is ineffective. The amount of lignin in corn stalks without pretreatment was 15% of the dry matter content. During the 50-minute treatment at a pulsation frequency of 6 kHz and a flow shear rate of  $60 \cdot 10^3$  s<sup>-1</sup>, the content of lignin decreased from 15% to 1.3%. Increase in treatment time to 70 minutes at pulsation frequencies of 2 kHz, 4 kHz, and 6 kHz, the content of lignin decreased slightly and reached 1%. Therefore, increasing the treatment time from 50 to 70 minutes is impractical and leads to an increase in energy consumption by 10–15%. The results obtained are explained by the fact that with an increase in the rotor speed, there is an increase in the frequency of flow pulsations. When opening and closing the holes of the stator and rotor, sharp pressure fluctuations are observed, resulting in microcavitation, which leads to explosive autohydrolysis, while the average particle size of lignocellulosic biomass decreases and the total contact area of the phase increases, ensuring high reactivity.

**Conclusion.** The use of discrete-pulse energy supply and the proposed modes of preliminary processing of corn stalk biomass allows increasing its reactivity during enzymatic conversion to 61% compared to the known method of explosive autohydrolysis, which provides an increase in conversion in the presence of H<sub>2</sub>SO<sub>4</sub> by 6–21%, and in the presence of Ca(OH)<sub>2</sub> by 25%.

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#### Corresponding author:

Valerii  
Myronchuk  
E-mail:  
mironchukvg@  
ukr.net

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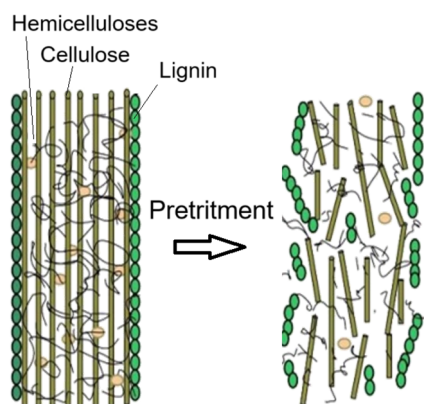
## Introduction

In recent years, there has been growing interest in the use of alternative fuels derived from plant biomass against the decline in crude oil production (Stabnikova et al., 2023a; Tran et al., 2019). Biofuels are considered a renewable energy source because they are produced from biomass, which can be replenished. One of the most common types of biofuel is bioethanol, which is produced from sugar- and starch-containing raw materials. A significant increase in bioethanol production, a reduction in its cost price and an increase in competitiveness are possible with the use of non-food raw materials, in particular lignocellulosic biomass, such as corn stalks (Shukla et al., 2023). The main components of lignocellulosic biomass are cellulose (23–50%), hemicellulose (12–29%), and lignin (13–31%) (Abolore et al., 2024; Tran et al., 2019).

Cellulose is a polysaccharide having a linear structure composed of d-glucose monosaccharides. The macromolecules of cellulose form fibrils. Hemicellulose has the form of a linear and branched heterogeneous polymer consisting of five saccharides – xylose, arabinose, mannose, galactose, and glucose. Lignin consists of phenylpropanoid alcohols, such as coniferyl, sinapyl, and coumaryl and contains different hydroxyl, methoxy, and carbonyl functional groups. The presence of lignin, due to its structure, significantly complicates the treatment of lignocellulosic biomass (Sheehan, 2001).

Lignin connects cellulose and hemicellulose, making the structure of lignocellulosic raw materials stronger and harder (Peciulyte et al., 2016). Lignocellulosic biomass is poorly suited for enzymatic degradation due to its low reactivity (Houfani et al., 2020). Cellulose is resistant to the action of enzymes due to its insolubility and strong structure, which is caused by its high crystallinity (Nitsos et al., 2019). Hemicelluloses in the form of lignocellulosic complexes have low reactivity (Karimi and Taherzadeh, 2016). Overall, the factors that contribute to the resistance of lignocellulosic biomass to enzyme action include a limited surface area available for enzyme interaction; a high degree of screening of cellulose by lignin; a high level of 'wrapping' of cellulose by hemicelluloses.

To increase the reactivity during the enzymatic conversion, pretreatment of lignocellulosic biomass is necessary to remove lignin and the breakdown of the structure of cellulose fibers. This is achieved by reducing the particle size of the raw material, increasing its porosity, and enhancing the surface area accessible to enzymatic action (Sun et al., 2014). Schematic of the breakage of the lignocellulosic material is shown in Figure 1 (adapted from Medeiros et al., 2023).



**Figure 1. Schematic of the structure of lignocellulosic material and its breakage during pretreatment**  
(adapted from Medeiros et al., 2023)

Corn is the most widely produced grain crop in Ukraine, and its production in 2023/2024 amounted to 31 million metric tons (Statista, 2024). It was estimated that approximately one ton of corn stover is generated for every ton of corn grain (Momayez et al., 2019), meanwhile stalks make up the largest part (from 32.9 to 39.4% of dry biomass) of corn stover (Berchem et al., 2017).

The cellulose content in corn stalks is within 40–50%, hemicellulose 20–30% and lignin 10–15% (Liang et al., 2023). The issue of recycling agricultural waste and turning it into useful products is receiving increased attention in Ukraine (Stabnikova et al., 2023b), and corn stalks can be an excellent raw material for producing biofuels, in particular bioethanol.

There are several methods for the pretreatment of lignocellulosic biomass: grinding (Hideno et al., 2009; Paudel et al., 2017); radiation methods (Kapoor et al., 2017; Yang et al., 2008); ultrasound (Bak et al., 2009); microwave radiation (Velmurugan and Muthukumar, 2011); acids (Mishra and Ghosh, 2019; Stabnikova et al., 2010); organosolv (Ostovareh et al., 2015); alkalis (Bali et al., 2015); ionic liquids (Karimi and Taherzadeh, 2016); explosive autohydrolysis (Alvira et al., 2014; Gogate et al., 2011; Putra, 2018); hydrothermal treatment (Putra, 2018); carbon dioxide explosive decompression (Kim and Hong, 2001; Gu et al., 2013), and biological treatment using enzymes (Dai et al., 2017; Rabemanolontsoa and Saka, 2016).

Methods for obtaining pure components from lignocellulosic biomass by its thermal treatment without the use of chemical reagents are effective and environmentally attractive. The aim of the present study is to determine the influence of discrete-pulse energy input on the reactivity of lignocellulosic biomass, specifically corn stalks, during their pretreatment for bioethanol fuel production.

## Materials and methods

### Materials

Corn stalks were used in the research. Lignin content and reactivity were determined in the original corn stalks and pre-treated ones. In both cases, the corn stalks were originally ground in a disintegrator to particles no larger than 500  $\mu\text{m}$ .

The reactivity was determined by hydrolysis of lignocellulosic biomass with a mixture of cellulase produced by *Penicillium verrucosum* B151 and cellobiase ( $\beta$ -glucosidase) produced by *P. verrucosum* F10 at 50 °C, pH 5.0 for 48 hours. The degree of conversion of lignocellulosic biomass into reducing saccharides because of enzymatic hydrolysis was taken as a criterion of reactivity.

### Methods

#### Pretreatment of corn stalks

Corn stalks ground in the disintegrator were mixed with 0.5% or 1% NaOH solution in a ratio of 1:10, and then processed in a rotary-pulsation apparatus. The processing was carried out in the recirculation mode for 10 to 70 min at a temperature of 50 °C. During the processing, the flow pulsation frequency was changed from 2 to 6 kHz and the flow displacement rate was changed within the range from  $20 \cdot 10^3$  to  $60 \cdot 10^3 \text{ s}^{-1}$ . The flow pulsation frequency and the flow displacement rate were regulated by the rotation speed of the rotor of the rotary-pulsation apparatus and the size of the gap between the stator and the rotors.

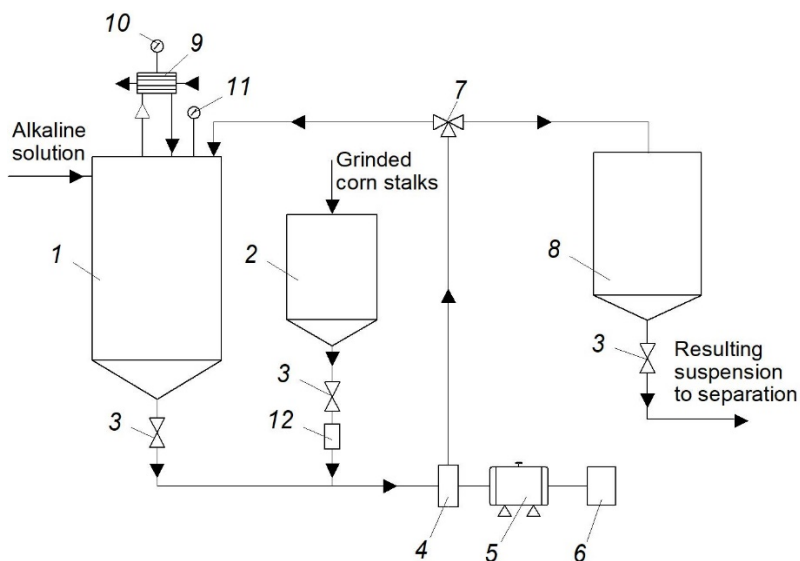
**Determination of lignin content.** Two samples of liquid from the mixture, 50 ml each, were taken before the start of treatment and every 10 minutes of mixture treatment. Sulfuric acid was added to one sample until pH 2 was reached to precipitate lignin, separate it from other components and stabilize the system after alkaline hydrolysis. Then the resulting

suspension was filtered through pre-weighed filter paper. The filter with the lignin sediment was dried and weighed on an Axis AN200 analytical balance with an accuracy of 0.0001 g. The mass of lignin was determined as the difference between the mass of the dried filter with sediment and the mass of the original filter.

Another sample was used to determine the total dry matter content of the solution according to the generally accepted method (drying the samples to a stable mass at a temperature of  $103 \pm 2$  °C (ISO 24196, 2022)). The lignin content was determined in% as the ratio of the mass of the obtained lignin to the mass of dry matter in each of the studied samples.

The Laff-Schurl method was used to determine reducing sugars (Ibrahima et al., 2020).

**Pretreatment of lignocellulosic raw materials.** The pretreatment of lignocellulosic raw material for hydrolysis was conducted at a pilot plant shown in Figure 2, which operates on the principle of discrete-pulse energy input.



**Figure 2. Schematic of pilot plant of discrete-pulse energy input:**

**1 – receiving tank; 2 – tank for raw materials; 3 – two-way valve; 4 – rotary-pulsation apparatus; 5 – electric motor; 6 – control unit; 7 – three-way valve; 8 – tank for pre-prepared raw materials; 9 – dephlegmator; 10 – pressure gauge; 11 – safety valve; 12 – vibratory batcher**

The pilot plant (Figure 2) consists of a receiving tank (1); a tank for raw materials (corn stalks) (2); a rotary pulsation apparatus (4); an electric motor (5); a container for pre-prepared raw materials (8); water supply and power supply systems, shut-off and control valves (3, 7); power supply systems and control devices (10).

The receiving tank with a useful volume of 200 l is designed to store plant biomass during processing. The tank is equipped with a heat exchange jacket with a volume of 27 l to maintain the required process temperature during processing. The tank lid is equipped with a pressure gauge and a safety valve. The jacket is equipped with a 5-liter flow-through water heater, an open-loop circulation pump with a capacity of 8 l/min with a controller for adjusting the temperature of the coolant heating from 0 to 95 °C. The heating element power is 2 kW.

The pilot plant operates as follows. The receiving tank (1) and rotary-pulsation device (4) are filled with alkaline solution. Corn stalks after mechanical grinding on the disintegrator are loaded into the container for raw materials (2), and the motor of the pump unit of the flow-through water heater is turned on.

After reaching the set temperature of the solution, the two-way valve (3) on the inlet pipe is opened and the vibratory batcher (12) is started. The alkaline suspension of grounded corn stalk biomass is circulated for the set processing time. When processing the lignocellulose complex, lignin and hemicellulose pass into solution. The resulting suspension is fed to a device for separating the solid phase (cellulose) from the liquid (lignin and hemicellulose) one. Phase separation can occur through filtration, ultrafiltration, membrane technologies, and separation.

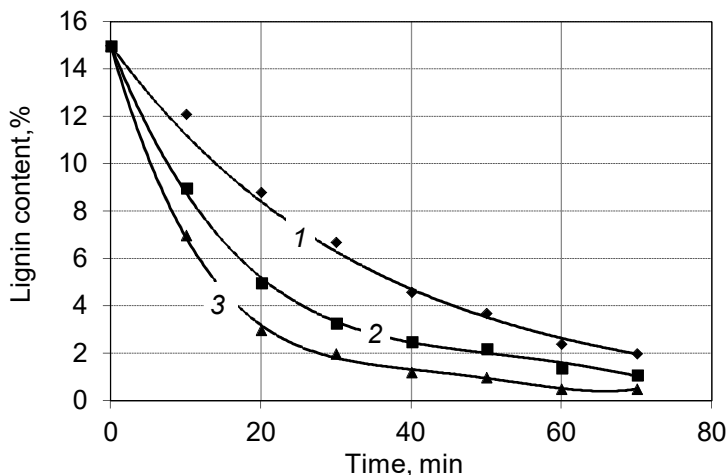
## Results and discussion

Compared to explosive autohydrolysis in the presence of acids and alkalis, the proposed method offers two key advantages: (a) it increases the reactivity without a significant rise in temperature (remaining below 50 °C), and (b) it intensifies the pretreatment process of corn stalks by generating uniformly distributed vapor–gas bubbles throughout the dispersed medium. Upon collapse, these bubbles convert stationary thermal, mechanical, or other energy forms within the system into powerful, discrete energy pulses in both time and space. The shock waves accompanying these phenomena, interphase turbulence, microcavitation, vortices, and penetrating cumulative microjets cause instabilities of the Rayleigh-Taylor or Kelvin-Helmholtz type at interphase surfaces, leading to intense fragmentation of disperse inclusions, significant increase in the total contact surface area of the phases, and enhancement of mass and heat transfer processes (Dolynskiy and Ivanyskiy, 2008; Dolinsky et al., 2012).

Results (Figures 3–6) were obtained in a pilot plant that enabled the production of a dispersed phase with an average particle size of lignocellulosic biomass ranging from 50 to 100 micrometers. The total surface area of contact between the phases of the dispersed system and the intensity of biomass conversion of corn stalks depend on the size of these particles. Pretreatment, after the disintegrator, of the original corn stalks using the method of discreet-pulse energy input through a rotor-pulsation apparatus for 50 minutes does not significantly reduce the size of the disperse phase particles. As a result, the reactivity does not increase, and the lignin content does not decrease.

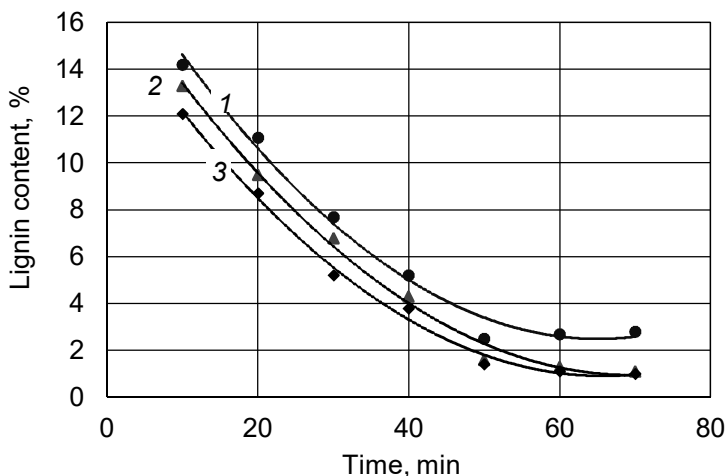
### Effect of treatment regimes on lignin content in corn stalks

At the first stage, the lignin content in corn stalks was determined depending on the operating parameters of their processing in a rotary-pulsation apparatus. The lignin content was determined depending on the duration of processing of corn stalks in a rotary pulsation apparatus of a heat and mass transfer unit at a pulsation frequency of 2 to 6 kHz for 0 to 70 min (Figure 3). The content of lignin in the original corn stalks was 15% of the dry matter (d.m.). With an increase in treatment duration to 70 minutes and a pulsation frequency ranging from 2 to 6 kHz, the lignin content decreased from 15% to 1.0%. During the treatment from 0 to 50 min. at a flow pulsation frequency of 6 kHz, the lignin concentration decreased from 15 to 1.3%. With an increase in the treatment duration to 70 min., the lignin concentration remained virtually unchanged and reached 1%. Finishing from 50 to 70 min. reduces the lignin content by only 0.3%, but leads to an increase in energy consumption by 10-15%.



**Figure 3.** Effect of treatment duration and flow pulsation frequency on lignin content in corn stalks. Pulsation frequency: 1 – 2 kHz, 2 – 4 kHz, 3 – 6 kHz

Dependence of the lignin content in corn stalks from the duration of treatment and the flow rate in the rotary pulsation apparatus are shown in Figure 4.

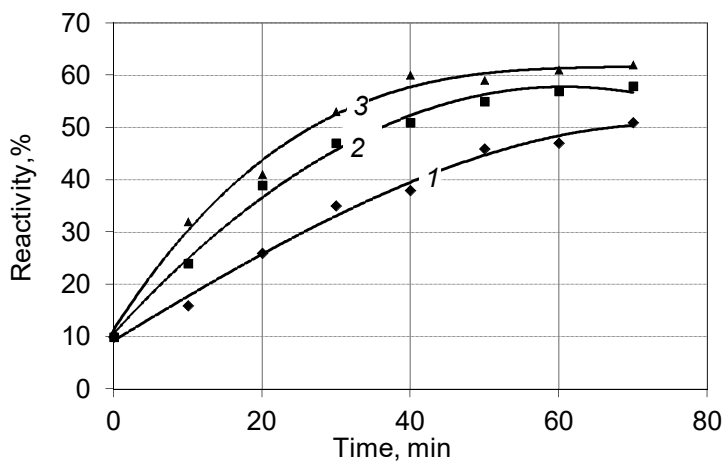


**Figure 4.** Effect of treatment duration and shear rate on lignin content in corn stalks. Shear rate: 1 –  $20 \cdot 10^3 \text{ s}^{-1}$ ; 2 –  $40 \cdot 10^3 \text{ s}^{-1}$ ;  $60 \cdot 10^3 \text{ s}^{-1}$

Increasing the treatment duration from 0 to 70 minutes and raising the shear rate from  $20 \cdot 10^3$  to  $60 \cdot 10^3 \text{ s}^{-1}$  leads to a reduction in lignin content from 15.0% to 1.0% from dry matter. The delignification process occurs most intensively over 50 minutes at a shear rate of  $60 \cdot 10^3 \text{ s}^{-1}$ , with a reduction of 1.3%. Extending the process duration from 50 to 70 minutes allows for a further decrease in lignin content by 0.3%, but results in a 10-15% increase in energy consumption.

### Effect of treatment regimes on reactivity (degree of conversion) of corn stalks

At the second stage, studies were conducted to determine the reactivity of corn stalks depending on the process parameters of their processing in a rotary pulsation apparatus (Figure 5).

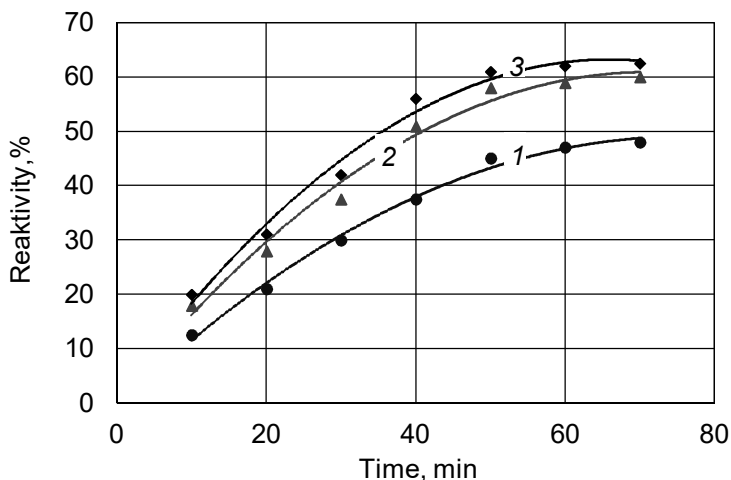


**Figure 5. Effect of treatment duration and flow pulsation frequency on reactivity (degree of conversion) of corn stalks.**  
Pulsation frequency: 1 – 2 kHz, 2 – 4 kHz, 3 – 6 kHz

The highest reactivity of 63% is achieved with a pulsation frequency in the rotary pulsation apparatus of 6 kHz and a processing duration of 70 min. The main increase in reactivity from 10% to 61% is observed in the time interval from 0 to 50 min. A further increase in the processing time from 50 to 70 min has little effect on the reactivity of corn stalks. Under these conditions, the lignin content decreased from 15% to 1% of the dry matter content in corn stalks. At the same time, with a processing time of 50 min, the lignin content decreased to 1.3%. Therefore, processing of corn stalk biomass in the range from 50 to 70 min reduces the lignin content by only 0.3%, while the energy consumption for the process increases by 10-15%.

Dependence of corn stalk reactivity on the duration of processing and the shear rate of the flow in the rotary-pulsation apparatus is shown in Figure 6.

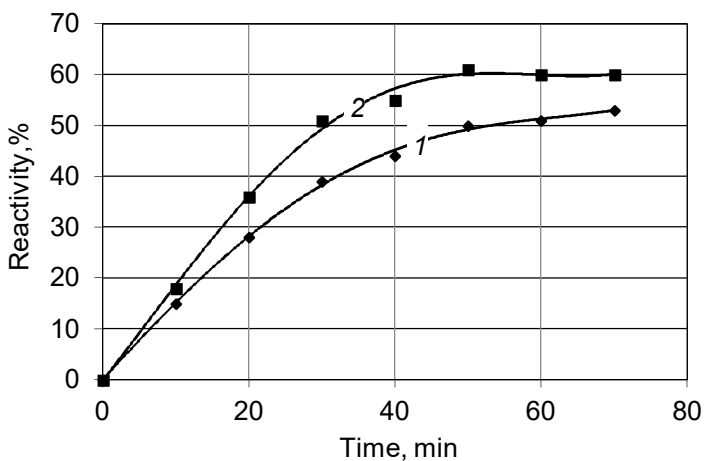
With an increase in the duration of treatment from 0 to 70 minutes and an increase in the shear rate from  $20 \cdot 10^3$  to  $60 \cdot 10^3 \text{ s}^{-1}$ , the reactivity reaches 61%. Among the investigated parameters, the most effective treatment of corn stalks in the rotor-pulsating apparatus is carried out for 50 minutes at a shear rate of  $60 \cdot 10^3 \text{ s}^{-1}$ . Under these treatment conditions, the reactivity increases to 61%. An increase in the treatment duration from 50 to 70 minutes results in a 10 – 15% increase in energy costs, while the reactivity only increases by 2%, which is economically unfeasible.



**Figure 6.** Effect of treatment duration and flow shear rate on the reactivity (degree of conversion) of corn stalks.

Shear rate: 1 –  $20 \cdot 10^3 \text{ s}^{-1}$ ; 2 –  $40 \cdot 10^3 \text{ s}^{-1}$ ; 60 ·  $10^3 \text{ s}^{-1}$

Dependence of the reactivity (degree of conversion) of corn stalks on the duration of processing in a rotary pulsation apparatus in the presence of NaOH solution (0.5% or 1.0%) at a pulsation frequency of 6 kHz is shown in Figure 7.



**Figure 7.** Effect of the duration of treatment and concentration of NaOH on the reactivity (conversion rate) of corn stalks.

1 – 0.5% NaOH; 2 – 1.0% NaOH.

Increasing the NaOH concentration to 1.0% with a treatment duration of 50 minutes leads to an increase in reactivity to 61%. Further increase in treatment duration does not lead in an increase in reactivity.

**Comparison of reactivity (degree of conversion) of original corn stalks pretreated by various methods**

The results of the researches on pretreated corn stalks using the method of discrete-pulse energy input in the presence of a 1% NaOH solution were compared with the results (according to literary sources) obtained from treatment through explosive autohydrolysis. The reactivity, i.e., the conversion degree of the original corn stalks after disintegration, is 10%, while those pretreated through explosive autohydrolysis in the presence of H<sub>2</sub>SO<sub>4</sub> range from 40% to 55%, and in the presence of Ca(OH)<sub>2</sub> reach 36% (Chen et al., 2023; Shukla et al., 2023).

Comparative characteristics of the reactivity (conversion degree) of original corn stalks and those pretreated through various methods for 50 minutes at a temperature of 50 °C, at a pulsation frequency of 6 kHz and a shear rate of 60·10<sup>3</sup> s<sup>-1</sup> are presented in Table 1.

**Table 1**  
**Reactivity (degree of conversion) of original corn stalks pretreated by various methods**

Indicator	Original corn stalks ground in disintegrator	Pretreatment method		
		Explosive autohydrolysis in presence of H <sub>2</sub> SO <sub>4</sub>	Explosive autohydrolysis in presence of Ca(OH) <sub>2</sub>	Discrete-pulse energy input method in presence of NaOH
Reactivity,%	10	40–55	36	61

The pretreatment of corn stalk biomass using the discrete-pulse energy input method in a rotor-pulsation apparatus under the specified conditions resulted in the highest reactivity, reaching 61%. This is 6–21% higher than that achieved through explosive autohydrolysis in the presence of H<sub>2</sub>SO<sub>4</sub>, and 25% higher than when using Ca(OH)<sub>2</sub>. This is explained by the fact that as the rotor speed increases, the frequency of opening and closing of the stator and rotor holes increases, i.e., the frequency of flow pulsations, which is defined as the product of the angular speed of the rotor's rotation and the number of stator holes. During the opening and closing of the stator and rotor holes, sharp pressure drops occur, which means microcavitation, resulting in explosive autohydrolysis. The frequency of explosive autohydrolysis occurring in the rotor-pulsation apparatus essentially corresponds to the pulsation frequency (Dolinsky et al., 2012; Myronchuk et al., 2019).

**Conclusions**

1. The conversion of lignocellulosic biomass, particularly corn stalks, is not very productive without pretreatment, i.e., separation into cellulose, hemicellulose, and lignin. The most effective method currently available for the conversion of lignocellulosic biomass in bioethanol production technology is explosive autohydrolysis in the presence of acids or bases, which allows for a maximum increase in the reactivity of corn stalks from 10 to 55%.

2. The use of discrete-pulse energy input method in a rotor-pulsation apparatus at a temperature of 50 °C and a treatment duration of 50 minutes enables an increase in reactivity to 61%. For this purpose, pre-ground biomass of corn stalks should be treated to a size of no more than 500 microns in a rotor-pulsation apparatus at a pulsation frequency of 6 kHz and a flow shear rate of  $60 \cdot 10^3 \text{ s}^{-1}$ . Meanwhile, the lignin content in the corn stalk biomass is reduced from 15% (original) to 1.3%.
3. The developed equipment for discrete-pulse energy input and the proposed technological modes of treatment enable an increase in the reactivity (conversion degree) of corn stalk biomass compared to explosive autohydrolysis in the presence of  $\text{H}_2\text{SO}_4$  by 6 to 21% and in the presence of  $\text{Ca}(\text{OH})_2$  by 25%.

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