

## NEWS

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**B. Sh. Kedelbaev<sup>1</sup>, K. M. Lakhanova<sup>2</sup>, Zh. B. Makhatov<sup>1</sup>**<sup>1</sup>M. Auezov South Kazakhstan State University, Shymkent, Kazakhstan,<sup>2</sup>Yassawi International Kazakh-Turkish University, Turkestan, Kazakhstan.E-mail: *kulzada.lakhanova@iktu.kz kulzada56@mail.ru***STUDY OF THE PROCESS OF SORBITOL PRODUCTION  
FROM WHEAT STRAW**

**Abstract.** The article presents the results of the study of the enzymatic combined (hybrid) hydrolysis-hydrogenation process for the production of sorbitol. This article describes the results of the pre-treatment and enzymatic hydrolysis of wheat straw, as well as the synthesis of sorbitol in order to increase the yield of valuable products needed for industry. Pretreatment of straw was carried out in the temperature range 190-250°C with a variation in the concentration of sulfuric acid from 0.6 to 2.5% by weight. The optimum temperature and duration of hydrolysis of straw with sulfuric acid were 160°C - 170°C and 30-80 minutes, respectively. In all straw hydrolysis experiments, the best results were achieved with a sulfuric acid concentration of 1.6% by weight and an experimental temperature of 150-160°C. Studies of the processes of enzymatic hydrolysis of straw have been carried out. Since the paper is practically pure cellulose, then, with the same kinetic parameters of the fermentolysis processes, the amount of formed sugars in the case of fermentolysis of straw would be 1.49 times less than when using paper. The resulting purified glucose hydrolyzate was subjected to hydrogenation in the presence of a nickel-aluminum-iron-chromium catalyst. The catalyst of the alloy with 7.0% chromium exhibits the greatest activity, the yield of sorbitol at which at 100°C and 6MPa for 60 minutes of hydrogenation is 50.5%, and the rate of hydrogenation of glucose is 1.46 times higher than for skeleton nickel without an additive. It is shown that with increasing hydrogen pressure from 2 to 12 MPa and 40-120°C, the rate of hydrogenation of glucose on titanium-promoted nickel-aluminum-iron catalysts increases.

**Key words:** wheat straw, sorbitol, cellulose, catalyst, enzymatic hydrolysis, biomass.

The growing interest in the use of plant biomass, rich in polysaccharides, determines the search for optimal methods for its processing. The main criterion in waste processing is their cost, volume, availability and localization, as well as chemical composition and technological properties. The processing of renewable plant materials in industrial-important substances is of great practical interest.

Cellulose-containing raw materials in the country are available in almost unlimited quantities in the form of wood, straw, solid household waste, etc. However, its effective conversion into biologically digestible sugars is a complex task, over which scientific collectives all over the world work. In this case, the possibilities of using directly microorganisms, complexes of cellulolytic enzymes, chemical hydrolyzing agents for the effective conversion of non-food raw materials into digestible sugars are explored.

According to the statistic data, wheat and cotton are the leaders in crop yields in the Republic of Kazakhstan. Gross harvest of wheat in 2011 amounted to 22.7 million tons, while in the fields of agricultural enterprises an average of 11.1 million tons of wheat straw was formed, only 10 percent of which is used for feeding livestock and as litter to animals, the rest of it is plowed up into the ground and burned in the fields. Thus, cereal straw is a large-capacity, affordable and promising secondary resource of agricultural production in the Republic of Kazakhstan. One of the cheapest and available types of cellulose-containing raw materials for large-tonnage production of energy carriers, in particular fuel alcohol, is straw. This is a huge potential raw material base. Finally, the existing agricultural infrastructure

makes it possible to solve the problem of delivering straw for processing, provided that the processing enterprise is located near the elevator [1].

Given the low degree of effective use of straw cereals at present, the main attention is paid to the problem of saccharification of this type of secondary raw materials. The plant cell wall has a high resistance to degradation. Fungi and bacteria that use cellulose as a source of carbon have developed a complex set of enzymes that hydrolyse cellulose, releasing glucose monomers. Enzymatic hydrolysis is a promising method of processing plant biomass. However, during the enzymatic hydrolysis of ligno-cellulosic materials in their native form, the yield of sugars reaches less than 20% of the theoretically possible yield. Overcoming the physico-chemical barriers that hamper the availability of cellulose for enzymes is an important issue, the solution of which is directly related to the search for low-cost pre-treatment methods for raw materials. The effectiveness of this process determines the yield of the target product in the process of enzymatic hydrolysis of cellulose and the economic feasibility of the entire technology as a whole. Despite the relatively high catalytic activity of sulfuric, hydrochloric and phosphoric acids, their use in the hydrolysis of lignocellulose is still not economically effective, since they have a strong corrosive activity, their cost is high, and the neutralization of their excess in hydrolyzates is associated with costs and environmental stress. Perspective is the use of sulfuric acid, which allows to reduce the consumption of hydrolyzing agent due to its recovery. In this regard, the determination of optimal pre-treatment regimes using sulfuric acid, as well as the study of the effect of its conditions on the efficiency of enzymatic hydrolysis of wheat straw, is an urgent task. The development of complex processing of wheat straw will not only improve the ecological situation, but also will provide raw materials and additional products for the industry.

The development of complex processing of carbohydrate-containing plant raw materials and waste will allow not only to improve the ecological situation, but also to obtain raw materials and additional products for the chemical industry and biotechnological productions. Thus, the processing of renewable carbohydrate-containing plant material and waste into industrially important chemicals is of great practical interest

Of particular interest is the search for catalytic technologies of a one-stage, combined (hybrid) process for obtaining valuable substances directly from this raw material, excluding the technological stages of separation and purification of intermediates. One-stage organization of the process makes it possible to obtain from the plant polysaccharide by the hydrolysis-hydrogenation reaction of compounds such as xylitol and sorbitol. Intensive research is being carried out in this direction in the world. A wide range of catalytic systems for the hydrolytic transformations of a renewable polysaccharide are proposed, for example, catalytic systems based on carbon, oxides, zeolites, ion exchange resins. Various technological schemes for the implementation of such processes have been developed, various variations of the methods for similar processing of polysaccharides of carbohydrate-containing plant material and waste are proposed.

However, the literature data published so far on the use of various catalytic systems in the conversion of these polysaccharides often contradict each other, and the catalysts proposed exhibit little activity or stability. It should be noted that most researchers have studied exclusively the hydrolysis of polysaccharides into glucose, and the number of works devoted to the possibility of obtaining, for example, from wheat straw of xylitol and sorbitol as a result of combined (hybrid) process processes is extremely small. Although, these compounds are substances that are highly demanded in many modern branches of the chemical, food, perfume, medical, and other industries. In particular, sorbitol and xylitol are widely used in the production of surfactants, synthetic resins, varnishes, drying oil, vitamin C, food products, etc.

To date, a fairly large number of experimental data on the hydrolysis of cellulose to glucose has been obtained, but information on single-stage hybrid processes for the production of sorbitol and xylitol is not enough, and the results described in the literature often do not agree with each other.

Analysis of the literature [2-11] on the topic of the work showed that in the field under study there are a number of unresolved problems, the search for solutions of which seems useful for the creation of effective catalytic technologies for the conversion of carbohydrate-containing plant material and waste into valuable chemicals.

In [12], a method is proposed that pertains to genetic engineering and can be used in the microbiological industry. Arabidopsis producing yeasts or fungi transform the DNA encoding the D-xylose-forming D-arabitol dehydrogenase and DNA encoding xylitol dehydrogenase. Then, transformed yeast or fungi under conditions that ensure the synthesis of xylitol. Xylitol is removed. Yeast is selected from *Lygosaccharomycesrouxii*, *Candidapolymorpha*, *Torulopsiscandida*, *Pichiafarinosa*, *Torulasporahansenii*. Mushrooms are selected from *Dendryphiellasalina* and *Schizophyllumcommune*. The method makes it possible to convert readily available sources of carbon, such as D-glucose to xylitol.

In [13], cellulose and hemicellulose in biomass are first decrystallized first with concentrated sulfuric acid and subjected to the first hydrolysis, resulting in a hydrolyzate saturated with sugar and acid. After that, the silicon oxides contained in the biomass are removed and sent to the processing. Then, the remaining solid residues are subjected to repeated decrystallization and re-hydrolysis. The resulting sugar solution is thereafter subjected to fermentation, using for this purpose microorganisms, preferably yeast and bacteria, providing fermentation of both hexoses and pentoses simultaneously. The method makes it possible to obtain sugars from biomass containing cellulose and hemicellulose, which is economically efficient and does not pollute the environment.

In [14] describes a process for pretreating a cellulose-containing feedstock for enzymatic hydrolysis, which involves preparing a feed slurry and treating it with an acid solution. A solution of nitric acid with a concentration of 2,8-6,5% is used as the acid solution. The solution is heated to a temperature of 98-100 °C and held for 1-5 hours. The product obtained is filtered, washed. And as a cellulose-containing raw material use miscanthus or fruit shells of cereals. The acid solution worked out after one cycle is strengthened with concentrated nitric acid to a solution concentration of 2,8-6,5% and repeatedly used again. The solution spent 18-25 cycles is neutralized with ammonia to obtain a solution of ammonium nitrate for use as a fertilizer.

A method related to the microbiological as well as the food industry and which can be used in the disposal of waste containing cellulose is described in [15]. The method involves pretreating cellulose-containing raw materials from which barley or rice grains are used, or cotton linters, or wheat straw to break down the crystal structure and increase its specific surface area. This treatment is carried out by extrusion. The obtained extrudate is subjected to enzymatic hydrolysis with enzymatic preparations of Asp. mix B 2000 or B1mix or celloviridine G20x at a mass ratio of the enzyme: extrudate 1-3: 1-9, respectively. The proposed method is non-waste, environmentally friendly and inexpensive, characterized by a high degree of bioconversion of raw materials with a significant reduction in the time required for its implementation.

A method was developed [16] comprising the step of hydrolyzing a cellulose-containing biomass to obtain an aqueous sugar solution. The resulting aqueous sugar solution is then filtered through a nanofiltration membrane and/or a reverse osmosis membrane. The purified sugar solution is collected from the inlet side and the fermentation inhibiting substances are removed from the filtrate side. These fermentation inhibiting substances are one or more compounds from organic acids, furan compounds and phenolic compounds. The invention makes it possible to obtain a sugar syrup purified from the fermentation-inhibiting compounds in a simple manner and to increase the efficiency of the fermentation of various chemical products.

Silveira et al [17] compared the activity of 6 strains of *Zymomonasmobilis* with respect to D-sorbitol and gluconic acid.

The strain of *Zymomonasmobilis* ATSS-29191 compared to others showed relatively good growth and high yield of products. For this strain, the effect of the initial concentration of substrates on the yield of D-sorbitol and gluconic acid in the batch process was investigated [18]. The equimolar concentrations of glucose and fructose ranged from 100 to 750 g / l. Increase in productivity occurred at an increase in the initial concentration of the substrate to 650 g / l. At 750 g / l, a drop in productivity was observed.

It was also found that with an initial concentration of 100 g / L, glucose and fructose were equally converted to sorbitol and gluconic acid, especially during the initial reaction period. However, after 20 minutes there was a shift in the metabolism of sugars, characterized by: an increase in glucose consumption, a strong decrease in fructose intake, a decrease in the yield of sorbitol, and a decrease in the concentration of gluconic acid. An increase in the concentration of substrates to 600 g / l led to an almost complete conversion of sugars to sorbitol and gluconic acid.

This article describes the results of the pre-treatment and enzymatic hydrolysis of wheat straw, as well as the synthesis of sorbitol in order to increase the yield of valuable products needed for industry. To develop the processing technology, wheat straw was formed, formed as waste in the agricultural sector of the Republic of Kazakhstan. Previously investigated plant raw materials were crushed and sorted. The individual monosaccharides in the hydrolysates were analyzed by paper chromatography using Filtrak FN-3, 11 and 14 in butanol-acetic acid-water solvents (4: 1: 5). The substances were detected by spraying first with a first developer of K<sub>2</sub>SO<sub>4</sub>, then a mixture of benzidine, acetone and hydrochloric acid in a ratio of 10: 2: 1. Alloys were prepared in the high-frequency melting furnace of the brand according to the technology developed by us. A calculated amount of aluminum was placed in a quartz crucible and gradually heated to 100-110°C, then the calculated amount of nickel, iron and titanium was introduced. As a result of the exothermic reaction, the temperature of the melt rose to 170-180°C, which was mixed for 3-5 minutes by an induction field. In graphite molds, the alloy was cooled in air and ground to 0.25 mm grains. Activation of the alloys was carried out by leaching with 10 aqueous solution of caustic sodium, taken in a volume of 40 cm<sup>3</sup> per 1 g of alloy in a boiling water bath for 1 hour, after which the catalyst was washed from alkali with water until neutral reaction with phenolphthalein. The catalysts thus obtained were used for the hydrogenation of glucose. For the rapid identification of the optimum catalyst and the study of kinetic regularities, the experiments were initially carried out in a modified batch reactor. The device is equipped with a hermetic drive with a power of 0,6 kW, the speed of rotation of the stirrer is 2800 rpm, which allows to remove the diffusion complications of the reaction.

Pretreatment of straw was carried out in the temperature range 190-250°C with a variation in the concentration of sulfuric acid from 0.6 to 2.5% by weight. Increasing the temperature to a greater extent, compared with the increase in the concentration of acid, contributed to a reduction in the processing time required to achieve the maximum yield of reducing substances (RS). more than decomposition of monosaccharides. The yield of monosaccharides therefore increases with the reaction temperature. The influence of the concentration of sulfuric acid at temperatures below 150 ° C is noticeable, but with increasing temperature to 160 ° C it disappears.

The optimum temperature and duration of hydrolysis of straw with sulfuric acid were 160°C - 170°C and 30-80 minutes, respectively. As the concentration of sulfuric acid increases, the rate of decomposition of sugars increases. The optimum concentration of sulfuric acid is 1.77% by weight. Pretreatment of straw with a variation of the hydromodule from 1: 3 to 1: 5 was carried out under conditions of 1.6% by weight. sulfuric acid and at a temperature of 150 ° C. The highest yield of RS was achieved with the hydromodule 1: 3.5, 1: 5 and 1: 5.8 and amounted to 26.8%, 27.0% and 29.2% respectively. The monosaccharide composition of the hydrolysates was mainly represented by glucose, the concentration of which reached 25 g / l in the hydrolysates.

Thus, the treatment of straw is 1.35% by weight. sulphurous acid at a temperature of 150°C, a hydromodule of 1:3 for 60 min allows to obtain hydrolysates with a concentration of reducing substances up to 7.6%, which will promote their further use in the microbiological industry. When using the hydromodule 1: 4.5, the maximum concentration of reducing substances in the hydrolyzate is reached at a temperature of 160°C, sulfuric acid concentration of 1.6% by weight. The yield of reducing substances was 25.57% of the absolutely dry substance. In all straw hydrolysis experiments, the best results were achieved with a sulfuric acid concentration of 1.6% by weight and an experimental temperature of 150-160°C.

#### Studies of the processes of enzymatic hydrolysis of straw

Model experiments for the study of enzyme kinetics were carried out using paper and cotton wool as the source of cellulose. In the experimental processes of fermentolysis, wheat straw that was pre-ground, screened and dried to constant value in a drying cabinet at a temperature of 120°C for 2 hours was used, which was pre-soaked in an autoclave at an excess pressure of 0.05-0.1 MPa for 0.5 - 1 hour.

The processes of fermentolysis were carried out while maintaining active acidity in the range of 4.9-5.0 units. pH and temperature of 49°C. The duration of fermentolysis was 7-10 hours.

Studies of the kinetics and stoichiometry of the reactions of enzymatic hydrolysis of disperse solid-phase vegetable substrates were carried out in a laboratory fermentolizer with automated pH control and thermostating.

The results of the experiment are given in Table 1.

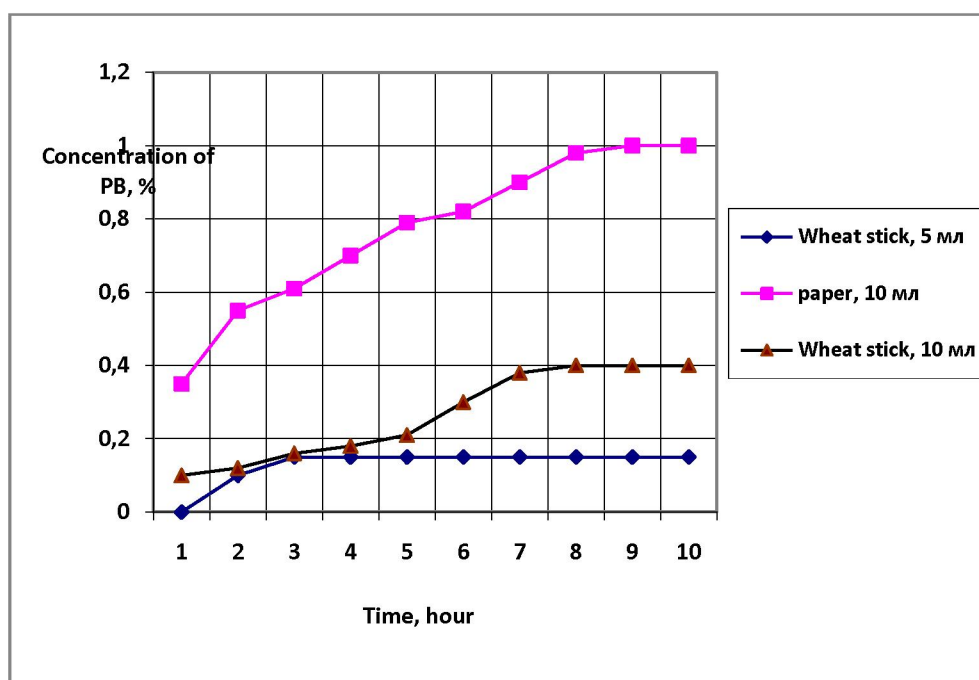
Table 1 – Results of fermentolysis of the wheat sticks in the fermenter

Process number	Number of enzymes, units activity	The maximum concentration of RS, %	Output RS, %
1	62-75.8	0.43	4.3
2	62-75.8	0.46	4.6
3	6.2-17.6	0.46	4.6
4	31-37.9	0.48	4.8
5	62-75.8	0.32	3.2
6	1.6-1.9	0.26	2.6
7	3.0-3.8	0.26	2.6
8	4.6-5.7	0.22	2.2

In order to reduce the error and verify the action of the enzyme on single-component substrates, a series of experiments were carried out on the fermentolysis of comminuted, de-impregnated paper into a 6-liter fermentolizer.

It is easy to verify that the dependence of the final concentration of RS (hence, the average rate of fermentolysis) on the average value of the enzyme activity is linear, which theoretically follows from the Michaelis-Menten model.

The results of comparative processes of fermentolysis of straw and paper are shown in Figure.



Variation in the concentration of RS in the processes of fermentolysis of paper and wheat straw

Since the paper is practically pure cellulose, then, with the same kinetic parameters of the fermentolysis processes, the amount of formed sugars in the case of fermentolysis of straw would be 1.49 times less than when using paper. However, in reality, the structure of the straw is characterized by a higher packing density of fibers and the presence of other interfering components, which leads to a slowing of the rate of the fermentolysis process by a factor of 2.5.

The resulting purified glucose hydrolyzate was subjected to hydrogenation in the presence of a nickel-aluminum-iron-chromium catalyst. From Table 2 it can be seen that the nickel catalysts under investigation, under the conditions studied by us, show high activity and stability with respect to sorbitol, the rate of formation of the latter varies with the number of doping metals in the initial alloys. The catalyst

Table 2 – The effect of the amount of added titanium additive on the activity of the nickel-aluminum-iron catalyst, (PN2 = 6MPa, Top-1000C)

Additive, %	t op., °C	Sorbitol yield (%) in time (min)					W*10 <sup>4</sup>
		0	10	20	40	60	
0	100	14.4	18.6	23.4	31.4	38.1	10.9
1.0	100	14.5	18.3	24.5	31.3	38.5	11.3
3.0	100	16.3	22.5	27.8	37.0	43.7	12.8
5.0	100	17.9	24.2	28.7	40.5	48.3	14.1
7.0	100	18.7	25.3	32.1	41.4	50.5	15.9
10.0	100	15.3	23.0	29.7	34.1	49.3	14.8

of the alloy with 7.0% chromium exhibits the greatest activity, the yield of sorbitol at which at 100°C and 6MPa for 60 minutes of hydrogenation is 50.5%, and the rate of hydrogenation of glucose is 1.46 times higher than for skeleton nickel without an additive.

It is shown that with increasing hydrogen pressure from 2 to 12 MPa and 40-120°C, the rate of hydrogenation of glucose on titanium-promoted nickel-aluminum-iron catalysts increases. However, the hydrogen pressure limit values have not been established. The "seeming" tendency of the reaction rate to is due to the lack of a hydrogenated compound on the surface of the catalyst, as indicated by the fractional order of the reaction for glucose. The order for hydrogen depends both on the temperature of the experiment and on the pressure of hydrogen. Experiments on the influence of the concentration of glucose and hydrogen show that the reaction order for the hydrogenated substance varies from zero to fractional, and in hydrogen, fractional.

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### **БИДАЙ САБАНЫНАН СОРБИТТИ АЛУ ҮДЕРІСІН ЗЕРТТЕУ**

**Аннотация.** Мақалада сорбитті алу мақсатында гидролиз – сутектендірудің ферментативті аралас (гибридті) процестерін зерттеудің нәтижелері көрсетілген. Дәл осы мақалада өндіріске қажетті бағалы өнімдердің шығымын жоғарылату мақсатында сорбит синтезін сонымен қатар бидай сабанының ферментативті гидролизін және қайта өңдеудің зерттеу нәтижелері көрсетілген. Сабанды қайта өңдеуді температурасы 190-250°C диапазонында күкірт қышқылы концентрациясының масс. 0,6 бастап 2,5 % түрлендіріп жүргізілді. Сабанды күкірт қышқылында гидролиздеудің оңтайлы температурасы және ұзақтығы 160–170°C және 30–80 минутты құрады. Сабанды гидролиздеудің барлық тәжірибелеріндегі ең жақсы нәтиже күкірт қышқылының концентрациясы масс 1,6% және температурасы 150–160°C кезінде жетті. Сабанды ферментативті гидролиздеу процестеріне зерттеулер жүргізілді. Қағаз тәжірибе жүзінде қаншалықты таза целлюлоза болып табылады, онда ферментализ процесінің кинетикалық параметрлері бірдей кезінде, сабан ферментализі жағдайында түзілетін қантмөлшері қағазды қолданғанға қарағанда 1,49 есе аз. Алынған тазаланған глюкоза гидролизаты никель-алюминий-темір-хромдық катализатордың қатысуымен сутектендірілді. 7,0% хром құймасынан жасалған катализатор сорбиттің шығуына үлкен белсенділік көрсетеді, 100°C және 6МПа, 60 минут кезіндегі сутектендіру 50,5%, ал глюкозаны сутектендіру жылдамдығы 1,46 есе жоғары қоспасыз қаңқалы никельге қарағанда. Көрсетілді, сутектің қысымының өсуі 2-ден 12 МПа дейін және 40-120°C глюкозаны сутектендіру жылдамдығы монтаждалған титан никель-алюминий-темір катализаторында үлкейеді.

**Түйін сөздер:** бидай сабаны, сорбит, целлюлоза, катализатор, ферментативті гидролиз, биомасса.

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### **ИССЛЕДОВАНИЕ ПРОЦЕССА ПОЛУЧЕНИЯ СОРБИТА ИЗ ПШЕНИЧНОЙ СОЛОМЫ**

**Аннотация.** В статье приведены результаты по изучению ферментативного совмещенного (гибридного) процесса гидролиз-гидрирование с целью получения сорбита. В настоящей статье изложены результаты исследования предобработки и ферментативного гидролиза пшеничной соломы, а также процесс синтеза сорбита с целью повышения выхода ценных продуктов, необходимых для промышленности. Предобработку соломы проводили в диапазоне температур 190–250°C при варьировании концентрации сернистой кислоты от 0,6 до 2,5 % масс. Оптимальная температура и продолжительность гидролиза соломы сернистой кислотой составили соответственно 160–170°C и 30–80 минут. Во всех экспериментах гидролиза соломы наилучшие результаты достигнуты при концентрации сернистой кислоты 1,6 % масс и температуре опыта 150-160°C. Проведены исследования процессов ферментативного гидролиза соломы. Поскольку бумага является практически чистой целлюлозой, то, при одинаковых кинетических параметрах процессов ферментализа, количество образовавшихся сахаров в случае ферментализа соломы было бы в 1,49 раза меньше, чем при использовании бумаги. Полученный очищенный глюкозный гидролизат подвергали гидрированию в присутствии никель-алюминий-железо-хромового катализатора. Катализатор из сплава с 7,0% хрома проявляет наибольшую активность, выход сорбита на котором при 100°C и 6 МПа на 60 минуте гидрирования составляет 50,5%, а скорость гидрирования глюкозы в 1,46 раза выше, чем на скелетном никеле без добавки. Показано, что с ростом давления водорода от 2 до 12 МПа и 40–120°C скорость гидрирования глюкозы на промотированных титаном никель-алюминий-железо катализаторах увеличивается.

**Ключевые слова:** солома пшеницы, сорбит, целлюлоза, катализатор, ферментативный гидролиз, биомасса.

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