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EFFECT OF HEAT TREATMENT CONDITIONS ON ENZYMATIC HYDROLYSIS EFFICIENCY OF RICE STRAW IN [BMIM] [CL] ENVIRONMENT

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Annotation: Enzymatic hydrolysis of isolated polysaccharide fractions and primary straw using Cello-lux A enzyme was performed to evaluate the efficiency of straw pretreatment in [BMIM][Cl] medium and to determine the optimal processing conditions that provide maximum sugar yield. The experiment was carried out for 4 and 48 hours in an acetate buffer medium (pH 4.7-4.9).

***Keywords*:** TS (technical cellulose) fraction, GS (grmycellulosic) fraction, SOS, [BMIM][Cl] (1-butyl-3-methylimidazole chloride), ultrasonic ionic liquid, rice straw, thermally treated straw.

The purpose of the study: to study the effect of thermal treatment process conditions (temperature, duration, ultrasound power) in [BMIM][Cl] medium on the yield and composition of rice straw biomass products and the reactivity of straw polysaccharides in acid and enzymatic hydrolysis.

Evaluation of efficiency of separation of low molecular products of heat treatment of straw and regeneration of [BMIM][Cl] by adsorption and extraction methods.

Research progress; Enzymatic hydrolysis of isolated polysaccharide fractions and primary straw using Cello-lux A enzyme was performed to evaluate the efficiency of straw pretreatment in [BMIM][Cl] medium and to determine the optimal processing conditions that provide maximum sugar yield. Experiment 4 and 48 hours during Acetate buffer (pH 4.7-4.9) was carried out.

Initial straw for 4 hours during fermentative from hydrolysis after returning of substances productivity am (absolute mass) is 6.3%, in 48 hours - 11.5% am did, this the straw trifluoroacetic acid acid with acidic from hydrolysis after sugar from productivity three even less Fermentolysis in the process from straw of sugar relatively low output, first in turn, the lignin in it and another of compounds existence with dependent being, this of enzymes to polysaccharides to enter hindrance does.

[BMIM][Cl] in the medium the straw heat with processing to give temperature and duration increase in TS fraction of lignin and hemicellulose take to throw with together will come.

Heat at 100°C (1 hour). with processing from giving after from the separated TS fraction of sugar productivity from straw received to harvest relatively almost 4 times increased. Treatment temperature increase from 100 to 150 °C (1 hour) and duration up to 2 hours increase with of sugar productivity increases, but acidic from hydrolysis next being below the harvest remains.

Technician of the cellulose (TS) fraction fermentative hydrolysis duration up to 48 hours increase sugar of the product to increase take comes (Table 1). in the range of 100-150 °C (1 hour) processing from giving after from straw isolated TS fractions for it to the original straw compared to 3 times increases. With that together, fermentative to hydrolysis able was fraction of polysaccharides percentage from 53 to 72%, processing at 140 °C (2 hours). from given after and up to 83% . As a result, the temperature and treatment duration increase of TS fraction with profitability to decrease despite the sugar product a little increased, maximum the value reaches 37.6% at 140°C (2 hours).

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Table 1 – Polysaccharide factions fermentative from hydrolysis sugar productivity (48 hours)

Straw again work conditions , °C/s	TS share , %		Share of GS , %		General profitability , % asm
	fraction per head	asm.	fraction per head	asm.	
100/1	52,59	32.74	70.35	12.75	45,49
120/1	56.11	34.00	83.67	16.82	50,81
140/1	60.95	31.88	83.66	20.58	52.46
150/1	72.18	35,40	84.61	22.01	57.40
140/2	82.95	37,58	83.89	23,18	60.76

Straw with processing of giving temperature and from duration strictly Look, it's easy hydrolyzable from GS fractions classified as polysaccharides of sugars product one different in the circumstances into isolated TS fractions than high. Your temperature increase with in the GS fraction fermentative to hydrolysis able of polysaccharides the share is from 70 to 85% changed, it 's worth it the same so in the circumstances into isolated TS fractions than high and of the faction acidic to hydrolysis according to polysaccharides to the composition near.

In relation to the noun of sugars the lowest yield was obtained at 100°C (1 h). of the faction Enzymolysis through received. Straw with processing to give temperature increase with GS (hemicellulose) fraction of sugar productivity almost 2 times increases. Maximum yield, 23.2% ad, obtained at 140 °C (2 hours).

Steam from the polysaccharides of cowpea straw the most high common sugar crop it in 1-butyl-3-methylimidazolium chloride at 140 °C for 2 h during again from working s right received. It is not refined straw fermentative hydrolysis as a result harvest was sugar from the fruit five from the bar is more.

In [BMIM] [Cl] medium ultrasonic heat with processing to give conditions fermentative hydrolysis efficiency effect.

Ultrasound with [BMIM][Cl]. together to apply wheat straw heat with processing of giving duration and temperature to reduce not but _ Enzymolysis conditions insulation done polysaccharide of fractions reactivity to increase possibility gave. Polysaccharide fractions heat with processing from giving after _ separated original straw and polysaccharide fractions than more to efficiency have enzymes by is hydrolyzed.

Again work from the conditions strict nazar, fractions of GS fermentative in the circumstances high to reactivity have. From ultrasound use because of in the fraction enzymes by hydrolysis possible was of polysaccharides share heat with processing from giving after more and 83-90% (asm. 16-23%).

Experimental in the circumstances isolated TS fractions fermentative hydrolysis conditions of low reactivity with is described. of sugars product each one in the fraction from 56 to 64 % or as from 27-40 % changed stands With that together, high to productivity have fraction TS to fraction GS than the original straw point of view in terms of of sugar high productivity provides.

Heat at 100°C (15 minutes) with processing to give during ultrasound _ power from 10 to 50 W increase with, polysaccharide from fractions of sugar output increases. Again work temperature increase with sugar of productivity increase only the GS fraction for observed . 50 W temperature and of power together effect from the TS fraction sugar productivity to decrease take will come It is above 120 °C at temperatures more it is felt. As a result, polysaccharide fractions of sugars common productivity decreases. Ultrasonic at 120 °C (30 W). processing to give the term change polysaccharide of fractions fermentative hydrolysis efficiency effect does not minutes at 100 °C 50 W

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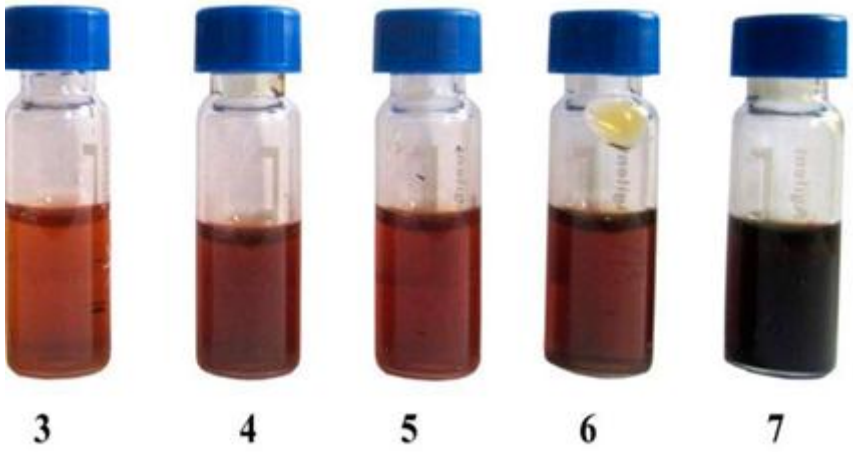
power during. Straw polysaccharides point of view in terms of sugar productivity theoretical in terms of possible 80 % of that organize did. In an IS environment without ultrasound the straw heat with processing to give 2 hours in the process of sugar at 140 °C during near productivity received.

So so, it's full of steam straw in [BMIM][Cl] medium ultrasound with treatment polysaccharide fermentative of sugars common 5 times the yield increase provides. From the GS fraction of sugar maximum yield was obtained at 140°C (50 W, 15 min) and from the TS fraction at 100 °C (50 W, 15 min).

Again use for [BMIM][Cl] regeneration

Starting point 1 -butyl-3-methylimidazolium chloride colorless is a liquid. From used then it 's amber to color have is the temperature and treatment duration increase with darkens (Fig. 1).



1 - initial , 2 - 100 °C (1 hour), 3 - 120 °C (1 hour), 4 - 140 °C (1 hour), 5 - 150 °C (1 hour), 6 - 140 ° C (2 hours), at 7 - 140°C (5 hours).

Figure 1 – [BMIM][Cl] . temperature and heat with processing to give duration increase with color Change in melanoidin formation to be and dehydration.

NPF faction from being separated then [BMIM][Cl] again recovery for organic with solvents (benzene, dioxane, TGF). liquid extraction and very critical CO_2 extraction , adsorption method with one in line . on activated carbon used _ Such without, low molecular weight in a heavy age products adsorbed and activated carbon layer the rest Straw heat with processing 2 hours at 140 °C during for IS used adsorption done increased. Activated carbon with processing from given After, cleaned IS of _ yield is 87.6% organize did _ It is in original IS color near to color have was.

Activated to carbon adsorption from being done then in the 13C NMR spectrum of purified [BMIM][Cl] impurity signals no, it's low molecular weight in weight products from IL complete take to throw shows of purified [BMIM][Cl]. ^{13}H YaMR compounds in the spectrum (Fig. 3.34). existence showing almost all signals also disappear: 2.69 ppm. (A1--CH_3), 2.83 ppm (-C=CH), 2.99 ppm (Alk -OH), 7.91 ppm (Ar -OH; ($CONH_2$)) and 8.23 ppm. (Ar -H).

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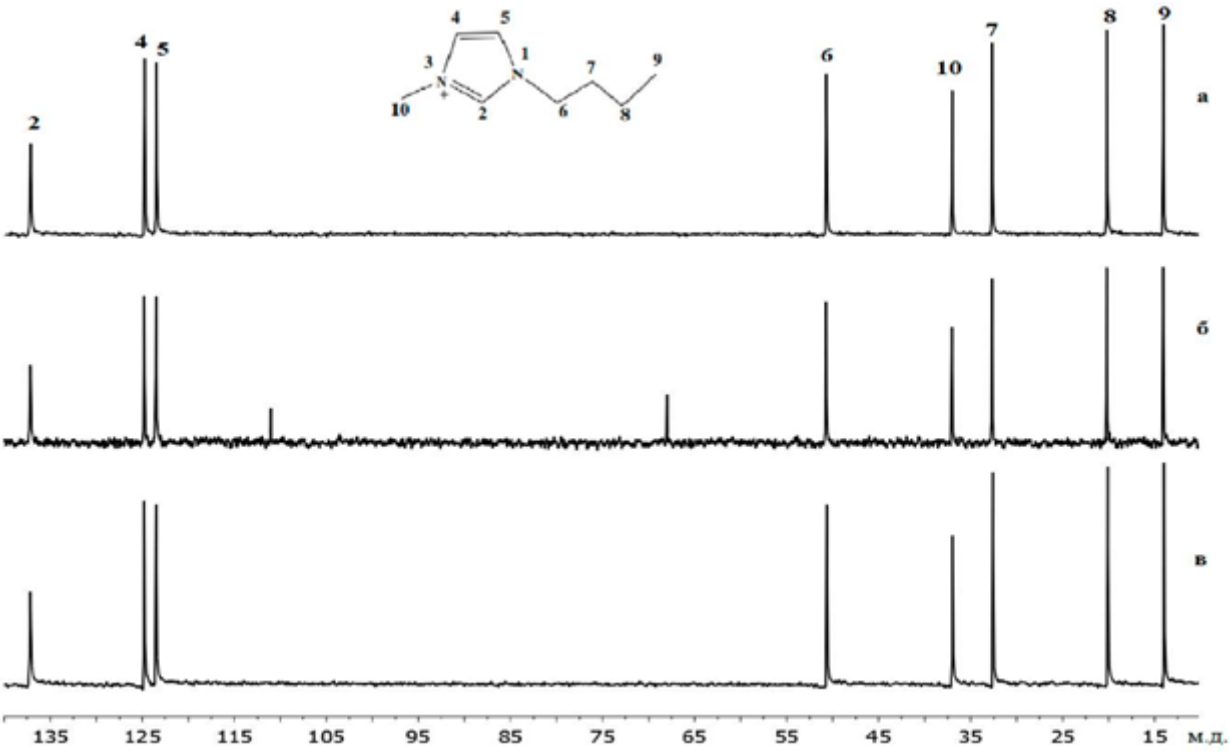
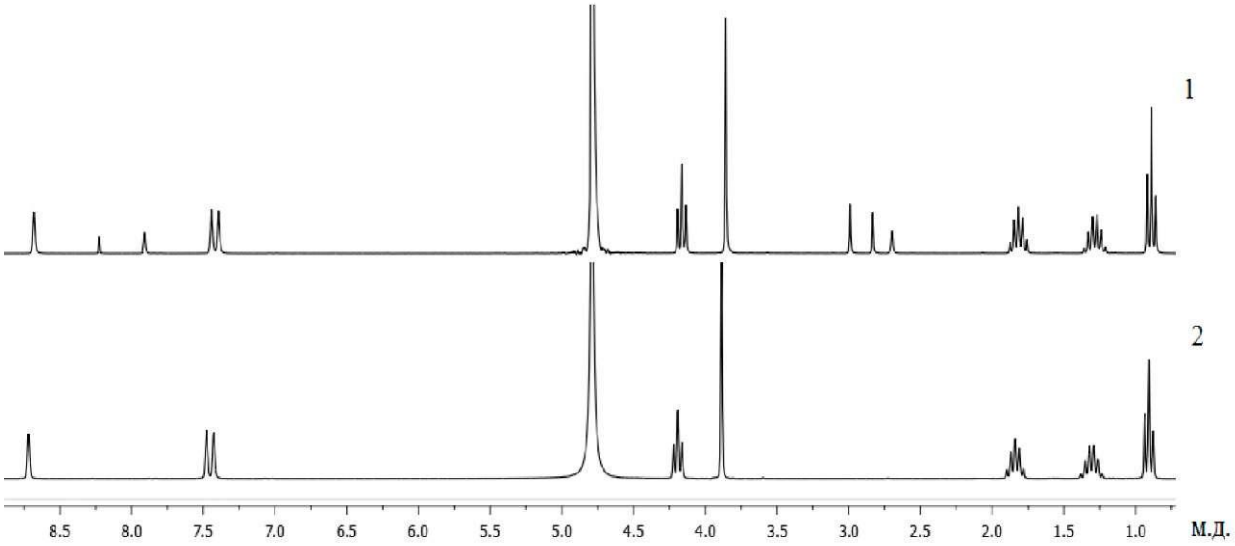


a - initial; b - used, c - activated carbon with cleaned

2 - *of [BMIM][Cl]. ^{13}C YMR spectra* i

Activated to carbon adsorption from being done of purified [BMIM] [Cl]. ^{13}C YMR in the spectrum impurity signals no, it's low molecular weight in weight full of products from IS take to throw shows.



1 - clean, 2 - used , 3 - activated carbon with cleaned

Figure 3 - of [BMIM][Cl]. YMR spectrum

the IR spectrum of purified [BMIM][Cl] at 2750 and 2450 cm $^{-1}$, the mixture swallowing of tapes intensity significant level decreased and carbonyl compounds special which is 1711 cm $^{-1}$ swallowing zone disappeared .

Original and cleaned active of carbon YMR ^{13}C- and ^{1}H-spectra

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Same with [BMIM][Cl] so.

Of all IS samples ^{13}C YMR in the spectra (Fig. 3.33) structural at 137.13 ppm for groups [BMIM][Cl]. characteristic signals there is (H(2)-C=), 123.48 ppm (H(5)-C=), 124.72 ppm (H(4)-C=), 50.55 ppm (-N_1-CH_2- CH_2-CH_2-CH_3),

of IS YMR from the spectra hydrogen of atoms relative content calculation is also activated in carbon adsorption way with IS cleaning high efficiency proves (Table 2).

Table 4 - Activated in carbon from adsorption after hydrogen of atoms relative composition [BMIM][Cl]

Chemical shift , ppm	Structural group	Ionic liquid	
		start drinking	cleaned
0.87(3)	-CH_2-CH_2-CH_2-C **H** $_3$	0.201	0.200
1.31(6)	-CH_2-CH_2-C **H** $_2$-CH_3	0.134	0.133
1.84(5)	-CH_2-C **H** $_2$-CH_2-CH_3	0.133	0.133
3.87(1)	-N_3-C **H** $_3$	0.201	0.200
4.19(3)	-N_1-C **H** $_2$-	0.134	0.136
7.42(2)	**H** (4,5)-C=	0.132	0.133
8.71(1)	**H** (2)-C=	0.065	0.066

Adsorption of the method advantages [BMIM][Cl] ni of mixtures cleaning relatively high effectiveness, disadvantages of [BMIM][Cl] . duration and significant losses own into takes.

Various methods with of purified [BMIM][Cl]. efficiency evaluation for full of steam to the straw heat with processing give 1 hour at 100 °C during, then fractionation through done increased. Experience for of purified IS three example used (Table 5).

Table 5 - Fractions output _

	Output , % a . s . m .		
IS sample	Technician celluloses	hemicelluloses	lignin
Get started	62.25	18,13	5.91
Cleared :			
Extraction of TGF	63.53	17.86	5.23
activated coal _	62,63	18.53	5.62
supercritical SO_2	63.05	17.93	5.44

Received information according to purified [BMIM] [Cl] straw biomass fractionation efficiency according to from the beginning almost difference does not Using purified [BMIM][Cl] the straw fractionation way with received polysaccharide factions from fermentolysis next sugar product too from the original [BMIM][Cl] when used to harvest near will be (table 6).

6 - table - 48 hours during from fermentolysis after of sugars output

IS sample	TS share , %		Share of GS , %		General profitability , % asm
	fraction per head	asm .	fraction per head	asm .	
Get started Cleared:	52,59	32.74	70.35	12.75	45,49
Extraction of TGF	51.23	32.55	68.56	12.24	44.79
activated coal _	52.01	32.57	69.51	12.88	45.45
supercritical SO_2	51.63	32.55	69.06	12.38	44.93

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So making the liquid extraction to do critical CO_2 extraction and activated in carbon adsorption methods regeneration of [BMIM][Cl]. for different different efficiency with use can.

Tetrahydrofuran, too critical carbonate anhydride or activated to carbon adsorption with liquid extraction with purified [BMIM][Cl] samples wheat straw biomass in the faction efficiency without losing use possible was shown.

Adsorption method of [BMIM][Cl] when using significant level to disappear and his duration account low molecular weight in weight wheat straw again work from products of chloride [BMIM][Cl]. cleaning for very critical CO_2 extraction method recommendation to do can.

Summary to do maybe 1-butyl-3-methylimidazolium **chloride** in the environment heat with processing to give acidic and fermentative hydrolysis during straw of polysaccharides reactivity increases. Straw of polysaccharides fermentative hydrolysis as a result harvest was of sugar the most high common productivity not cleaned from straw received sugar 5.3 times the yield a lot and theoretical in terms of possible 83% of which organize Heat at 140 °C (2 hours) with processing to give through received. Ultrasound and heat with processing to give, from the original straw sugar from productivity five even high has been of sugar maximum efficiency of 50 W ultrasound power treatment at 100 °C (0.25 h). given straw polysaccharides fermentation way with received.

As it turns out, straw heat with processing low molecular weight products organic solvents with get their 1-butyl-3-methylimidazolium chloride contained the amount by only 40-45%, too critical CO_2 - extraction and activated to carbon adsorption - 1-butyl-3-methylimidazoline chloride again when used efficiency without losing in practice complete again recovery.

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