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DETERMINATION OF HOLOCELLULOSE AND LIGNIN CONTENT IN WOOD ON THE BASIS OF HEAT OF COMBUSTION MEASUREMENT

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Relation between holocellulose and lignin content of wood degraded by chemical and biotical agents and its calorific value has been determined. This dependence is expressed in form a straight line equation passing through two points. Results of calculations of lignin and holocellulose contents based on heat of combustion measurements are to a great extent similar to the results of their determinations by the chemical way.

INTRODUCTION

First methods developed in the wood chemistry analytics were the direct determinations of its chemical composition and functional groups. Those methods are encumbered with errors, since they are based on chemical reactions destroying the composition and chemical structure of wood. The aim of almost all analytical methods in wood chemistry is to overcome those errors. The main directions of analytical methods development during the last decades are the physico-chemical methods such as thermoanalysis and spectrophotometry. Among the thermoanalytic values one of the basic is the heat of combustion.

The heat of combustion had been previously mainly used for determination of the calorific value of wooden raw materials (Parr, Davidson 1922, Kollmann 1951, Szturma, Woltyńska 1967, Murphy, Cutter 1974). There were also applied calculations of the heat of combustion of wood from its elementary composition based on the Dulong's formula (Stohman, Kerl 1893). Gradually the heat of combustion begun to be used in the analytics of wood chemistry. Those applications had been used in the investigations of the wood decayed by fungi (Vanin 1949, Kawase 1962, Salmi 1964, Rypáček, Coufalikova 1981). The heat of combustion was also correlated with the extractives contained in wood (Urbanik 1965, Howard 1973), with the wood density (Pavić 1965) and with the ecological conditions of the tree growth (Madgwick 1970, Dadykin, Kononienko 1975, Boversox, Blankenhorn, Murphey 1979, Nečesany, Oberländerova 1982). In the last years attempts were undertaken to interpret the heat of combustion

as the function of the chemical wood composition (Doat 1977, Murphy, Masters 1978, Dziurzyński, Owczarzak, Surmiński 1978). It was showed in last investigations, that the heat of combustion was a linear function of the chemical wood composition (Dziurzyński 1984, Dobry, Dziurzyński, Rypáček 1986). Those investigations formed a basis for elaboration of dependence allowing to calculate the holocellulose and lignin content in the wood as a result of measurements of heat of combustion. Elaboration of such dependence is the scope of this paper. With the application of such relation it is possible to determine holocellulose and lignin contents in wood in the relatively short time of 0.5 - 1.0 hours.

MATERIALS AND METHODS

EXPERIMENTS

The tests were made on the samples of Scots Pine (*Pinus silvestris* L.) and Beech (*Fagus silvatica* L.) wood. Dimensions of the samples were $3 \times 20 \times 30$ mm. They have been extracted with the ethanol-benzene mixture 1 : 1, and then exposed to the procedure of decomposition such as: alkaline pulping in NaOH, acid hydrolysis in H_2SO_4 and fungal decay.

The alkaline pulping was performed in Hagglund's bombs using 1.2 mol/dm^3 NaOH at a temperature of 165°C and liquor to wood ratio equal to 5. The acid hydrolysis was made in glass flasks under a reflux condenser 0.6 mol/dm^3 of H_2SO_4 in temperature of 100°C and the liquid ratio equal to 5. The solid products obtained in the alkaline pulping and acid hydrolysis were washed with hot water to the neutral reaction.

The fungal decays were obtained with the brown rot fungus (*Coniophora puteana* Schum. ex Fr.) and white rot fungus (*Polystictus versicolor* L. ex Fr.) (Seifert 1962, 1966). The prepared wood samples were sterilized with steam in an autoclave and then placed in the Kolle's flasks containing the agar-agar medium with addition of malt extract. The samples were placed on glass stics. The fungal decay had been performed in a box at temperature of 22°C, relative humidity between 65 and 70% and a diffused daylight.

The decayed wood was ground into particles of 0.5 - 1.0 mm, and then its chemical composition was determined. The analysis were made accordingly to the methods described by Browning 1967, namely: the solubles in 0.1 mol/dm^3 NaOH were determined accordingly to Tappi T 4 m method for 1% NaOH (0.25 mol/dm^3). The only modification was the reduction of the NaOH concentration to 0.1 mol/dm^3. The lignin was determined on the material extracted with 0.1 mol/dm^3 NaOH accordingly to Tappi T 13 m. The holocellulose content was determined on the parallel batch of material with the use of 10% peracetic acid in temperature of 90°C during 20 minutes for beech and 45 minutes for pine (Haas, Schoch, Ströhle 1955). The determination of the solubles in NaOH and lignin with a parallel determination of holocellulose brings in case of the degraded wood a total percent of them

close to 100 (Tichy 1967). The lignin obtained from wood extracted with 0.1 mol/dm^3 NaOH can be also more easily filtered in the crucible (Rypáček 1979).

The heat of combustion was measured with a calorimeter type KL-5 production of the Cooperative „Precission" in Bydgoszcz (Poland). The results of those determinations were expressed in Joules per gram of oven dry wooden material calculated as a pure organic substance.

CALCULATIONS

The main factor causing the variability of the heat of combustion are resinous substances. A feature of the extracted wood within the same species is a great stability of the heat of combustion, which is closely dependent upon the holocellulose and lignin content (Dziurzyński 1984). The holocellulose and lignin content in wood is expressed by a dependence:

$$x_h = \frac{100 - x_h^0}{q_h^0 - Q^0}(Q - Q^0) + x_h^0 \quad (\%) \tag{1a}$$

$$x_l = \frac{100 - x_l^0}{q_l^0 - Q^0}(Q - Q^0) + x_l^0 \quad (\%) \tag{1b}$$

where Q, x_h, x_l – heat of combustion (Q) and holocellulose (x_h) or lignin (x_l) percentage in the analysed sample of degraded wood; Q^0, x_h^0, x_l^0 – heat of combustion (Q^0) and holocellulose (x_h^0) or lignin (x_l^0) percentage in an original wood sample after extraction in ethanol-benzene mixture; q_h^0, q_l^0, 100 – heat of combustion of holocellulose (q_h^0) or lignin (q_l^0) preparations isolated from the origina wood *in vitro*, and percentage of those constituents (100%) in those preparations

Table 1

Heat of combustion and holocellulose and lignin content of wood and its preparations

Wood species	Kind of sample	Content %		Heat of combustion (kJ/kg)
		holocellulose	lignin	
Pine	wood*	67.42	27.64	20 120
	holocellulose	100.0	–	17 790
	lignin	–	100.0	28 200
Beech	wood*	69.49	23.51	19 920
	holocellulose	100.0	–	17 780
	lignin	–	100.0	26 820

* extracted with ethanol-benzene

The relation presented above is a straight line equation passing through two points. Their coordinates are (Q^0, x_h^0) and (q_h^0, 100) in the case of calculation of holocellulose percentages – equation 1a, or (Q^0, x_l^0) and (q_l^0, 100) in the case of

calculation of lignin percentages – equation 1b. This dependence is based on the assumption, that the heats of combustion of wood constituents *in situ* are additive. This assumption results from the additivity of chemical bounds energy known in thermochemistry (Świętosławski 1928). The application of the principle of additivity of chemical bonds energy for the calculation of the heat of combustion of the chemical constituents of wood causes that the results of those calculations are compatible with the experimental ones (Dziurzyński, Owczarzak, Surmiński 1978).

The numerical values of coordinates of the fixed points through which straight lines described by equations 1a and 1b passes are presented in table 1.

RESULTS AND DISCUSSION

Results of chemical determinations of holocellulose and lignin in wood decomposed by chemical and biotical agents are tabulated in tables 2 and 3. It results from them that the holocellulose and lignin contents increase or decrease depending upon the kind of process and the degree of wood degradation.

Reactions of the carbohydrates decomposition prevail in the H_2SO_4 hydrolysis processes and the decay by *C. puteana* fungus. On the other hand reactions

Table 2

Lignin and holocellulose contents in pine wood determined by chemical methods and on the basis of heat of combustion measurements

Decomposition parameters			Results of chemical analysis (%)				Heat of combustion (kJ/kg)	Results on the basis of the heat of combustion (%)		
environment	time	mass loss Δm(%)	solubles in NaOH (0.1 mol/dm³)	lignin	holocellulose	total percent		lignin	holocellulose	total percent
1	2	3	4	5	6	7	8	9	10	11
–	–	0	5.72	27.64	67.42	100.78	20120	27.64	67.42	95.06
NaOH	1 min	21.0	3.99	28.70	67.00	99.69	20200	28.36	66.30	94.63
	2 hours	33.3	4.28	21.99	74.60	100.87	19460	21.73	76.65	98.38
	6 hours	47.8	4.27	10.31	88.59	103.17	18230	10.72	93.85	104.57
H_2SO_4	20 min	10.2	12.98	29.79	64.11	106.88	20410	30.23	63.37	93.60
	6 hours	22.6	7.20	34.41	65.17	106.78	20750	33.28	58.61	91.89
	30 hours	31.6	6.73	36.42	63.54	106.69	21210	37.40	52.18	89.58
Polystictus versicolor	30 days	6.0	18.96	20.84	65.84	104.84	19580	22.80	74.97	97.77
	70 days	14.0	20.87	20.42	63.22	104.51	19550	22.54	75.39	97.93
	300 days	28.7	24.27	19.75	61.87	105.89	19700	23.88	73.29	97.17
Coniophora puteana	10 days	12.0	35.16	25.45	50.40	111.01	20030	26.84	68.68	95.52
	30 days	23.0	36.58	28.18	48.76	113.52	20210	28.45	66.16	94.61
	90 days	38.0	36.35	29.82	45.04	111.21	20670	32.57	59.73	92.30
	220 days	63.0	58.27	34.43	11.47	104.17	23050	53.88	26.45	80.33

of delignification take the major part in the process of pulping with NaOH. The activity of the fungus *P. versicolor* decomposes the constituents of wood evenly. In the wood decayed by fungi, and especially by *C. puteana* the content of solubles in NaOH increases. Those substances testify the depolimerisation of holocellulose

Table 3

Lignin and holocellulose contents in beech wood determined by chemical methods and on the basis of heat of combustion measurements

Decomposition parameters			Results of chemical analysis (%)				Heat of combustion (kJ/kg)	Results on the basis of the heat of combustion (%)		
environment	time	mass loss Δm(%)	solubles in NaOH (0.1 mol/dm³)	lignin	holocellulose	total percent		lignin	holocellulose	total percent
1	2	3	4	5	6	7	8	9	10	11
–	–	0	12.95	23.51	69.49	105.95	19920	23.51	69.49	93.00
NaOH	1 min	25.5	4.75	25.40	64.38	94.53	20010	24.51	68.21	92.72
	2 hours	52.0	2.14	8.18	84.31	94.63	18260	5.11	93.16	98.27
	6 hours	58.7	3.68	2.11	88.94	94.73	17630	−1.87	102.14	100.27
H_2SO_4	20 min	13.8	23.11	25.16	57.15	105.42	20100	25.50	66.92	92.42
	2 hours	24.0	16.95	29.38	59.33	105.66	20290	27.61	64.21	91.82
	6 hours	42.7	10.56	34.48	62.49	107.53	20770	32.93	57.37	90.30
Polystictus versicolor	10 days	3.0	18.78	21.12	64.19	104.09	19860	22.84	70.31	93.15
	30 days	24.0	20.81	20.65	63.99	105.45	19960	23.95	68.92	92.87
	50 days	33.0	21.81	19.60	61.60	103.01	19800	22.18	71.20	93.38
	120 days	63.0	27.41	17.47	60.51	105.47	19570	19.63	74.48	94.11
Coniophora puteana	10 days	2.5	26.94	19.90	56.21	103.05	19930	23.62	69.35	92.97
	30 days	16.0	49.08	15.68	40.80	105.56	19980	23.18	69.92	93.10
	50 days	23.0	52.88	14.64	38.66	106.18	20060	25.06	67.49	92.55
	120 days	36.0	59.70	13.47	35.74	108.91	20110	25.62	66.78	92.40
	250 days	57.0	75.11	10.79	21.23	107.13	21590	42.02	45.68	87.70

and lignin (Tichy 1974, Rypáček 1977, Blankenhorn et all. 1980). Small amounts of substances soluble in NaOH in wood after the pulping processes in NaOH and the hydrolysis in H_2SO_4 are due to the fact, that those processes were conducted in a liquid medium and most of the depolimerized wood constituents dissolved

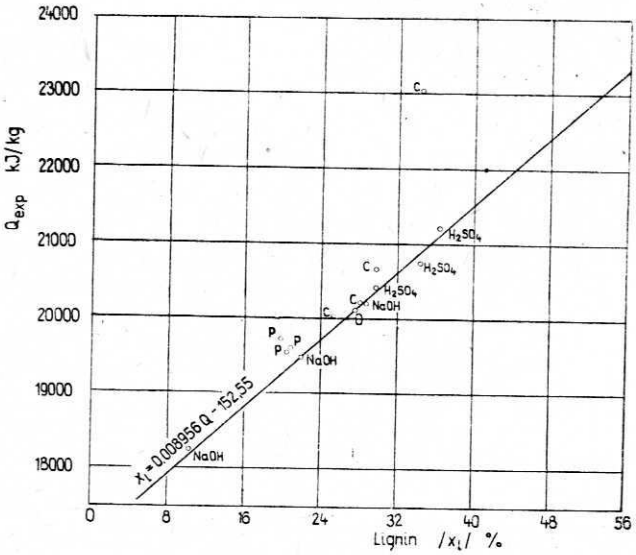

Fig. 1. Dependence of the heat of combustion upon the lignin content in pine wood decayed by chemical (NaOH and H_2SO_4) and biotical (*C. puteana*-C, *P. versicolor*-P) agents

in the reagents. Results of the heat of combustion measurements are also presented in tables 2 and 3. In the last three columns there are given results of calculations of lignin and holocellulose contents and the total of their percentage.

Determinations of lignin and holocellulose contents on the chemical way and on the basis of heat of combustion measurements are equal for the original wood

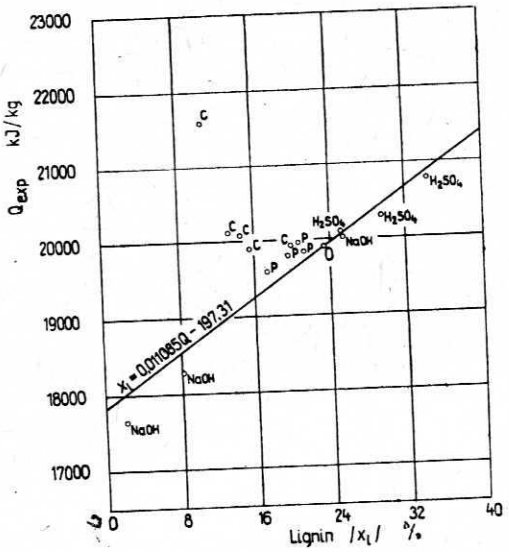

Fig. 2. Dependence of the heat of combustion upon the lignin content in beech wood decayed by chemical (NaOH and H_2SO_4) and biotical (*C. puteana*-C, *P. versicolor*-P) agents

according to the assumption in the equations 1a and 1b (tab. 2 and 3). In degraded wood, obtained values differ each to other depending upon the kind of the process of wood degradation (Fig. 1 - 4). Those differences are greatest for the wood decayed by *C. puteana* fungus.

Values of differences between the determinations of lignin and holocellulose contents with the use of chemical methods and on the basis of heat of combustion measurements are illustrated by the following statistics (tab. 4):

$$\text{arithmetic mean } \overline{\Delta x} = \frac{1}{n}\sum_{l}^{n}(x_{\text{chem}} - x_{\text{comb}})_l$$

$$\text{standard deviation } \bar{\rho} = \sqrt{\frac{1}{n}\sum_{l}^{n}(x_{\text{chem}} - x_{\text{comb}})_l^2}$$

$$\text{statistics } \chi^2 = \sum_{l}^{n}\frac{(x_{\text{chem}} - x_{\text{comb}})_l^2}{x_{\text{comb}_l}}$$

where x_{chem} – percentage of lignin (x_l) or holocellulose (x_h) determined with the use of chemical methods; x_{comb} – percentage of lignin (x_l) or holocellulose (x_h)

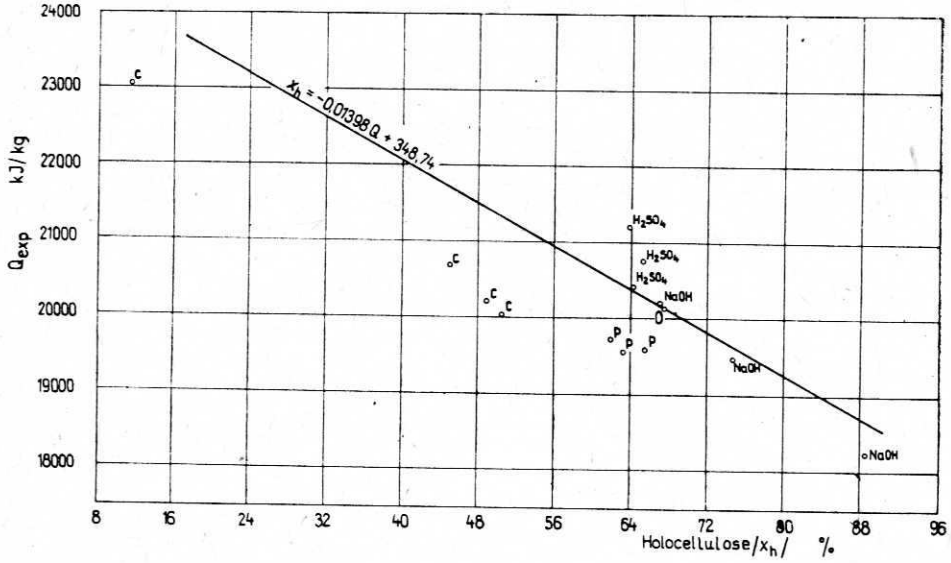

Fig. 3. Dependence of the heat of combustion upon the holocellulose content in pine wood decayed by chemical (NaOH, H_2SO_4) and biotical (*C. puteana*-C, *P. versicolor*-P) agents

determined on the basis of heat of combustion measurements; l – index of sample of wood degraded by chemical or biotic agent – mass losses (tab. 2 and 3); n – sum of samples of wood degraded by chemical or biotic agent.

An addendum to the mentioned statistical values are accuracies, the so called „errors of repeteability" of heat of combustion measurement and holocellulose and lignin determinations with the use of chemical methods. Their values are: $\pm 0.23\%$ for heat of combustion, $\pm 0.24\%$ for lignin, $\pm 0.37\%$ for holocellulose (Dziurzyński

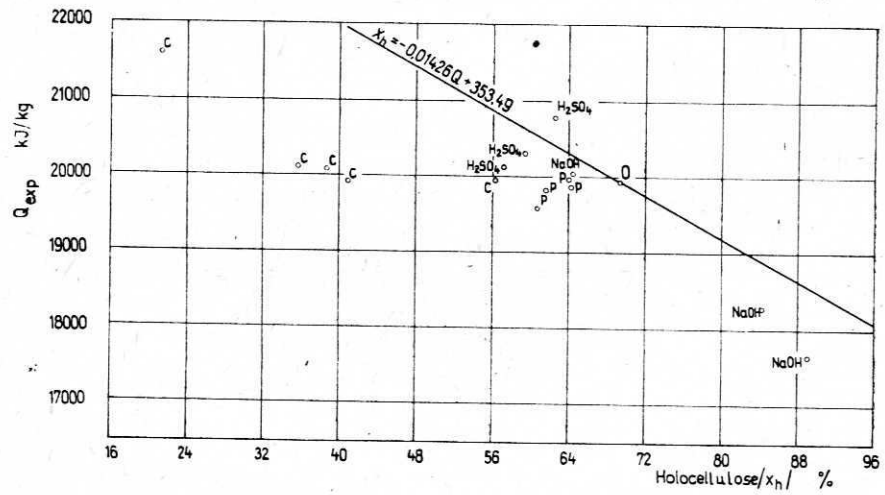

Fig. 4. Dependence of the heat of combustion upon the holocellulose content in beech wood decayed by chemical (NaOH, H_2SO_4) and biotical (*C. puteana*-C, *P. versicolor*-P) agents

1981). Those accuracies are also valid for constant values (Q_o^0, q^0, x^0) applied in equations 1a and 1b. Accordingly to the classical rules of errors calculations, it can be stated that values of mean deviations $\bar{\rho} < 2.0\%$ (tab. 4) are in the limits of accuracy of performed determinations.

The compatibility of percentage contents of lignin determined by chemical methods and on the basis of measurements of heat of combustion are illustrated

Table 4

The statistical characteristics of differences between the holocellulose and lignin content determinations with the use of chemical methods and on the basis of heat of combustion measurements

	Statistical value	Differences between chemical determinations and determinations on the basis of heat of combustion			
		pine		beech	
		lignin	holocellul.	lignin	holocellul.
*	$\overline{\Delta x}$	−0.70	−2.30	0.12	−7.00
	$\bar{\rho}$	1.64	7.94	2.38	8.73
	χ^2	1.01	8.43	11.66	7.93
	$\overline{\Delta x}$	−0.89	−5.92	−2.33	−12.29
**	$\bar{\rho}$	1.68	9.64	5.23	15.98
	χ^2	1.32	18.82	19.00	49.29

* Results for samples of wood decomposed in three processes: alkaline pulping, hydrolysis by sulphuric acid, decay by *P. versicolor* fungus, $n = 9$ for pine, $n = 10$ for beech.

** Results for samples of wood decomposed in all four investigated processes with exception of the most decayed samples by *C. puteana* fungus, $n = 12$ for pine, $n = 14$ for beech.

by statistical characteristics calculated for three and four processes of wood decomposition (tab. 4). For processes: pulping in NaOH, hydrolysis in H_2SO_4 and the decay by *P. versicolor* fungus, standard deviations ($\bar{\rho}$) between two kinds of lignin determinations are 1.64% for pine wood and 2.38% for beech wood. Taking into account all four investigated wood decomposition processes of wood, that is together with decay by *C. puteana*, those deviations rise respectively into: 1.68% for pine wood and 5.23% for beech wood. Those deviations are caused mainly by extraction with NaOH of the wood samples preceding determinations of lignin according to the TAPPI method. The contents of the extractives in 0.1 mol/dm^3 NaOH of wood decayed by *C. puteana* fungus are considerably greater than in other processes (tab. 2 and 3). The relinquishment of extraction with NaOH could create some difficulties in filtering of lignin preparations and also some increase of obtained data of lignin contents by the products of furfural polymerisation (Browning 1967).

The compatibility of percentage of holocellulose contents – determined with the chemical methods and on the basis of heat of combustion measurements are presented by statistical characteristics calculated similarly as for lignin. For the processes of pulping in NaOH, hydrolysis H_2SO_4 and decay by *P. versicolor* fungus,

standard deviations ($\bar{\rho}$) among the two methods of holocellulose content determinations are: 7.94% for pine wood and 8.73% for beech wood. For all four investigated processes those deviations are increasing alike as for lignin. Those deviations are caused by lowering of results of holocellulose determinations with chemical method, that is too severe conditions of its isolation. Such lowering can be observed especially in beech wood. The lowering of the holocellulose determinations results in case used there chemical method, can be eliminated by the application of more mild parameters of action of peracetic acid. The shortening of the time of action of peracetic acid from 20 to 15 minutes could increase results of determinations of holocellulose in beech wood about 3% (Haas, Schoch, Ströhle 1955). But in such case the accuracy of holocellulose determinations is lowering due to decrease of repeteability of CH_3COOH action onto the analysed wooden material.

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ОПРЕДЕЛЕНИЕ СОДЕРЖАНИЯ ХОЛОЦЕЛЛЮЛОЗЫ И ЛИГНИНА В ДРЕВЕСИНЕ НА ОСНОВЕ ЕЁ ТЕПЛОТЫ СГОРАНИЯ

Резюме

Разработана зависимость между содержанем холоцеллюлозы и лигнина в древесине разложенной химическими и биологическими факторами и теплотой её сгорания. Эта зависимость представлена в форме прямой линии переходящей через две точки

$$x_h = \frac{100 - x_h^0}{q_h^0 - Q^0}(Q - Q^0) + x_h^0 \quad (\%) \tag{1a}$$

$$x_l = \frac{100 - x_l^0}{q_l^0 - Q^0}(Q - Q^0) + x_l^0 \quad (\%) \tag{1b}$$

где Q, x_h, x_l — теплота сгорания (Q) и содержание холоцеллюлозы (x_h) или лигнина (x_l) в анализируемом образцу разложенной древесины, Q^0, x_h^0, x_l^0 — теплота сгорания (Q^0) и содержание холоцеллюлозы (x_h^0) или лигнина (x_l^0) в образцах здоровой древесины экстрагированной смесью этапол-бензол, q_h^0, q_l^0, 100 — теплота сгорания образцов холоцеллюлозы (q_h^0) или лигнина (q_l^0) изолированных со сдоровой древесины, *in vitro* и содержание этих компонентов в их препаратах (100%).

Выследки расчётов содержания холоцеллюлозы и лигнина в большой степени сходные с обозначениами этих компонентов химическими методами.

OZNACZANIE ZAWARTOŚCI HOLOCELULOZY I LIGNINY W DREWNIE NA PODSTAWIE POMIARÓW CIEPŁA SPALANIA

Streszczenie

Określono zależność między zawartością ligniny i holocelulozy w drewnie zdegradowanym czynnikami chemicznymi i biotycznymi a ciepłem spalania tego drewna. Zależność tę przedstawiono w formie równań linii prostej przechodzącej przez dwa punkty

$$x_h = \frac{100 - x_h^0}{q_h^0 - Q^0}(Q - Q^0) + x_h^0 \quad (\%) \tag{1a}$$

$$x_l = \frac{100 - x_l^0}{q_l^0 - Q^0}(Q - Q^0) + x_l^0 \quad (\%) \tag{1b}$$

gdzie Q, x_h, x_l – ciepło spalania (Q) i zawartość procentowa holocelulozy (x_h) lub ligniny (x_l) w analizowanej próbce drewna zdegradowanego; Q^0, x_h^0, x_l^0 – ciepło spalania (Q^0) i zawartość procentowa holocelulozy (x_h^0) lub ligniny (x_l^0) w próbkach drewna zdrowego wyekstrahowanego mieszaniną etanol-benzen; q_h^0, q_l^0, 100 – ciepło spalania preparatów holocelulozy (q_h^0) lub ligniny (q_l^0) wyizolowanych z drewna zdrowego, *in vitro* i zawartości procentowe tych składników w ich preparatach (100%).

Wyniki obliczeń zawartości holocelulozy i ligniny są w dużym stopniu zbieżne z oznaczeniami chemicznymi.

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