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ИССЛЕДОВАНИЕ КОМПОНЕНТНОГО СОСТАВА ПРОДУКТОВ ОКИСЛИТЕЛЬНОЙ ТЕРМОДЕСТРУКЦИИ ТОПЛИВНЫХ ГРАНУЛ ИЗ БИОМАССЫ *HERACLEUM SOSNOWSKYI* MANDEN МЕТОДОМ ХРОМАТО-МАСС-СПЕКТРОМЕТРИИ

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Проведено исследование компонентного состава продуктов окислительной термодеструкции топливных гранул из биомассы Heracleum sosnowskyi Manden методом хромато-масс-спектрометрии. Биомасса борщевика Сосновского была собрана на территории села Выльгорт республики Коми, высушена до атмосферно-сухого состояния, измельчена до d_m < 0,25 мм и запрессована с усилием 10 кН. Топливные гранулы были сожжены в установке, продукты окислительной термодеструкции уловлены и проанализированы на газо-жидкостном хроматографе с масс-селективным детектором. В результате анализа масс-спектров из 47 уловленных соединений было идентифицировано 39 низкомолекулярных продуктов со временем удерживания от 6 до 79 мин и процентным содержанием от 0,4 до 12%. Качественный и количественный анализ компонентного состава продуктов деструкции показал, что их образование напрямую связано с компонентным составом биомассы и определяется содержанием и строением целлюлозы, гемицеллюлоз и лигнина. Анализ зависимости соединение – время удерживания показал, что в первую очередь деструкции подвергаются целлюлоза и гемицеллюлозы образца биомассы, а лигнин вследствие ароматической природы является термически более стабильным. Анализ зависимости компонент биомассы – продукты окислительной термодеструкции показал, что в массовом отношении полисахариды и лигнин образуют примерно одинаковое количество продуктов (45 и 49% соответственно) при этом состав продуктов деструкции полисахаридов разнообразнее (23 и 16 наименований соответственно). Показано, что перечень продуктов окислительной термодеструкции биомассы Heracleum sosnowskyi Manden сопоставим с продуктами термического разложения лигноцеллюлозных материалов другого ботанического происхождения. Результаты представленного исследования могут быть использованы при расчетах процессов и установок для сжигания топливных гранул из биомассы Heracleum sosnowskyi Manden или комбинированного состава, а также оценки комплексного экологического влияния технологии энергетического использования биомассы борщевика Сосновского на атмосферный воздух.

Ключевые слова: биомасса *Heracleum sosnowskyi* Manden, топливные гранулы, окислительная термодеструкция, хромато-масс-спектрометрия, время удерживания, компонентный состав продуктов деструкции

INVESTIGATION OF THE COMPONENT COMPOSITION OF THE OXIDATIVE THERMAL DEGRADATION PRODUCTS OF FUEL PELLETS FROM THE *HERACLEUM SOSNOWSKYI* MANDEN BIOMASS BY CHROMATOGRAPHY-MASS-SPECTROMETRY

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The component composition of oxidative thermal degradation products of Heracleum sosnowskyi Manden biomass was studied by chromatography-mass-spectrometry. The hogweed Sosnovskyi biomass was collected on the territory of the village of Vylgort in the Komi Republic, dried to an atmospheric-dry state, crushed to $d_m < 0.25$ mm and pressed with a force of 10 kN. Fuel pellets were burned in the special device. The products of oxidative thermal degradation were captured and analyzed on a gas-liquid chromatography with a mass-selective detector. As a result of mass spectrums analysis, 39 low-molecular-weight products with a retention time of 6 to 79 min and a percentage content of 0.4 to 12% were identified from 47 captured compounds. Qualitative and quantitative analysis of the component composition of the degradation products showed that their formation is directly related to the component composition of the biomass and is determined by the content of cellulose, hemicellulose and lignin. Analysis of data on retention time of the degradation products showed that cellulose and hemicellulose of the biomass sample are degraded priority, while lignin is thermally more stable due to its aromatic nature. Analysis of data on products of oxidative thermal degradation showed that in mass terms, polysaccharides and lignin are formed as a percentage 45 and 49 respectively, while the composition of the products of destruction of polysaccharides is more diverse (23 and 16 names). It is shown that the list of products of oxidative thermal degradation of the Heracleum sosnowskyi Manden biomass is comparable to the products of thermal decomposition of lignocellulosic materials of other botanical origin. The results of the presented study can be used in the calculation of processes and installations for burning fuel pellets from the biomass of the Heracleum sosnowskyi Manden or a combined composition fuel pellets. As well as the results can be used to assess the complex environmental impact of the technology of the energy use of the biomass of the hogweed Sosnowskyi on the atmospheric air.

Key words: *Heracleum sosnowskyi* Manden biomass, fuel pellets, oxidative thermal degradation, chromatography-mass-spectrometry, retention time, component composition of the oxidative thermal degradation products

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INTRODUCTION

From the standpoint of the priority of high environmental and economic efficiency, plant raw materials have established themselves as an efficient energy fuel. Pellets and briquettes have strongly entered into use, and become an integral part of the fuel and energy complex. Thus, the environmental problems of the woodworking and agricultural industries are successfully solved by recycling the resulting waste. Despite the universality of the technology of energy use of plant raw materials and its renewable nature, the lack of data on the adverse effects of combustion products on atmospheric air limits the possible use of Sosnovsky hogweed [1]. Thus, the study of the composition and amount of products of the oxidative thermal destruction of the biomass fuel of the *Sosnovskyi* hogweed is actual.

OBJECT AND METHODS

Fuel pellets from shredded biomass of *Sosnovskyi* hogweed, dried to an atmospheric dry state, dm < 0.25 mm, pressed with a force of 10 kN, were combustion in installation for the experiment consisting of an electric tube furnace (quartz tube diameter 10 mm, heating rate 20 °C per minute for 30 min up to 600 °C – minimum combustion temperature) and cryo-trap (methanol, chemically pure, 2 ml, temperature -25 °C). The products of oxidative thermal destruction were analyzed on a gas-liquid chromatography with a mass-selective detector GCMS-QP2010 ULTRA, Shimadzu. Conditions: carrier gas – helium, flow rate – 1.1 ml / min, temperature gradient from 60 to 200 °C for 5 min, capillary column - DB-5ms, Agilent (d = 0.25 mm, 60 m, 0.25 μm), maximum detector sensitivity – El, SCAN: 1 pg OFN m/z 272 S/N > 1500. The components were identified automatically by analogy with the known mass spectra of the samples stored in the NIST 08 databases. The biomass of *Heracleum sosnowskyi* Manden, in addition to biopolymers - cellulose – 44% and lignin – 21-23 %, contains polysaccharides, proteins, tannins (tannins), essential oils, glutamine, carotene, vitamins C and E, folic acid, galactan, araban, furanocoumarins, macro- and microelements [2-6].

Pectin polysaccharides ~ 17%, of which 12.5% extracted by ammonium oxalate contain 83% glycuronic acids (up to 85% galacturonic acid), 4.7% galactose, 2.6% arabinose, 3.7% rhamnose, 0.6% xylose, 2.6% glucose, 0.5% mannose, as well as arabinogalactan proteins ~ 2% in the pectin-protein complex [7, 8], 4% furanocoumarins (xanthotoxin – 1.15%, bergapten – 1.04%, umbelliferone – 0.83%, angelicin – 0.63%, sphondin – 0.35) [9], essential oils of most cow hogweeds are represented mainly by monoterpenes, sesquiterpenes and esters.In Sosnovsky hogweed, their number is up to 1%, the main components are p-cymol – 0.48%, terpinolene – 0.17%, limonene – 0.14%, and γ-terpinene 0.082. In all species, by the end of the growing season, the content of esters increases and the amount of monoterpenes decreases [10].

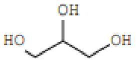
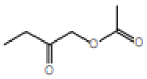
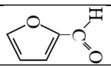
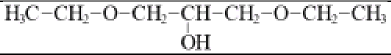
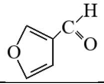
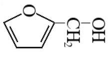
RESULT AND DISCUSSION

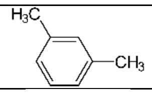
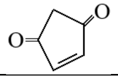
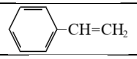
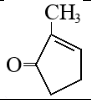
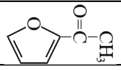
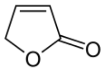
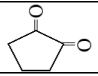
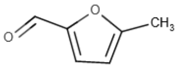
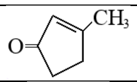
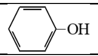
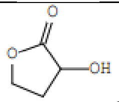
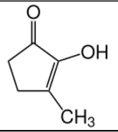
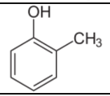
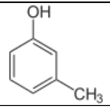
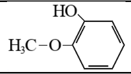
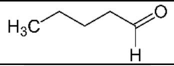
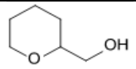
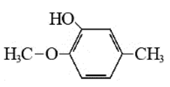
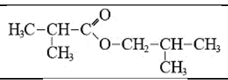
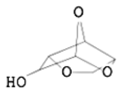
Low molecular weight products mixture composition of oxidative thermal destruction and their percentage were estimated by the retention time and the peak area on the ion chromatogram (Table 1, 2).

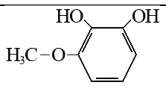
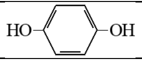
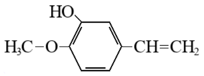
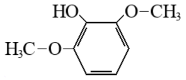
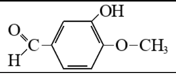
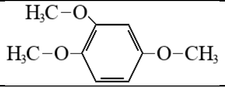
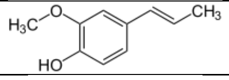
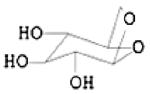
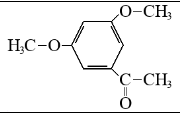
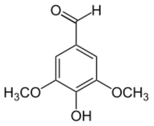
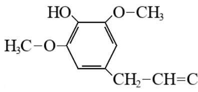
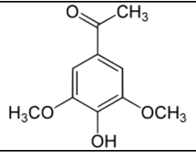
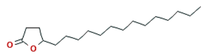
Table 1

Retention time of the oxidative thermal degradation products of *Heracleum Sosnowskyi* Manden biomass

***Таблица 1.* Время удерживания компонентов смеси продуктов окислительной термодеструкции биомассы борщевика Сосновского**

Compound	Molecular Formula	Structural formula	Retention time, min,
1	2	3	4
glycerin	$C_3H_8O_3$		5.94
1-acetoxy-2-butanone	$C_6H_{10}O_3$		6.63
furfural	$C_5H_4O_2$		7.73
1,3-diethoxy-2-propanol	$C_7H_{16}O_3$	 $H_3C-CH_2-O-CH_2-\underset{OH}{CH}-CH_2-O-CH_2-CH_3$	8.22
furancarbaldehyde-3 (furfural isomer)	$C_5H_4O_2$		8.33
2-hydroxymethylfuran	$C_5H_6O_2$		8.94

1	2	3	4
xylol	C_8H_{10}		9.60
2-cyclopentene-1,4-dione	$C_5H_4O_2$		10.11
styrene	C_8H_8		10.42
2-methyl-cyclopentene-2-one-1	C_6H_8O		11.03
2-acetylfuran	$C_6H_6O_2$		11.19
γ-crotonolactone	$C_4H_4O_2$		11.28
not identified	–		11.44
cyclopentanedione-1,2	$C_5H_6O_2$		11.67
5-methylfurfural	$C_6H_6O_2$		13.64
3-methyl-cyclopentene-2-one-1	C_6H_8O		13.78
phenol	C_6H_6O		14.34
3-hydroxydihydro-2(3H) -furanone	$C_4H_6O_3$		14.81
2-hydroxy-3-methyl-2-cyclo-pentene-1-one	$C_6H_8O_2$		17.02
o-cresol	C_7H_8O		18.60
m-cresol	C_7H_8O		19.84
guaiacol	$C_7H_8O_2$		20.93
pentanal	$C_5H_{10}O$		21.11
tetrahydropyran-2-methanol	$C_6H_{12}O_2$		27.13
4-methylguayacol	$C_8H_{10}O_2$		27.68
2-methylpropyl ether of 2-methylpropanoic acid	$C_8H_{16}O_2$		27.79
1,4:3,6-dianhydro-α-D-glucopyranose	$C_6H_8O_4$		28.60
not identified	–	–	29.83
not identified	–	–	31.50

1	2	3	4
2,3-dihydroxy-1-methoxybenzene	$C_7H_8O_3$		32.13
hydroquinone	$C_6H_6O_2$		32.70
4-vinylguayacol	$C_9H_{10}O_2$		35.69
syringol	$C_8H_{10}O_3$		38.09
vanillin	$C_8H_8O_3$		41.13
1,2,4-trimethoxybenzene	$C_9H_{12}O_3$		44.17
isoevgenol	$C_{10}H_{12}O_2$		44.35
levoglucosan	$C_6H_{10}O_5$		45.52
not identified	–	–	49.04
not identified	–	–	49.30
3,5-dimethoxyacetophenone	$C_{10}H_{12}O_3$		51.30
not identified	–	–	53.52
not identified	–	–	56.30
lilac aldehyde	$C_9H_{10}O_4$		56.70
6-methoxyevgenol	$C_{11}H_{14}O_3$		59.00
acetosyringol	$C_{10}H_{12}O_4$		60.76
not identified	–	–	62.79
γ-stearolactone	$C_{18}H_{34}O_2$		78.91

A total of 47 compounds were caught, of which 39 were identified. All compounds are degradation products of cellulose, hemicelluloses, and lignin. Analysis of the compound – retention time dependence showed that hemicelluloses and cellulose undergo destruction first of all, while lignin is thermally more stable due to its aromatic nature. The thermal stability of cellulose depends on the degree of crystallinity and the content of low molecular weight polysaccharides and lignin. The higher the degree of crystallinity, the lower the degree of delignification and the content of polysaccharides, the higher the thermal stability of the sample [11-13]. The degree of crystallinity of cellulose from the biomass of *Heracleum sosnowskyi* Manden averages 70 ± 2% [3], a typical value for cellulose samples of various botanical origin is 50-80% [14-16].

The chemical nature of the components of the biomass of *Heracleum sosnowskyi* Manden certainly predetermines the component composition of the products of oxidative thermal destruction (Table 2).

45% of all captured compounds are formed due to the destruction of hemicelluloses and cellulose, forming the largest amount of furancarbaldehyde-3, 2-methylpropyl ester of 2-methylpropanoic acid, γ-crotonolactone, 2-hydroxymethylfuran, pentanal and levoglucosan, etc. The component composition of the destruction products is comparable with the literature data for polysaccharides [17-19].

49% are lignin degradation products, most of which are syringol, guaiacol, 3,5-dimethoxyacetophenone and 4-vinyl guaiacol, etc.The component composition of the destruction products of this part of the biomass is comparable with the literature data for lignins of various origins [18-22]. The reliability of the identification results is also indicated by the ratio of the main monomeric structural units of lignin G:S:H = 45.2:45.0:9.8, calculated on the basis of Table 2, which correlates with the data [4], confirmed, among other things, by other methods.

Table 2

Component composition of the oxidative thermal degradation products of *Heracleum sosnowskyi* Manden biomass

***Таблица 2*. Компонентный состав смеси продуктов окислительной термодеструкции биомассы борщевика Сосновского**

Original compound	COMPOUND	QUANTITY, %	∑, %
1	2	3	4
cellulose, hemicelluloses	*furans*		
	furancarbaldehyde-3 (furfural isomer)	6.37	13.14
	2-hydroxymethylfuran	4.24	
	3-hydroxydihydro-2(3H) - furanone	1.10	
	2-acetylfuran	0.58	
	5-methylfurfural	0.46	
	furfural	0.39	
	lactones		
	γ-crotonolactone	4.38	6.35
	γ-stearolactone	1.97	
	ketones		
	cyclopentanedione-1,2	0.80	2.34
	2-cyclopentene-1,4-dione	0.53	
	3-methylcyclopentene-2-one-1	0.52	
	2-methylcyclopentene-2-one-1	0.49	
	aldehydes		
	pentanal	3.43	3.43
	esters		
	2-methylpropyl ether of 2-methylpropanoic acid	4.94	9.21
	1-acetoxy-2-butanone	2.44	
	tetrahydropyran-2-methanol	1.83	
	alcohols		
	1,3-diethoxy-2-propanol	2.39	3.65
	glycerin	1.26	
	enols		
	2-hydroxy-3-methyl-2-cyclopentene-1-one	1.73	1.73
	carbohydrates		
	levoglucosan	3.10	4.16
	1,4:3,6-dianhydro-α-D-glucopyranose	1.06	
	hydrocarbons		
	styrene	0.67	1.29
	xylol	0.62	
	∑		45.30
lignin	*G-unit*		
	guaiacol	7.30	18.49
	4-vinylguayacol	4.95	
	isoevgenol	2.40	
	vanillin	1.58	

Продолжение таблицы

1	2	3	4
lignin	4-methylguayacol	1.28	
	2,3-dihydroxy-1-methoxybenzene	0.98	
	S-unit		
	syringol	11.98	18.39
	6-methoxyevgenol	2.92	
	acetosyringol	1.92	
	lilac aldehyde	1.57	
	H-unit		
	phenol	1.36	4.00
	m-cresol	1.11	
	hydroquinone	1.02	
	o-cresol	0.51	
	other		
	3,5-dimethoxyacetophenone	5.87	8.12
	1,2,4-trimethoxybenzene	2.25	
	∑		49.00
∑			94.30

CONCLUSIONS

The qualitative and quantitative composition of the products of the oxidative thermal destruction of the biomass of *Heracleum sosnowskyi* Manden was determined by the method of gas chromatography-mass spectrometry.

Identified 39 (94.30%) low molecular weight components, which are products of the destruction of hemicelluloses, cellulose and lignin.

The largest number of identified compounds: furancarbaldehyde-3 (6.37%), 2-methylpropyl ester of 2-methylpropanoic acid (4.94%), γ-crotonolactone (4.38), 2-hydroxymethylfuran (4.24%), pentanal (3.43) and levoglucosan (3.10%), syringol (11.98%), guaiacol (7.30%), 3,5-dimethoxyacetophenone (5.87%) and 4-vinyl guaiacol (4.95 %).

It was shown that hemicelluloses and cellulose undergo destruction first of all, while lignin is thermally more stable.

The component composition of the products of oxidative thermal destruction directly depends on the component composition of the biomass and is determined by the content and structure of cellulose, hemicelluloses and lignin.

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