CHEMICAL STUDIES OF PARAMETERS AND COMPOSITION OF LIGNOCELLULOSE RAW MATERIAL SAMPLES OF MUNICIPAL ORIGIN

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Abstract. As a result of analytical studies, the main types of lignocellulose raw materials of communal origin were established (using the example of the city of Kyiv). It was found that the most common and promising for use in biofuel technologies are the following types of lignocellulose raw materials in groups: 1) lawn grass; roadside grass; 2) green trimmings of maple, linden, poplar, chestnut, oak, birch; 3) fallen leaves of maple, linden, poplar, chestnut, oak, birch. These types of trees make up 91.6% of the number of all perennial plantations, excluding coniferous trees (based on the analysed data of municipal enterprises of Kyiv). The largest share of fallen leaves (92.9%) is formed by three types of trees - poplar, maple and chestnut. Chemical studies were conducted on the composition of these types of raw materials, as well as three types of miscanthus and sugar sorghum, as plants that can take a promising place in urban landscapes. As a result of chemical studies, it was found that the highest ash content (mineral component) was observed in the dry matter of roadside grass (31.63%), fallen birch leaves (31.17%) and lawn grass (17.53%), the highest content of lignin is in fallen poplar leaves (30.29%), oak trimmings (29.41%) and fallen oak leaves (27.63%), the highest cellulose content is in miscanthus plants of all species (42.23-46.00%), sugar sorghum (34.07%) and fallen birch leaves (31.79%), hemicelluloses – in fallen maple leaves (36.66%), roadside grass (33.97%) and sugar-flowering miscanthus (33.96%), water soluble substances – in plants of sugar sorghum (33.62%), trimmings of linden (22.81%) and poplar (22.57%), pitches and fats – in fallen birch leaves (14.71%), fallen oak leaves (9.82%) and trimmings of birch (8.73%). The obtained results allow us to determine the most promising types of raw materials of communal origin for use in technologies of liquid and gaseous biofuels.

Keywords: lignin, cellulose, chemical composition, fallen leaves, tree trimmings.

Introduction

Modern biotechnology opens up great opportunities for the efficient use of lignocellulosic plant biomass [1-3], a significant proportion of which is municipal waste [4-6]. The use of lignocellulosic raw materials as a substrate for the production of biofuels by microbiological methods [7-9] involves the study of the influence of physical and mechanical chemical and biological methods of destruction of lignocellulosic complexes on the efficiency of biofuel production [10-12]. Studies [13-16] on increasing the efficiency of using lignocellulosic raw materials of various origins in liquid and gaseous biofuel technologies have shown that the use of effective methods of preparing raw materials and producer strains [17; 18] can increase the efficiency of fermentation. In turn, the search for and justification of effective methods of processing lignocellulosic raw materials [19] requires in-depth knowledge of the composition and parameters of these raw materials [20], in particular, the content of the main biopolymers – cellulose, hemicellulose, and lignin.

From the point of view of ensuring the efficient production of second-generation biofuels, especially by fermentation methods, lignocellulosic raw materials [7; 16], which are formed and accumulated with sufficient frequency and in the required quantities, are of the greatest interest for use. For countries with developed agricultural production, such raw materials are crop residues [7; 16; 20], the volume of which is comparable to the gross yield of grain crops and, for example, for Ukraine is more than 40 million tons per year [20]. Modern technologies ensure the collection, compaction and storage of this raw material. At the same time, most municipal utilities also provide centralized management of lignocellulosic raw materials of municipal origin, grinding and accumulating waste from tree trimming, grass mowing and fallen leaves.

In view of this, the overall objective of the research is to increase the efficiency of the use of municipal lignocellulosic raw materials in biofuel technologies by studying the chemical composition and determining the content of the main biopolymers that determine the efficiency of further fermentation of this raw material.

Previous studies [13; 18] have identified promising types of raw materials of agricultural origin, the most promising of which for the conditions of Ukraine is rapeseed straw. Further research on the application of promising methods of processing raw materials allowed to increase the yield of biobutanol per unit of dry matter by about 2.5 times [13; 19]. Ongoing research is aimed at identifying the most promising feedstocks of municipal origin for further efficient use in biofuel technologies.

Materials and methods

The predominant botanical (species) composition of lignocellulosic raw materials of municipal origin was determined in the example of the city of Kyiv [21], for which we used the information of the Unified State Web Portal of Open Data, in particular, the data of municipal enterprises [22] on the maintenance of green spaces in the districts of Kyiv. For the analysis, we selected datasets that contained information on the species composition of landscaping objects.

The data analysis revealed that 82.7% of all plantings (or 91.6% excluding conifers) are made up of only six tree species: maple, linden, poplar, chestnut, oak, and birch. The current practice of creating and repairing lawns does not exclude the use of different types of lawn grasses on the same site. On the contrary, the industry-specific municipal standards of Ukraine provide for the formation of grass mixtures for the creation and repair of different types of lawns. The structure of green infrastructure facilities includes objects that belong to both public plantations (parks, squares, etc.) and special-purpose plantations (traffic interchanges, protective strips, etc.). With this in mind, two generalized types of grass raw materials were identified for further research: lawn grass and roadside grass. Giant miscanthus and sugar sorghum were identified as promising plantation species [23] that can perform decorative and protective functions in urban landscapes. At the same time, the predominant propagation of giant miscanthus by rhizomes has both advantages, which include the possibility of creating and maintaining a viable perennial plantation of the species and improving the soil structure, and disadvantages, one of the main ones being the higher costs of forming a new plantation.

For chemical studies, organic raw materials of certain types of native moisture content were harvested in June-November 2023. Freshly cut branches up to 1.5-2.0 m long (up to 2 cm in diameter) with green leaf mass were harvested as tree trimmings. Fallen leaves of the relevant species were harvested during leaf fall in dry weather, without precipitation. Mowers with hopper storage were used to harvest the grass. Plants of giant miscanthus and sugar sorghum were cut whole. The raw materials were ground using a laboratory mill MILLER-2000 and, without storage, were immediately sent for chemical analysis (Fig. 1).

The main parameters of lignocellulosic raw materials were determined by known methods. The moisture content was determined according to [24] by the thermographic method using electronic moisture scales ADGS-50, which are registered in the State Register of Measuring Instruments of Ukraine under the number U1214-06.

The content of raw ash was determined according to the method [25] by drying the samples at a temperature of 60-65 ºC in an air-dry state, followed by calcining the crucible in a muffle furnace at a temperature of 525 ± 25 ºC for 2 hours, cooling in an evaporator, and weighing. The process is repeated until a constant crucible weight is achieved, when the difference in the results of two consecutive weighings does not exceed 0.001 g.

The cellulose content was determined by the nitrogen-alcohol method (Kürschner-Hoffer method) [26], for which a nitrogen-alcohol mixture consisting of one volume of concentrated nitric acid (density 1.4 $g \cdot mL^{-1}$) and four volumes of 95% ethanol was used. The lignin content was determined according to the method [27]. The content of hot water-soluble substances was determined by prolonged (3 hours) extraction of a 2% suspension of raw materials in distilled water, followed by filtration on a porous glass filter previously dried to a constant mass, drying the filter to a constant mass in an oven at 103 ± 2 ºC, and weighing. The total amount of hemicelluloses was determined by the method [28].

The determination of the content of pitches and fats was carried out using a Soxhlet-type unit according to the method [29]. The composition of ash was determined using a precision analyser "Expert 3L" (model U168), designed for direct operational non-destructive measurement of the mass fraction of chemical elements in samples. The measurements were performed by non-destructive energy dispersive X-ray fluorescence analysis.

The accuracy of the obtained results of measuring the parameters of the chemical composition of raw materials was due to the methods used to determine them.

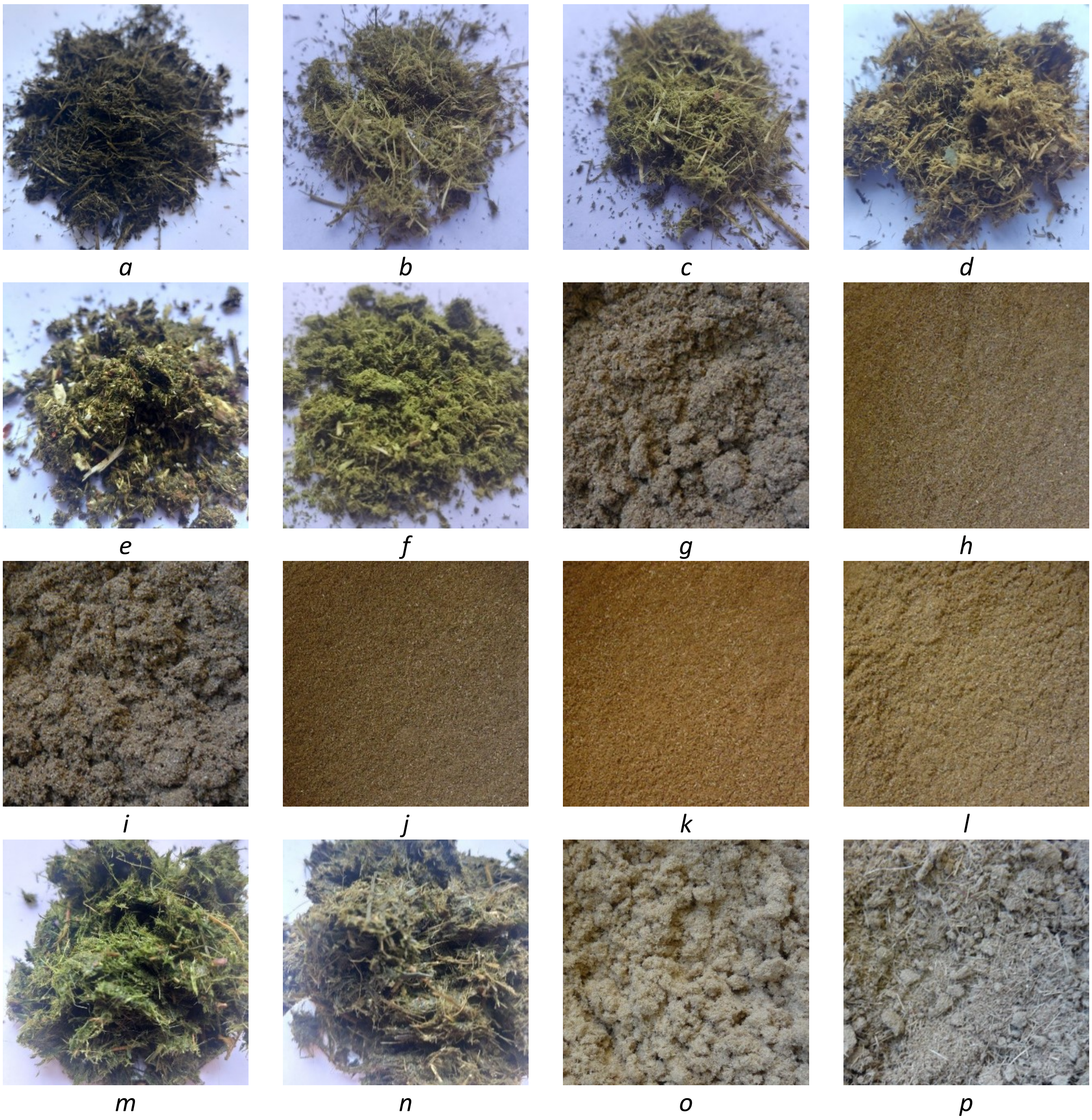

Fig. 1. **Experimental samples of lignocellulosic raw materials of municipal origin:** a – poplar (trimmed branches); b – oak (trimmed branches); c – birch (trimmed branches); d – linden (trimmed branches); e – chestnut (trimmed branches); f – maple (trimmed branches); g – poplar (fallen leaves); h –oak (fallen leaves); i – birch (fallen leaves); j – linden (fallen leaves); k – chestnut (fallen leaves); l – maple (fallen leaves); m – lawn grass clippings; n – roadside grass clippings; o – giant miscanthus; p – sugar sorghum

Results and discussion

The results of the raw material composition analysis are presented in Tables 1 and 2 and Fig. 2.

The chemical composition of the raw materials was analysed by the following groups: green branch trimmings, fallen leaves, and grass raw materials. Giant miscanthus and sugar sorghum were analysed separately. As it can be seen from the data in Tables 1, 2 and Fig. 2, the samples of giant miscanthus (44.71%), poplar leaves (25.71%), poplar branches (23.24%), and lawn grass (21.12%) have the highest cellulose content. The highest content of hemicellulose was found in maple leaves (31.84%), maple branches (28.73%), giant miscanthus (25.31%) and roadside grass (23.23%); pitches and fats – in birch leaves (10.12%), birch branches (8.73%) and lawn grass (4. 94%); water soluble substances – in sugar sorghum (30.75%), linden branches (22.81%) and oak leaves (17.61%); lignin – in oak branches (29.41%), poplar leaves (25.76%) and lawn grass (17.06%); ash – in roadside grass (31.63%), birch leaves (31.17%) and linden branches (11.14%).

Table 1

Composition of lignocellulosic biomass of municipal origin, %

Raw material	Moisture	Ash	Pitches and fats	Water-soluble substances	Cellulose	Hemi-cellulose	Lignin	Other
Poplar branches	61.21	6.73	5.12	22.57	23.24	21.06	20.85	0.43
Oak branches	53.18	6.94	4.00	21.07	21.71	16.35	29.41	0.52
Birch branches	46.32	5.79	8.73	18.42	16.95	27.87	21.65	0.59
Linden branches	56.03	11.14	5.35	22.81	20.67	19.85	20.00	0.18
Chestnut branches	65.13	9.98	3.29	17.96	18.95	25.81	24.00	0.34
Maple branches	51.06	9.01	4.73	17.97	19.43	28.73	20.36	0.20
Poplar leaves	21.57	14.94	5.33	11.70	25.71	16.74	25.76	0.62
Oak leaves	17.18	8.44	9.82	17.61	16.77	21.88	25.30	1.01
Birch leaves	31.03	31.17	14.71	8.67	21.88	15.38	12.58	0.20
Linden leaves	16.82	18.53	5.68	4.68	25.65	24.37	21.60	0.54
Chestnut leaves	17.41	14.07	7.44	8.08	21.97	24.89	23.90	0.70
Maple leaves	18.49	13.14	9.44	10.98	19.74	31.84	14.62	0.24
Lawn grass	73.59	17.53	5.99	16.02	21.12	23.22	17.06	0.11
Roadside grass	70.83	31.63	6.00	13.76	16.88	23.23	10.25	0.15
Giant miscanthus	15.65	2.80	0.29	13.01	44.71	25.31	13.77	0.12
Sugar sorghum	44.27	8.54	0.64	30.75	31.16	19.84	9.03	0.09

Table 2

Ash composition of lignocellulosic biomass

Raw material (ash)	Content of main components (mass fractions), %										
	SiO_2	K_2O	CaO	TiO_2	Fe_2O_3	SO_2	SrO	MnO_2	P_2O_5	ZnO	Cl
Poplar branches	9.344	33.570	43.708	0.094	0.303	4.829	0.071	0.087	6.365	0.256	1.372
Oak branches	16.113	23.874	48.742	0.118	0.373	1.569	0.051	0.233	8.793	0.134	–
Birch branches	3.157	19.005	65.293	–	0.416	1.908	0.120	0.177	8.947	0.977	–
Linden branches	7.415	19.096	61.866	0.119	0.332	1.332	0.136	–	7.826	ppm 479	1.875
Chestnut branches	8.494	32.976	41.095	0.113	0.443	2.137	0.059	–	9.587	ppm 373	5.059
Maple branches	6.962	19.166	63.141	0.116	0.334	1.365	0.120	–	6.873	ppm 479	1.876
Poplar leaves	20.157	17.380	56.851	–	0.596	2.153	0.097	0.144	2.322	0.299	–
Oak leaves	13.504	22.432	43.910	0.208	0.844	2.273	0.059	0.167	9.426	–	–
Birch leaves	60.741	5.816	25.476	0.420	2.841	0.959	–	0.235	–	0.120	–
Linden leaves	36.098	12.199	33.091	0.603	3.404	–	–	0.157	13.565	0.059	0.634
Chestnut leaves	21.550	12.529	41.091	0.383	2.500	2.457	–	0.158	13.519	–	2.814
Maple leaves	23.672	17.016	49.186	0.110	0.481	1.459	–	–	4.526	–	3.550
Lawn grass	26.229	37.402	14.817	0.263	1.472	3.408	–	–	6.058	0.073	10.268
Roadside grass	38.178	32.218	8.505	0.542	1.465	2.167	–	–	8.085	ppm 241	8.815
Giant miscanthus	47.908	28.260	8.901	0.098	0.720	–	–	–	13.043	–	0.643
Sugar sorghum	54.918	12.377	14.672	0.131	0.668	1.235	–	0.121	5.435	0.160	–

Analysis of the ash content shows the presence of soil contaminants (SiO_2), the content of which is significantly higher in fallen leaves compared to tree trimmings, the ash composition of which is dominated by calcium oxide (CaO). The presence of ZrO_2 in the studied samples was determined only in lawn grass in the amount of 105 ppm. In other samples of lignocellulosic raw materials, the ZrO_2 content was not recorded.

Comparative analysis shows that, compared to green branch trimmings, the total cellulose and hemicellulose content in fallen leaves increases in birch (7.56%) and poplar (1.85%) and decreases in other analysed species: linden (-9.50%), maple (-3.42%), chestnut (-2.10%), and oak (-0.59%).

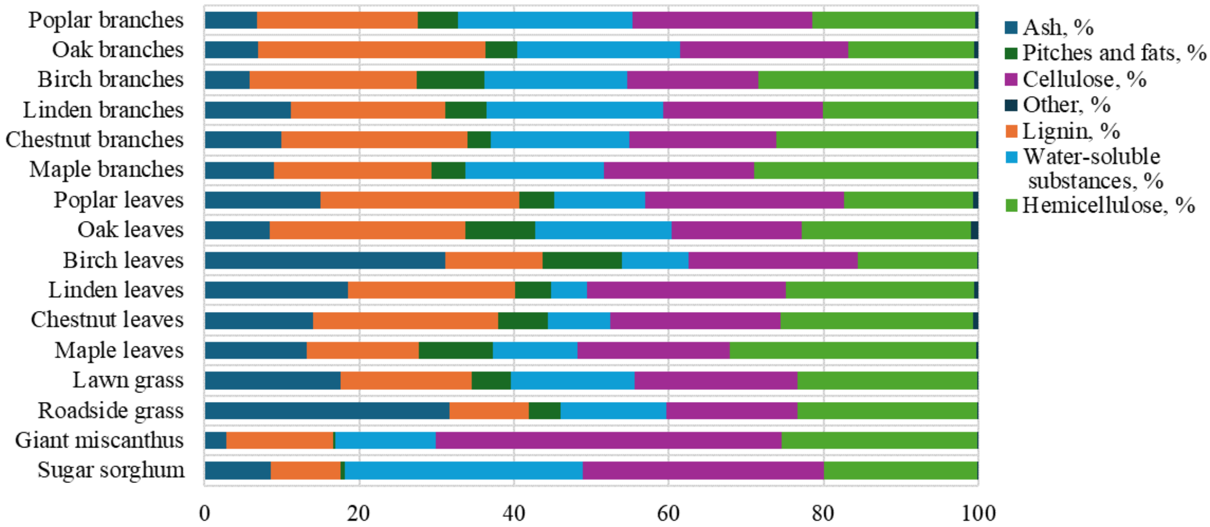

Fig. 2. **Composition of lignocellulosic biomass of municipal origin**

In general, the highest total cellulose and hemicellulose content is found in giant miscanthus (70.02%) and sugar sorghum (51.00%), among fallen leaves – in maple leaves (51.58%) and linden leaves (50.02%), among green branch trimmings – in maple branches (48. 16%), as well as in birch, chestnut, and poplar branches with the total cellulose and hemicellulose content in the range 44.30-44.82%, which allows us to conclude that these types of raw materials have a higher potential for further use in biofuel technologies produced by fermentation.

The data obtained correlate well with the known results of studies of the composition of biopolymers of lignocellulosic raw materials [30-32]. For example, the study [33] found the lignin content of palm grass leaves to be 20.30-21.66% and the cellulose content in fibres to be 64%, which makes this raw material comparable to that of giant miscanthus. The lignin content in municipal solid waste of 44.16-47.60% reported in [34] indicates the predominance of wood in its composition. The lignin and cellulose content of different poplar species studied in [35; 36] also correlates well with the obtained data.

Conclusions

1. Based on the first comprehensive analysis of open data sets of municipal enterprises, carried out on the example of the city of Kyiv, the main groups and species of plants that form lignocellulosic raw materials of municipal origin were identified, and the chemical composition of these species was analysed.
2. Based on an analysis of open data from Kyiv municipal enterprises, it was found that 82.7% of all tree plantations (or 91.6% excluding coniferous plantations) are made up of six tree species: maple, linden, poplar, chestnut, oak, and birch.
3. According to the results of the analysis of the chemical composition of raw materials, which was carried out in such groups as green branch trimmings, fallen leaves and grass raw materials, it was found that the samples of giant miscanthus (44.71%), poplar leaves (25.71%), poplar branches (23.24%) and lawn grass (21.12%) have the highest cellulose content. The highest content of hemicellulose was found in maple leaves (31.84%), maple branches (28.73%), giant miscanthus (25.31%) and roadside grass (23.23%).
4. The highest total cellulose and hemicellulose content is observed in giant miscanthus (70.02%) and sugar sorghum (51.00%), among fallen leaves – in maple leaves (51.58%) and linden leaves (50.02%), among green branch trimmings – in maple branches (48. 16%), as well as in birch, chestnut, and poplar branches, in which the total cellulose and hemicellulose content is in the range

(44.30-44.82%), which allows us to conclude that these types of raw materials have a higher potential for further use in biofuel technologies produced by fermentation.

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Author contributions

Conceptualization, V.B., T.T., S.S.; methodology, T.T., V.B. and S.S.; validation, T.T., O.T. and V.B; formal analysis, T.T. and O.T.; investigation, V.B., T.T., S.S. and O.T.; data curation, T.T., V.B. an O.T.; writing - original draft preparation, V.B., T.T.; writing - review and editing, S.S. and O.T.; visualization, V.B., T.T.; project administration, V.B.; funding acquisition, V.B., S.S. All authors have read and agreed to the published version of the manuscript.

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