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# CHEMICAL AND ENERGETICAL PROPERTIES OF SELECTED LIGNOCELLULOSIC RAW MATERIALS

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SYNOPSIS. The presented studies included young shoots of basket willow (*Salix viminalis* L.), stalks of woodland sunflower (*Helianthus tuberosus* L.), straw of perennial grass of miscanthus (*Miscanthus sinensis* (Thunb.) Anders.) and stalks of Virginia mallow (*Sida hermaphrodita* Rusby). In the studied lignocellulosic raw materials the following determinations were carried out: chemical composition (contents of soluble substances, cellulose, lignin and pentosans), elementary composition (contents of carbon, nitrogen, hydrogen and sulphur), heat of combustion as well as calorific value and ash content.

KEY WORDS: *Helianthus tuberosus* L., *Sida hermaphrodita* R., *Miscanthus sinensis* (Thunb.) Anders., *Salix viminalis* L., chemical properties, energetic properties

## INTRODUCTION

Poland, being a member of the European Union, is obliged to increase its share of heat and electric energy obtained from renewable resources to the level of 12.9% by the year 2017. In the geographical and climatic conditions of our country, the plant biomass, including wood, is the only accessible resource of renewable energy (Dyrektywa 2001/77/WE, Rozporządzenie... 2000, 2008). The undertaken obligations awake fears that some wood assortments (pulpwood) and wood waste (sawdust) will be directed to the energetic sector creating a deficit of these raw materials for the production of cellulose and paper, as well as wood-based boards. Since the Polish State Forests do not plan any increase in the currently existing cutting plans set for a 10-year period, there is an urgent need to implement lignocellulosic raw materials as an alternative material to wood both in industrial processing and for energy purposes.

The expected deficit of industrial wood raw material could be supplemented by obtaining lignocellulosic biomass originating from plant cultivations on agricultural areas: agro-based resources (Stolarski et al. 2006, Majtkowski and

Majtkowska 2008). Trees and perennial plants such as *Salix* spp. and *Populus* spp., Virginia mallow (*Sida hermaphrodita* Rusby), woodland sunflower (*Helianthus tuberosus* L.), giant knotwood (*Reynoutria sachalinensis* F. Schmidt), Japanese rose (*Rosa multiflora* Thunb.), grasses like *Miscanthus giganteus*, *Miscanthus sinensis* Thunb., and annual plants like different cereals belong to plants which in a short vegetation period can supply a significant amount of biomass.

Lignocellulosic raw materials such as cereal straw, reed, bamboo, bagasse, hemp, flax, jute plant have been utilized for many years in the cellulose-paper industry (Surmiński 1989). Recent reports from our country inform about searches being carried out in order to find poplar clones being most suitable for production of cellulose and paper (Wnorowska 2008).

Industry of wood-based materials is also interested in agro-based resources which can replace wood. Among others, trials are made to implement into the production of wood-based panels, such raw materials as miscanthus, straw of cereals and of rape, robinia, willow, woodland sunflower and maize (Frąckowiak 2007, Kowaluk et al. 2008, Meinlschmidt et al. 2008, Danecki et al. 2008).

So far, the greatest interest as an energetic raw material has been evoked by willow, mainly because of its quick growth and small requirements in reference to the soil. Wood of different willow species has been applied in the basket-making and plaiting industry as well as in paper industry, while the bark of willow (*Salix* spp.) – because of its high content of salicylates and tannins – is suitable raw material for the pharmaceutical and chemical industries. Thanks to the ability to absorb heavy metals from the environment, willow is used for the purification of sewages, for the recultivation of areas damaged by industry and for the creation of protective zones around drinking water intakes and at roads with a high intensity of car traffic (Kluska 1996, Pestka 1996, Prądzyński et al. 1996, Obarska-Pempkowiak and Kołecka 2005). According to Szczukowski (2006), the output of basket willow (*Salix viminalis* L.) amounts from 11.0 to 26.4 t/ha per year, whereas every 3 years the harvest of this species gives the best results – on the average 21.6 t/ha per year. However, Majtkowski and Majtkowska (2008) report that the productivity of willow plantation has shown to be lower than the experimental yields.

Similarly as basket willow, also woodland sunflower (*Helianthus tuberosus* L.), which has been known in Poland since the 19th century under the name of 'topinambur', is characterised by low requirements regarding the habitat. This species finds a wide application as a fodder plant. In the industry, this raw material is used for the production of bioethanol and biogas. Tubers of woodland sunflower, because of their inulin content, are recommended as a diet component for diabetics. In Poland, the average yield of green parts of *Helianthus tuberosus* range from 31 to 75 t/ha, while the yield of tubers may amount from 12 to 36 t/ha, whereby the obtaining of the green parts of this plant radically decreases the yield of its subterranean parts (Piskier 2007).

Perennial grasses from *Miscanthus genus* (*M. giganteus*, *M. sinensis*, *M. sacchariflorus*) can be cultivated in Polish climatic conditions (Majtkowski 2007). These grasses do not require soils of high quality and give yields already in the first year after the establishment of the plantation. The obtained yields increase with the age of the plantation and they range from several tons per ha during the first harvest to 20-30 t/ha in the third year of the plantation.

Similarly as other species of perennial plants, Virginia mallow (*Sida hermaphrodita* Rusby), in the third and fourth year of plantation reaches the highest yields. Borkowska (2007) reported that the yields of dry matter on sandy soils amounted from 9 to 11 t/ha, while on more tight soils, they ranged from 11 to 17 t/ha.

The life of plantations of perennial energetic plants reaches even 20 years. Cultivations in plantations are exposed to the danger of pests and plant diseases. However the biggest threat to the environment in Poland is uncontrolled spreading of plants which escaped from captivity of cultures, especially plantations. An example of that is *Helianthus tuberosus* L. which escaped from the control of planters and occupied fallows as well as municipal areas forcing out other native plant species (Alien Species in Poland). In order to prevent this phenomenon, stress must be placed on a careful removal of any rests of plant biomass on the field after harvest, particularly in case of perennial plants, and care should be taken for the further management of the remains.

A proper and effective use of the lignocellulosic plant biomass as industrial and energetic raw materials depends on their chemical and physical properties.

## OBJECTIVE OF THE RESEARCH PROJECT

The objective of the presented work was the determination of physico-chemical properties of selected lignocellulose materials designed for industrial use (as composite materials) or for energetic purposes. In the scope of our research the fast growing plants were used as raw materials.

## MATERIALS AND METHODS

The presented studies included one-year-old basket willow (*Salix viminalis* L.), woodland sunflower known also as Jerusalem artichoke (*Helianthus tuberosus* L.), perennial grass *Miscanthus sinensis* (Thunb.) Anders. and Virginia mallow (*Sida hermaphrodita* Rusby). Material for studies was obtained from experimental cultivations of the University of Warmia and Mazury in Olsztyn. Biomass samples (willow shoots in bark, stalks of sunflower and mallow and straw of miscanthus) were air-dried until they acquired a dry condition, then they were cut into 5-cm long segments which were disintegrated in a knife-mill or impact mill into grains of the desired size.

In samples of 0.5-1.0 mm grain size, the method of Prosiński (1984) was applied to obtain the following determinations:

- moisture by oven-dry metod,
- content of extraction substances by Soxhlet method (ethanol-benzene mixture in 1:1 proportion),

– content of substances soluble in cold and hot water,
– content of substances soluble in 1% water solution of NaOH,
– content of cellulose by Seifert method,
– content of Klason lignin by Tappi method,
– content of pentosans by Tollens method.

Content of ash was determined at 550°C according to DIN 51731.

In samples of grain sizes < 0.20 mm, the elementary composition was determined (content of carbon, nitrogen, hydrogen and sulphur) using the elementary analyser Flash 1112 of Thermo Electron Corporation. The determination was done in three replications, the weighed portions of samples showed 3-4 mg each. For the calibration of the instrument, sulfanilamide standard was applied.

Heat of combustion and calorific value were determined according to the standard PN-81/G-04513 using the calorimeter KL-12 Mn of Precyzja Bit Company.

## RESULTS AND DISCUSSION

Results of the chemical composition of the studied raw materials: one-year-old shoots (unbarked) of basket willlow, stalks of woodland sunflower and Virginia mallow as well as straw of miscanthus are shown in Table 1. Data taken from literature and referring to the chemical composition of mature wood from selected hardwood species (white willow and white poplar) and softwood species (common pine) (Prosiński 1984), as well as agro-based materials, such as *Miscanthus sinensis*, *Helianthus tuberosus* and *Sida hermaphrodita* (Han and Rowell 1997) are presented in Table 2.

Table 1. Chemical composition of biomass from selected lignocellulose raw materials

| Components | | *Helianthus tuberosus* | *Miscanthus sinensis* | *Sida hermaphrodita* | *Salix viminalis* |
|---|---|---|---|---|---|
| | | the percentage of oven dry mass | | | |
| Moisture | | 8.11 | 7.06 | 7.54 | 6.22 |
| Substances soluble in | ethanol-benzene mixture | 3.00 | 1.16 | 1.21 | 5.34 |
| | cold water | 2.12 | 5.07 | 1.18 | 6.60 |
| | hot water | 4.68 | 7.59 | 3.12 | 10.37 |
| | 1% NaOH | 25.66 | 36.34 | 25.62 | 34.47 |
| Seifert Cellulose | | 40.95 | 43.18 | 41.02 | 39.29 |
| Pentosans | | 22.65 | 25.20 | 24.77 | 17.05 |
| Klason Lignin | | 20.48 | 23.02 | 20.52 | 26.04 |

The studied raw materials differed both in their main and secondary wood components. Wood of basket willow and stalks of woodland sunflower, in our own studies contained more substances soluble in ethanol-benzene mixture (5.34%

Table 2. Chemical composition of selected wood and agro-based raw materials – data from literature (Prosiński 1984, Han and Rowell 1997)

| Components | | *Salix alba*[a] | *Pinus sylvestris*[a] | *Populus alba*[a] | *Miscanthus sinensis*[b] | *Sida* spp.[b] | *Helianthus tuberosus*[b] |
|---|---|---|---|---|---|---|---|
| | | the percentage of oven dry mass | | | | | |
| Substances soluble in | ethanol-benzene mixture | 2.2[c] | 4.6[c] | 5.7 | 4.0 | 4.0 | 6.4 |
| | cold water | 1.3[c] | 2.1[c] | 1.2 | – | – | – |
| | hot water | 2.7[c] | 6.4[c] | 2.0 | – | – | – |
| | 1% NaOH | – | – | – | 37.8 | 28.3 | 38.7 |
| Seifert Cellulose | | 43.6[ce] | 48.1[ce] | 52.4[e] | 45.5[d] | 51.2[d] | 48.5[d] |
| Pentosans | | 21.5[c] | 9.8[c] | 21.8 | – | – | – |
| Klason Lignin | | 25.0[c] | 28.8[c] | 20.4 | – | – | – |

[a]Prosiński (1984), [b]Han and Rowell (1997), [c]mean value, [d]Cross and Bevan cellulose, [e]Seifert cellulose.

and 3.00%, respectively) than miscanthus (1.16%) and Virginia mallow (1.21%) (Table 1). However, the mean content of extraction substances in the mature wood of pine, willow and poplar (Table 2) was in the range of 2.2-5.7%, while for agro-based raw materials the range was from 4.0% to 6.4%.

In miscanthus, the content of substances soluble in cold water was 5.07% and the content of substances soluble in hot water was 7.59%, in willow, the values were 6.60% and 10.37%, respectively. Thus, the quoted values were higher than in mature wood of pine (on the average: 2.1% and 6.4%, respectively) or in poplar (1.2% and 2.0%, respectively). On the other hand, in the stalks of *Helianthus tuberosus* and particularly in *Sida hermaphrodita*, they were similar to the contents of these substances in mature wood of hardwood tree species (Tables 1 and 2).

In our own studies, the content of substances soluble in 1% solution of NaOH was similar in the stalks of *Helianthus tuberosus* and *Sida hermaphrodita* (25.66% and 25.62%, respectively), while for willow, the value was 34.47% and for miscanthus 36.34%. Han and Rowell (1997) reported higher content of substances soluble in 1% NaOH for *Sida hermaphrodia* and *Helianthus tuberosus* (28.3% and 38.7%, respectively), while for miscanthus – 37.8% (Table 2). The differences may result from the age of the raw material, from the time and place of cultivation, as well as from other cultivars of the studied plants.

The content of Seifert cellulose in the tested resources ranged from 39.29% for basket willow to 43.18% for miscanthus (Table 1). In mature wood of willow and pine, the content of Seifert cellulose was on the average 43.6% and 48.1%, while in poplar, it was 52.4%. In the biomass of lignocellulosic plants, the content of cellulose determined by the Cross and Bevan method oscillated between 45.5% for miscanthus and 51.2% for *Sida hermaphrodita* (Table 2). This method gives higher cellulose content results than the Seifert method (Prosiński 1984).

The content of pentosans in the studied lignocellulose raw materials: *Miscanthus sinensis*, *Sida hermaphrodita* and *Helianthus tuberosus* showed the following values: 25.20%, 24.77% and 22.65%, respectively (Table 1) and the values were higher than in mature white willow wood (on the average 21.5%) and in white poplar wood (21.8%) (Table 2). One-year-old shoots of basket willow contained less pentosans than the studied wood-free plants (17.05%) (Table 1) and the mature wood of white willow (Table 2).

The content of lignin in unbarked wood of basket willow (26.04%) was comparable with the mean content of this component in mature wood of white willow (25.0%) (Table 1 and 2). On the other hand, straw of miscanthus contained 23.02% of lignin, while stalks of *Sida hermophrodita* and Helianthus tuberosus contained 20.52% and 20.48% respectively. The latter values are at the same level as the lignin content in white poplar (20.4%) (Table 2).

The results of the studies on the properties of lignocellulose raw materials, which are significant for the energetic industry, are shown in Table 3. The values have been compared with the data quoted in the literature for pine wood, willow wood and brown coal (Table 3). Table 4 presents the typical properties of selected solid biofuels (Technical Specification 2007).

Carbon content in the studied raw materials oscillated from 45.0% in willow to 47.1% in miscanthus and it was lower than typical values of this parameter

Table 3. Elementary composition and energetic values (heat of combustion, calorific value, ash content) of the studied lignocellulosic raw materials in comparison with mature wood of pine and willow, and brown coal

| Raw material | Heat of combustion (analytical) | Calorific value in state | | | Elementary composition | | | | Ash |
|---|---|---|---|---|---|---|---|---|---|
| | | analytical | working | dry ash-free | C | H | N | S | |
| | MJ/kg | | | | the percentage of oven dry mass | | | | |
| *Helianthus tuberosus* | 16.128 | 14.588 | 14.699 | 16.653 | 45.8 | 6.1 | 0.3 | n.d. | 2.5 |
| *Miscanthus sinensis* | 17.698 | 16.233 | 16.093 | 18.272 | 47.1 | 6.0 | 0.5 | n.d. | 3.9 |
| *Sida hermaphrodita* | 17.224 | 15.717 | 15.768 | 18.300 | 47.0 | 6.0 | 0.2 | n.d. | 1.8 |
| *Salix viminalis* | 18.150 | 16.715 | 16.876 | 18.489 | 45.0 | 5.8 | 0.7 | n.d. | 1.8 |
| *Pinus sylvestris** | – | – | – | 19.2 | 53.2 | 5.9 | 0.1 | 0.06 | 0.4 |
| *Salix* spp.* | – | – | – | 18.4 | 47.1 | 6.1 | 0.5 | 0.05 | 2.0 |
| Brown coal* | – | – | – | 22.0-27.0 | 63.0-74.0 | 4.9-6.0 | 0.7-1.9 | 0.3-3.9 | 1.3-15.0 |

*CICHY (2007).
n.d. – not detected.

Table 4. Typical values of solid biofuels for Europe – data obtained from Swedish, Danish, Dutch and German studies (Technical Specification 2007)

| Raw material | Heat of combustion dry, ash free state | Calorific value dry, ash free state | Elementary composition | | | | Ash |
|---|---|---|---|---|---|---|---|
| | | | C | H | N | S | |
| | MJ/kg | | the percentage of dry ash free state | | | | |
| Grasses, general | 19.4 | 18.4 | 49 | 6.3 | 1.4 | 0.2 | 7 |
| *Miscanthus sinensis* | 19.8 | 18.4 | 49 | 6.4 | 0.7 | 0.2 | 4 |
| Rape straw | 19.8 | 18.5 | 50 | 6.3 | 0.8 | 0.3 | 5 |
| Wheat, rye, barley straw | 19.8 | 18.5 | 49 | 6.3 | 0.5 | 0.1 | 5 |
| Bark of hardwood | 21 | 20 | 55 | 6.1 | 0.3 | 0.10 | 5 |
| *Salix* spp. | 20.3 | 18.8 | 49 | 6.2 | 0.5 | 0.05 | 2.0 |
| Softwoods | 20.5 | 19.2 | 51 | 6.3 | 0.1 | 0.02 | 0.3 |
| Hardwoods | 20.2 | 19.0 | 49 | 6.2 | 0.1 | 0.02 | 0.3 |

(about 49%) for agro-based solid fuels. Hydrogen content (5.8-6.1%) in the tested lignocellulosic materials was similar to the content of this element in agro-based solid fuels (ca. 6.3%) (Table 3 and 4).

The greatest differences between the studied agro-based resources occurred in the content of nitrogen (sida – 0.2%, sunflower – 0.3%, miscanthus – 0.5% and willow – 0.7%). The content of this element in the tested materials was lower in reference to the data typical of biofuels (0.5-1.4%) (Table 3 and 4).

The content of sulphur in the studied materials was below analytical detection level of method used for elementary components determination (Table 3). Sulphur content in agro-based resources and wood is usually very low and ranged from over 0.01 to 0.3% according to Technical Specification (2007).

The average ash content in woodless solid biofuels ranges from 4 to 7% and for decidous (hardwood) and coniferous (softwood) is 0.3%, for bark of decidous trees – 5% and for willow wood – 2% (Table 4). Ash content in the studied biomass samples was 1.8% in willow and in sida, 2.5% in woodland sunflower and 3.9% in miscanthus, and it was lower than the quoted values typical of plant raw materials and than ash content in brown coal (Table 3 and 4).

Combustion heat of the studied raw materials oscillated between 16.128 MJ/kg for woodland sunflower (*Helianthus tuberosus* L.), and 18.150 MJ/kg for basket willow (*Salix viminalis* L.) (Table 3).

Calorific value was calculated for the studied materials in the moist analytic state and in the working state (as received) as well as in dry ash-free state – on the basis of a biomass sample from which the total moisture and the ash have in theory been removed (Table 3). Fuel calorific value depends on the content of moisture and ash. The higher the content of water and of non-combustible substances in the fuel, the lower is the calorific value of the fuel. In practice, the content of ash and moisture are important in the moment of fuel dozing into the stove furnace, however, for comparative purposes, it is accepted to quote the calorific value in the dry ash-free state (theoretical value). Comparison of the calorific value of the studied raw materials has shown that this parameter is low for moist material, and it shows the highest value for material in the dry ash-free state. The calorific values of the studied samples of *Miscanthus* and *Sida hermaphrodita* showed in the dry ash-free state the following values: 18.272 MJ/kg and 18.300 MJ/kg, respectively, for willow the value was: 18.489 MJ/kg and for *Helianthus tuberosus*: 16.653 MJ/kg (Table 3). Thus, *Helianthus tuberosus* was characterised by the lowest calorific value, while basket willow (*Salix viminalis*) showed the highest calorific value among the studied materials. The average calorific value in the dry ash-free state for monocotyledonous plants (grasses, straw, cereals, reed), according to Technical Specification (2007) is 18.4-18.5 MJ/kg, for deciduous trees (hardwood) and coniferous trees (softwood) the value is 19.0-19.2 MJ/kg and for basket willow it is 18.4-18.8 MJ/kg (Table 3 and 4). Among the studied plants only *Helianthus tuberosus* showed a lower calorific value than the range presented in the specification (Table 3 and 4), but the value was also lower than the requirements of the standard DIN 51731 for fuels obtained from pressed wood particles (17.5-19.5 MJ/kg). Calorific value of the other raw materials, and particularly of willow (18.489 MJ/kg) complied with the requirements of DIN 51731 (1996) standard for wood. However, the content of nitrogen (0.7%) and of ash (1.8%) in willow exceeded the limits given in the standard (over 0.3% of nitrogen and over 1.5% of ash). The other agro-based materials also did not meet the requirements of DIN 51731 standard in the range of the contents of ash and nitrogen, in spite of the fact that the content of these substances was lower in the studied plants than the average content presented in Technical Specification (2007) (Table 4).

In comparison with brown coal, biofuels are characterised by a lower content of ash (2-4 times lower) and by a lower calorific value (by about 40%) (Table 4). Therefore, one can believe that in reference to an energy unit obtained in the combustion process the amount of ash obtained from biofuel might be higher than that obtained from brown coal. Kalembasa (2006) reported that ash from biomass combustion contains macro- and microelements brought together with the yield from the soil and it can be utilized for the fertilization of agricultural cultivations.

## CONCLUSIONS

1. The greatest amounts of cellulose, pentosans and lignin were contained in straw of Miscanthus sinensis (43.18%, 25.20% and 23.02%, respectively). Wood of willow contained less of Seifert cellulose (39.29%) than *Helianthus tuberosus* (40.95%) and *Sida hermaphrodita* (41.02%), a smaller amount of pentosans 17.05%, as compared with 22.65% and 24.77% for the two earlier mentioned materials, and the greatest amount of lignin (26.04%).
2. The elementary composition (C, H, N, S) of the studied raw materials (C: 45-47%, H: ca. 6%, N: 0.2-0.7%, S: not detected) did not deviate from elementary composition typical for organic matter (C: ca. 49%, H: ca. 6%, N: 0.5%, S: ca. 0.05%).
3. Calorific values of Miscanthus and Sida hermaphrodita in dry ash-free state amounted to 18.3 MJ/kg; willow *Salix viminalis* showed 18.5 MJ/kg and *Helianthus tuberosus* – 16.7 MJ/kg.
4. On the basis of chemical composition and energetic properties of Virginia mallow (*Sida hermaphrodita* Rusby), miscanthus (*Miscanthus sinensis* Thunb.), woodland sunflower (*Helianthus tuberosus* L.) and basket willow (*Salix viminalis* L.) it was found that these species are potential sources of the valuable lignocellulose raw materials which are adequate both for the industrial and energetic use.

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