

## EXAMINATION OF ASH CONTENT FOR DIFFERENT SPECIES OF SHORT ROTATION COPPICE

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### Abstract

*The conversion of lignocellulose biomass to liquid biofuels requires a far more complex technology than food crop-based first generation biofuel production does. Conversion rate and production capacity are also lower, but for advanced biofuel plants, the investment cost can be up to ten times higher. However, recent technological developments have made it possible to produce biofuels without endangering the continuous food supply. Lignocellulose biofuels can be produced from a wider range of raw materials such as forest residues and agricultural by-products. Unfortunately, collecting woody and herbaceous by-products is costly; therefore, we investigated the usability of short rotation coppices (SRC) as base material for biofuel production. The plantations are suitable for producing a large amount of biomass in a small area, and even in non-utilized agricultural areas. The dendromass from SRC is characterized by high bark content, which generates a high ash amount during utilization. Several studies confirm that bark content adversely affects both biochemical and thermochemical processes; our studies examined the importance of cutting-cycle selection and tree species on ash content during thermal treatment.*

**Keywords:** bark, ash, bioenergy, dendromass, SRC

### 1. INTRODUCTION

Oil is the most widely-consumed energy source, amounting to 32.3% of global energy consumption in 2016. Its use in the transport sector is continuously increasing [1]. To achieve Paris World Conference targets, it is necessary to create a new technological and political framework to convert the energy structure. Over the past decade, the EU has set numerous biofuel guidelines for Member States. Crop-based biofuels were encouraged initially, but policymakers eventually recognized the dangers of land use change on sustainable food supply. According to a survey, the cultivation area of base materials for biofuels covered approximately 2.9% of the total agricultural area in 2014 [2]. Today, crop yield loss due to climate change already disrupts the sustainable raw material supply of first generation biofuel plants. This is complemented by an expected doubling of cereal consumption in the 21st century due to population growth and the change of consumption patterns [3]. For the above reasons, the European Union intends to limit the use of food crops for energy purposes in the future. As an encouragement, the energy content of biofuels produced from lignocelluloses is counted by double multiplication according to RED II (Renewable Energy Directive II).

Lignocellulose biofuels can be produced from a wide range of raw materials including agricultural by-products, forest residues, and other non-food crops such as short rotation coppices (SRC). The literature supports establishing SRC on low-quality agricultural areas with the provision of appropriate tree species selection and careful soil preparation [4]. Drought and flood-affected areas, abandoned grasslands with shallow crop layers, and areas prone to erosion and deflation exemplify low-quality agricultural areas [5-7]. Moreover, experiments have proven that mining areas awaiting re-cultivation and denominated areas affected by red sludge pollution are also appropriate for short rotation woody energy plantation cultivation [8-10]. This avoids the risk of a food conflict and reduces agricultural sector carbon emissions by binding carbon into the soil and into the dendromass over the growing period.

The conversion of lignocellulose materials into biofuels requires a far more complex technology than first-generation biofuel production does. Conversion rate and production capacity are lower, but the

investment cost can be up to ten times higher for advanced biofuels plants. Conversion methods for lignocelluloses for liquid biofuels may be biochemical and/or thermochemical. Generally, thermochemical processes like pyrolysis, gasification, and direct thermal liquefaction show greater efficiency and shorter reaction time than biochemical processes [11-13]. The ash content generated by inorganic components and contaminations of biomass reduces the efficiency of the biofuel production processes [14]. According to literature sources, the comparison of biomasses with high lignocellulose content shows great differences in the typical parameter of thermal treatment (see Table 1.). Many factors, including biomass type, age, growing area, cultivation technology, etc., influence the properties [15].

	<b>Ash content [%]</b>
<b>SRC Salix Spp.</b>	1.0-2.8
<b>SRC Populus Spp.</b>	1.9-3.0
<b>SRC Robinia pseudoacacia</b>	2.1- 2.9
<b>Forest tree species</b>	0.4-0.9
<b>Vágástéri apadék</b>	1.0-10.0
<b>Wheat straw</b>	5.8-8.3
<b>Corn stalk</b>	3.5-8.8
<b>Rape stalk</b>	2.8-5.1
<b>Miscanthus</b>	2.6-9.5
<b>Energiafű</b>	4.8-10.0

**Table 1.** Ash content of different high lignocellulose content biomass [16-21]

According to Molnár-Bariska (2002), about 80-85% of the ash content is generated by the bark during the firing process [22]. The bark contains a higher inorganic element than wood. Hence, the ash content of debarked wood is generally less than 1%, while the ash content of wood with bark is between 1.5% and 3.5% [23]. These values for hardwood logging waste is 2.3–3.5%, while for poplar tree plantations, they are 0.9–3.2% [24]. The high ash content with the high alkali metal (Na, K) and high alkaline earth metal content (Mg, Ca) can cause unstable flames and combustion inefficiencies as well as emissions. In addition to bark content, the generated amount of ash is greatly influenced by the presence of soil residues that may be formed during harvesting. The ash content of highly contaminated dragged tree bark can reach values ranging from 6.0% to 14% [23]. Excessive contamination causes increased wear and tear of treatment and processing systems, and increased instability of pyrolysis oil. The alkali metal compounds can react with the silica present in the ash and produce a sticky, mobile liquid phase, which can lead to airway blockages in the furnace [25]. Moreover, the biomass components determines the ash melting point, the high Si content of base material increase the slag formation [26]. Proper selection and pre-treatment of the raw material and applied growing technology during the cultivation play an important role in ash content reduction [27]. Furthermore, reactor design can also greatly facilitate the processing of raw materials with high ash content [28-29].

Dendromass use from SRC for direct firing is already widespread. At the same time, the usability of the thin shoots with high bark content for large-scale thermochemical biofuel production should be examined. According to the results of Tröger et al. (2013), the bio-oil yield decreases in parallel with increasing ash content when tested in a 10 kg/h reactor [30]. Dou et al. (2017a) reached a similar conclusion with the compared pyrolysis oil yield of a 2-year-old poplar SRC plantation and a 12-year-old poplar short-rotation forestry plantation. According to their results, the yield of pyro-oil from SRC was 5% lower [31].

Sebestyén (2014) determined that steam explosion pre-treatment effectively removes sodium and potassium from wood samples. The process is always carried out in an aqueous medium, so part of the sodium and potassium ion content of the dendromass is dissolved by the structural changes. An even more efficient method is the 60 °C water wash, which extracted 90% of the potassium ions and 80% of the sodium ions during their experiment [32]. Mészáros et al. (2004) applied thermogravimetric/mass spectrometric analysis for comparing the thermal decomposition characteristics of wood and of bark for young shoots (poplar, willow, black locust) from SRC. According to their measurements, the mineral content of the bark is significantly higher, of which a great part has been removed by a washing procedure [33]. Raavendran et al. (1995) used a two-step washing procedure. In the first step, the base material was treated with 10% hydrochloric acid at 60 °C for 48 hours with continuous stirring and then with a second wash with 5% NaOH at 90 °C for 1 hour. Then the biomass was washed with distilled water, filtered, and dried. This treatment improved the yield, but it is not economical under industrial conditions [34]. Recent research suggests a more environmentally-friendly and less costly procedure. Oudenhoven et al. (2013) removes minerals from biomass with the help of organic acids from pyrolytic processes. In their experiments, the vapors produced by rapid pyrolysis of pine wood are condensed in two steps, and with the resulting liquid at 90 °C the alkali metals and alkaline earth metals from the raw material are removed in 2 hours. This process increased the liquid phase yield from 48% to 56% while avoiding the use of external solvents [35].

Other promising experiments are being implemented within the framework of the EU project “residue2heat” (2016-2019). The project aim is to produce liquid fuel in a sustainable way using agricultural and forestry by-products and to reduce the industry’s CO<sub>2</sub> emissions.

## 2. MATERIALS AND METHODS

### 2.1 Materials

The measurements were completed in December 2013 on the Bajti Experimental Plantation of the Forest Research Institute (NAIK ERTI) in Hungary. The plantation was installed in April 2007. The planting scheme is a simple row design with 0.40 m plant spacing and 3.00 m row spacing. One parcel contains 65 rows with different clones in each row. The plantation was completed in three repetitions and cultivated with a coppicing technology.

During the winter of 2007/2008, the area suffered wild game damage; thus, at the beginning of the year 2008, the second and third repetitions were cut before the planned time. From here onward, another parcel will be harvested each year with a three-year cutting-cycle. The advantage of the plantation is that several species and clones are present in the area in the first, second, and third vegetation year under the same soil and climatic conditions; see Figure 1. This experiment gives us an excellent opportunity to compare the energetic characteristics of different species and clones.

The test area is a clayloam forest soil in hornbeam-oak climate in medium deep topsoil, which is independent from extra water. Nutrient replenishment during the cultivation or watering after planting is not used.



**Fig. 1.** The plants in the experimental plantation in the first, second and third vegetation year

For the investigation, we have selected the species/clones with the highest yield in the area. The other aspect was the examination of the most widely used clones in Hungary.

The analysed species/clones are the following:

- *Populus x euramericana* 'Koltay'
- *Populus x euramericana* 'I-214'
- *Salix alba* 'Drávamenti'
- *Robinia pseudoacacia*

## 2.2 Methods

We conducted our examination with the Höker LS-12 laboratory muffle furnace according to the requirements of Hungarian standard MSZ EN ISO 18122: 2016; see Figure 2. During the measurements, we separately determined the ash content of tree in bark and bark in triple repetition.



**Fig. 2.** Laboratory muffle furnace

During the measurements, we determined the moisture content of the shredded samples using a BOECO SMO 01 moisture meter. Over the laboratory measurements, we determined the analytical moisture content, which is usually the moisture content removed at 105 °C. Thereafter, we measured sample weight in an absolutely dry state using an analytical balance. The samples were placed in heat-resistant cups and placed in the muffle furnace; see Figure 3.



**Fig. 3.** Determination of ash content in laboratory muffle furnace

At the end of the measurement, the ash weight was again measured with an analytical balance. Finally, the ash content was determined using the following formula:

$$AS = m_o / m_1 * 100 [\%]$$

where

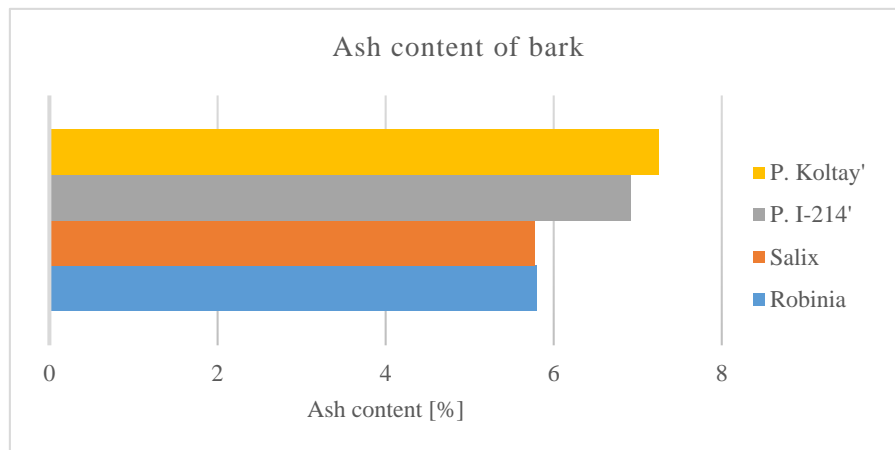
AS – ash content,

$m_1$  – dry sample weight,

$m_o$  – ash weight.

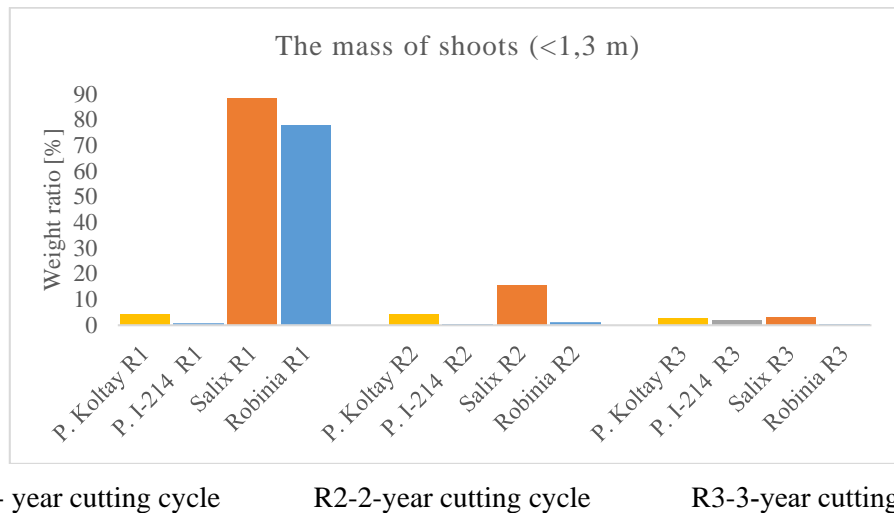
### 3. RESULTS

Base material ash content is an important feature from the perspective of thermal processes. Therefore, we examined this property for 4 tree species/clones from SRC for the biofuel production applicability. The high ash content was mainly caused by the high bark rate. Thus, as a first step, we determined bark ash content. The result shows that the ash content varied between 5.8 and 7.2% depending on tree species. The lowest value was found for salix and robinia; the highest value for poplar; see Figure 4.



**Fig. 4.** The mean ash content of bark for the different wood species/clones

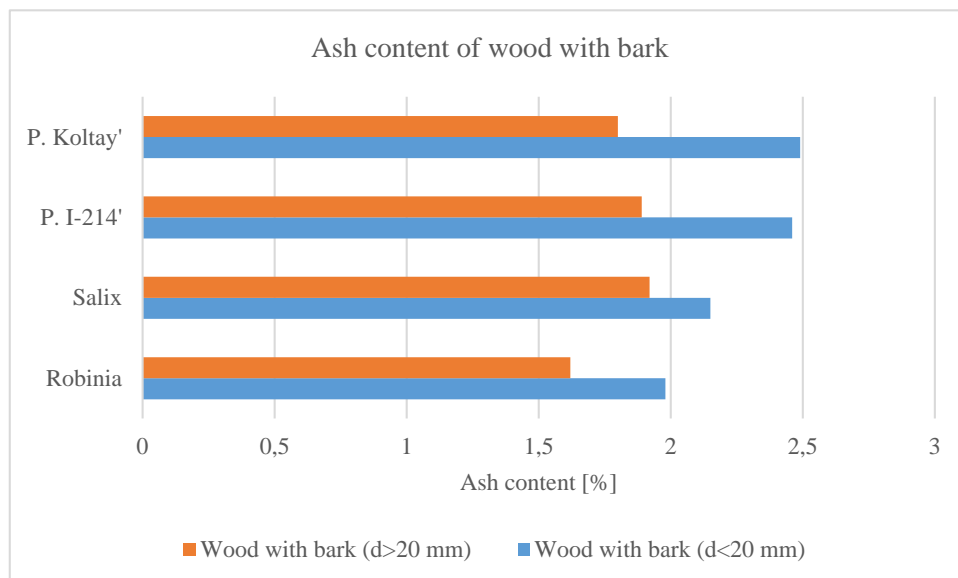
Choosing the right cultivation technology is a significant factor in bark content reduction. A shorter cutting cycle would ensure a continuous supply for the user plant and easier harvesting. However, over this short growing time, the shoots often do not reach a height of 1.3 meters and diameter of 8 mm. In the first vegetation year especially, the weight ratio of the small shoots is more than 80% in the case of salix and robinia [36], see Fig. 5.



**Fig. 5.** The mass of shoots with the height of less than 1,3 m

During our earlier examination, we found a relationship between the diameter of shoot and the bark content. At the smallest diameter ( $d < 8$  mm) the bark content reaches 15-34%, and at larger diameter ( $8 \text{ mm} < d < 48 \text{ mm}$ ) this varied between 2.7% and 15%. The smallest bark value was found at the shoot diameter of 28-32 mm with the Robinia tree species [36].

Ash content was also determined for wood in bark for all 4 species/clones, see Fig. 6. The results shows 3 to 4 times lower bark content for wood in bark than in the case of bark.



**Fig. 6.** The results of the ash content measurement for the different tree species

The ash content of the base material from SRC cultivated with a shorter cutting-cycle is between 2% and 3%. The carbon content remaining in the ash is also higher than in wood from the forest. According to the latest experiments, carbon content can be returned to the soil as fertilizer. It should be noted that during ash content measurement, only natural contamination that appeared on the bark during the growth was present. The high ash content in firing technology is mainly caused by external physical contaminants adhering to the bark during the harvest [37]. Preventing external physical contaminants is an important task to reduce the weight of generated ash.

One possible solution is the use of one-way plantation harvesting. A self-propelled walking chipper executes the felling of individual plantation trees, places the felled trees into a chipping chamber, chips the felled trees, and loads the wood chips onto the forwarder. The Claas Jaguar is an example of such a machine, and it can help the dendromass avoid soil contact. However, using these chippers is only economical in larger contiguous areas where the implementation of high mechanization is worthwhile.

Another option is the use of a vibratory screen with a wind tunnel, a process originally used as an agricultural crop cleaner. The method helps clean the chopped base material from the contamination. The two-stage screening first picks out the pollutants at the 3 cm level and then the below 0.3 cm level. However, the residual sawdust contamination mixture still contains many usable materials. The screening combined with a windtunnel is able to separate further small contaminants from the usable biomass. The disadvantage of the method is it requires small particle-size material, which increases the energy demand for shredding. The method is even more useful for forestry waste where there is a high amount of contamination present due to the harvesting technology.

From a technological point of view, it is important that meso element content in the base material is as low as possible. Measurements of meso element content of different tree species/clones in the Bajti energy plantation were performed by ERTI staff, see Table 2.

	<b>Ca</b> [mg/g (dm)]	<b>Mg</b> [mg/g (dm)]	<b>Na</b> [mg/g (dm)]
<b>Robinia pseudoacacia debarked wood (lower level)</b>	1.98	0.32	
<b>Robinia pseudoacacia bark (lower level)</b>	21.50	0.94	
<b>Robinia pseudoacacia bark (middle level)</b>	22.70	1.06	
<b>Robinia pseudoacacia wood in bark (top level)</b>	11.20	0.82	
<b>Populus x euramericana debarked wood (lower level)</b>	2.01	0.62	
<b>Populus x euramericana bark (lower level)</b>	17.60	1.97	<0,10
<b>Populus x euramericana debarked wood (middle level)</b>	3.27	0.65	
<b>Populus x euramericana bark (middle level)</b>	18.10	2.82	
<b>Populus x euramericana wood in bark (top level)</b>	13.50	2.17	
<b>Salix alba debarked wood (lower level)</b>	3.12	0.67	
<b>Salix alba bark (lower level)</b>	21.70	1.99	
<b>Salix alba wood in bark (top level)</b>	12,26	0.51	

dm - dry matter

**Table 2.** Meso element content of the examined tree species based on measurement of Forest Research Institute (NAIK ERTI)

The data does not show significant differences between tree species for calcium. On the other hand, bark contains a 7 to 10 times higher element concentration than debarked wood, which rises slightly towards the smaller diameters. Robinia shows the smallest element content for magnesium, followed by willow and by poplars. The sodium content was below 0.1 mg/g in all cases. The meso element content increases in parallel with the bark content, which can be reduced by raising the cutting-cycle.

#### **4. DISCUSSION**

In recent years, intensive biomass pre-treatment research studies have been launched to utilize the widest possible base of raw materials. Alternatively, it is possible to wash the biomass alkaline metals and alkaline earth metals content to below the critical level before feeding the biomass into the combustion plant. Commercial implementation seems unrealistic as the process also affects the organic matter content of biomass, and pyrolytic technology can process biomass with a maximum moisture content of 5 to 10%. Thus, it necessary to insert a further drying process into the technology line, one which deteriorates the total energy balance during the life cycle of bio-oil production.

Element reduction is still important from the point of view of combustion equipment, especially due to ash softening and melting. The fluidized bed equipment is engineered to remain below 950 °C. Above this temperature, the ash starts melting and the contamination sticks to the furnace wall. On the other hand, in pyrolytic processes, the high ash and meso element content reduce the oil yield and shifts the reaction towards gas output. Although the produced gas and tar can be used for energy purposes, it does not enrich the main product.

The conversion of base material from short rotation coppices to biofuels by thermochemical process is made difficult first by its high bark ratio. The high bark ratio results an ash content of 2-3%; this value depends on the tree species and on the cultivation technology. The pyrolysis plant in Hengelo is able to tolerate the ash content of biomass with a maximum value of about 2% [38]; however, this results in the reduction of oil yield. Above the critical concentration of alkali metal and alkaline earth metal to be supplied, the current Empyro technology needs to be modified. The diameter of SRC shoots above 20 mm are below the critical 2% ash content, but the optimum 1.5% is only approached by robinia in the case of larger shoots. The smaller shoots exceed the critical value. Thus, it is definitely recommended to grow SRC with a longer cutting-cycle. Adding more debarked wood, which is a generated by-product in the wood industry, to the base material during mixing is also recommended. A further important task is to reduce the external contamination, which greatly limits the usability of the SRC dendromass in pyrolytic processes.

#### **5. CONCLUSIONS**

The use of biomass in Hungary is still very important today. With some assistance, we can meet the targets for renewable energy consumption by 2020. Hungarian biofuel production is primarily based on food plants, despite the availability of significant lignocellulose materials. Second generation biofuels are produced only at a laboratory scale. Their use is hampered by the difficulty inherent in collecting the by-products, the high investment costs of the producer plants, and the inefficiency of available technology. Nonetheless, biofuel use is imperative in the near future because it plays a key role in climate change prevention by reducing the carbon footprint of biofuel consumption compared to fossil fuels.

Short-rotation energy plantations can be planted on unused agricultural areas, which can lead to intensive biomass production and the avoidance of food conflicts. However, high ash content hinders their utilization for liquid biofuel. The use of right cultivation and harvesting techniques can decrease the ash content, but the further development of the pyrolysis process is necessary for higher ash content tolerance. The development of different ash and alkali element reducing pre-treatment processes is ongoing, but they are still too expensive for commercial-sized use.

The dendromass from SRC use in combined heat and electricity power plant is a well-established method, so their utilization is expected to be mainly for this purpose. Electric vehicles represent exciting opportunities for the EU.



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