

Article

Calorific Value and Ash Content of Extracted Birch Bark

Lukas Gruber ^{1,*}, Lukas Seidl ¹, Michela Zanetti ²  and Thomas Schnabel ^{1,3} 

¹ Department of Forest Products Technology and Timber Construction, Salzburg University of Applied Sciences, Markt 136a, Kuchl, 5431 Salzburg, Austria; lukas.seidl@fh-salzburg.ac.at (L.S.); thomas.schnabel@fh-salzburg.ac.at (T.S.)

² Department of Land, Environment, Agriculture and Forestry, University of Padova, Viale dell'Università 16, 35020 Legnaro, PD, Italy; michela.zanetti@unipd.it

³ Faculty of Furniture Design and Wood Engineering, Transilvania University of Brasov, B-dul. Eroilor nr. 29, 500036 Brasov, Romania

* Correspondence: lukasalexander.gruber@fh-salzburg.ac.at

Abstract: Wood bark is one of the main residues in the forest industry worldwide. Currently, the bark is used to produce process heat and energy. A major amount of this is mainly used in power and heating plants. Due to the fact that the demand for heating energy is seasonal, the storage of bark is necessary. The storage process of bark and therefore related problems (like biomass loss, increase of ash content etc.) were discussed in previous studies. Different approaches to increase the economic and ecologic value of wood bark through gaining extractives from the bark were investigated in order to revalue this by-product as well. This study shows the change in calorific value and ash content of birch bark based on results of ultrasound-assisted extraction (UAE). Regarding the energy content (gross calorific value), a comparison was made that showed that due to the extraction process, the energy content of the birch bark is decreased. The extraction yield of the methanol extracts results as 17.74%. The total phenolic content (TPC) of this extract was 447.75 mg GAE/g of oven-dried bark extracts. The amount of ash increased by 23.74% after the extraction. The gross calorific value of the birch bark decreased by 6.98%. The calculated energy content decreased from 2.48 MWh/m³ before extraction to 1.61 MWh/m³ after extraction, which is a loss of 35.08%. The obtained results show that through the extraction of birch bark via ultrasound-assisted extraction, valuable substances can be produced. At the same time, the ash content of extracted birch bark increased whereas the energy content decreased.

Keywords: birch bark; ultrasonic-assisted extraction; energy content; calorific value; ash content



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1. Introduction

The utilization of lignocellulosic biomass for energy generation is an important pillar for the contribution to achieve environmental requirements in the future, such as lowering the greenhouse gas emissions, and to reach independence from fossil fuel sources [1,2]. The by-product bark from sawmill, wood panel, and pulp industry is commonly used as fuel [3,4]. The amount of industrial roundwood (all roundwood used without energy wood) and wood fuel was stated to be nearly 4000 million m³ worldwide (Africa, Asia-Pacific, Europe, Latin America and Caribbean, North America) in 2018 according to the Food and Agriculture Organization of the United Nations [5]. Therefore, the amount of bark (calculated with a ratio of 10%) was approximately 400 million m³. In Austria, according to Linser [6], 3.2 million cubic meters of bark were produced in the year 2018. A certain amount of the bark is directly used for producing process energy and heat at processing companies, but the main part is sold to power or heating plants [3]. Since the demand for energy out of forest residues like bark is seasonal, the storage of bark is necessary for supplying households with energy in the heating season [3,7].

During the conventional biomass storage (wood chips, wood bark, and wood residues), the biomass is subjected to biotic and abiotic factors, affecting its chemical properties [8]. Due to such degradation processes, biomass producers and traders are faced with material losses and thus economic consequences [9]. Several studies investigated the problem with dry matter loss during the storage of biomass. Dry matter loss varies between 5% and 30% depending on different factors like storage condition, season, pile height etc. [3,10,11]. Regarding the storage of biomass, dry matter loss influences the potential economical revenues significantly. According to Anerud et al. [3], the accessible energy of 1 kg dry bark is reduced by approximately 7% of the potential energy after 3 months of storage. This energy loss increases by time and after a storage of 6 months only 85% of the initial accessible energy remains [3]. In comparison to wood, bark has a higher energy content due to a different anatomical and chemical structure than wood and mainly because of a higher content of fatty acid esters and lignin (21–24 MJ/kg) [9,12].

Although bark is mainly used to produce energy, the increasing potential as a material for biorefineries is discussed in different studies as well [9]. Bark mainly consists of cellulose, lignin, hemicelluloses, and different extractives. The exact composition varies regarding different species (softwood, hardwood) [13], and various interesting chemical compounds are detected in their extractives [14,15]. According to Abyshev [16], extracts of the outer bark consist out of several compounds like e.g., ether oils, tannins, flavonoids, terpenoids, etc. depending on the birch species. Regarding the review of Routa et al. in 2017, the extractive content of inner bark was observed in a range of 14.3–18.9% and for outer bark in a range of 32.1–56.9% on a dry mass basis for *Betula pendula* [17]. Furthermore, according to Hasssegawa and Achim [18], the main phenolic components of yellow birch bark (*Betula alleghaniensis* Britt.) are betulin (5.4 ± 3.1 mg/g), lupenone (7.2 ± 3.6 mg/g), lupeol (14.8 ± 5.7 mg/g), stigmasta-3,5-dien-7-one (1.3 ± 0.6 mg/g), and β -sitosterol (4.6 ± 1.2 mg/g). Those numbers can only be seen as reference value, hence the fact that the exact amount of extractives varies due to extraction method and wood species. Those phenolic extractives can be used for different applications in different industry sectors like the pharmaceutical industry or in medical practice [16,19,20]. Also, different methods for extracting sustainable materials (e.g., lignin, tannins, extractives, etc.) can be used as substitutes to oil-based products to reduce their environmental impact, and they are already applied [21].

This study deals with the investigation of the energy and ash content of bark (*Betula pendula*) before and after ultrasound-assisted extraction (UAE). UAE is a green extraction method that can lead to higher extraction yields, a selectivity improvement of extracted compounds, and a higher stability of the obtained product in comparison to classic procedures like conventional maceration [22]. Furthermore, this study aims to show if useful extractives can be obtained from birch bark without major influences on the thermal energy generation and ash content.

2. Materials and Methods

2.1. Bark Preparation

Birch (*Betula pendula* [Roth]) bark was collected from harvested trees of a local forest in Hallein, Salzburg, Austria, in April 2020. The research material consists of bark from five birch trees with an age of approximately 50 years and was collected in a stem height range of 1–8 m. The bark was manually chopped into chips and air-dried at room temperature for 2 weeks. Afterwards, the air-dried bark chips were grinded with a cross-beater mill to powder. The powder was screened with a vibration-screening machine to obtain a fraction with a particle size smaller than 500 μ m, which was the base for the analytical extraction. Three samples of birch bark powder were prepared according to this procedure.

2.2. Chemicals

To obtain bioactive substances, the bark material was extracted using Ultrasound-assisted extraction (UAE). The solvent for the extraction method was methanol from

VWR International (Fontenay-sous-Bois, France). Folin-Ciocalteu reagent from Merck (Darmstadt, Germany) and sodium carbonate from Carl Roth (Karlsruhe, Germany) were used for the analysis.

2.3. Ultrasound-Assisted Extraction (UAE)

For ultrasound-assisted extraction (UAE), a Bandelin Sonorex RK 100 H ultrasonic device (Bandelin electronics, Berlin, Germany) was used. Five grams of birch bark powder and 50 mL of methanol were filled into an Erlenmeyer flask. The extraction was carried out for 3 hours with an ultrasonic frequency of 35 kHz and 50 °C. Afterwards, the powder was separated from the obtained extract with a ROBU-glass-filter Por.4, pore size 10–16 µm (ROBU, Hattert, Germany). The powder was oven-dried and stored in zip bags after the extraction process.

2.4. Extract Processing

Methanol extracts were further processed with a rotary vacuum evaporator to replace the methanol in the extract with water. Methanol was evaporated at a temperature of 60 °C and a vacuum of 337 mbar until the extractives remained in dry matter.

2.5. Yield and Extractives Content

Yield was calculated by subtracting the weight of extracted bark from the initial bark weight using the following formula:

$$\text{yield [\%]} = \frac{m_1 - m_2}{m_1} \times 100 \quad (1)$$

where:

- m_1 is the weight of initial bark [g] (dry base) and
- m_2 is the weight of extracted bark [g] (dry base).

The extractives content of the extracts is calculated by the weight of solid components of a sample divided by the initial weight of the sample.

To determine the extractives content, 10 mL of each extract are filled in a beaker and put into the drying kiln. The extractives content is then calculated by the following formula:

$$\text{extractives content} \left[\frac{\text{mg}}{\text{mL}} \right] = \frac{c_2}{c_1} \quad (2)$$

where:

- c_1 is the initial amount of liquid [mL] and
- c_2 is the weight of solid components [mg].

2.6. Total Phenolic Content

Folin-Ciocalteu method, based on the procedure described by Ozturk et al. [23], was used with slight modifications to determine the total phenolic content of birch bark extracts. Two-hundred microliters of the extract, with a diluted concentration of 1 mg/mL (solid content), were filled in a test tube and mixed with 3000 µL deionized water and 500 µL of the Folin-Ciocalteu reagent. The mixture was shaken and after 3 minutes, 2000 µL of 20% (w/v) sodium carbonate was added. Following, the mixture was put in the dark for 1 hour to react. Absorbance was measured with the Shimadzu UV mini 1240 UV/VIS spectrophotometer at a wavelength of 756 nm. The tests were carried out in triplicates and the average of each extraction method/solvent was calculated. Results are expressed as mg Gallic acid Equivalent per g Extract (mg GAE/g).

2.7. Ash Content Determination

For the determination of ash content, EN ISO 18122:2015 was followed. Samples of original and extracted particles were dried to anhydrous. A minimum of 1 g was put into a

melting pot and weighed. The sample in the melting pot was put into an oven. The oven was heated for 40 min to reach a temperature of 250 °C. This temperature was held for 60 min and afterwards the temperature was raised to 550 °C and held for 120 min. After the combustion process, the oven was turned off and the melting pot with the sample was put into a desiccator to cool down to room temperature. The sample was weighed and the ash content was calculated by the following formula:

$$\text{ash content [\%]} = \frac{a_3 - a_1}{a_2 - a_1} \times 100 \times \frac{100}{100 - M_{ad}} \quad (3)$$

where:

- a_1 is the weight of the empty melting pot [g],
- a_2 is the weight of the melting pot with dried sample [g],
- a_3 is the weight of the melting pot with ash [g], and
- M_{ad} is the moisture content of the sample.

2.8. Gross Calorific Value and Energy Content

Gross calorific value was determined following EN ISO 18125:2018. The samples were weighed and burned in high-pressure oxygen using an IKA C-200 calorimeter that gives the heating value of the sample as received. The average of two replications was calculated. During the combustion process, the corrected temperature rise was measured. The gross calorific value [MJ/kg] is calculated by the corrected temperature rise and the effective heat capacity of the calorimeter.

To calculate the net calorific value of fresh birch bark (wet) and extracted birch bark (air dried), the following formula, used by Routa et al. [9], was applied:

$$q_{p,net,ar} = q_{p,net,d} \times \frac{100 - M_{ar}}{100} - 0.02443 \times M_{ar} \quad (4)$$

where:

- $q_{p,net,ar}$ is the net calorific value as received [MJ/kg],
- $q_{p,net,d}$ is the net calorific value on a dry basis [MJ/kg],
- M_{ar} is the moisture content as received, and
- 0.02443 is the correction factor of the enthalpy of vaporization at 25 °C.

For the calculation of the energy content, the formula of Routa et al. [9], is applied:

$$E_{ar} = \frac{1}{3600} \times q_{p,net,ar} \times BD_{ar} \quad (5)$$

where:

- E_{ar} is the energy content as received [MWh/m³],
- $q_{p,net,ar}$ is the net calorific value as received [MJ/kg], and
- BD_{ar} is the bulk density as received [kg/m³].

2.9. Bulk Density

The bulk density for the initial and extracted sample is determined based on the norm EN ISO 17828:2016. The bark powder is filled in a cylindrical container with a known volume and the weight is measured. The bulk density is calculated with the following formula:

$$BD_{ar} = \frac{m_1 - m_2}{V} \quad (6)$$

where:

- BD_{ar} is the bulk density [kg/m³],
- m_1 is the weight of the container and bark [kg]
- m_2 is the weight of the container [kg], and

- V is the volume of the container [m^3]

3. Results and Discussion

3.1. Analysis of Extraction

In general, the methanol solvent is used frequently to extract polyphenols from different plant materials [24] and yields a higher total amount of bark extractives than water as solvent [25]. Furthermore, some other compounds could be found in such methanol extracts (e.g., fatty acids, simple sugar, and resin acids).

In the first step of this study, the yield of solid material after extraction is determined by drying the material at 103 °C to constant weight. The total solid compound amount of the methanol extracts was 177.4 g/kg of oven-dried birch bark powder, which corresponds to 17.74% extraction yield by using this ultrasound-assisted method. According to Chemat et al. [22], the yield of ultrasound extraction is higher in a shorter period of time compared to a classical maceration process. The total phenolic content (TPC) of the methanol extract resulted in the mean of 447.75 mg GAE/g, expressed in mg equivalents of gallic acid unit/g of dried bark extracts. Various TPC-yields have been reported for the solvent extraction of birch bark. Comparable values for white birch bark, in the range of 429–435 mg GAE/g from ultrasound-assisted extraction with ethanol and water, were obtained by Sillero et al. [26]. Diouf et al. [27] concluded that the possible content of total phenolics in the birch bark (inner bark) using ultrasound-assisted extraction, with half an hour extraction time, is approximately 303 mg GAE/g of oven-dried extracts. Moreover, the extract of birch bark contains different phenolic compounds including for example phenolic acids, flavanols, lignans, and procyanidins [28,29]. Different compounds of the polyphenols affect the properties of plant extracts, including the antimicrobial activity [25,30], and thus, polyphenols have attracted considerable attention for many applications in the field of life sciences [20].

Concerning the antimicrobial properties of bark, a problem with the storage duration of bark is observed. According to Routa et al. [9], the amount of extracted compounds is strongly influenced during the biomass storage within the first 24 weeks, which decreases to 7% extractives content—based on oven dry material—compared to the initial state of 28%. It is observed that not only substances like diglycerides are degraded in the birch bark during storage but also bioactive components like betulin and other triterpenoids. For this reason, the bark should be extracted as soon as possible after the harvesting process. [17]

After the extraction process, the extracted material remains and can be used for energy generation through combustion. Due to the extraction of solid components (17.74%), a loss of energy content and an increase of ash content is expected. In the following chapter, this assumption is examined.

3.2. Calorific Value and Ash Content of Sample Material

Table 1 shows the important parameters of the fresh and extracted birch bark regarding the application in bioenergy generation. The term “fresh” refers to a storage duration of the bark of 2 weeks after harvesting. The gross calorific value was 22.21 MJ/kg for fresh birch bark and 20.66 MJ/kg for the extracted bark. According to Shirmohammadli et al. [20], condensed tannins can be extracted from bark with the use of methanol as solvent, and Laitinen et al. [31] describes condensed tannins with a content of 28.59 mg/g (dry weight) as the main phenolic compound group in birch bark methanol extracts. The decrease of the energy content after extraction could be explained by the removal of condensed tannins, which according to Zanetti et al. [32], have a higher energy content than wood.

Table 1. Means (\pm standard deviation) of the results of the birch bark before and after extraction.

Sample	Gross Calorific Value (MJ/kg)	Ash Content (% \pm SD)	Yield (% \pm SD)
fresh birch bark	22.21	2.12 * \pm 0.02	17.74 * \pm 1.64
extracted birch bark	20.66	2.78 * \pm 0.06	-

* mean value of 3 samples.

An increased ash content is determined for the extracted bark sample. Fresh birch bark has an ash content of 2.12% whilst extracted birch bark has a value of 2.78%, which means an increase of 0.66 percent points. This coincides with the results of Routa et al. [9] that determined an increase of ash content after storage of spruce bark after 8 weeks: The ash content increased from 3.29–3.74%, whereas the extractive content decreased from 11.83–7.83%. For the stem bark of downy birch, Dibdiakova et al. [33] received an ash content of 2.0–2.5% dependent on stem height. For the combustion process, ash content is one of the most important parameters. Higher values can lead to problems with ash removal, slagging, pollution of the combustion chamber, and equipment corrosion [34].

In comparison to most other hard wood species, birch bark has a lower ash content (2.12%) and a higher gross calorific value (22.21 MJ/kg), even after extraction (2.78% and 20.66 MJ/kg). According to Kamperidou et al. [34], beech bark has an ash content of 7.73% and a gross calorific value of 18.60 MJ/kg, paulownia bark 2.99% and 18.12 MJ/kg, and black locust bark 7.03% and 19.66 MJ/kg.

From the results in Table 1, it could be seen that material loss due to the extraction is 17.74% and the gross calorific values of the fresh and extracted oven dried bark sample are different by 1.55 MJ/kg (6.96%). Accordingly, the material weight of 1.0 kg fresh birch bark was reduced to 0.8226 kg of extracted material.

However, these results represent only the theoretical interpretations but show a clear result between the material loss due to extraction (e.g., artificial and/or natural processes) and reduction of energy content. The natural effects for material loss may be rain, snow, or microorganisms by storing the biomass in outdoor areas. Therefore, this practical approach was investigated by using Equations (4) and (5). Table 2 shows the determined bulk density of the initial sample and the extracted sample, as well as the calculated energy content of these samples.

Table 2. Bulk density and calculated energy content (means \pm standard deviation) of birch bark before and after extraction.

Sample	Bulk Density (kg/m ³ \pm SD)	Calculated Energy Content (MWh/m ³)
oven-dried birch bark	402.45 * \pm 13.9	2.48
Oven-dried extracted birch bark	280.15 * \pm 7.64	1.61

* mean value of 6 repetitions.

The calculated energy content of the oven-dried birch bark, based on oven-dried mass and a bulk density of 402.45 kg/m³, results in 2.48 MWh/m³ and for the extracted birch bark with a bulk density of 280.15 kg/m³ an energy content of 1.61 MWh/m³ is obtained. Furthermore, the extraction process leads to a decrease of 30.39% of the bulk density, which results in a decrease of the energy content of the bark by 35.08%. It should be noted that the bulk densities of the fresh and the extracted bark have a significant difference, which leads to this decrease of calculated energy content. The decrease of the gross calorific value between initial and extracted sample results as 6.96%.

4. Conclusions

Bark is one of the main by-products of the forest industry and is currently mainly used to produce energy and heat. Therefore, the extraction of different components out of the bark could lead to new products, substitution of current products, and in the long term to new business models and companies. Also, the extraction of components could help to increase greenhouse gas emissions by substituting oil-based materials. Since the price of bark is relatively low and the availability of bark is high, the extraction of bio-active compounds could increase the value of bark. An important aspect would be that through the extraction process, combustion parameters like ash content and energy content are not negatively influenced.

This study shows that there is the possibility of increasing the value of wood residues like birch bark by using an ultrasound-assisted extraction (UAE). Due to the extraction, the energy content of the extracted bark materials is lower than the fresh birch bark, whereas the ash content increases after the extraction. Nevertheless, as studies already explained, most of the extractives of wood bark are lost due to biotic and abiotic processes during the storage.

Material extraction could be implemented within a bio-economy concept to improve the value chain of biomass by extracting bio-active compounds from bark before combustion. Such bark compounds are interesting for different industrial sectors and may be used for bio-active applications [25,35]. Therefore, an investigation of the emerging costs and potential benefits of this extraction process and the following combustion should be conducted. Ash content and calorific value have to be investigated further to compare different species and the possible economic advantages of ultrasound-assisted extraction (UAE). Further research in parallel experiments on different extraction methods and a determination of the extracted components in terms of type and quantity should be performed to understand their influence on the calorific value and ash content. This should help to find technically feasible possibilities that can be implemented in accordance with ecological aspects.

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