Effect of Thermochemical Treatment on the Physicochemical Properties of Fiber Hemp (*Cannabis sativa* L.) by-Product

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The growing demand for biomass is forcing attention toward alternative resources, including agricultural by-products such as fiber hemp (*Cannabis sativa* L.). Growing hemp for seed produces about 10 tons of dry matter by-products per hectare. Processing-conversion technologies used in practice change the properties of primary raw materials, give them higher added value, and prepare them for various needs. In addition to economic benefits, biochar contributes to carbon sequestration and increases the sustainability of agricultural production. This study evaluated the effect of thermochemical treatment in an oxygen-free environment on some physicochemical properties of the fiber hemp by-products of the Felina 32 variety. At a heating temperature of 700 °C, the biomass yield of biochar from stems was 28.4 ± 0.5% and from seed husks was 30.6 ± 0.6%. At that time, the carbon concentration in fiber hemp stems biochar increased 1.82 times, from 45.0 ± 2.26% to 81.9 ± 8.46%, and in seed husk biochar by 1.20 times, from 56.5 ± 11, 6% to 67.9 ± 7.47%. Furthermore, the changes in the fractional composition, density, calorific value, ash content, and ash melting characteristics that occurred during the conversion of primary raw material of fiber hemp into biochar were evaluated.

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Keywords: Fiber hemp (Cannabis sativa L.); By-products; Biochar; Fractional composition; Calorific value; Ash content

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INTRODUCTION

The developing world is facing one of its biggest problems: increasing consumption. It is predicted that by 2050, consumption on Earth will increase by an average of three times; the need for materials such as biomass, fuel or metals will increase by 200%, and the generation of waste will increase by 70% (European Commission 2020). The problem is exacerbated by the desire to abandon fossil raw materials and fuels, which are needed to meet the growing consumption. Therefore, the use of alternative resources and the development of waste-free production are needed.

One of the alternatives to conventional resources is biomass, including agro-biomass and agricultural by-products (production waste). In the European Union, the yield of agricultural crops increases by an average of 0.9% per year. It is predicted that in 2050, the sustainable biomass grown in Europe will be 340 million tons of dry mass. The main sources are cereal straw, agricultural residues, and lignocellulosic plants (Imperial College London 2021). In Lithuania, according to 2020 data, 4.2 million tons of straw waste and 59.4 thousand tons of grain cleaning waste were generated in agriculture. Of this waste, only 5.1 thousand tons of straw and 1.4 thousand tons of grain cleaning waste was used for energy production, and the rest was recycled/used in farms in one way or another (Official Statistics Portal 2021). Biomass is one of the most abundant renewable resources in the world. It is especially relevant in temperate climate zones where, compared to other renewable resources, the potential of plant biomass is relatively high. There are also favorable conditions for growing biomass, such as fiber hemp (*Cannabis sativa* L.).

In 2021, fiber hemp (*Cannabis sativa* L.) crops occupied 31,870 hectares in the European Union. Almost 58% of the crop is in France. It is the largest grower of fiber hemp in the European Union (European Commission 2021). In Lithuania, the cultivation of fiber hemp grew in popularity just as rapidly. In 2019, Lithuania was in second place in the European Union in terms of fiber hemp crop areas. They comprised 9182 ha. In the following years, there was a significant decrease in crop areas and only from 2022 they started to increase again. Currently, fiber hemp crops in Lithuania occupy 5,334 ha (Statista 2022).

Fiber hemp is mostly grown for its seeds. According to Eurostat data, the harvest of fiber hemp seeds in the European Union countries is very uneven - from less than 1 ton per hectare to 7.81 tons according to the results of 2021 (Eurostat 2022). In parallel, large amounts of secondary products are formed. This is about 10 tons of dry hemp plant biomass per hectare (Zvicevičius *et al*. 2021), which can be used to replace traditional resources or for other uses. All parts of the fiber hemp plant can be used, and products suitable for various applications are produced: from construction materials to cosmetics and pharmaceutical products (Ahmed *et al*. 2022). Currently, the most popular field of use of plant biomass is energy production. In the European Union in 2020 domestic biomass energy production amounted to 142 million tons of oil equivalent (Mtoe). Using agricultural products, by-products and waste produced an average of 22 Mtoe of energy in each branch. Energy production from wood amounted to 32 Mtoe (European Commission 2019).

The versatility of both fiber hemp biomass and other plant biomass allows it to be used in virtually all industries. Modern processing-conversion operations and technologies make it possible to effectively manage the properties of the primary raw material and prepare it as a quality raw material or product for various needs. One such area could be biofuels and energy, where the use of fiber hemp raw material in extraction of these products is considered a positive energy balance (Prade *et al.* 2012). Even biofuels such as biomethane, biodiesel, bioethanol can be obtained from industrial hemp, as well as bioproducts such as microbial proteins and biopolymers during valorization (Moscariello *et al.* 2021). Specific processing-conversion methods are selected considering the further purpose. Mechanical processing - grinding, pressing, make it possible to confer upon biomass the desired fractional composition, bulk, sample structure, density, and other properties. Chemical, thermochemical, and biochemical processing methods can fundamentally change the raw material and its chemical composition. They are used in the paper, chemical, cosmetic, pharmaceutical industries (Paraskeva *et al*. 2008; Shen *et al*. 2015; Dahal *et al*. 2019; Jeyasubramanian *et al*. 2021; Wang *et al*. 2021; Atta *et al*. 2022; Patwa *et al*. 2022). Thus, biomass processing-conversion expands the range of raw materials/products from biomass, reduces their cost price, creates prerequisites for producing products with high added value, as biofuel, the price of which can reach 60 to 380 €·ha^{-1} per year for the production of methane or 510 to 868 €·ha^{-1} per year for the production of biodiesel, efficient use of production waste, and development of waste-free production (Moscariello *et al.* 2021; Ahmed *et al.* 2022).

Biochar is one of the products of thermochemical conversion of biomass with high added value. According to 2022 US market data, the price of a ton of wood ranges from 31.6 to 36.2 dollars depending on the type of wood. At that time, the market value of biochar in the US was about 1,000 dollars per ton, which is 28 times higher than the price of primary biomass. Biochar production costs can range from 450 to 1800 dollars per ton. It depends on the used equipment, technological process and modes, as well as primary raw material (Sahoo *et al.* 2019; Nematian *et al.* 2021; Seow *et al.* 2022; Statista 2022). Apart from the economic benefits, biochar can contribute to preserving and improving soil productivity, carbon sequestration, reducing GHG emissions, and increasing the sustainability of agricultural production. One of the goals of reducing greenhouse gas emissions is to reduce the amount of nitrogen released into the environment. Biochar has this feature, because when using biochar made from straw, compared to ordinary straw, about 15 to 19% less nitrogen was released from chicken manure into the environment during composting (Chen *et al.* 2023). Also, biochar contributes to carbon sequestration, as it can remain in the soil for a long time and partially decomposes in more than 500 years (Wang *et al.* 2015).

The primary raw material, technological process, and modes are also the main indicators that determine the yield and properties of biochar. A higher biomass heating temperature reduces the biochar yield and increases the amounts of liquid and gaseous conversion products (Mlonka-Mędrala *et al.* 2021). However, the effect and its effectiveness are specific to different types of biomasses. The biomass of herbaceous plants reacts sensitively to the increase of heating temperature: increasing the temperature by 300 °C (from 400 to 700 °C), the biochar yield of sesame stalks decreases by as much as 17% (Ateş *et al.* 2004) and from sunflower straw biochar yield decreases from 40% to 28% at 300 and 700 °C respectively (Yue *et al.* 2018). At that time, the yield of biochar from hazelnuts shells decreased by only 10% (Pütün *et al.* 2000) and from woody willow biomass by 4.6% (Jin *et al.* 2017; Kończak *et al.* 2019). Thermochemical conversion conditions also influence the internal structure of the biochar and its quality. As the heating temperature increases, the internal porosity and active surface area of biochar increase, but the pore diameter decreases (Ateş *et al.* 2004; Sbizzaro *et al.* 2021). A very significant effect was recorded on the biochar structure of the pecan shells. Its active surface area increased from 0.71 to 311.7 m^2·g^{-1}, as much as 439 times. This is 4.67 times more than branch biochar and 12.9 times more than leaf biochar: from 2.62 m^2·g^{-1} to 248 m^2·g^{-1} and from 1.53 to 52.72 m^2·g^{-1}, respectively (Liu *et al.* 2023). The internal structure and active surface area of biochar are essential parameters that determine its hygroscopic and chemisorption properties (Sbizzaro *et al.* 2021). The high porosity and comparative surface area of biochar improves the retention of water and nutrients in the soil and their availability (Liu *et al.* 2022; Yadav *et al.* 2023), immobilization of chemicals and heavy metals in the soil (Sun *et al.* 2020; Gao *et al.* 2022). Incorporation of 20% mixed woody biochar by volume into soil increased soil water capacity by an average of 1.55 times: from 0.56 mm to 0.83–0.91 mm in sandy soil and from 0.56 mm to 0.79–0.96 mm in sandy loam soil (Verheijen *et al.* 2019). When evaluating biochar, it is equally important to pay attention to its chemical composition and the regularities of its formation (Novak *et al.* 2013; Jin *et al.* 2016). Balmuk *et al.* (2023) recommends paying attention to the amounts of ammonium nitrogen (NH_4-N) and nitrate nitrogen (NO_3-N) in biochar, which influence the ability of plants to absorb nitrogen. Those amounts in biochar also depend on biomass heating temperatures. In addition, a significant difference in temperature effects was recorded for different types of biomasses. By increasing the pyrolysis temperature from 300 to 500 ℃, the amounts of NH_4-N in both grapevine waste and corn stalk biochar decreased, from 41.8 to 24.1 $mg \cdot kg^{-1}$ and from 88.6 to 45.2 $mg \cdot kg^{-1}$ respectively. However, the effect of temperature on NO_3-N levels was opposite: its concentration decreased from 97.0 to 20.1 $mg \cdot kg^{-1}$ in grapevine waste biochar and increased from 27.8 to 41.1 $mg \cdot kg^{-1}$ in corn stalk biomass (Balmuk *et al*. 2023).

Thus, the properties of biochar formed during the thermochemical conversion of biomass depend on both the primary raw material and its heating regimes and conditions. In addition, the effects of these factors are complex. This complicates the selection of modes, but also creates assumptions for modeling the properties of the produced biochar by choosing an appropriate combination of factors.

The aim of the research is to evaluate the effect of thermochemical treatment in an oxygen-free environment on the quantitative-qualitative transformation of the physico-chemical properties of fiber hemp (*Cannabis sativa* L.*)* by-products—husks and stems biomass.

EXPERIMENTAL

Materials

The research was carried out in cooperation with the Laboratory of Biomass Preparation, Logistics and Solid Fuel Processes of the Vytautas Magnus University Agriculture Academy and the Agrobiology Laboratory of the Lithuanian Research Center for Agriculture and Forestry.

The stems (without leaves and inflorescences) and seed husks of the fiber hemp (*Cannabis sativa* L.) variety Felina 32 were used. Fiber hemp is grown in soil that was plowed in the autumn and germinated and cultivated in the spring, without the use of additional fertilization of the crop with either mineral or organic fertilizers.

The 1 kilogram stems and seed husks of air-dried hemp were ground with a rotary mill Retsch ZM 200 to particles of 1 mm size. The raw materials prepared in this way were later used in biochar production, chemical composition, fractional composition, sample density, active surface area, calorific value, and ash content studies. In addition, the fractional composition and sample density of the non-ground biomass of the husks of fiber hemp seeds and the biochar produced from them were determined.

Methods

Biochar preparation and yield

Thermochemical conversion of ground fiber hemp stems and seed husks of biochar production was carried out in a Nabertherm RS tubular heating furnace with a 10.65-liter internal volume heating chamber that was sealed on both sides with cooling flanges. To remove combustion products and volatile compounds, the combustion chamber was ventilated with nitrogen gas throughout the process. Conversion process modes were as follows: heating temperature 700±10 °C, temperature rise rate 6.7 °C per min, holding time 2 hours, and nitrogen gas flow 100 liters per hour. At the same time, three crucibles with a diameter of 90 mm and a capacity of 200 mL were placed in the heating chamber with about 20 g of ground primary raw material. A total of at least 8 conversion cycles were performed with each fiber hemp feedstock. In order to calculate the mass yield of biochar from fiber hemp stems and seed husks, the moisture content of the primary raw materials used was determined in the MEMMERT UF450PLUS drying cabinet, the weights of empty crucibles, crucibles with primary raw material and biochar were recorded using KERN ABJ scales and calculated as follows:

$$B = \frac{M_2 - M_0}{(M_1 - M_0) \cdot (100 - \omega)}, \tag{1}$$

where B is the yield of biochar from the dry mass (%), M_0 is mass of the empty crucible (g), M_1 is the mass of crucible with primary raw material before burning (g), M_2 is the mass of crucible with biochar after burning (g), and ω is raw material moisture content (%).

Fractional composition, sample density and active surface area studies

Fractional compositions of ground primary raw materials and biochar produced from them were determined using a Haver EML Digital plus shaker with a set of sieves consisting of metal 200 mm diameter certified Retsch (Certificate of compliance EN 10204) sieves with round holes of 3.15, 2.5, 1.7, 1, and 0.315 mm in diameter. Molded samples of 40 to120 g were spread on the top sieve and sieved through a set of collected sieves. Shaker working modes were as follows: shaking duration 3 min, shaking interval - 10 seconds, shaking amplitude - 2 mm. After each test, the amount of raw material accumulated on each sieve was weighed on a Scaltec SPO 51 scale. The percentage of each fraction in the sample and the average particle diameter of the entire sample were calculated as follows,

$$m_i^{\%} = \frac{m_i}{\sum_{i=1}^{n} m_i} \cdot 100, \tag{2}$$

$$d = \frac{\sum_{i=1}^{n}(d_i \cdot m_i)}{\sum_{i=1}^{n} m_i} = \frac{\sum_{i=1}^{n}\left(\frac{d_{i1} + d_{i2}}{2} \cdot m_i\right)}{\sum_{i=1}^{n} m_i}, \tag{3}$$

where $m_i^{\%}$ is fractional part (%), m_i is amount of fraction *i* (kg), *i* is fraction number, and *n* is number of fractions, *d* is the average particle diameter of the entire sample (mm), d_i is particle diameter of *i* fraction (mm), d_{i1} is the diameter of the holes of the sieve through which the measured fraction *i* passed (mm), and d_{i2} the diameter of the holes of the sieve on which the measured fraction *i* remained (kg).

A cylindrical vessel of 50 mm internal diameter and 0.1295 mL volume and known mass was used to measure sample densities. After filling the vessel with crushed primary raw material or biochar produced from it, the vessel was weighed on a Scaltec SPO 51 scale. The weighing results were used to calculate the density of the corresponding sample as follows,

$$\rho = \frac{M - m_b}{V_b}, \tag{4}$$

where ρ is the density of the ground primary raw material or biochar sample made from it (kg·m^{-3}), *M* is mass of container with tested raw material (kg), m_b is mass of the used vessel (kg), and V_b is the volume of the used vessel (m^3).

The surface area of the capillaries and cavities that carved out the biochar particles was determined by the B.E.T. (Brunauer–Emmett–Teller) method with a gas sorption analyzer NOVAtouch LX (Quantachrome), consisting of four degassing and two analyzing stations. During the research, the biochar samples were first cleaned of moisture and other impurities: in the degassing chamber, the glass tubes with the biochar samples were heated to a temperature of 300 °C and kept in a vacuum under these conditions for 6 hours. After that, the tubes with the biochar samples were transferred to the analysis station and immersed in a container with liquid nitrogen. During the measurement, nitrogen was also directed inside the tubes and the pressure change in them was recorded. After evaluating the mass of purified biochar in the tubes, the B.E.T. method was used to calculate the comparative active surface area [$m^2 \cdot g^{-1}$] of the biochar used in the research.

Calorific value and ash content studies

Calorimeter measurements were made with an IKA C2000 calorimeter according to the methodology specified in the LST EN 14918 standard. Before that, the moisture content of the samples prepared for research was determined (standard EN 14774-3), and combustible capsules, whose weight was between 0.3 and 0.5 g, were formed with ground masses of stem, seed husks, and biochar. After burning the capsules in a calorimetric bomb, the upper calorific values of the analyzed samples Q_{gr}^{v} were determined. After evaluating the moisture content of the raw materials used, their upper calorific values $Q_{gr,d}^{v}$ for dry mass were calculated by Eq. 4,

$$Q_{gr,d}^{v} = Q_{gr}^{v} \cdot \left(\frac{100}{100-\omega}\right), \qquad (5)$$

where $Q^{v}_{gr,d}$ is the upper calorific value of dry raw material ($kJ \cdot kg^{-1}$), Q^{v}_{gr}. is the upper calorific value of the studied raw material ($kJ \cdot kg^{-1}$), and ω is the moisture content of the studied material (%).

Ash content tests were performed according to the methodology described in the LST EN 14775 standard. Crucibles with at least 1 g of ground raw material were uniformly heated to 250 °C and then to 550 °C in the CZYLOK burning furnace. After evaluating the moisture content of the raw materials used, the masses of empty crucibles, crucibles with raw materials before burning and after burning, the ash content A_d of fiber hemp stems, seed husks and the biochar produced from them was calculated by Eq. 5,

$$A_d = \frac{(m_3-m_1)}{(m_2-m_1)} \cdot \left(\frac{100}{100-\omega}\right), \qquad (6)$$

where m_1 is the mass of the empty crucible (g), m_2 is the mass of crucible with raw material before heating (g), and m_3 is the mass of crucible with ash (after burning) (g).

The temperature characteristic of ash melting was determined according to the methodology presented in the standard LST CEN/TS 15370-1. After using the raw materials used in the research (fiber hemp stems and seed husks) and the biochar ash produced from them, cylindrical samples of 4 mm in diameter and 5 mm in height were formed with a hand press. They are placed on special plates in the heating furnace Carbolite CAF Digital. Eight samples were tested at a time. During the study, the heating furnace chamber was continuously ventilated with nitrogen gas and heated uniformly (at a rate of 7.0 °C per min) until the samples were completely poured, periodically every 1 °C with a surveillance camera and recording their pictures on the computer. The heating of the chamber and the capture of photos were continued until all the samples loaded into the furnace reached the flow state (FT state). Then, by analyzing the photos with a special program, the temperature characteristics of ash melting were determined - the temperatures of the characteristic points (shrinking (state ST), deformation (state DT), hemispherical formation (state HT), flow (state FT)) at which the states of the ash samples occur changes.

Total carbon studies

The tests were carried out using the elemental combustion system ECS 4010. Samples of fiber hemp stems and seed husks and biochar made from them were poured into tin capsules, 10 mg each, and placed in the autosampler of the elemental analyzer. During the measurement, the samples were burned in a modified gas environment and the gas flow rate was 110 mL/min, nitrogen gas – 110 mL/min, and oxygen – 180 mL/min. Using the computer program "Elemental analysis software", the amounts of total carbon (%) in the raw materials used in the research were calculated based on the obtained results.

Statistical analysis

The results of the studies were determined after at least eight replicates and evaluated statistically using the Microsoft Excel ANOVA (analysis of variance) subroutine of the data package and estimating the standard error using the correlation coefficient, when significance level was $p \leq 0.05$. Tukey's HSD test was used to test for significant differences between mean values. Values that were not significantly different are marked with the same letters in the graphs.

RESULTS AND DISCUSSION

Analysis of Biometric Characteristics

The fiber hemp by-products – stem biomass and seed husks, ground by a rotary mill, had an average particle diameter of 2.83 mm and 1.02 mm, respectively. The size fractions of 1.0 mm and larger dominated (Figs. 1 and 2). They made up more than 50% of the mass of the sample.

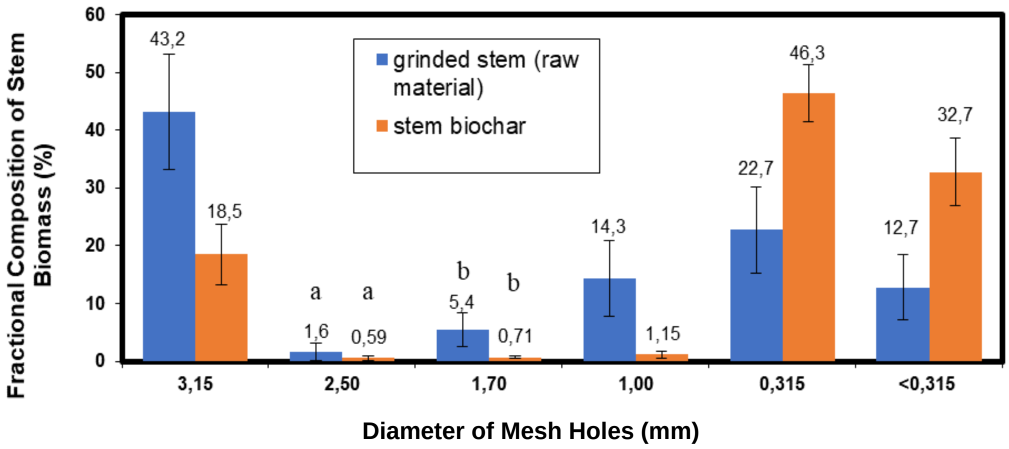

Fig. 1. Fractional composition of ground fiber hemp stems and biochar (there is no significant difference between the columns marked with the same letters (a and b))

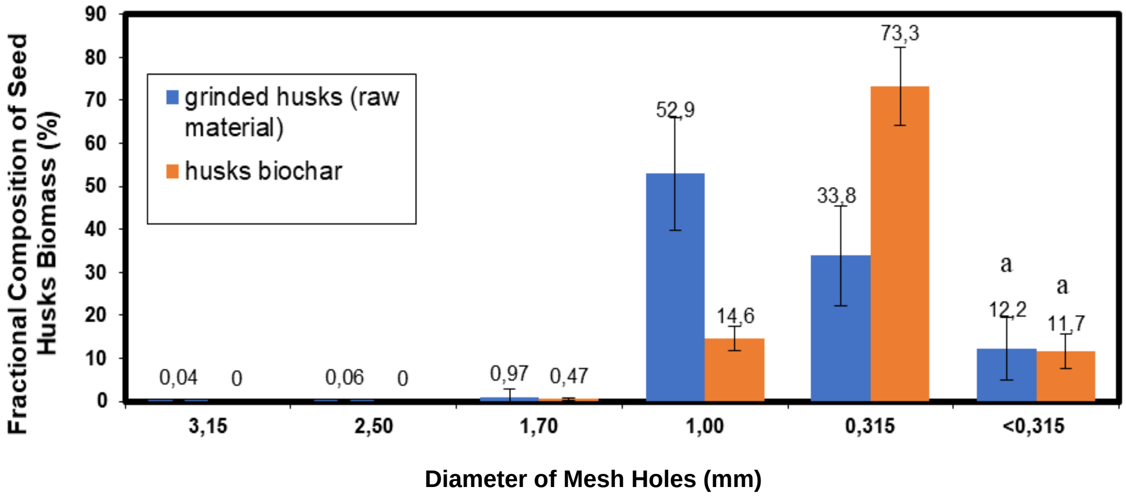

Fig. 2. Fractional composition of ground fiber hemp seed husks and biochar (there is no significant difference between the columns marked with the same letter (a))

The fractional composition of ground stems was greatly influenced by the fiber contained in the stem. During grinding of the raw material and especially during sieving, the fiber particles tend to get tangled. A felt-like structure is formed, which accumulates on sieves with holes of a larger diameter, and additionally retains particles of a finer fraction. Therefore, the 1 mm and larger size fraction was mainly composed of the mass that accumulated on the sieve with 3.15 mm diameter holes: the particles of the 1 mm and larger size fraction made up 64.5% (43.2% + 1.6% + 5.4% + 14.3%) of the sample, and the particles of the 3.15 mm and larger size fraction – 43.2% of the sample. At that time, only 1.07% (0.04% + 0.06% + 0.97%) of ground hemp seed husks accumulated on sieves with holes of 3.15 mm, 2.5 mm, and 1.7 mm in diameter.

The thermochemical conversion process fundamentally changed the fractional composition of the raw material. The 3.15 mm fraction was predominant in the ground stem raw material, which accounted for 43.2 ± 9.8%, while stem biochar consisted of 46.3 ± 4.9% of the 0.315 fraction particles. The smallest part in both raw materials was 2.50 mm fraction particles, 1.6 ± 1.5% in chopped stem and 0.59 ± 0,4% in stem biochar. Compared to the raw material, the average particle diameter of stem biochar decreased to 1.36 mm, and the biochar of ground seed husks decreased to 0.71 mm. These are 2.08 and 1.44 times, respectively. However, a smaller dispersion of the fractional composition of the particles was recorded. Biochar particles with a size of 0.315...0.999 mm dominated. The fraction of 1 mm and larger size in biochar from ground stems decreased by 3.08 times (from 64.5% to 20.9% (18.5% + 0.59% + 0.71% + 1.15%)), and in biochar from ground seed husks by 3.58 times (from 53.9% (0.04% + 0.06% + 0.97% + 52.9%) to 15.1% (0% + 0% + 0.47% + 14.6%)). A significant change in the amount of the fraction smaller than 315 mm was recorded only in the sample of ground stems: after thermal conversion, the share of the smallest fraction in the sample increased from 12.7 ± 5.6% to 32.7 ± 5.9%. It was observed that biochar from the biomass of fiber hemp stems is prone to decay: fractions accumulated on sieves with 3.15, 2.5, 1.7, and 1.00 mm holes decreased by 2.34, 2.71, 7.61 and 12.4 times, respectively. However, the felt-like structure characteristic of the primary raw material of ground stem biomass has not completely disappeared.

The thermochemical conversion process of non-ground fiber hemp seed husks also resulted in a decrease in particle size mean diameter. It changed from 4.81 to 2.7 mm, *i.e.*, 1.78 times. Instead of one fraction (3.15 mm and larger) dominating the primary raw material, and amounting to 91.1 ± 4,5%, two main fractions were separated in the biochar: 2.5...3.149 mm and 1.7...2.499 mm (Fig. 3). They accounted for 51.3 ± 5.1% and 29.5 ± 1.5% of the sample mass, respectively. Based on the research results, it can be said that during conversion, both the biochar from non-ground seed husks and the biochar from ground seed husks are prone to crumbling, in contrast to stem biochar, which is prone to rotting. After thermochemical conversion, the number of particles larger than 3.15 mm in the samples decreased 8.76 times. The biochar from non-ground seed husks was distributed on sieves with finer holes. However, for fractions smaller than 315 mm, *i.e.*, flour-dust fraction, the increase was not significant, from 0.45 ± 0.3% in non-ground husks to 0.52 ± 0.5% in husks biochar.

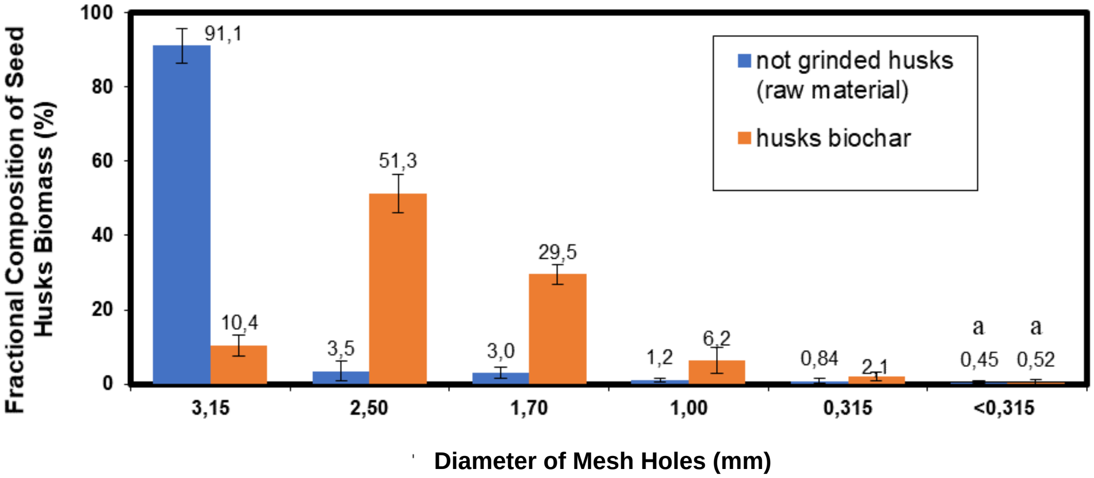

Fig. 3. Fractional composition of not ground fiber hemp seed husks and biochar produced from them (there is no significant difference between the columns marked with the same letter (a))

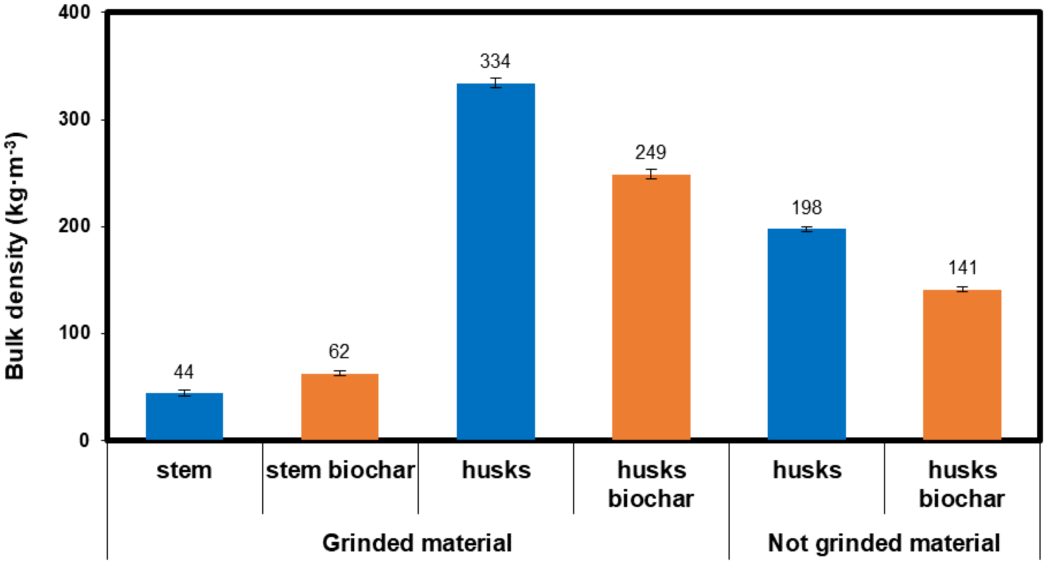

Fig. 4. Effect of thermochemical conversion on density of fiber hemp stems and seed husks

The density of a sample of non-ground fiber hemp seed husks was 198 ± 2.4 kg·m^{-3} (Fig. 4). Their grinding with a rotary mill through a sieve with holes of 1 mm in diameter led not only to changes in the fractional composition, but also to an increase in the density of the sample by 1.69 times, up to 334 ± 4.5 kg·m^{-3}. The effect of the thermochemical conversion process on the densities of the seed husks biochar sample was lower. The density of biochar decreased on average by 1.37 times: the density of biochar of ground husks decreased to 249 ± 3.9 kg·m^{-3}, and biochar of not ground seed husks decreased to 141 ± 2.2 kg·m^{-3}.

The B.E.T. method was used to evaluate the internal structure of biochar particles. At a temperature of 700 °C, an active surface area of 53.4 ± 6.51 m^2·g^{-1} was recorded in the biochar made from fiber hemp stems (Fig. 5). At that time, the active surface area of biochar of ground seed husks was only 4.11 ± 0.39 m^2·g^{-1}. But its average capillary radius was 1.05 times larger: 0.902 ± 0.035 nm in seed husks biochar and 0.861 ± 0.074 nm in stems biochar.

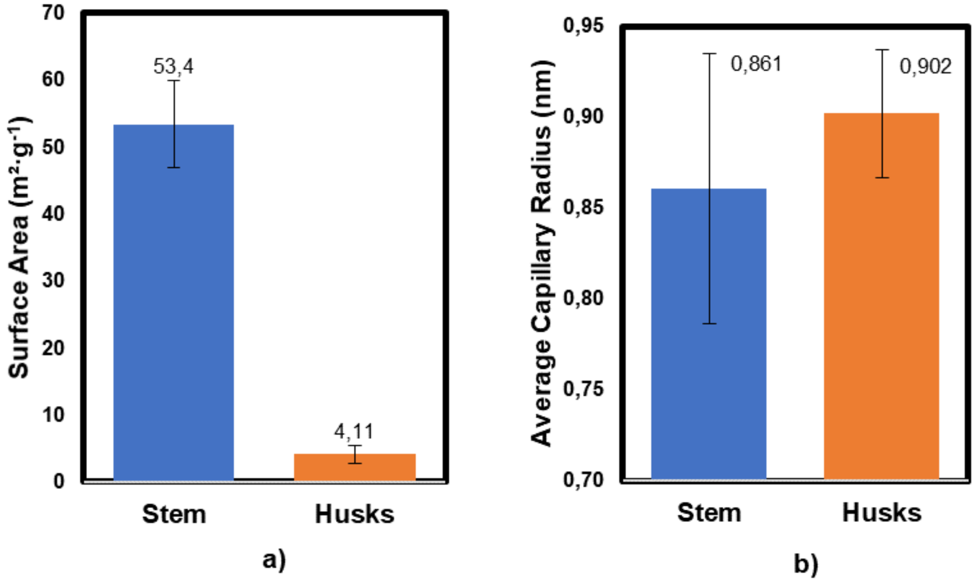

Fig. 5. Biochar active surface area (a) and average capillary radius (b) of ground fiber hemp stems and seed husks

Analysis of chemical composition and thermal properties

Thermochemical conversion of fiber hemp stems and seed husks was performed at a temperature of 700 °C in a specially prepared tubular heating furnace Nabertherm RS, using nitrogen gas purging of the heating chamber. The mass of the tested samples decreased by more than 50%. The biomass yield of biochar from ground stems was 28.4 ± 0.53%, and from ground seed husks was 30.6 ± 0.59%. A fixed difference of 2.2 percentage points is small but statistically reliable and substantial.

During thermochemical conversion, carbonization of the primary raw material takes place. The carbon concentration in the composition of the burned raw material increases (Fig. 6). Carbon in the chemical composition of fiber hemp seed husks was 56.5 ± 11.61%. In the biomass of the plant stem, it was accumulated 1.26 times less, only 45.0 ± 2.26%. After thermal conversion, a higher carbon concentration was fixed in stem biochar. The carbon concentration in it increased to 81.9 ± 8.46%. At that time, the carbon in the biochar of the seed husks was 67.9 ± 7.47%. This was 1.21 times less. After thermochemical conversion, the dispersion of the results of measurements of carbon concentrations in stem biochar also increased. The uneven composition and structure of the stem led to an increase in the value of the confidence interval as much as 3.74 times, from 2.26% in the primary raw material to 8.46% in biochar. Therefore, in contrast to the primary feedstock, the reliability of the difference between the carbon amounts in seed husks biochar and stem biochar was not established.

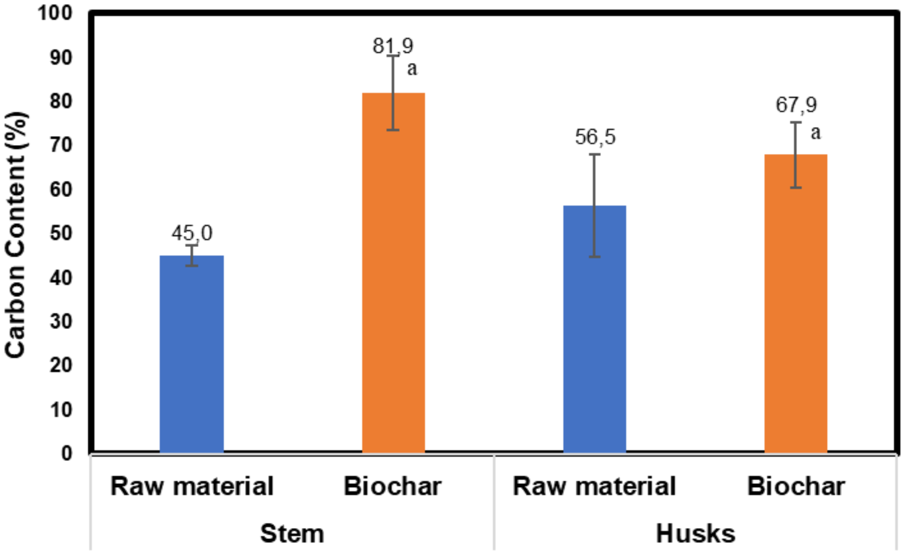

Fig. 6. Carbon concentration in primary fiber hemp raw material and in biochar (there is no significant difference between the columns marked with the same letter (a))

Changes in carbon concentrations were correlated with changes in the calorific value of the used raw materials (Fig. 7).

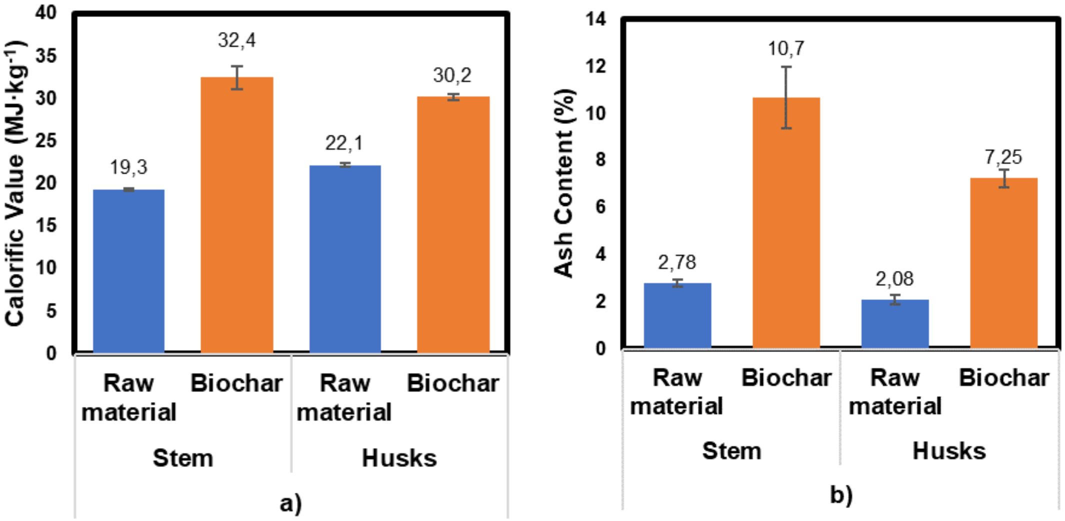

Fig. 7. Calorific values (a) and ash contents (b) of fiber hemp raw material and biochar

A higher carbon content in the seed husks resulted in a higher calorific value. The maximum calorific value of the seed husks for dry mass was 22.2 ± 0.21 MJ·kg^{-1}. At that time, the calorific value of fibrous hemp stalks was 19.3 ± 0.17 MJ·kg^{-1}. Due to a more significant change in the carbon concentration in the raw material during thermochemical conversion, the change in the calorific value of stem biochar was also recorded higher. Compared with the original raw material, its upper calorific value increased by 1.68 times, up to 32.5 ± 1.31 MJ·kg^{-1}. The calorific value of seed husks increased only 1.36 times, from 22.2 ± 0.21 to 30.2 ± 0.38 MJ·kg^{-1}.

However, the ash content of fiber hemp stems was higher than that of seed husks, 2.78 ± 0.12% and 2.08 ± 0.13%, respectively. The same trend was also observed after converting raw materials into biochar. The ash content of biochar from stems biomass increased to 10.7 ± 0.50% and biochar from seed husks increased to 7.25 ± 0.20%. Thermochemical conversion increased the ash content of fiber hemp by-products by an average of 3.7 times. In addition, the difference between the ash content of the stem biomass and seed husks raw material, and the biochar produced from them increased: the ash content of the raw materials differed by 1.34 times and that of the biochar by 1.47 times.

When comparing the ash melting characteristics of primary raw materials (fiber hemp stems and seed husks) and biochar produced from them (Table 1), no significant difference was recorded, except between seed husk primary raw material and biochar.

Table 1. Comparison of Ash Melting Characteristics

Ash Melting Stage	Fiber Hemp Stem Biomass		Fiber Hemp Seed Husks Biomass	
	Raw Material	Biochar	Raw Material	Biochar
DT	714 ± 18 °C	725 ± 20 °C	814 ± 23 °C	871 ± 6 °C
ST	1524 ± 62 °C	1522 ± 40 °C	1025 ± 23 °C	1005 ± 7 °C
HT	1529 ± 54 °C	1531 ± 12 °C	1192 ± 7 °C	1182 ± 8 °C
FT	1544 ± 35 °C	1547 ± 7 °C	1209 ± 17 °C	1228 ± 10 °C

Note: DT – deformation temperature, ST – shrinking temperature, HT – hemisphere temperature, FT – flow temperature

The onset temperature of deformation of seed husks and biochar ash produced from them was found to be higher than that of fiber hemp stem and biochar produced from them. However, fiber hemp stems and biochar produced from them were characterized by larger differences between DT and ST temperatures, 810 °C for the raw material and 797 °C for biochar. At that time, the deformation temperatures of the seed husk raw material and biochar ash were 814 ± 2 °C and 871 ± 6 °C, respectively, and the shrinking temperatures of the ash were 1025 ± 23 °C and 1005 ± 7 °C, respectively. The temperature differences decreased to 211 °C and to 134 °C, *i.e.*, 3.84 and 5.95 times compared to the results of stem biomass research. Thus, the thermal conversion of fiber hemp stems biomass and seed husks to biochar did not significantly affect their ash melting characteristics.

Effect of the Thermochemical Conversion Process

More than 2/3 of the primary raw material mass is lost during thermochemical conversion. The yield of the solid residue biochar depends on the conditions of the technological process, the properties of the biomass, and its preparation. The yield of biochar decreases by lengthening the duration of biomass retention at high temperature (Wang *et al*. 2019) and reducing the rate of overheating of its particles (Goia *et al*. 2011), as well as by increasing the temperature of the conversion process (Ateş *et al*. 2004; Jin *et al*. 2017; Kończak *et al*. 2019) and the rate of temperature increase (Goia *et al*. 2011). The biochar yield of fiber hemp stem biomass and seed husks used in the research and heated at 700 °C was 28.4 ± 0.5% and 30.6 ± 0.6%, respectively. Anand *et al.* (2023) found that the biochar content between rice straw and rice husk produced at 800 degrees differed by 4.5%, 31.00% and 35.50%, respectively. And the total yield of biochar from biomass usually reaches 25 to 40 percent (Nguyen *et al.* 2021; Iamsaard *et al.* 2023; Kwoczynski and Čmelik 2021). High temperature fundamentally changes the chemical composition of the raw material: it breaks down thermally less resistant compounds and increases the concentration of carbon in the raw material, which is characterized by a stronger C-C bond signal. Compared to the raw material, the carbon content of fiber hemp stem biochar increased 1.82 times, from 45.0 ± 2.26% to 81.9 ± 8.46%, and seed husk biochar increased 1.20 times, from 56.5 ± 11.6% to 67.9 ± 7.47%. At high temperatures, an intensive process of carbonization of plant biomass takes place: the carbon concentration in the raw material increases to the level of lignite/hard coal. However, the physical amount of carbon decreases: part of the carbon stored in plant biomass is lost. During the conversion, it is removed together with volatile compounds. Higher carbon losses were incurred during heating of the seed husks. Carbon losses amounted to 63.2%. At that time, the carbon loss in stem biomass was only 48.3%, *i.e.*, was 1.31 times lower. This confirms the statement (Demirbas 2004; Fassinou *et al*. 2009; Biswas *et al*. 2014) that the impact of factors determining biochar yield on each type of biomass is individual and complex.

Carbon is the main chemical element of materials that are used as solid fuel or fuel. A higher carbon concentration results in a higher calorific value of the raw material. Therefore, the calorific value of fiber hemp seed husks, which had a carbon concentration 1.26 times higher than that of stem biomass, was 22.15 ± 0.21 MJ·kg^{-1}, *i.e.* it was 1.15 times higher than stem biomass. The calorific value of fiber hemp biomass is said to be between 15 and 19 MJ·kg^{-1} (Poiša and Adamovičs 2011; Komlajeva *et al*. 2012; Mankowski *et al*. 2014; Černiauskienė *et al*. 2021). Studies of the calorific value of biomasses of various varieties of fiber hemp grown in Lithuanian conditions confirmed this: the values of the upper calorific value ranged from 17.53 ± 0.12 MJ·kg^{-1} (variety Felina 32) to 18.52 ± 0.14 MJ·kg^{-1} (variety Bialobrzeskie) (Černiauskienė *et al*. 2017). Fiber hemp stems without leaves were used in the research, so their calorific value was fixed higher. The increase in carbon concentration caused by the high temperature processing of the primary raw material further increased their calorific value. The calorific value of fiber hemp stems converted to biochar increased 1.68 times, from 19.29 ± 0.17 to 32.45 ± 1.3 MJ·kg^{-1}. Compared with the primary raw material, the calorific value of seed husk biochar increased by 1.36 times, from 22.15 ± 0.21 to 30.19 ± 0.38 MJ·kg^{-1}. Higher carbon loss and lower increase in carbon concentration after thermochemical treatment resulted in lower increase in calorific value of seed coats. However, the energy value of both fiber hemp stem biomass and seed husks increased significantly, becoming close to the calorific value of coal, which can reach up to 35 MJ·kg^{-1} (Yao *et al*. 2017). Thus, the thermochemical conversion of biomass in an oxygen-free environment has been established as an effective means to increase the comparative energy value of biomass.

However, the thermochemical conversion of biomass to biochar does not eliminate or mitigate the disadvantage of combustion technologies: the increase in the concentration of undesirable compounds. The concentration of carbon in biomass increases along with its ash content: the concentration of non-combustible mineral impurities. High-quality burning fuel is considered to contain no more than 5% ash content, and the predominant ash content in agricultural residues exceeds this limit and reaches 6.26 ± 3.23% (Sun *et al.* 2017). Therefore, fiber hemp plant material has a lower ash content than other agricultural residues. High temperature processing does not reduce the physical ash content, unlike the physical carbon content. Compared to the starting material, the ash content of seed husks biochar increased by 3.49 times to 7.25 ± 0.2% and that of fiber hemp stems by 3.83 times to 10.66 ± 0.5%. A similar increase in ash content can be observed in the results reported in the literature. In the biochar made from rice and oat husks and hazelnut and walnut shells, the ash content increased respectively by 2.7 times (from 3.43 ± 0.11% to 13.49 ± 0.43%), 3.9 times (from 17.39 ± 0.24 % to 46.76 ± 0.24%), 4.5 times (from 0.50 ± 0.08% to 2.80 ± 0.32%) and 5.6 times (from 0.35 ± 0, 03% to 1.58 ± 0.22%) (Bardha *et al.* 2023). Biochar produced from herbaceous plant waste also has a high ash content, with 12.0% from straw, 14.5% from leaves, 13.2% from grass and 19.2% from grass straw (Mitchell *et al.* 2013). In addition, the ash content from *Achnatherum splendens* (Trin.) raw material and biochar produced from it increased 2.6 times (from 10.02% to 26.59%) (Irfan *et al.* 2016). The type/morphological part of the biomass and its chemical composition, fertilization, and in the conditions of industrial production, as well as the harvesting and processing technology, are the main factors that determine its ash content (Černiauskienė *et al*. 2021; Zajac *et al*. 2018; Tumuluru *et al*. 2012). Fiber hemp stems without leaves were used in the research, so their ash content was relatively low, 2.78 ± 0.12%. Researchers state that the ash content of fiber hemp is 2.5 to 4.3% (Poiša and Adamovičs 2011; Komlajeva *et al*. 2012; Sausserde *et al*. 2013; Černiauskienė *et al*. 2021), and the average ash content of biomasses of various varieties of fiber hemp grown in Lithuanian conditions is 3.36 ± 0.23% (Černiauskienė *et al*. 2017). The ash content of seed husks was lower, only 2.08 ± 0.13%: 0.7 percentage points lower than the ash content of stem biomass. It was found that processing the primary raw material at high temperature increased the difference between fiber hemp stems biochar and seed husks biochar to 3.4 percentage points. However, it had almost no significant effect on the melting characteristics of the ash. The melting characteristic of ash is an important property that is considered in evaluating the quality of biofuels and which often causes problems in the thermal conversion of biomass. At low values of ash deformation and softening temperatures, slag, sticky and hard-to-remove impurities are formed (Mankowsk *et al*. 2014; Čepauskienė and Pedišius 2018; Čepauskienė and Pedišius 2019). It is desirable that the ash maintains its state up to at least 1100 °C. The values (temperatures) of the critical points of the melting temperature characteristics of ash depend on the chemical composition of the raw material, the fertilizers used in growing biomass, the hydrometeorological conditions during the plant vegetation and other factors (Poiša and Adamovičs 2011; Poiša *et al*. 2013; Zajac *et al*. 2018). This determines the amount of micro- and macroelements in biomass, which, remaining in the ash, participate in chemical reactions at high temperatures and influence the temperature characteristic of ash melting. The onset temperature of fiber hemp biomass ash deformation is reported to be very variable. It is usually indicated in the range from 648 to 890 ºC, but it can reach up to 1450 °C (Černiauskienė *et al*. 2017). The temperature of onset of deformation (DT) of ash used in the research was found to be 714 ± 18 °C for fiber hemp stems, and 814 ± 23 °C for hemp seed husks. Accordingly, the DT temperatures of biochar ash were 725±20 °C and 871±6 °C. The thermochemical conversion of the primary raw material to biochar did not change the melting characteristics of the ash: a significant difference was recorded only between the starting temperatures of seed husk primary raw material and seed husk biochar ash deformation. Thus, treatment of fiber hemp stems biomass and seed husks at high temperature in an anoxic environment was not an effective means of altering the melting characteristics of their ash.

Analyzing the changes in the fractional composition of the biochar particles of fiber hemp stems revealed the effect of their fiber structure. Fiber particles tend to tangle. A felt-like structure is formed, which gives structure to the sample, increases its porosity, and holds and prevents the raw material from collapsing. The density of the sample of ground fiber hemp stems was 44 kg·m^3, and the sample of ground seed husks was 334 kg·m^3, *i.e.*, 7.59 times lower. Grinding is a frequently used operation in biomass post-processing technologies. Grinding is aimed at increasing the homogeneity of the raw material and improving its technological properties, as well as during thermochemical conversion: better conditions are created for faster reheating of the raw material particles. This results in higher biochar yield and better quality (Goia *et al*. 2011) In addition, biochar with a finer (less than 2 mm size) fractional composition has better sorption properties, higher moisture absorption, better retains nutrients and water in the soil (Dubrovina 2021). It has been established that during processing at high temperature, the particles of the primary raw material are naturally crushed. Compared to the starting material, the diameter of the produced biochar particles decreased by an average of 1.77 times: ground fiber hemp stems – 2.08 times, ground hemp seed husks – 1.44 times and whole hemp seed husks – 1.78 times. Differences in the principles of decomposition of biochar particles of different origins were also observed. Biochar from fiber hemp seed husks tends to crumble. A mass of particle-flour (larger than 315 mm) fraction is formed. At that time, the biochar of the stem tends to decay. A substantial increase in the flour-dust fraction (smaller than 315 mm) was recorded. In addition, the thermochemical conversion did not destroy the fiber structure of the hemp stem: the felt-like structure characteristic of the primary raw material was also preserved in the biochar. Therefore, the difference between the biochar sample density of fiber hemp stems (62 ± 2.2 kg·m^3) and the biochar sample density of ground seed husks (249 ± 3.9 kg·m^3) decreased, but it still remained significant.

Based on the B.E.T. evaluation of the internal structure of biochar using the method, it was found that the average capillary diameter (0.86 ± 0.07 nm) was lower in stem biochar and the active surface area (53.4 ± 6.5 m^2·g^{-1}) was higher than in seed husk biochar. The active surface area formed by the walls of capillaries and cavities participates in hydrosorption and chemosorption processes, so it is a very important indicator that determines and determines the quality of biochar. It can be tentatively stated that biochar from fiber hemp stems had higher qualitative values. The average capillary diameter of fiber hemp seed husk biochar was 4.65% larger and reached 0.9 ± 0.03 nm. However, the active surface area was fixed as much as 12.99 times smaller, *i.e.*, 4.11 ± 0.3 m^2·g^{-1}. The obtained results were very different from the results published by other authors: active surface area of biochar from date palm biomass 249 m^2·g^{-1} (Elnour *et al*. 2019), pecan branch biochar – 248 m^2·g^{-1} (Liu *et al*. 2023), buckwheat husk biochar – 39-43 m^2·g^{-1} (Lim *et al*. 2022), walnut shall biochar – 150,3-45,4 m^2·g^{-1}, sunflower seed husk biochar – 303.7 m^2·g^{-1} (Han *et al*. 2022), pecan shells biochar – 311.66 m^2·g^{-1} (Liu *et al*. 2023) jujube pit biochar – 246.9 m^2·g^{-1} (Chun *et al*. 2021). This confirms the complexity of biochar production and the differences between researchers who emphasize various factors and their importance for the internal structure of biochar. However, it can be stated that the spread of active surface area values in biochar from husks/shells biomass was found to be greater than in biochar from large-stemmed plant biomass.

CONCLUSIONS

1. The thermochemical process affected both the physical and chemical properties of the fiber hemp material. One of the observed physical changes was evident in the fractional composition. When the largest part of the fraction of the raw materials of ground stems was 3.15 mm particles - 43.2 ± 9.8%, then in the biochar of this raw material, particles with a size of 0.315 mm fraction prevailed - 46.3 ± 4.9%. In the biochar of seed husks, the largest part was also made up of particles of 0.315 mm fraction (73.3 ± 8.9%), but the largest part of the primary raw material was made up of particles of 1 mm fraction (52.9 ± 12.8 %).
2. The change in fractional properties during the thermochemical process also affected the density indicators. The density of chopped stalks increased from 44 to 62 after the thermochemical process, and the density of husk raw material, both chopped and unchopped, decreased from 334 ± 4.5 $kg·m^{-3}$ to 249 ± 3.9 $kg·m^{-3}$and from 198 ± 2.4 $kg·m^{-3}$ to 141 ± 2.2 $kg·m^{-3}$, respectively.
3. The different characteristics of raw biochar from ground stems and husks are also determined in the surface area indicators. The determined surface area in the husks was even 12.9 times smaller than in the stem, while the average capillary size in the husks was higher, but only 1.05 times.
4. During thermochemical conversion, the carbon concentration in the biochar of fiber hemp stems and seed husks changes disproportionately to the biochar yield: the biochar yield of fiber hemp stems heated at 700 °C for 2 hours was 1.08 times lower than the biochar yield of seed husks (respectively, 28.4 ± 0.5% and 30.6 ± 0.6%), and the increase in carbon concentration was 1.52 times higher (from 45.0 ± 2.26% to 81.9 ± 8.46% in the biochar of fiber hemp stems and in seed husks biochar from 56.5 ± 11.6% to 67.9 ± 7.47%). The thermochemical conversion promoted the crumbling or wear of the biochar particles, but it did not destroy the fiber structure of the fiber hemp stem: the felt-like structure characteristic of the original raw material remained in the biochar and affected its density and fractional composition.
5. High temperature treatment increased the ash content of fiber hemp seed husk biochar by 3.49 times, from 2.08 ± 0.1% to 7.25 ± 0.2% and fiber hemp stem biochar by 3.83 times, from 2.78 ± 0.1% to 10.66 ± 0.5%. However, the increase in ash in biochar had no effect on ash for the melting characteristics, where the change was fixed by 2 degrees in stem raw material and biochar, respectively 1524 ± 62 °C and 1522 ± 40 °C at ST stage, and by 20 degree comparing seed husk raw material with biochar, respectively 1025 ± 23 °C and 1005 ± 7 °C at ST stage.

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