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Investigation of Pressed Solid Biofuel Produced from Multi-Crop Biomass

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Abstract: The paper presents the preparation and use of pressed solid biofuel of multi-crop plants (fibrous hemp (*Cannabis sativa* L.), maize (*Zea mays* L.) and faba bean (*Vicia faba* L.)) as mono, binary and trinomial crops. The results of the investigation show that three main chemical elements (carbon, oxygen and hydrogen) accounted for 93.1 to 94.9% of the biomass pellet content. The moisture content varied from 3.9 to 8.8%, ash content from 4.5 to 6.8% and calorific value from 16.8 to 17.1 MJ·kg^{-1}. It was found that the density (DM) of all variants of pellets was very similar; the faba bean biomass pellets had the highest density of 1195.8 kg·m^{-3} DM. The initial ash deformation temperature (DT) of burning biomass pellets was detected, which varied from 976 to 1322 °C. High potassium (K), calcium (Ca) and phosphorus (P) concentrations were found in all types of biomass ash. The quantities of heavy metals in pellet ash were not large and did not exceed the permissible values according to Lithuanian legislation. These chemical properties of multi-crop biomass ash allow them to be used in agriculture for plant fertilization.

Keywords: multi-crop; biomass; solid biofuel; pellet properties; ash utilization

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1. Introduction

The goal of reducing dependence on fossil fuels and the need to reduce the negative impact on the environment are encouraging the increasing use of renewable energy sources. Various types of biomass can be used for this purpose. Biomass is considered to be an environmentally friendly energy source that can reduce carbon emissions [1]. Compaction technologies, such as granulation, briquetting and others, produce fuels with desired physical, mechanical, chemical, thermal and combustion characteristics [2].

Ensuring the quality of biofuel is paramount, especially for pellets, as there are many different feedstocks that can be granulated [3]. Various studies suggest that durable pellets can be produced not only from wood or agricultural residues, but also from energy-bearing herbaceous plants [4]. However, pellets made from non-wood biomass are often inferior to pellets made from wood biomass in terms of quality. They have a lower bulk density, high ash content and lower calorific value. Thus, solutions are needed that allow the more efficient use of non-wood biomass as solid fuel, such as blending different types of biomass, to obtain better pellet quality [3].

Compaction makes it possible to produce more compact products of the same shape and size, which can be used more easily with existing handling and storage equipment, thus reducing transport, handling and storage costs [5].

The compaction of biomass allows the production of fuels with a higher bulk density and high calorific value, ensures better composition quality through the fractionation of components, and allows the adaptation of the product to specific conversion technologies [6]. Granulation is currently one of the most popular methods of biomass compaction [7].

Reducing the size of biomass requires mechanical energy to overcome the decomposition and friction of materials. The energy demand of machines can be an important factor in this process [8–10].

European Green Deal commitments in the agricultural sector, such as reducing the use of fertilizers and pesticides and increasing the number of organic farms, are likely to lead to an increase in the area of multifunctional crops. Multifunctional crops could also be a potential source of solid biofuel.

Polyculture (multi-crop) is when two or more crops are grown in the same field. The interlocking of two or more types of crops increases resource efficiency and often results in higher yields per unit area [11]. In addition, catch crops improve soil fertility due to the biological fixation of nitrogen using legumes, increase soil conservation by covering the soil and provide greater protection against fallout compared to monoculture [12].

Polyculture can not only increase yields, but is also a means of combating pests, pathogens and soil degradation, and is a preventive measure against deteriorating environmental conditions [13–16].

In some countries, agriculture is dominated by polyculture. In Latin America, for example, small farmers grow 70–90% of their beans together with maize, potatoes and other crops, while in Colombia 90% of beans are grown in multifunctional crops [13].

It is recommended to grow mixtures of legumes and non-legumes to obtain more and better-quality products with lower costs. Good complementary mixtures include maize (*Zea mays* L.) with Chinese beans (*Vigna unguiculata*), kidney beans (*Phaseolus vulgaris*) or field beans (*Vicia faba* L.). Growing a mixture of maize and legumes produces significantly higher yields than using monoculture. Studies show that this saves 16–29% of land and 19–36% of fertilizers [17,18].

Hemp (*Cannabis sativa* L.) is a suitable plant for polyculture. Lately, cannabis cultivation has been gaining popularity in many countries, such as the United States, Germany, France, Sweden, the Netherlands, the United Kingdom, Spain and Italy, among others. Cannabis biomass can be processed into many products, including biofuel, and as a result cannabis has an advantage over other industrial crops from which only one type of raw material is extracted [19,20]. Industrial cannabis can compete with energy crops in global markets as a feedstock for many bioenergy products, allowing emission reduction [21].

Maize is a plant suitable for both food and feed production as well as the production of bioenergy. However, climate change, plant pathogens, insect pests and other biotic and abiotic stressors, such as heat and drought, adversely affect maize production [22]. Corn stover, which accounts for 80% of agricultural residues in the United States, has received significant attention as a renewable energy source [23].

Soil properties can be improved by growing beans. They add nitrogen and thus increase the sustainability of growing systems. Their strong root systems loosen the subsoil, improve soil structure and promote the activity of soil microorganisms [24]. Beans not only improve the soil, but also reduce the use of chemical fertilizers for future crops and contribute to reducing environmental pollution in agriculture [25].

Maize and bean crops, which are widely cultivated around the world, are high in residues. Studies show that a mixture of bean stalk and maize cob biomass can be used to produce fuel briquettes that meet German standards [26]. The lignin, cellulose, hemicellulose, protein, lipids and moisture in biomass feedstock are the main chemical components that affect granulation. Proper operating parameters (temperature, pressure and proper moisture content, usually 10–12% depending on the type of feedstock) also have a significant impact on granulation, but the impact of these parameters on pellet quality is complex. The granulation of different feedstocks of biomass can improve the physical quality (e.g., density, durability) of biomass fuel pellets while reducing the energy consumption of the pelleting process [27].

The developed standards allow for control over the quality of biomass pellets supplied to the market. The International Organization for Standardization (ISO) has developed international quality standards for pellets. Several European countries have also developed regulations and standards for pellet quality certification, among them Austrian standard ÖNORM M 7135, Swedish standard SS 187120, German standards DIN 51731 and DIN EN 15270, Italian standard CTI-R04/05 and the French recommendation ITEBE [3,28].

The combustion of biomass pellets poses another problem: large amounts of ash are generated, which in most cases becomes landfill waste. According to Zhai et al., global ash production from various types of biomass (agricultural residues, energy crops, wood biomass, forest residues, recovered wood, paper sludge, sewage sludge and municipal solid waste) is 170 Mt per year and could increase to 1000 Mt per year if all available biomass resources were exhausted [29].

Researchers suggest a variety of uses for biomass ash, and some argue that the most sustainable way is to return it to the soil. According to Silva et al. [30], the disposal of biomass ash in landfills is not only costly but also wastes valuable resources. As this ash contains important macronutrients and can be used as a soil liming agent, returning it to the soil would contribute to sustainable energy production [30]. As pointed out by Voshell, ash disposal in landfills not only will be undesirable due to high costs, but may not be possible at all once the principles of the circular economy are finalized [31].

However, the use of ash still raises many questions, as its elemental composition varies greatly depending on the type of biomass and many other factors, and even heavy metals can be detected in it. The aim of this study was to evaluate the suitability of multi-crops (maize, hemp and beans) for solid biofuel and the potential use of ash from the combustion of biomass pellets for fertilization.

2. Materials and Methods

2.1. Selection and Preparation of Biomass for Granulation

A stationary field experiment was carried out at the Experimental Station of Vytautas Magnus University Agriculture Academy. Cultivation of fibrous hemp (*Cannabis sativa* L.), maize (*Zea mays* L.) and faba bean (*Vicia faba* L.) as mono, binary and trinomial crops was investigated. The experiment had 7 combinations: (1) maize, (2) fibrous hemp, (3) faba bean, (4) maize and fibrous hemp, (5) maize and faba bean, (6) fibrous hemp and faba bean and (7) maize, fibrous hemp and faba bean. The scheme of experiments can be seen in Figure 1.

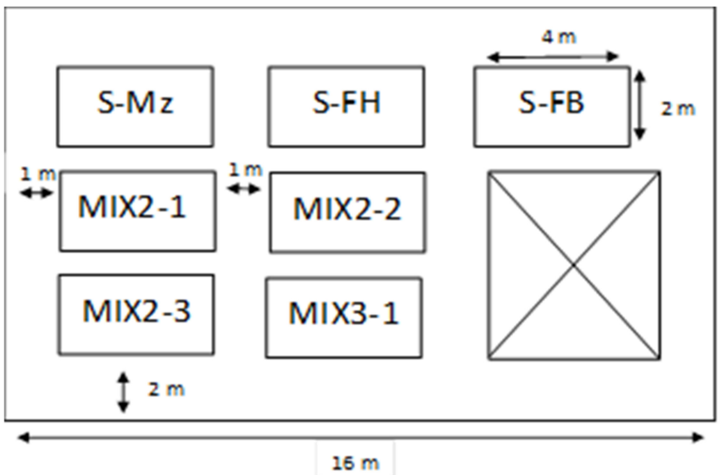

Figure 1. Scheme of planting arrangements. Abbreviations: S-Mz—maize (mono), S-FH—fibrous hemp (mono), S-FB—faba bean (mono), MIX2-1—maize and fibrous hemp (binary), MIX2-2—maize and faba bean (binary), MIX2-3—fibrous hemp and faba bean (binary) and MIX3-1—maize, fibrous hemp and faba bean (trinomial).

The investigated biomass was divided into 7 groups depending on its nature, and biomass types were evaluated.

Figure 2 shows the multi-crop fields of the experiments, from which biomass was used for research. The physical–mechanical, chemical and thermal properties of multi-crop biomass pellets were investigated to determine the suitability of multi-crop plants for biofuel.

Figure 2. Trinomial crop (MIX3-1) cultivation (photos by authors).

These investigations were carried out in stages: first, the harvested plants were chopped and milled, the produced flour was then pressed into pellets, and the pellets were burned, eventually producing heat energy and ash.

At first, the plant biomass was chopped with the drum chopper of a MARAL-125 forage harvester (Germany). The harvested plants were then milled using a Retsch SM 200 hummer mill (Germany). The fraction composition of the produced mill was determined with a Retsch AS 200 sieve shaker (Germany) using sieves with holes 0.1, 0.25, 0.5, 0.63, 1.0 and 2.0 mm in diameter. The weight of each sample was 100 g, and the following parameters were determined when sieving: height, 1 mm; sifting time, 1 min; interval, 10 s. After the sample was sifted, the remaining mass was weighed and the percentage of sample particles was calculated. Each test was repeated 3 times for every sample.

2.2. Investigation of Biofuel Pellets and Determination of Their Properties

Physical–mechanical properties of pellets. A low-power (7.5 kW, up to 200–300 kg·h^{-1}) ZLSP200B granulator (Poland) with a horizontal matrix with 6 mm holes was used for pellet production. Investigation of physical–mechanical, chemical and thermal properties of biomass pellets and determination of ash melting temperatures and chemical composition were performed in the laboratories of the Vytautas Magnus University Agriculture Academy and the Lithuanian Energy Institute according to the standard methodology valid in Lithuania and other European countries.

The volume of the pellets was calculated according to their size (diameter and length). A Limit 150 mm digital vernier calliper (PRC) was used (accuracy up to 0.01 mm). For the experiments, 10 pellets were randomly picked to obtain the mean value and error. After determining the volume and weight of the pellets, the density of all investigated pellet samples was calculated according to ISO 18847:2016 standard [32]. Samples were weighed on a KERN ABJ scale (Germany) with accuracy up to 0.01 g. Each test was repeated 3 times.

The moisture content of the pellets was determined according to the standard methodology. The weighed samples (7 combinations, 3 repetitions) were dried for 24 h at 105 °C, and then weighed again. The empty containers were also weighed, and the moisture content of each sample was calculated as a percentage. Analysis of variance with a 3-replicate design was performed on the experimental data to determine significance at the 95% confidence level.

Analysis of chlorine and sulphur. The samples were milled to a homogeneous powder (1 mm sieve) by using an IKA MF 10.2 cutting mill. Approximately 1 g of milled powder for each combustion was weighed into a compressed tablet with a pressure of 10 t per square centimetre. The samples were put into a quartz combustion crucible and closed in a calorimetric bomb. The bomb was then pressurized with oxygen to 35 atm. The bomb was immersed in a water bucket and ignited via an electrical discharge. After the ignition and cooling steps, the released gases were dissolved after passing through an Erlenmeyer flask filled with deionized water. All internal parts of the bomb were rinsed with deionized water, and all washings and solutions were collected in 50 mL volumetric flasks. All samples were analysed with a Dionex ICS 5000 ion chromatography system.

Elemental analysis. The samples were mineralized by the same method according to ISO 16967:2015 standard [33], with 8 mL of concentrated nitric acid, 1 mL of hydrofluoric acid and 3 mL of hydrogen peroxide. Mineralisation process was carried out with Anton Paar Multiwave 3000 microwave at 800 W power and 6 MPa pressure, with a pressure rate of 50 $kPa \cdot s^{-1}$. After mineralisation, the solution was poured into 50 mL flasks and diluted to 50 mL with deionized water. Analysis of solutions (Al, Ca, Cd, Cu, Fe, K, Mg, Na, P, Pb, Si and Zn) was performed using an ICP-OES device (Perkin Elmer). Quantitative analysis was performed to collect sample data. The scanning of each individual sample was repeated 3 times to obtain sufficiently good results. Efforts were made to avoid a memory effect during the measurements, so a wash-out time of 1 min was used.

Determination of calorific value. The samples were ground to a homogeneous powder (1 mm sieve) by using an IKA MF 10.2 cutting mill. Calorific value was determined in 2 test sub-samples, examined in the form of pellets. The prepared sample was pressed with a hydraulic press at a force of about 10 t to a diameter of about 13 mm and weight of 1.0 ± 0.2 g. The calorific value was determined by an IKA C6000 automatic bomb calorimeter in adiabatic mode.

2.3. Determination of Ash Content and Essential Properties

Determination of ash content. After drying, the samples were ground to a nominal upper particle size of 1 mm. The prepared samples were tested and ash content and calorific value were determined. Ash content was determined in 2 test portions with a minimum of 1 g each. The weighed samples were placed in the furnace and heated for approximately 4 h in 2 steps. The first step was to remove volatiles at 250 °C (heating rate 7.5 °C/min) for approximately 1 h, and the second step was to totally oxidize the organic matter at 550 °C (heating rate 10 °C/min) for approximately 2 h. After the heating procedure, the dishes with samples were placed in a desiccant and allowed to cool to ambient temperature. The cooled dishes with samples were weighed.

CHN analysis. The total carbon, hydrogen and nitrogen contents were determined using a Flash 2000 CHNS-O analyser from Perkin Elmer. For the CHN analysis, the dried and crushed samples were weighed (0.8–1.5 mg) in a tin capsule, which was combusted in a reactor at 1000 °C. The sample and container melted and promoted a violent reaction (flash combustion) in the atmosphere temporarily enriched with oxygen. Combustion products CO_2, H_2O and NO_2 were carried by a constant flow of carrier gas (helium) passing through the column. Oxygen content was determined by the equation O = 100-Ash-C-H-N-S-Cl.

Ash melting behaviour. The obtained ash was crushed with a pestle to minimize the particle size. The ash was moistened with ethanol (purity > 95%) to obtain a paste consistency. The processed mass was compressed by hand into cylindrical samples 5 mm high and 5 mm in diameter. The compressed samples were placed vertically to dry for about 24 h. The test was carried out in an oxidizing reducing atmosphere using a mixture of carbon monoxide and carbon dioxide in a volume ratio of 60 to 40%. First, the oven temperature was raised to 550 °C, and then was raised evenly by 2 °C/min and photographs were taken. When the ash melted, the temperature at which the phase of the sample changed was recorded visually according to the melting phase.

There are 4 ash melting phases: starting shrinkage temperature (SST), at which the dimensions of the prepared ash sample decrease and the sample area does not shrink below 95% at 550 °C; ash deformation temperature (DT), at which the sample is shaped into an oval with a height equal to half of the base diameter; fusibility temperature (FT), at which the melting ashes become liquid and spread on the plate in such a manner that the height of the layer is equal to half the height of the ash hemisphere; and ash hemisphere temperature (HT) [34,35].

MS Office Excel was used for statistical processing of the results. From the obtained results, averages were derived, and diagrams with standard deviations were made. Mean values and their confidence intervals (CI) were calculated at a probability level P of 0.95.

3. Results and Discussion

3.1. Physical–Mechanical, Chemical and Thermal Properties of Multi-Crop Mill and Pellets

The research results of multi-crop plant cultivation in 2019–2020 are presented in Figure 3. The results show that the highest biomass yield was obtained in trinomial crop plots (2081.06 g·m^{-2}). Compared with mono-crop yields, higher biomass yields were obtained in binary plots with maize and hemp (1219.73 g·m^{-2}) and hemp and beans (1097.48 g·m^{-2}).

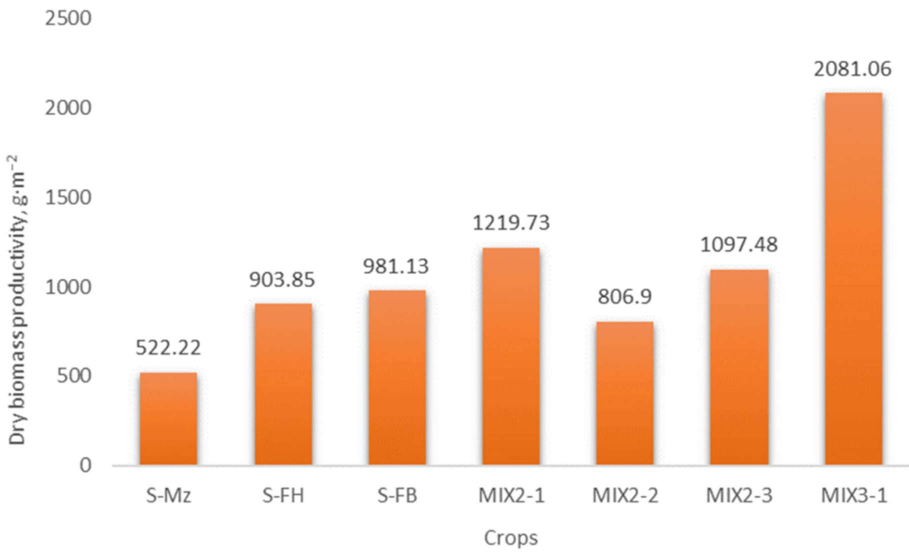

Figure 3. Yield of biomass from different crops.

The quality and properties of the produced pellets depend mainly on the particle size and moisture of the raw material, as well as the conditions of the process, i.e., the pressure and temperature of the working parts of the granulator [36]. When preparing solid biofuel pellets, it is important that they are of uniform shape and size. This makes it easier to load them into heating systems and avoids the formation of excess smoke that can be caused by pellet cracks. When granulating different types of biomass, it is important to obtain high-density pellets with good particle adhesion. It was determined that high-quality biomass pellets do not form dust fraction particles and are resistant to crushing when stored under suitable conditions [37].

As the quality parameters of the pellets are influenced by the fineness of the mill, the fractional composition of the mill was determined during the study. The results of the fractional composition of the multi-crop mill are presented in Table 1.

Table 1. Fractional composition of multi-crop mill.

Type of Biomass	Average Fraction Remaining on Sieve, with Error, % (Diameter of Sieve Holes, mm)						
	2.0	**1.0**	**0.63**	**0.5**	**0.25**	**0.1**	**0.0**
S-Mz	0.23 ± 0.04	42.90 ± 17.10	18.64 ± 14.60	8.46 ± 4.68	18.91 ± 1.52	5.34 ± 1.51	5.52 ± 0.34
S-FH	75.20 ± 14.19	1.44 ± 2.07	6.00 ± 3.58	4.54 ± 3.35	7.86 ± 3.68	2.32 ± 1.12	2.66 ± 0.70
S-FB	3.96 ± 2.15	36.92 ± 6.41	28.53 ± 4.24	5.45 ± 2.19	13.76 ± 1.85	4.31 ± 0.62	7.07 ± 0.77
MIX2-1	41.81 ± 2.94	6.49 ± 2.27	25.66 ± 10.07	5.41 ± 3.48	13.83 ± 2.55	2.96 ± 0.65	3.84 ± 0.51
MIX2-2	66.95 ± 8.92	2.16 ± 0.83	9.10 ± 3.79	5.21 ± 1.09	9.87 ± 1.47	2.75 ± 1.44	3.95 ± 0.67
MIX2-3	0.23 ± 0.08	32.41 ± 7.68	32.15 ± 14.50	5.12 ± 1.87	20.02 ± 4.13	5.21 ± 1.90	4.86 ± 1.16
MIX3-1	20.59 ± 10.58	23.14 ± 12.02	29.02 ± 0.89	4.56 ± 0.49	14.18 ± 1.23	3.75 ± 0.70	4.77 ± 0.22

After investigating the biomass of all seven combinations, it was found that the largest fractions of milled biomass (2 mm till) were obtained by milling the biomass of hemp mono-crop (S-FH) and hemp and bean multi-crop (MIX2-2): 75.20 ± 14.19% and 66.95 ± 8.92% of mill, respectively, remained on the 2 mm sieve. The highest fraction of dust was formed when milling biomass of bean mono-crop (S-FB; 7.07 ± 0.77%), and the lowest from biomass of hemp mono-crop (S-FH; 2.66 ± 0.70%).

Whittaker et al., stated that smaller particles can be used to produce more durable pellets, in that they increase friction in the mill and can occupy voids more efficiently than larger particles [4].

Pradhan et al., provided some insights from the generalized findings of their research. Initially, the large particle size of the biomass acts as a predetermined breaking point for the pellets. The finer the grind, the greater the durability, as smaller particles have more surface contact during granulation. Small particles absorb more moisture than large particles, making them more conditioned. A mixture of particles of different sizes would ensure optimal pellet quality, as the particles would form bonds between them with almost no gaps [8].

This study shows that a fraction of all seven biomass samples by fractional composition was suitable for granulation. The optimal size of the fraction has to be 1–2 mm. In the pre-granulation studies, the plant biomass was milled finely enough, with 60–80% of particles up to 1 mm in size, and the remaining particles were 2 mm.

The results of the studies show that three main chemical elements—carbon (C), oxygen (O) and hydrogen (H)—made up 93.1 to 94.9% of the biomass pellets. Based on an assessment of the Cl content in the biomass pellets, it can be stated that the content in multi-crop pellets was almost twice as low as that in the mono-crop pellets. The results of the chemical composition of the biomass pellets are presented in Table 2.

Table 2. Chemical composition of biomass pellets.

Type of Biomass	Chemical Composition, %					
	C	**O**	**H**	**S**	**N**	**Cl**
S-Mz	45.61 ± 1.13	42.21	5.59 ± 0.46	0.06 ± 0.46	0.81 ± 0.32	0.17 ± 0.04
S-FH	42.47 ± 2.15	47.11	4.96 ± 0.05	0.04 ± 0.01	0.41 ± 0.18	0.20 ± 0.02
S-FB	46.57 ± 0.56	41.32	5.47 ± 0.05	0.06 ± 0.03	0.91 ± 0.16	0.22 ± 0.02
MIX2-1	45.88 ± 1.13	43.32	5.66 ± 0.45	0.04 ± 0.33	0.65 ± 0.31	0.12 ± 0.02
MIX2-2	45.80 ± 0.67	41.05	5.60 ± 0.05	0.04 ± 0.01	0.76 ± 0.16	0.10 ± 0.02
MIX2-3	45.88 ± 0.67	41.88	5.59 ± 0.05	0.06 ± 0.01	0.88 ± 0.16	0.12 ± 0.02
MIX3-1	45.35 ± 1.31	42.33	5.41 ± 0.09	0.05 ± 0.01	0.82 ± 0.30	0.11 ± 0.02

Data from other research also confirm that the main elements in non-wood biomass pellets are C, O and H. For example, Fusi et al., found that the contents of these elements in miscanthus were C, 45.40%; O, 46.25%; and H, 4.10% [38]. Jasinskas et al., found that their content in faba bean biomass pellets was C, 46.00%; O, 40.16 to 42.89%; and H, 1.3 to

1.6% [39]. The study of Sulaiman et al., showed that the biomass mixture of maize stems and cobs in pellets accounted for C, 48.57%; O, 44.19%; and H, 6.22% [40].

According to the IWPB standard, solid fuels from biomass pellets for industrial use must contain ≤0.04% S, ≤1.5% N and <0.1% Cl [4]. It was determined that the content of both S and N in the tested mono- and multi-crop biomass pellets did not exceed the established maximum limits. In variant MIX2-2, the Cl content did not exceed the set limit, while in the remaining variants, the upper limit was exceeded. In the case of MIX3-1, the excess was very small, only 10%; in the case of MIX2-1 and MIX2-3, it was 20%, and in the S-FB variant, the limit was exceeded by 2.2 times. The authors suppose that fields with multiple crops could have a negative influence on chlorine accumulation and that different species inhibit chlorine accumulation in others.

Data from other research on a variety of plants are similar. Miranda et al., found that the nitrogen and sulphur contents in maize cob pellets were very low (0.32 and 0.04% of dry matter, respectively) and 80% below the upper limit. However, chlorine values were slightly above the upper limit (0.1% dry matter) [7].

Some researchers found that pellets of equal length ensured smooth and even movement in boiler feed transportation facilities, while pellets of smaller diameter provided more uniform combustion compared with pellets of larger diameter [8,41].

According to the German standard DIN 51731, the diameter of the pellets must be 4 to 10 mm and the length less than 5 × D mm [28]. The biometric and other properties of all investigated biomass pellets are presented in Table 3.

Table 3. Biometric properties of biomass pellets.

Type of Biomass	Moisture Content, %	Length, mm	Diameter, mm	Weight, g	Density, kg·m^{-3}	Density, kg·m^{-3} (DM)
S-Mz	4.61 ± 0.59	26.78 ± 1.31	6.18 ± 0.05	0.98 ± 0.06	1215.60 ± 30.48	1159.57 ± 29.08
S-FH	3.86 ± 0.05	26.05 ± 1.20	6.17 ± 0.16	0.97 ± 0.06	1243.75 ± 48.94	1195.75 ± 47.05
S-FB	6.15 ± 0.28	23.69 ± 1.13	6.08 ± 0.06	0.88 ± 0.05	1273.77 ± 36.40	1195.42 ± 34.16
MIX2-1	4.44 ± 0.22	26.36 ± 1.48	6.14 ± 0.03	0.95 ± 0.06	1215.27 ± 37.03	1161.30 ± 37.30
MIX2-2	8.78 ± 0.43	25.93 ± 1.64	6.10 ± 0.13	0.92 ± 0.08	1212.90 ± 46.70	1106.46 ± 42.60
MIX2-3	8.30 ± 0.15	23.43 ± 1.94	6.13 ± 0.08	0.85 ± 0.09	1230.30 ± 58.80	1128.12 ± 53.88
MIX3-1	5.63 ± 0.23	26.30 ± 1.56	6.18 ± 0.07	0.95 ± 0.06	1211.44 ± 31.87	1143.26 ± 30.08

The results of pellet density show that S-FB and S-FH had the highest density S-FB and S-FH (1195.75 ± 47.05 and 1195.42 ± 34.16 kg·m^{-3} of dry matter (DM), respectively). MIX2-2 pellets had the lowest density, 1106.46 ± 42.60 kg·m^{-3} of DM. However, the density of all investigated types of pellets exceeded 1100 kg·m^{-3}. The recommended density of pellets specified in the standards is 1000–1400 kg·m^{-3}; thus, according to this parameter the pellets of all tested samples met the standard and can be transported without the fear of crushing and dust formation, which is undesirable in the combustion of any biofuel.

Moisture content is among the dominant factors affecting pellet quality. The moisture content of pellets can be associated with safe storage and efficient combustion [8]. According to German standard DIN 51731, the moisture content of biofuel pellets must be lower than 12%, and according to European standard DIN EN 15270, lower than 10%.

The moisture content of MIX2-2 biomass pellets reached 8.78%, which was slightly higher than that of MIX-3 and S-FH (8.30 and 6.15%, respectively). The lowest moisture content was found in MIX2-1, at 3.86%. It can be stated that all investigated pellets met the requirements of moisture content for granulated biofuel (Figure 4).

According to other research, pellets from wood industry waste, agricultural and forestry residues, and energy crops have high density and very low humidity (<10%), thus ensuring high energy conversion efficiency (about 75%) [8].

Fusi et al., found that the moisture content of pellets produced from miscanthus biomass was 8.29%, and according to the data provided by Picchio et al., the moisture content of pellets from Pinus spp. was 7.30–9.60%, Picea spp. was 7.84% and Tsuga spp. was 5.79 [3,38]. Thus, the pellets of all seven combinations tested met the quality requirements in terms of moisture content, as did pellets of softwood biomass.

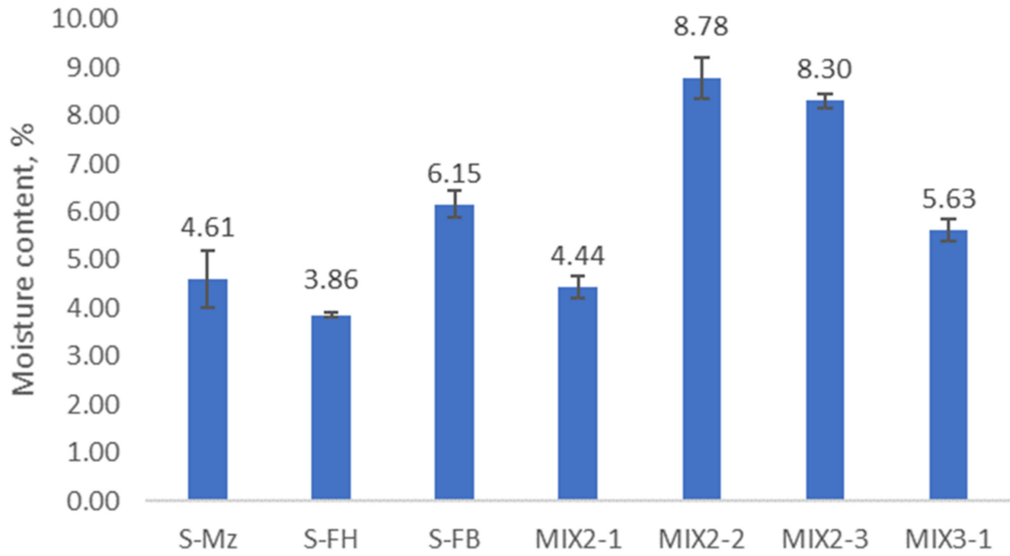

Figure 4. Moisture content of biomass pellets.

The research results regarding the calorific value of multi-crop biomass pellets are presented in Figure 5. These results show that the lower calorific value of dry fuel pellets varied from 16.80 ± 0.35 MJ·kg^{-1} (MIX-2) to 17.14 ± 0.57 MJ·kg^{-1} (S-FH).

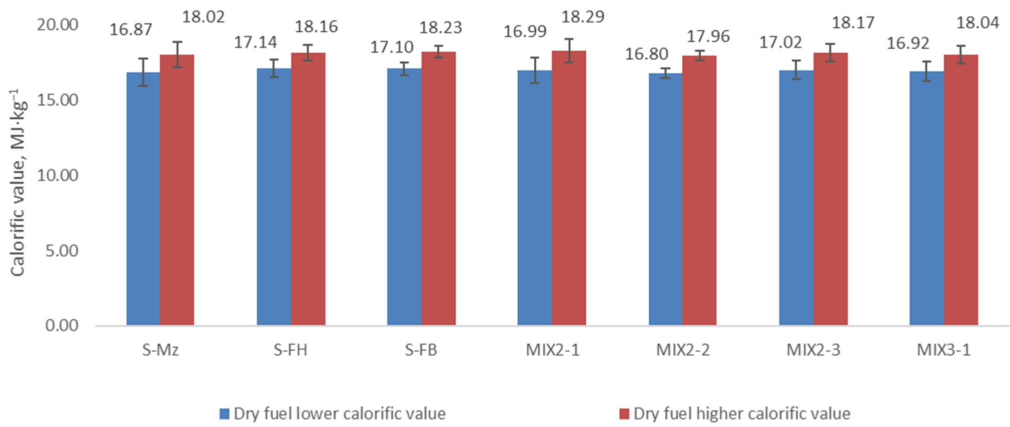

Figure 5. Calorific value of multi-crop biomass pellets.

As mentioned earlier, the moisture content of the granules was obtained, which differed slightly and varied from 3.86 to 8.78%, and did not exceed 10% (<12% moisture content of wood pellets is recommended according to standard DIN 51731) [42]. The moisture content of the granules differed because in the technological process of pellet production, the mill was irrigated with the required amount of water, and the moisture of the produced pellets differed slightly due to the operation of the working parts of the granulator and their heating, the uniformity of fineness and the feeding of mass, etc. All of these properties also slightly affected the properties of the granules, such as density and thermal properties, which were calculated as dry mass (DM) calorific value.

Based on the results of other studies on solid biofuel calorific value, Nunes et al., showed that the lower calorific value of wood pellets was sufficiently high and reached 20 MJ·kg^{-1}, but the lower calorific value of rice husk pellets was significantly lower at only 12–14 MJ·kg^{-1} [43].

According to Osman et al., the calorific value of a corn cob was 17.3 MJ·kg^{-1}, and that of corn stover was only 10.73 MJ·kg^{-1} [44]. According to Jasinskas et al., the lower calorific value of dry biofuel from faba bean waste pellets was similar in all samples and ranged from 16.9 to 17.1 MJ·kg^{-1}. The net calorific value of fibrous hemp varied from 17.37 to 17.45 MJ·kg^{-1} [24,39].

The lower calorific value of the seven investigated combinations of biomass pellets was close to that of some types of wood and willow pellets at 17.2 MJ·kg^{-1} [45].

3.2. Ash Content of Burned Biomass Pellets, Elemental Composition and Melting Temperatures

The combustion of biofuel pellets produces ash. It is important to quantify this ash, as it might be problematic to utilize large quantities of it. The results of burning the seven combinations of pellets show that ash content ranged from 4.49 to 6.78%. The lowest ash content (4.49%) was found in MIX2-1 biomass pellets (maize and fibrous hemp) and the highest ash content (6.78%) was found in MIX2-2 biomass pellets (fibrous hemp and faba bean) (Figure 6).

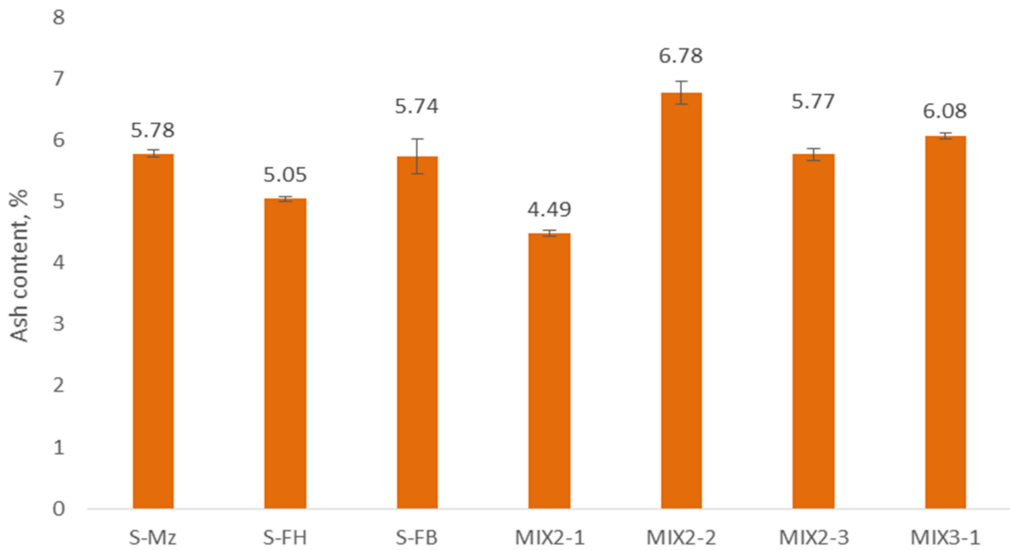

Figure 6. Ash content of burned multi-crop biomass pellets.

It has been noted that the permissible ash content after burning pellets of mixed biomass (i.e., non-wood biomass) is <7% [43]. That content was not exceeded when pellets were produced from all seven combinations of biomass.

Zając and other scientists found that the ash content in agricultural biomass (wheat straw, triticale straw, oat straw, barley straw, buckwheat straw and hay) ranged from 6.88 to 9.20% [46]. After burning energy crops such as Virginia mallow (*Sidahermaphorodita* R.), Jerusalem artichoke (*Helianthus tuberosus* L.), multiflorous rose (*Rosa polyantha*), Miscanthus giganteus (*Miscanthus sinensis gigantea*), Miscanthus sacchariflorus (*Miscanthus sacchariflorus*), prairie cordgrass (*Spartina pectinata*), common reed (*Phragmites australis*) and switchgrass (*Panicum virgatum*), the ash content ranged from 2.36 to 7.72%, while the ash content from traditional maize ranged from 4.7 to 8.4% [47].

Vassilev et al., summarized the data of ash content from different types of biomass: corn crop, 6.7%; corn (whole plant), 5.2%; corn straw, 10.0%; fibrous hemp, 3.8%; bean, 15.4%; and bean stalks, 5.4% [48].

The ash content of energy plant biomass pellets is higher than that of wood or woody biomass pellets. According to the data on wood pellets made from birch and pine sawdust from the wood processing industry presented by Manić et al., the ash content of pine pellets was 0.96%, and that of the two sawdust samples was 1.76 and 1.45%, respectively [49].

The melting characteristics of ash are important during the combustion of biofuel. They influence the chemical composition of the ash and the formation of slag on the surfaces of burning implements. When burning crop biomass pellets, their initial ash deformation temperature (DT) was determined to be 976 to 1322 °C. The highest temperature was recorded for hemp pellets (S-FH), indicating that the ash clogged the least on the surfaces of the incinerator. The ash melting temperatures of biomass pellets are presented in Table 4.

Table 4. Ash melting temperatures.

Type of Biofuel	Melting Temperature, °C			
	Ash Softening Temperature (ST)	Initial Ash Deformation Temperature (DT)	Ash Hemisphere Temperature (HT)	Ash Fusibility Temperature (FT)
S-Mz	940 ± 0.30	976 ± 0.29	1070 ± 0.20	1089 ± 1.69
S-FH	741 ± 0.57	1322 ± 1.07	1408 ± 0.40	1432 ± 0.20
S-FB	1062 ± 1.60	1141 ± 0.87	1163 ± 0.12	1178 ± 0.72
MIX2-1	1007 ± 0.63	1061 ± 0.93	1118 ± 0.76	1135 ± 0.37
MIX2-2	1029 ± 0.27	1051 ± 0.13	1137 ± 0.37	1177 ± 0.36
MIX2-3	935 ± 0.76	969 ± 0.73	1104 ± 0.26	1184 ± 0.96
MIX3-1	968 ± 0.58	1007 ± 1.12	1129 ± 0.88	1199 ± 0.83

The most important DTs of investigated biofuel pellets of S-FB, MIX2-1, MIX2-2 and MIX3-1 were close to the DTs of herbaceous plants (tall fescue, festulolium and timothy) and equalled 1020–1270 °C [50]. The DT of S-FH was only slightly lower than the DT of birch bark, which was 1350 °C [51].

Jasinskas et al., found that three varieties of fibrous hemp had an ST of 733–747 °C, a DT of 762–770 °C, an HT of 775–783 °C and an FT of 849–863 °C. By comparison, for all of our biomass samples, the melting point of the biomass ash of both fibrous hemp and mixtures with fibrous hemp was higher [24].

The chemical composition of ash from biomass is an important property that can be used to assess the movement of chemical elements during combustion and subsequent use. Returning the ash to the soil is a relatively ecological and sustainable way of using it, as it returns a significant proportion of the macro- and micronutrients absorbed by the plants and closes the mineral circulation [46].

Investigations of the elemental composition of the seven combinations of biomass ash showed that the predominant macronutrients were K, Ca, Mg and P, and the predominant microelements were Si, Fe, Zn and Cu (Table 5). The content of elements such as Cd, Cu, Pb and Zn did not exceed the highest permitted concentration in wood biomass ash used in agriculture or for the reclamation of damaged areas established by Lithuanian legislation.

Table 5. Chemical composition of multi-crop biomass ash.

Chemical Element	Types of Multi-Crops						
	S-Mz	S-FH	S-FB	MIX2-1	MIX2-2	MIX2-3	MIX3-1
	Chemical Composition of Multi-Crop Ash, mg kg^{-1}						
Cd	<0.51	<0.51	<0.51	<0.51	<0.51	<0.51	<0.51
Pb	<1.20	<1.20	<1.20	<1.20	2.68 ± 0.95	<1.20	<1.20
Cu	67.40 ± 3.21	85.06 ± 0.91	64.82 ± 3.45	80.63 ± 1.86	71.88 ± 3.74	63.17 ± 1.55	66.62 ± 7.44
Zn	522.00 ± 20.44	679.83 ± 17.49	154.64 ± 19.06	581.38 ± 3.74	721.02 ± 19.08	1339.21 ± 14.71	400.17 ± 40.65
Mg	31,638.10 ± 16.11	27,555.96 ± 10.77	21,018.50 ± 9.33	37,380.16 ± 24.94	23,870.85 ± 12.64	27,882.13 ± 6.70	27,466.08 ± 11.20
Fe	8848.28 ± 14.30	9029.58 ± 10.71	11,672.95 ± 10.39	8880.02 ± 16.22	21,934.71 ± 11.53	15,869.02 ± 8.71	7671.48 ± 12.28

Table 5. *Cont.*

Chemical Element	Types of Multi-Crops						
	S-Mz	S-FH	S-FB	MIX2-1	MIX2-2	MIX2-3	MIX3-1
	Chemical Composition of Multi-Crop Ash, mg kg^{-1}						
Ca	51,549.95 ± 18.09	156,890.20 ± 13.41	117,840.35 ± 10.63	96,122.58 ± 17.03	118,996.52 ± 12.50	89,728.14 ± 8.79	104,086.17 ± 14.26
K	265,865.78 ± 2.00	197,921.93 ± 10.99	85,964.19 ± 13.62	231,545.58 ± 7.87	123,579.76 ± 14.39	159,210.76 ± 13.98	144,021.32 ± 12.11
Si	95,668.66 ± 8.98	62,609.65 ± 11.40	248,960.29 ± 33.63	81,779.35 ± 25.03	216,689.14 ± 17.04	260,773.74 ± 9.22	228,605.42 ± 20.63
Al	1375.16 ± 8.24	1551.06 ± 2.68	6482.69 ± 7.98	1520.43 ± 7.34	4836.99 ± 8.60	4207.60 ± 6.26	4650.34 ± 7.22
Na	630.64 ± 5.73	2082.72 ± 15.08	1604.98 ± 6.94	1080.55 ± 26.39	9302.97 ± 9.58	8454.99 ± 2.20	5878.78 ± 16.30
P	28,847.45 ± 18.81	30,359.01 ± 13.92	16,922.97 ± 11.41	30,771.96 ± 33.61	22,088.97 ± 15.92	26,305.29 ± 23.73	24,665.83 ± 16.71
	Chemical Composition of Multi-Crop Ash, %						
Cl	0.27 ± 0.05	1.07 ± 0.21	0.40 ± 0.11	2.41 ± 0.47	0.70 ± 0.12	2.47 ± 0.74	1.76 ± 0.27
S	0.08 ± 0.02	0.62 ± 0.11	0.55 ± 0.11	0.67 ± 0.12	0.49 ± 0.08	0.87 ± 0.18	0.63 ± 0.13

Lithuanian legislation does not define the use of wood biomass ash in agriculture; however, the maximum levels in mg/kg of dry weight that are regulated for the handling and use of wood fuel ash are: Cd, 5 mg/kg; Cu, 200 mg/kg; Pb, 50 mg/kg; and Zn, 1500 mg/kg (Table 6).

Table 6. Maximum concentrations of chemicals in ash used in forestry and agriculture for remediation of damaged areas.

Chemical Element	In Forestry	In Agriculture/Remediation of Damaged Areas
	Concentration, mg kg^{-1} in Dry Matter	
Boron (B)	200	250
Vanadium (V)	150	150
Nickel (Ni)	20	30
Chrome (Cr)	20	30
Cadmium (Cd)	3	5
Lead (Pb)	40	50
Copper (Cu)	100	200
Zinc (Zn)	1000	1500
Arsenic (As)	3	3
Mercury (Hg)	0.2	0.2
Benz(a)pyrene, µg·kg^{-1}	0.5	0.5

The results of other studies are similar. Zając et al., found that, in agricultural biomass and energy crops, there was mostly calcium–potassium–phosphorus (Ca–P–K) ash or potassium–calcium–phosphorus (K–Ca–P) ashes. The content of toxic elements (As and Pb) was the lowest, and these elements were included at the end of the series [46].

Studies by Vassilev et al., show that the group of wood biomass is mostly enriched in Ca, Mg, Mn and S, while the group of herbaceous and other agricultural crops is generally rich in ash, Cl, K, Na, P and Si. It has also been found that fast-growing biomass and agricultural and herbaceous waste contain more Cl, K, Na, S and Si than wood [52].

Previous research has shown that biomass ash can replace mineral fertilizers. Wierzbowska et al., found that willow ash can be used instead of phosphorus, potassium and magnesium fertilizers for the cultivation of willows for energy. The uptake of P, K and Mg from ash did not differ from the uptake of plants supplied with mineral salts. Using this alkaline ash, the soil was enriched with trace elements such as Zn, Cu and Mn, indicating that the ash can be successfully used in agriculture as a fertilizer [53].

According to a study by Cruz-Paredes et al., the use of biomass ash as a fertilizer is a sustainable solution to maintain the available P content in the soil. The use of ash in the soil increased the P content for two seasons, similar to the use of triple superphosphate (TSP) fertilizer; the plants did not accumulate the cadmium in the ash, and the plant yield did not decrease [54].

Vassilev et al., noted that the main disadvantage of using biomass as a solid biofuel besides the high ash content is the high content of alkali metals such as K and Cl. These metals can cause ash deposition, sintering, contamination, dissolution, slag formation and corrosion, as well as dust and metal emissions and other problems [52].

The examination of all seven combinations of biomass ash revealed that their Cl content ranged from 0.27% (S-Mz) to 2.47% (MIX2-3). Vassilev et al., provided generalized research data showing the following Cl levels in different types of biomass ash: hemp, 1.28%; corn straw, 5.40%; corn cobs, 1.24%; and corn stoves, 6.58% [48].

Some countries, such as Germany, Austria, Denmark, Sweden and Finland, have developed national legislation allowing the use of biomass ash in soil. These guidelines set maximum levels for potentially toxic elements and minimum levels for nutrients (Ca, K and P) [30].

In order to use biomass ash for plant fertilization, many issues need to be addressed in order to ensure that it contains the right amount of nutrients, does not exceed the allowed content of heavy metals, is easy to transport, and that its effects on soil and plants are optimal. The scientific literature indicates that the use of ash as a fertilizer requires controlling the rate of nutrient release to prevent plants from experiencing stress after receiving large amounts of nutrients. Therefore, it is proposed to granulate the ash, thereby slowing down the release of nutrients and preventing the formation of dust [55]. However, due to the high diversity of biomass, no generalized conclusions can be made. Further studies are needed to determine the effects of ash resulting from the combustion of concrete multi-crop biomass on soil and plants.

4. Conclusions

The need to find new biofuel feedstocks is driving more research to support the use of different types of biomass for biofuel. Although many studies have been carried out to substantiate the suitability of herbaceous plants and agricultural waste for biofuel, possibilities for preparing multi-crop plants for biofuel have not yet been analysed. The suitability of multi-crop plants and their combinations for solid biofuel is analysed in this work. In the experimental studies, most of the dry biomass was obtained from a trinomial crop (MIX3-1), so it is worth noting the possibility of growing crops of three different plants.

Seven types of solid biofuel pellets were produced from hemp, maize and seed beans and biomass mixtures from these plants. The biometric and physical–mechanical properties of all mixtures met the requirements of pellet quality standards according to DIN 51731. The highest density of pellets was found in S-FB and S-FH samples (1195.75 $\pm$ 47.05 and 1195.42 $\pm$ 34.16 kg$\cdot$m^{-3} dry matter (DM), respectively). The moisture content of the produced pellets ranged from 3.86 to 8.78%, the ash content from 4.49 to 6.78% and the calorific value from 16.80 to 17.14 MJ$\cdot$kg^{-1}. The analysis of the elemental composition and other properties indicates that these pellets can be used for solid biofuel; compared with monocultures, the elemental composition was found to be 1.4 to 2.2 times lower in Cl. The content of Cl in MIX2-1, MIX2-2, MIX2-3 and MIX3-1 pellets met or only slightly exceeded

(10–20%) the requirements of the standards. When using pellets from monoculture crops with a higher Cl content, the use of plants suitable for combustion should be considered.

Although the ash content of all multi-crop pellets met the requirements of the standards, it was higher than that of wood or woody plant pellets. The elemental composition of this ash suggests that the use of ash from this biofuel as a fertilizer should be considered in order to return nutrients to the soil. High concentrations of potassium (K), calcium (Ca) and phosphorus (P) were detected in all types of biomass ash. When evaluating the content of heavy metals (copper (Cu), zinc (Zn), tin (Al) and cadmium (Cd)) in pellet ash, it was found that their quantities were not large and did not exceed the permissible values.

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