

Article

Modification of Energy Parameters in Wood Pellets with the Use of Waste Cooking Oil

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Abstract: Biomass is one of the most important sources of renewable energy. It is expected that in the coming decades, biomass will play a major role in replacing fossil fuels. The most commonly used biofuels include wood pellet, which is a cost-effective, uniform and easy-to-use material. In view of the growing interest in this type of resource, novel methods are being investigated to improve the quality of pellet. This article presents the results of a laboratory study focusing on wood pellets refined with waste sunflower cooking oil applied by spraying. In this work, authors attempted to modify the energy parameters of wood pellets with the use of waste cooking oil. Addition of waste cooking oil, applied at the rates of 2%, 4%, 6%, 8%, 10% and 12% relative to the weight of pellets, increased the calorific value of the pellets without decreasing their durability. The highest dose of the modifier (12%) on average led to a 12–16% increase in calorific value. In each case, the addition of sunflower oil resulted in decreased contents of ash in the pellets; on average a decrease of 16–38% was observed in the samples treated with the highest dose of the modifier. The treatment led to a higher content of elements affecting the heating value, i.e., carbon and hydrogen, which on average increased by 7.5–12%, and 7.0–10.0%, respectively. The presented method seems to be a promising way of increasing the calorific value of pellets. Further research on refining the method and the possibility of using it in industry is necessary.

Keywords: wood pellets; calorific value; carbon; ash; volatile substances; waste cooking oil



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1. Introduction

Changing climate conditions and increasing emissions of greenhouse gases, as well as the excessive exhaustion of non-renewable resources resulting from their unsustainable exploitation, have led to the urgent need for transformation in the energy sector. Programs which have recently been launched worldwide and in Poland provide support for operations aiming to protect the climate and to promote a sustainable, low-emissions economy. Renewable energy sources (RES) are of key importance from this viewpoint [1]. The most important sources of renewable energy include biomass. In Poland, nearly 70% of the renewable energy produced in 2018 was obtained from solid biofuels (Central Statistical Office). Biomass is recognised as a clean source of energy, with a total globally installed power estimated at a level of 87 GW [2]. It is expected that during the coming decades, biomass will play a crucial role in efforts to replace fossil fuels in global energy systems and to reduce greenhouse gas (GHG) emissions [3,4]. Experts estimate that by 2035, biomass may provide 120 exajoules (EJ) (50% of the global demand for energy) for production of heat, 15% EJ for transport and 18 EJ (7%) for production of electricity [5,6]. Biomass can be defined as all biodegradable organic material derived from animals, plants or microorganisms. Biomass resources include natural materials and their derivatives. These are plants grown for energy purposes, i.e., Giant miscanthus, Basket willow and Virginia mallow. Biomass includes agricultural raw materials and their residues (e.g., straw, cereal grains), wood and its residues, forest and mill residues, municipal waste and sewage, as well as

animal manure and residues. Popular as biomass are banana peels, rice and coffee arches, sugar cane pomace as well as palm oil processing residues and animal faeces [7].

Bioenergy (energy from biomass) is predominantly obtained from plants. The calorific value of pellets made of wood biomass and straw is within the limits of 16.5–20.5 MJ kg⁻¹; the calorific value of pellets from sewage sludge is lower and amounts to approximately 11.37 MJ kg⁻¹ [8]. Bio-oil is obtained from biomass in the pyrolysis process; the calorific value of bio-oil is 39–41 MJ kg⁻¹ [9]. Improved bio-oil can be used as transport fuel, in turbines and engines generating electricity, or for heat-generating boilers [10].

Wood pellet is a type of solid fuel produced for energy-related purposes. This uniform, easy-to-use solid biofuel is obtained through a densification process known as pelletisation [11–13], which involves pressing of the milled wood. Pelletisation is an extrusion process in which refined dry biomass is subjected to high pressure and high temperature, and pressed through an opening a few metres long, whereby small cylinders are produced; these are finally cut to the required length and cooled down [14,15]. Wood pellet comes in the form of granules with a diameter of 6–8 mm and length of 35–40 mm. Compared to wood waste, pellets have a higher density of energy per unit of weight and volume. Pellets are easier to store and handle as they are more uniform in physical and chemical terms [16,17]. Pellets vary in terms of quality. Calorific value is the most important quality of wood pellet; a higher calorific value corresponds to a better energy efficiency for the same quantity of the product, and consequently to a lower cost for the consumer [18]. The calorific value of pellet depends on the duration of the drying process applied to the biomass (responsible for the moisture level), and is related to the compactness of the granulate, and—most importantly—to the origin and type of the raw material [19–21]. The calorific value (KJ kg⁻¹) is related to the concentrations of carbon, hydrogen, oxygen, sulphur, nitrogen and ash in a sample [18]. Carbon and hydrogen are the essential elements contained in wood. These elements are the primary reagents in the heating process—they determine the calorific value of fuels and are a source of heat in combustion processes [22]. Ash content reflects percent weight of ash contained in the pellet, relative to the weight of the fuel [23]. Ash content, ash melting point, and elemental composition are important properties of biofuel. As reported by Obernberger et al., 2010 in his textbook, these parameters may adversely affect the combustion and conversion processes [20]. Obernberger et al., 2010 and Trasov et al., 2013, report that high ash content will decrease the stove efficiency, and make the stove require cleaning more often [20,23]. In accordance with EU standards, ash content in premium class wood pellets should be less than 0.7%, whereas according to the Pellet Fuels Institute (PFI) the value of this parameter should be lower than 1% [3,20]. According to the EU standard, the ash melting point has to be higher than 1200 °C. High concentrations of chlorine, potassium and sodium also enhance the likelihood of corrosion of the inner components of the stove or boiler system [23]. Furthermore, lower ash content results in a less frequent need to remove ash, which is important for the automatic operation of biomass boilers and stoves [3]. Nitrogen, like sulphur, is an inorganic element which is involved in formation of ash; additionally, emissions of NO_x gases occur during combustion [24]. An important parameter characterizing pellets are volatile parts; volatile parts are formed as a result of the thermal decomposition of organic compounds in a combustible mass. Such gases include hydrogen, hydrocarbons, carbon oxides and carbon dioxide. Volatile contents determine the process of fuel combustion, including its ignition. Fuels with low volatile contents are more difficult to get ignited. [25,26]. A very important quality feature of pellets is their resistance to shocks and abrasion during treatment processes and in transport [27]. It is assumed that the durability of pellets is high if the related value exceeds 80%, moderate if the value is in the range between 70% and 80%, and low if the value is less than 70%. [28]. Pellets with a durability exceeding 97.5%, measured using a defined tumble testing method, are recognised among high quality biofuels [29,30].

Many studies by scientists prove that lignin is the binding agent in wood pellets. The literature reports that its higher content corresponds to the better quality of the pellet produced. Experts in many studies have proved that coniferous trees tend to have a

higher content of lignin than most deciduous trees. The highest contents of lignin are found in pine, fir and beech wood. Pellets are predominantly produced from coniferous wood waste. Scientists report that deciduous wood wastes, because of the hardness of such materials, are generally less suitable for production of pellets [31,32]. Nevertheless, experts and manufacturers have presented evidence that if adequate conditions are applied in the pelletisation process, it is possible to produce granulate from deciduous wood wastes [33]. Recent literature reports that today in the European Union, wood pellet is widely used as heating fuel in detached houses, where it competes with oil and electric heating systems. Pellet combustion is also a realistic alternative for heating plants and even power stations [13,34,35]. According to Sarcaglu & Gunduz (2009), wood pellets are tomorrow's fuel for Europe [36].

Currently, it is estimated that the energy produced from wood pellets meets 14% of the global demand. It is also estimated that starting from 2020, demand for pellet will amount to approximately 50 million tonnes per year [37], and consumption of industrial pellet will systematically grow at a rate of 21% per year [13]. In the European Union, the year 2017 saw a considerable increase in production and use of wood pellets, amounting to over 10 million tonnes. Europe is the main consumer and producer of wood pellets, and is followed in this respect by the USA, and then the rest of the world. Europe is also a global net importer of wood pellets [38,39]. The United Kingdom, Finland and Sweden are known for the highest consumption of wood pellets; the material is mainly used there in production of electricity and heating. Other important consumers of pellets in Europe include Belgium, the Netherlands, Denmark and Italy [40]. Producers, and simultaneously the largest exporters of wood pellets, include Portugal and Latvia, followed by Germany, Lithuania, Estonia, Finland and Sweden [41,42]. The main producers of pellets in the EU include Germany, Sweden, Latvia and Austria [43]. The use of pellets in Europe mainly depends on governmental regulations. The bioenergy policies implemented by the specific EU member states stipulate tax exemptions, targets to be achieved and subsidies, as well as biomass sustainability policies, which stimulate growth of production and increased use of wood pellets [34]. In the Netherlands, UK and Belgium, pellets are widely used in major power plants. In Scandinavia, wood pellet is used in medium-size heating plants and in private houses. In Germany, France, Italy and Austria, wood pellets are mainly used to provide heating in small farms and in industry. The European market for wood pellets used in small heating systems has significantly grown over the last decade [35].

In Poland, currently less than one million tonnes of this type of material are used. The pellet sector in Poland slightly differs from those in other EU countries, in terms of both production and use of this ecological fuel. In Poland, pellet is produced from biomass, which is acquired from local sources. In fact, there is a constantly increasing interest in pellets in the country [44].

Because of the increasing popularity of pellets, researchers look for ways to enhance their quality, i.e., heating value. The most frequently investigated are thermochemical and biochemical methods, i.e., torrefaction, pyrolysis and gasification. Various additives, such as glycerine and waste cooking oil, may be applied to improve the heating value. These substances may be applied by spraying or soaking the pellets; they may also be added at the stage of pellet formation [44].

In this work, we attempted to modify the energy parameters of wood pellets with the use of waste cooking oil. This research shows that waste cooking oil introduced into wood pellets by the spraying method caused an increase in the calorific value of the analysed pellets. At the same time, the oil did not reduce the strength of the pellets, which is very important. More research is needed to fine-tune the optimal oil dosage and to fine-tune the technique in order to use this method in industry. Waste cooking oils (WCO) are classified among used vegetable oils (UVO), which, according to the Waste Catalogue Regulation of the Minister of Environment dated 27 September 2001, constitute waste hazardous for the environment [45,46]. In line with Art. 39 of the Waste Act dated 27 April 2001, waste oils should in the first place be recovered through regeneration. If this is

impossible, such oils should be subjected to combustion with energy recovery [47]. In terms of chemical composition, WCOs are quite similar to the original cooking oils; they differ in the products of decomposition and in leaching [48]. Changes in the chemical composition of oil during the frying process may be associated with contamination and decomposition of food products and kitchen tools. Nevertheless, the relative amounts of contamination produced during the cooking process are not increased. Research has shown that WCOs are very similar in chemical composition to the original cooking oils [49]. In recent years, increased interest in the processing of WCOs can be observed. WCOs are geographically dispersed and produced in large numbers. The total annual production of used vegetable oils exceeds 190 million metric tons, including the production of the European Union, which is about 1 million tons/year. In Europe, WCOs are collected by the urban waste system and delivered to an authorized recycling point. WCOs are considered an economic and green raw material. The possibility of using waste vegetable oil in a valuable way, combined with the advantage of avoiding its dispersion into the environment and its very high availability, has encouraged scientists to look for a way to reuse waste. The most common uses of WCOs are the production of bio-lubricants or fuel as animal feed, or as additives for asphalts and for energy production [45].

This study aimed to assess the effects of used sunflower oil applied to selected types of wood pellets on the physicochemical parameters of the materials. The present study is the first to discuss findings of experiments in which various doses of used sunflower oil were applied to wood pellets by spraying. If positive results of such treatment are identified, it may be beneficial in two ways, since this would be an efficient method for both enhancing the value of wood pellets, and for the recycling of WCOs.

2. Materials and Methods

2.1. Research Material

The samples designated for this study were processed to achieve an air-dry state and then were refined by grinding. The research material comprised various types of pellets obtained from various producers operating in different regions of Poland:

- Beech wood pellet: Pomorskie Region
- Oak wood pellet: Wielkopolskie Region
- Oak wood pellet: Podkarpackie Region
- Coniferous wood pellet: Świętokrzyskie Region
- Coniferous wood pellet: Podkarpackie Region
- Coniferous and deciduous wood pellet (70% coniferous sawdust, 30% deciduous sawdust): Podkarpackie Region

Figure 1 shows the research material.



Figure 1. Research materials.

The wood pellets were treated with waste sunflower cooking oil acquired from a restaurant located in Rzeszów, Podkarpackie Region, Poland.

2.2. Sample Collection and Analysis

The research material was delivered to the laboratory of the University of Rzeszów in commercially available 15 kg packaging. The pellets had a diameter of 6 mm and a length of 35–40 mm.

Each batch of the pellets (of different type and origin) was divided into seven samples and placed into separate containers. Subsequently, waste sunflower cooking oil was added, by spraying, at the rates of 2%, 4%, 6%, 8%, 10% and 12% relative to the weight of the pellets. The control sample comprised pellets without a modifier added. The samples prepared this way were left for 24 h so that the oil could be absorbed by the pellets. Finally, the samples were put through a sieve with 1 mm openings, in order to remove any potential contamination and excess oil. Figure 2 shows the scheme of the experiment.

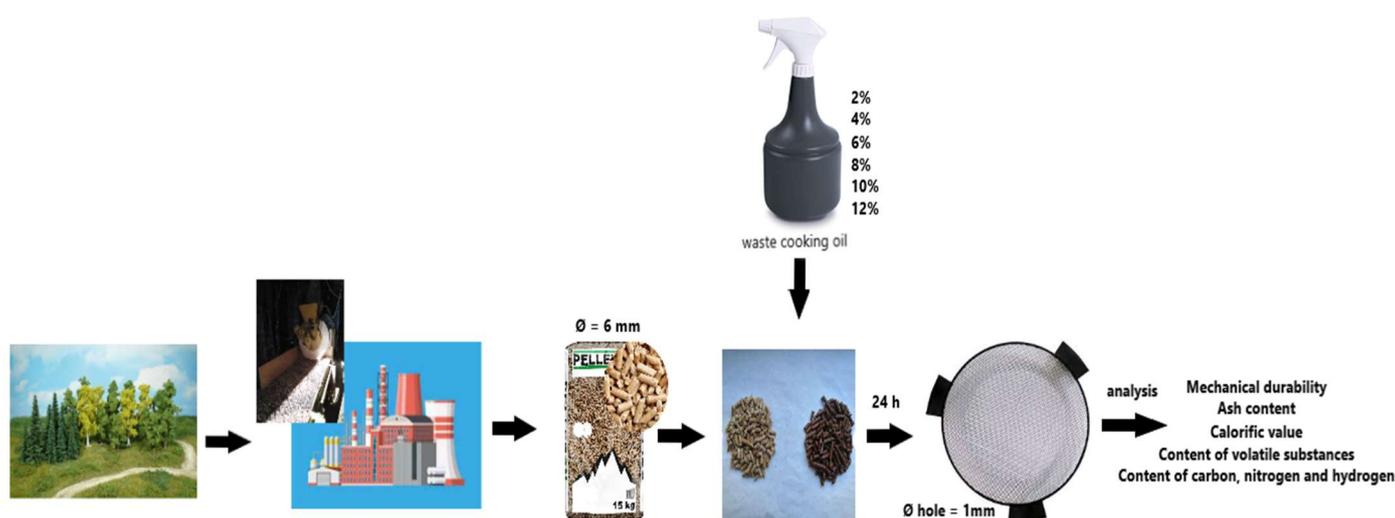


Figure 2. The scheme of the experiment.

2.3. Samples Analysis

Assessments were carried out to identify the effects of waste vegetable oil on the stability and physicochemical properties of wood pellets of various origins. Samples were subjected to laboratory analyses using current analytical standards (Table 1).

Table 1. Parameters analysed with research methods.

Item	Parameter	Testing Method
1.	Mechanical durability	PN-EN 17831-1:20169-02 [50]
2.	Content of carbon, nitrogen and hydrogen	PN-EN 15104:2011 [51]
3.	Ash content	PN-EN 14775:2010 [52]
4.	Content of volatile substances	PN-EN 15148:2011 [53]
5.	Calorific value	PN-EN 14918:2010 [54]

Mechanical durability was tested using a Tumbler 1000 apparatus. The part subjected to the test, (mE) $500 \pm 0.1\text{g}$, was placed in the drum and them tumbled for 10 min with a speed from 500 rotations/min to 0 rotations/min. Subsequently, the sample was manually put through a sieve with 3.15 mm openings, in accordance with BS ISO 3310-2:2013 [55]. Subsequently, the sample left on the sieve was weighed (mA). The test was performed in

three replications for each variant. The mechanical durability of pellets (DU) was calculated following the formula:

$$DU = mA/mE \times 100$$

where:

Du—mechanical durability of pellets

mA—pellet weight following the test (g)

mE—pellet weight before the test (g).

Analyses of the contents of ash and volatile substances in the samples were performed using a thermogravimetric method, with a TGA 701 apparatus from LECO (LECO Corporation, Saint Joseph, MI, USA). The contents of total carbon and nitrogen were tested using a TrueSpec CHN analyser from LECO (LECO Corporation, Saint Joseph, MI, USA). An AC500 calorimeter from LECO (LECO Corporation, Saint Joseph, MI, USA) was used to determine the calorific value of the materials analysed.

2.4. Names of Tests

For further identification, the samples of biomass are described using letters of the alphabet, to distinguish the types and origins of the materials, and the rate of modifiers.

A—Beech wood pellet from the Pomorskie Region

B—Oak wood pellet from the Wielkopolskie Region

C—Oak wood pellet from the Podkarpackie Region

D—Coniferous wood pellet from the Świętokrzyskie Region

E—Coniferous wood pellet from the Podkarpackie Region

F—Coniferous/deciduous wood pellet from the Podkarpackie Region

0—No modifier

1—Modifier added at a rate of 2% relative to the weight of pellets

2—Modifier added at a rate of 4% relative to the weight of pellets

3—Modifier added at a rate of 6% relative to the weight of pellets

4—Modifier added at a rate of 8% relative to the weight of pellets

5—Modifier added at a rate of 10% relative to the weight of pellets

6—Modifier added at a rate of 12% relative to the weight of pellets

2.5. Statistical Analysis

The effects of the experimental factors reflected by the relevant parameters, and the relationships between these, were examined using Analysis of Variance (ANOVA) by means of the Duncan test. Statistica 12 software was applied to compute the statistical analyses. A significance threshold of ≤ 0.05 was set for all analyses. The data were analysed separately for each type of pellet.

3. Results and Discussion

In order to assess the effects produced by waste cooking oil applied by spraying to wood pellets, first it was necessary to perform tests to determine whether the modifier affected the durability of the granulate. For this purpose, a mechanical durability test was carried out. The results are shown in Table 2.

The results of durability tests related to pellets of one type are very similar. The findings show that addition of used vegetable oil to pellets, applied by spraying, does not significantly affect the durability of pellets. Oil applied at weight rates of 8%, 10% and 12% increased the durability of the pellets; however, these results are not statistically significant. The highest durability was found in the pellets made from coniferous wood chips, i.e., 98.34% and 98.20% in the control sample and 98.91% and 98.82% following treatment with the highest dose of used oil. The lowest durability was identified in coniferous–deciduous pellet; 95.28% in the control sample, and 95.80% in the pellets with the highest addition of the modifier. At present, the literature does not report on the modification of pellets with waste cooking oil using the spraying method. The literature reports on the method of refining pellets by immersing them in oil; this method was used Craven et al. (2015). The

researchers applied hydrophobic coatings to wood pellets by submerging them in various types of oil for up to 1800 s, to assess the effects of such treatment on the stability of the pellets. They found that the strength of the granules processed after they were submerged in water for 60 s was not significantly decreased [56]. The literature reports on attempts to add used cooking oil to wood biomass during the production of pellets. The findings show that addition of vegetable oil during the pelletisation procedure led to decreased sawdust compressibility and the reduced strength of the finished pellets. The authors suggested that the oil reduced the force of friction in the pellet–die wall contact area, which resulted in decreased durability of the pellets [57]. These results show the superiority of the method presented by the authors over the WCO addition method during pelletisation. Many researchers have investigated the effects of various substances on the durability of pellets. It has been shown that starch and carbohydrates soluble in water improve the durability of biomass pellets [58]. Ahn et al. (2014) found that rapeseed flour and coffee meal, as well as lignin, considerably improved the durability of larch and tulipwood pellets [59]. Another team showed that a 2% addition of potato starch significantly improves the hardness of pellets made from sawdust and miscanthus [60]. The related literature also discusses other additives, unrelated to food products. It was shown that 2% addition of sulfonated lignin and lignosulfonate, by-products in paper industry, increases the durability of granulate from European red pine by 0.8% [61] and European spruce by 1.4% [62]. Addition of glycerol, aluminium silicate, as well as lignin (at weight rates of 5%:2%:2%) to pellets made from wheat straw increases the durability of the final material [63]. Addition of glycerol alone to straw from wheat, barley, oat and rapeseed did not produce visible changes in the durability of pellets [64]. Increased durability of pellets was also produced by the addition of algae [65]. Effects of waste cooking oil addition on the quality of wood pellets produced were assessed by performing a number of physicochemical analyses; their results are shown in Table 3.

Table 2. Durability of pellets—unprocessed and treated with waste cooking oil, added by spraying.

Sample	DU [%]	Sample	DU [%]
A0	95.30 ^a ± 0.36	D0	98.34 ^a ± 0.35
A1	95.38 ^a ± 0.37	D1	98.47 ^a ± 0.26
A2	95.42 ^a ± 0.28	D2	98.33 ^a ± 0.24
A3	95.35 ^a ± 0.19	D3	98.48 ^a ± 0.28
A4	95.65 ^a ± 0.23	D4	98.97 ^a ± 0.36
A5	96.17 ^a ± 0.21	D5	99.03 ^a ± 0.41
A6	96.10 ^a ± 0.33	D6	98.91 ^a ± 0.31
B0	96.58 ^a ± 0.29	E0	98.20 ^a ± 0.28
B1	96.74 ^a ± 0.37	E1	98.17 ^a ± 0.19
B2	96.68 ^a ± 0.34	E2	98.28 ^a ± 0.37
B3	96.70 ^a ± 0.37	E3	98.32 ^a ± 0.34
B4	97.04 ^a ± 0.24	E4	98.83 ^a ± 0.16
B5	97.00 ^a ± 0.12	E5	98.75 ^a ± 0.29
B6	96.98 ^a ± 0.19	E6	98.82 ^a ± 0.43
C0	97.87 ^a ± 0.37	F0	95.28 ^a ± 0.26
C1	97.85 ^a ± 0.28	F1	95.36 ^a ± 0.32
C2	97.89 ^a ± 0.27	F2	95.27 ^a ± 0.41
C3	97.96 ^a ± 0.42	F3	95.36 ^a ± 0.29
C4	98.22 ^a ± 0.31	F4	95.70 ^a ± 0.39
C5	98.20 ^a ± 0.26	F5	95.82 ^a ± 0.33
C6	98.25 ^a ± 0.17	F6	95.80 ^a ± 0.29

Statistically significant differences marked by different letters ($p \leq 0.05$). Differences between average values marked with the same letters are not statistically significant at the level of $p \leq 0.05$ according to the Duncan test. The data were analysed separately for each type of pellet.

Table 3. Contents of total nitrogen, total carbon, hydrogen, ash, and volatile substances in selected pellets, before and after waste cooking oil was added by spraying.

Sample	C	H	N	Ash	Volatile Substances
[%]					
A0	50.35 ^a ± 0.08	5.97 ^a ± 0.01		3.16 ^c ± 0.09	20.43 ^b ± 0.09
A1	52.58 ^{a,b} ± 0.14	6.15 ^a ± 0.01		2.78 ^b ± 0.04	20.24 ^b ± 0.10
A2	53.74 ^{a,b} ± 0.12	6.22 ^a ± 0.00		2.66 ^b ± 0.05	19.74 ^{a,b} ± 0.04
A3	54.25 ^{a,b} ± 0.09	6.37 ^a ± 0.01		2.46 ^{ab} ± 0.06	19.19 ^{a,b} ± 0.08
A4	54.51 ^{a,b} ± 0.20	6.44 ^a ± 0.00		2.46 ^{ab} ± 0.06	19.03 ^{a,b} ± 0.11
A5	55.45 ^b ± 0.20	6.51 ^a ± 0.00		2.43 ^{ab} ± 0.04	18.49 ^{a,b} ± 0.06
A6	56.59 ^b ± 0.17	6.56 ^a ± 0.02		2.37 ^a ± 0.03	18.10 ^a ± 0.13
B0	50.73 ^a ± 0.21	6.22 ^a ± 0.00		0.36 ^d ± 0.01	17.77 ^a ± 0.05
B1	53.66 ^a ± 0.11	6.31 ^a ± 0.01		0.35 ^d ± 0.01	17.74 ^a ± 0.09
B2	54.75 ^a ± 0.08	6.37 ^a ± 0.01		0.33 ^d ± 0.005	17.45 ^a ± 0.07
B3	55.17 ^a ± 0.09	6.45 ^a ± 0.01		0.31 ^c ± 0.005	17.06 ^a ± 0.12
B4	55.42 ^a ± 0.12	6.52 ^a ± 0.01		0.29 ^{b,c} ± 0.01	16.55 ^a ± 0.12
B5	55.73 ^a ± 0.06	6.63 ^a ± 0.00		0.28 ^b ± 0.005	16.27 ^a ± 0.08
B6	55.89 ^a ± 0.08	6.72 ^a ± 0.01		0.25 ^a ± 0.01	16.04 ^a ± 0.10
C0	51.36 ^a ± 0.12	5.98 ^a ± 0.00		0.47 ^e ± 0.02	18.18 ^a ± 0.09
C1	52.20 ^a ± 0.07	6.24 ^a ± 0.01		0.41 ^d ± 0.01	18.07 ^a ± 0.11
C2	52.34 ^a ± 0.07	6.28 ^a ± 0.01		0.37 ^c ± 0.01	17.99 ^a ± 0.09
C3	53.16 ^a ± 0.10	6.33 ^a ± 0.00		0.33 ^b ± 0.01	17.84 ^a ± 0.11
C4	53.32 ^a ± 0.13	6.38 ^a ± 0.01		0.30 ^a ± 0.01	17.74 ^a ± 0.12
C5	53.51 ^a ± 0.06	6.40 ^a ± 0.01		0.29 ^a ± 0.02	17.51 ^a ± 0.11
C6	55.28 ^a ± 0.13	6.51 ^a ± 0.01	<0.04	0.29 ^a ± 0.02	17.14 ^a ± 0.09
D0	50.52 ^a ± 0.12	6.17 ^a ± 0.01		1.35 ^b ± 0.06	17.82 ^a ± 0.11
D1	52.36 ^{a,b} ± 0.22	6.25 ^a ± 0.01		1.26 ^{a,b} ± 0.03	17.58 ^a ± 0.09
D2	55.30 ^{a,b} ± 0.12	6.33 ^a ± 0.01		1.26 ^{a,b} ± 0.04	17.36 ^a ± 0.04
D3	56.29 ^{a,b} ± 0.56	6.44 ^a ± 0.01		1.25 ^{a,b} ± 0.06	17.19 ^a ± 0.04
D4	56.23 ^b ± 0.05	6.53 ^a ± 0.01		1.21 ^{a,b} ± 0.03	16.96 ^a ± 0.08
D5	56.52 ^b ± 0.08	6.66 ^a ± 0.01		1.17 ^a ± 0.01	16.67 ^a ± 0.11
D6	56.71 ^b ± 0.09	6.77 ^a ± 0.01		1.14 ^a ± 0.10	16.37 ^a ± 0.10
E0	53.14 ^a ± 0.16	6.21 ^a ± 0.01		0.32 ^c ± 0.03	17.24 ^b ± 0.13
E1	53.82 ^a ± 0.17	6.27 ^a ± 0.01		0.31 ^c ± 0.01	16.83 ^{a,b} ± 0.10
E2	55.12 ^a ± 0.11	6.32 ^a ± 0.01		0.30 ^{b,c} ± 0.02	16.53 ^{a,b} ± 0.08
E3	55.37 ^a ± 0.23	6.36 ^a ± 0.01		0.28 ^b ± 0.01	16.46 ^{a,b} ± 0.10
E4	55.64 ^a ± 0.12	6.68 ^a ± 0.01		0.28 ^b ± 0.01	15.80 ^{a,b} ± 0.16
E5	56.22 ^a ± 0.11	6.78 ^a ± 0.01		0.26 ^{a,b} ± 0.02	15.78 ^{a,b} ± 0.08
E6	58.26 ^a ± 0.14	6.82 ^a ± 0.01		0.24 ^a ± 0.02	15.64 ^a ± 0.09
F0	52.77 ^a ± 0.14	6.15 ^a ± 0.01		0.29 ^d ± 0.01	17.42 ^a ± 0.18
F1	53.10 ^a ± 0.12	6.32 ^a ± 0.01		0.27 ^c ± 0.01	17.35 ^a ± 0.07
F2	53.47 ^a ± 0.09	6.38 ^a ± 0.00		0.27 ^c ± 0.01	17.18 ^a ± 0.08
F3	54.59 ^a ± 0.14	6.46 ^a ± 0.01		0.25 ^{b,c} ± 0.01	16.73 ^a ± 0.12
F4	55.16 ^a ± 0.14	6.56 ^a ± 0.02		0.25 ^b ± 0.01	16.55 ^a ± 0.08
F5	56.81 ^a ± 0.11	6.57 ^a ± 0.00		0.22 ^a ± 0.01	16.33 ^a ± 0.06
F6	57.76 ^a ± 0.22	6.57 ^a ± 0.01		0.21 ^a ± 0.01	16.17 ^a ± 0.08

Statistically significant differences marked by different letters ($p \leq 0.05$). Differences between average values marked with the same letters (a–e) are not statistically significant at the level of $p \leq 0.05$ according to the Duncan test. Detection limit of nitrogen < 0.04. The data were analysed separately for each type of pellet.

The analyses show that in each case, the addition of waste oil as a modifier led to a decrease in ash content in the pellets. In all the types of pellets the same relationship was observed: higher addition of waste cooking oil corresponded to a greater decrease in the contents of ash, in comparison to non-processed pellets. Addition of waste cooking oil,

even at a rate of 2%, resulted in a statistically significant decrease in ash contents in beech wood pellet from the Pomorskie Region, in coniferous wood pellet from the Świętokrzyskie Region and in coniferous and deciduous wood pellet from the Podkarpackie Region. The most pronounced decrease in ash contents following addition of the highest dose of waste cooking oil was observed in oak wood pellet from the Podkarpackie Region—ash content decreased by 38.0%—and then in oak wood pellet from the Wielkopolskie Region—a decrease of 30.6%. Similarly, ash contents decreased by 25.0% in coniferous wood pellet from the Podkarpackie Region and in beech wood pellet from the Pomorskie Region. After the highest dose of modifier was applied, the lowest decrease in ash content, amounting to 15.6%, was identified in coniferous wood pellet from the Świętokrzyskie Region.

Numerous related studies have focused on the effects of various types of substances on ash content. Craven et al. (2015) reported that treatment of wood pellet with the use of paraffin, mineral and linseed oil produced no changes in ash content. Conversely, addition of castor oil led to an increase in ash content in wood pellets [56]. Bala-Litwiniak & Radomiak (2019) investigated the association between glycerine addition and ash content in wood pellets. The findings reported by those researchers show that the addition of glycerine at the weight rates of 2.0%, 4.5% and 7.0% led to reduced ash contents in the relevant pellets [66]. Chavalparit et al. (2013) showed that the addition of glycerine is related to increased content of ash in the pellets [67]. Gądek (2019) reported that addition of halloysite at a rate of 4% led to over a 50% reduction in ash contents in wheat and barley straw pellets [68].

The tests showed increases in the contents of carbon and hydrogen in the pellets corresponding to higher quantities of modifier added. The highest increase in carbon and hydrogen content was observed in the samples in which the modifier was added at a weight rate of 12%. The highest contents of carbon and hydrogen were found in coniferous wood pellets treated with the highest dose of waste sunflower oil. Pellets from the Podkarpackie Region contained 58.28% of carbon and 9.45% of hydrogen, and coniferous wood pellets from the Świętokrzyskie Region were found with contents of 56.71% and 6.77%, respectively. This means that in these respective pellets, the contents of carbon increased by 9.45% and 12.25%, and the contents of hydrogen increased by 9.80% and 9.72%. The highest increases in the contents of carbon and hydrogen, amounting to 12.39% and 10.38%, respectively, were observed in beech wood pellets from the Pomorskie Region with waste oil added at the highest weight rate of 12%. In the group of samples treated with the highest dose of the modifier, oak wood pellets from the Podkarpackie Region were found with the lowest increase in carbon content (by 7.63%), and a low increase in hydrogen content—whereas coniferous and deciduous wood pellets from the Podkarpackie Region were found with the lowest increase in hydrogen content.

The pellets assessed in the study contained relatively high levels of C and H, which corresponded to their high heating potential.

No nitrogen contents were identified in the samples subjected to the analyses (<0.04%). Very low contents of ash-forming elements suggest that pellets may be used in industrial heating systems [69].

Numerous researchers have examined the contents of key elements in the process of biochar combustion. Kazimierski & Kardaś [70] carried out a study focusing on pyrolysis of wood chips and showed that higher temperatures in the process corresponded to greater increases in carbon and hydrogen contents in the produced pellets. Research reports also show that the application of a torrefaction process leads to increased carbon content and reduced hydrogen contents in the produced pellets [25,71]. Craven et al. (2015) reported that wood pellets treated with paraffin, mineral, linseed and castor oils were found with decreased contents of carbon [56]. Chevalparit et al. (2013) assessed the effects of glycerine on the properties of pellets made from palm fronds, and they observed a decrease in carbon content in the samples containing 15–45% of glycerine by weight [67].

Analysis of volatile contents in the pellets before and after waste sunflower oil was added showed that in each case, the modifier reduced the contents of volatile substances in

the pellets. The changes in the volatile contents resulting from addition of oil were small, even when the highest dose of modifier was applied. Only in two cases, did addition of sunflower oil at the highest rate of 12% lead to statistically significant decreases in volatile contents; these changes were observed in coniferous wood pellets from the Podkarpackie Region. In this case, the control sample was found to have a 17.24% content of volatile substance. With increasing weight rate of waste sunflower oil added, the related values were in the range between 16.83% and 15.64%. In beech wood pellet from the Pomorskie Region, a volatile content of 20.43% was identified in the control sample, and following addition of the modifier, the values ranged from 20.24 to 18.1%.

In the related literature, many articles discuss volatile contents in biomass. As an example, Kratofil et al. (2014) and Poskart et al. (2018) reported that a torrefaction process leads to decreases in volatile contents in biomass [25,26]. Poskart et al. (2018) observed a 40% decrease in the contents of volatile substances following a torrefaction process applied to Virginia mallow biomass, in comparison to the raw biomass [25]. Craven et al. (2015) observed increases in volatile contents in wood pellets treated with paraffin oil, castor oil, mineral oil and linseed oil [56]. Sirrajudin et al. (2016) and Chavalparit et al. (2013) reported that the contents of volatile substances in pellets made from palm fronds increased after glycerine was added [67,72].

Calorific value is the most important parameter describing the quality of fuels from plant biomass. The findings related to the calorific value of the pellets, before and after the modifier was added, are shown in the Figures 3 and 4.

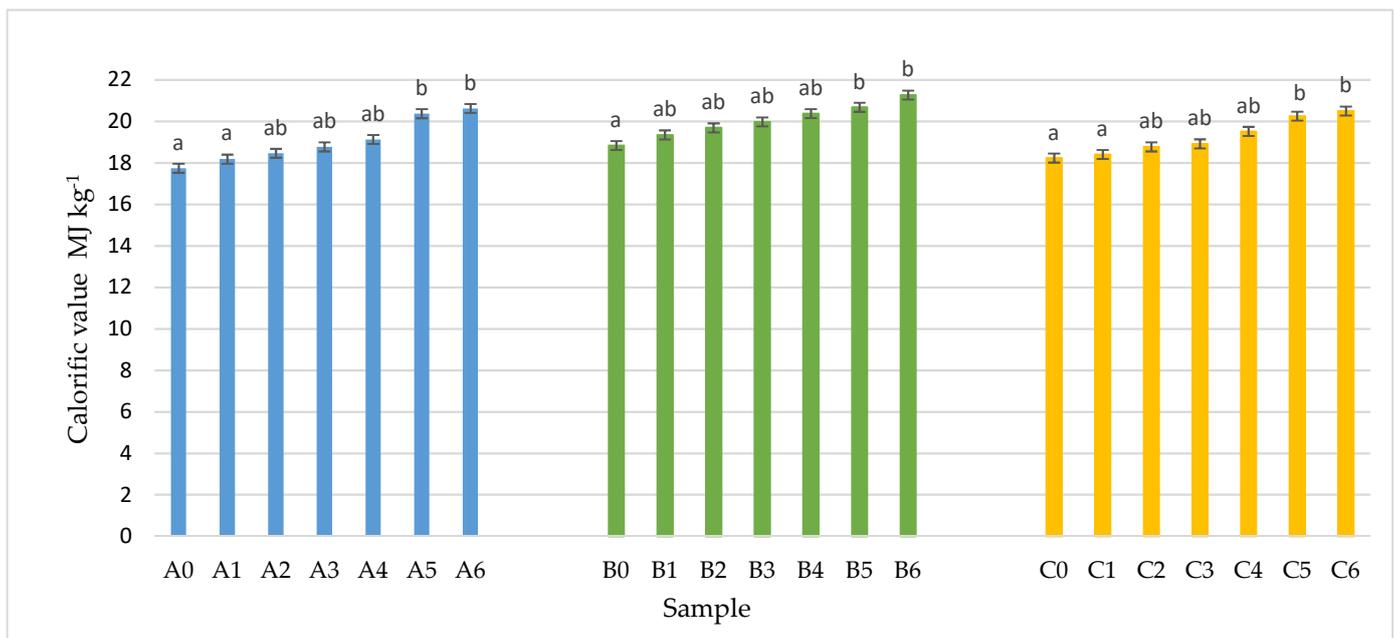


Figure 3. Calorific value of samples of deciduous woods pellets before and after waste cooking oil was added by spraying. Statistically significant differences marked by different letters ($p \leq 0.05$). Differences between average values marked with the same letters (a,b) are not statistically significant at the level of $p \leq 0.05$ according to the Duncan test. The data were analysed separately for each type of pellet.

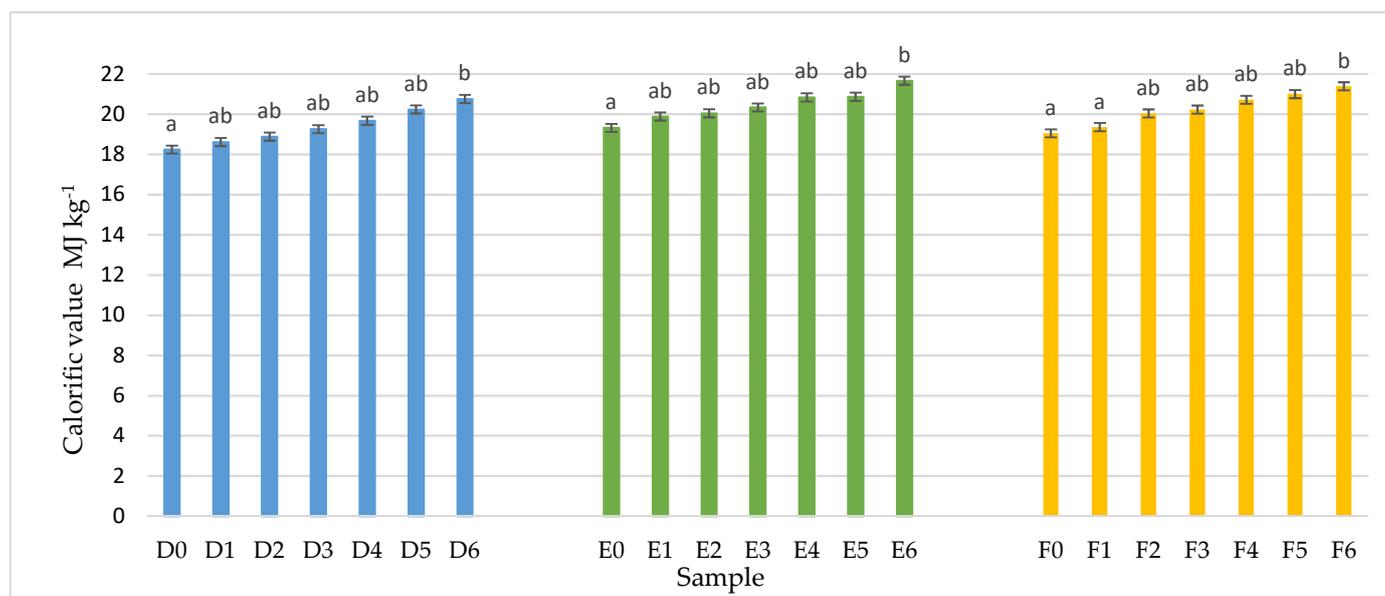


Figure 4. Calorific value of samples of coniferous and coniferous–deciduous wood pellets before and after waste cooking oil was added by spraying. Statistically significant differences marked by different letters ($p \leq 0.05$). Differences between average values marked with the same letters (a,b) are not statistically significant at the level of $p \leq 0.05$ according to the Duncan test. The data were analysed separately for each type of pellet.

The highest calorific value was found in coniferous wood pellets from the Podkarpackie Region, in both the control and the treated samples. The calorific value in the sample with sunflower oil added at a rate of 12% was 21.67 MJ kg^{-1} , which reflects a 12% increase in relation to the initial value of 19.32 MJ kg^{-1} . In this case, addition of oil at a low rate of 2% by weight led to a nearly 3% increase in the calorific value. Similar calorific values were found in coniferous and deciduous wood pellets from the Podkarpackie Region. In this case, the calorific value of untreated pellets was 19.05 MJ kg^{-1} , and following the addition of oil at a rate of 2% by weight, the value increased by 1.5% and amounted to 19.36 MJ kg^{-1} . Addition of the modifier at a rate of 12% by weight led to an over 12% increase in the calorific value, which in the relevant sample amounted to 21.39 MJ kg^{-1} . The lowest calorific value was identified in beech wood pellets from the Pomorskie Region— 17.74 MJ kg^{-1} . However, this type of pellet was found with the most pronounced increase in calorific value, which in the sample treated with the highest dose of oil, was 16% higher. The findings presented in this study show that the changes in the calorific value of the pellets were related to the quantity of the waste sunflower oil applied as the modifier. The highest values in each type of the wood pellet were identified in the samples treated with the highest dose of the modifier, i.e., oil added at a weight rate of 12% in each type of wood pellet corresponded to an over 12% increase in the calorific value. The highest increase of the calorific value, exceeding 16%, was identified in beech wood pellet from the Pomorskie Region. Furthermore, statistically significant increases in the calorific value were identified in all the types of pellets following treatment with oil at a weight rate of 4%. These findings also suggest that wood pellets produced in the Podkarpackie Region have higher calorific value than the other pellets taken into account in this study.

According to the related literature, wood from coniferous trees generally has a higher calorific value than wood from deciduous trees [73]. In the present study, pellets made from conifer wood had the highest calorific value. Various attempts to improve the calorific value of biomass pellets have been reported in the literature. Researchers have shown that there is an association between lignin content and the heating value of the wood biomass [74]. The calorific value of pellets is also significantly affected by conditions during transport and in storage [75,76]. Kuokkanen et al. (2011) investigated the calorific value of coniferous wood pellets and found that this parameter is not affected by addition of lignosulphonate,

potato flour or potato peel waste [61]. Nosek et al. (2011) demonstrated that addition of motor oil or vegetable oil at a weight rate of 0.5% corresponds to a higher calorific value of pellet, whereas addition of maize starch at the weight rate of 0.5% leads to a decrease in this parameter [77]. Civitarese et al. (2019) reported that, compared to non-pelletised wood, the calorific value of pelletised wood on average is 3.95% higher [11]. Król et al. (2017), in their study, found that the hydrophobic compound DEPOL 40 added to sawdust from conifer wood, at a rate of 10, 20 and 30% by weight, considerably increased the calorificity of the materials [78]. Craven et al. (2015) investigated the effects of selected types of oil on calorific value of pellets; addition of oil during the production process led to increases in this parameter on average of 5.7% [56]. Sirrajudin et al. (2016) and Chavalparit et al. (2013) showed that a combination of oil palm fronds and glycerine may be an alternative method for producing a source of energy from biomass [67,72]. The teams assessed the effects of glycerine added at a rate ranging between 15 and 45% by weight to pellets made from oil palm fronds. The researchers showed that ash contents were higher than the reference value applied as a criterion; however, because of its chemical composition, the ash could be used as an alternative material for other purposes—hence the pellets produced this way may be a good replacement for conventional sources of energy [67]. The Table 4 compares the physicochemical properties of different biomasses.

Table 4. The physico-chemical parameters of selected biomasses [79].

Biomass	Volatile Matter	Fixed Carbon	Ash	Hydrogen	Nitrogen	Calorific Value	Reference
			%			MJ kg ⁻¹	
Corn cob	85.7	9.4	4.9	6.4	0.4	18.7	Tian et al. [80]
Corn stalk digest	63.2	16.7	20.00	4.9	2.0	15.3	Zhang et al. [81]
Eucalyptus tree residues	63.3	24.5	6.1	5.1	<1.0	18.1	Cardona et al. [82]
Oil palm frond	79.4	20.6	25.6	5.5	1.4	20.3	Matali et al. [83]
Rice husk	70.6	12.9	16.5	3.9	2.5	22.8	Zhang et al. [84]
River tamarind	72.7	15.3	12.00	6.8	1.4	17.9	Matali et al. [83]
Scots pine	86.4	-	0.3	6.7	0.1	20.6	Rudolfsson et al. [85]
Steam wood	88.1	11.6	0.3	6.0	0.1	22.6	Wang et al. [86]
Stump	86.7	14.7	0.4	6.5	0.1	19.5	Wang et al. [86]
Wheat straw	77.0	19.5	3.5	5.8	0.9	17.5	Bai et al. [87]

4. Conclusions

The calorific value of plant biomass pellets is in the range between 16 and 19 MJ kg⁻¹. Wood pellets are among the most commonly used biofuels. Given the growing interest in pellets and a need to enhance the efficiency of such materials, scientists began to look for novel solutions to make it possible to improve their heating value.

This study provided promising results; the positive effect of wood pellet modification with used sunflower oil using the spraying method was confirmed. The aim of the study was achieved: the authors increased the calorific value of the pellets, leaving them with a high durability. Treatment with waste oil at a weight rate of 12% resulted in a 12–16% increase in the calorificity of the wood pellets—the calorific value of pellets is their most important quality feature. The durability of pellets is a particularly important feature during their transport and storage. The results of the experiment are unambiguous—they show that the analyzed method causes an increase in heat-generating elements in pellets, which results in an increase in their heating potential. The introduction of the modifier did not increase the ash-forming elements. The amount of ash in pellets after the experiment was reduced. On the basis of the obtained results, it is stated that the method of pellet refining proposed by the authors may be competitive in relation to other available technologies. However, the very process of oil dosing in the spraying method and its filtration on an industrial scale can be cumbersome and requires refinement. More

research, along with government subsidies and technology transfer, is needed to tackle future challenges in the development of pellet valorisation technology.

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