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Application of Post-Flotation Dairy Sludge in the Production of Wood Pellets: Pelletization and Combustion Analysis

Sławomir Obidziński ¹, Magdalena Joka Yildiz ^{1,*}, Sebastian Dąbrowski ², Jan Jasiński ³
and Wojciech Czekala ^{3,*}

¹ Department of Agri-Food Engineering and Environmental Management, Białystok University of Technology, Wiejska 45E Street, 15-351 Białystok, Poland

² BSH Dąbrowscy General Partnership, Piotrkowska 28 Street, 95-080 Syski, Poland

³ Department of Biosystems Engineering, Poznań University of Life Sciences, Wojska Polskiego 50, 60-637 Poznań, Poland

* Correspondence: m.joka@pb.edu.pl (M.J.Y.); wojciech.czekala@up.poznan.pl (W.C.)

Abstract: The amount and variety of waste increases every year. One of the places where biodegradable waste is generated is the agri-food industry, where it is possible to utilize it for the purpose of energy production. The aim of this research was to determine the possibility of using post-flotation dairy sludge as a raw material for co-pelletization with sawdust. The scope of this work included physical and chemical characterizations of the feedstock, the co-pelletization process, and the combustion of the produced pellets, combined with an exhaust analysis. The obtained values of the pellets' density at each level of sludge addition allowed us to conclude that the obtained pellets had a good market quality and constituted a full-fledged, innovative solid fuel, in accordance with the guidelines of the latest, currently applicable ISO 17225 standard. Furthermore, adding ca. 20%wt of sludge to sawdust resulted in a 30% decrease in the pelletization power demand, and still, the combustion characteristics of the pellets met the European Ecodesign emission limitations in terms of the CO and NO_x content in the exhaust. The addition of post-flotation dairy sludge to sawdust has a beneficial influence on the production of fuel pellets by decreasing the energy consumption of the pelletization process and improving the pellets' kinetic durability. Due to legal requirements and the pursuit of the circular economy principle, one should expect an increased interest in the use of agri-food waste for the production of biofuels.

Keywords: pellets; biofuels; waste to energy; circular economy; kinetic durability; energy consumption; post-flotation dairy sludge



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1. Introduction

Agriculture and the agri-food industry in Poland generate over 10 million tons of waste each year [1]. Moreover, the problem of improper waste appears more and more often. The uncontrolled decomposition of waste from agriculture and the agri-food industry generates significant amounts of pollutants, including hazardous compounds and substances [2]. Currently, it is necessary to develop new, rational systems for processing waste from food production and processing [3,4]. One of the methods of managing all kinds of plant waste is its processing with the use of biological processes. This includes the process of anaerobic digestion, as a result of which, biogas and digestate are obtained [5,6], and composting, the product of which is compost, which is a valuable fertilizer [7]. An alternative to biological processes is the processing of biodegradable waste into solid biofuels, for example the pressure agglomeration process allowing the homogenization and reduction of the bulk density of used feedstock [8,9]. As a result of the pressure agglomeration process, pellets, granules, or briquettes can be produced [10,11]. Among the many types of biomasses, wood is the main raw material for the production of pellets [12]. Due to the fact that before the process of cutting (sawing) the wood, even small impurities are removed by removing the bark and washing the logs, sawdust is a suitable raw material for the production of clean

pellets [13]. However, with the growing demand for wood pellets and the growing popularity of pellet fuels, the supply of all kinds of waste and by-products related to this sector of the economy has increased. Therefore, there is huge interest in the production and use of solid biofuels, including pellets produced from feedstocks other than sawdust [14–16]. However, the most important features that should characterize these raw materials are primarily their availability, lack of contamination, homogeneity, and high content of organic matter.

Nowadays, one of the possible solutions to improve the quality of pellets is co-pelletization, i.e., mixing two or more raw materials [17]. While producing pellets from alternative raw materials, the general trend is to add wood materials, for example, sawdust, to improve the quality of the pellets obtained. This causes an increase in the lignin content in the pellets, and consistently improves their quality, mainly in terms of their calorific value, bulk density, durability, and ash content [18].

Garcia et al. [19] produced fuel pellets from mixtures of pine sawdust with many alternative agricultural raw materials and assessed the quality of the obtained pellets in accordance with ISO standards for industrial pellets. As part of the research, they found that pellets made of pine sawdust with the addition of almond shells (30%wt), olive pits (30%wt), or amount cones (15%wt) resulted in I1 class pellets.

Recently, co-pelletization has been one of the most studied methods of producing fuel pellets. Harun et al. [20] produced high-quality pellets from mixtures of reeds, timothy (*Phleum pratense* L.), and millet (*Panicum virgatum* L.) with pine and spruce sawdust. They also noticed that mixing pine sawdust with other types of biomasses reduced the energy demand during pelletization, which resulted in a reduction in the pellet production costs. Liu et al. [21] produced granules from blends of bamboo and spruce sawdust. They found that adding pine sawdust to bamboo was an effective way to improve the bulk density of pellets. Adding 40% pine sawdust to bamboo increased the pellets' bulk density from $0.54 \text{ g} \cdot \text{cm}^{-3}$ to $0.60 \text{ g} \cdot \text{cm}^{-3}$. The pellets obtained from such a mixture had a slightly lower calorific value, which, however, could still meet the requirements of DIN 51731 [22] ($>17,500 \text{ J} \cdot \text{g}^{-1}$).

Further research related to the production of wood pellets with additives was carried out by Barbanera et al. [23]. They produced pellets from the wood waste from olive tree pruning with the addition of olive pomace. The results of their conducted research allowed them to state that the best mixtures, resulting in pellets that met the requirements of the EN 17225-6 standard [24], were a mixture of olive wood waste with the addition of 25% olive pomace from the second pressing and a second mixture with the addition of 50% olive pomace from third pressing. The pellets obtained from these mixtures were characterized by a greater durability and a lower ash content (up to 2.4%) compared to the pellets from olive pomace alone, although they had extremely high values of ash-forming elements, such as Fe, Mg, and K.

Obidziński et al. [25] conducted a detailed study to determine the impact of a rye bran addition to sawdust (mixed spruce and pine in a 50/50% ratio) for pellet production. It was found that by increasing the addition of rye bran from 10 to 20%wt, the power demand of the pelletizer decreased from 11.06 to 8.89 kW; while pelletizing the sawdust alone, the unit required 13.06 kW. Moreover, the obtained pellets had an improved kinetic durability reaching up to 98.08% and an improved particle density ($1218.6 \text{ kg} \cdot \text{m}^{-3}$) for the 20%wt rye bran addition. However, a decrease in the bulk density was observed from $646.5 \text{ kg} \cdot \text{m}^{-3}$ (10% rye bran) to $584.73 \text{ kg} \cdot \text{m}^{-3}$ (20% rye bran).

Sludge, such as sewage sludge or post-flotation dairy sludge, is a possible binder that has been reported to improve the mechanical properties of pellets and decrease the process energy consumption during co-pelletization. The main obstacle to its use for fuel pellet production is attributed to the high ash content, which limits the amount of sludge that can be mixed with solids [26]. Therefore, it is suggested to limit its content; however, detailed chemical and process research should be performed to precisely classify the sludge-to-pellet possibilities and benefits from its addition during solid fuel production.

The aim of the research was to determine the possibility of using post-flotation dairy sludge as a raw material for co-pelletization with sawdust. The scope of the work included

a physical and chemical characterization of the feedstock, the co-pelletization process, and the combustion of the produced pellets, combined with an exhaust analysis.

2. Materials and Methods

2.1. Materials

The basic raw material used during this research was wood waste in the form of sawdust (deciduous tree wood) obtained from BSH Dąbrowscy Sp.j. (Syski, Poland). The company specializes in fuel pellet and briquette production. Additionally, for binding purposes, post-floatation dairy sludge was added to sawdust in amounts of 10, 15, or 20%wt. The sludge was obtained from an industrial dairy plant located in Wysokie Mazowieckie, Poland. The tested materials are shown in Figures 1 and 2, respectively.



Figure 1. Sawdust used in the research.

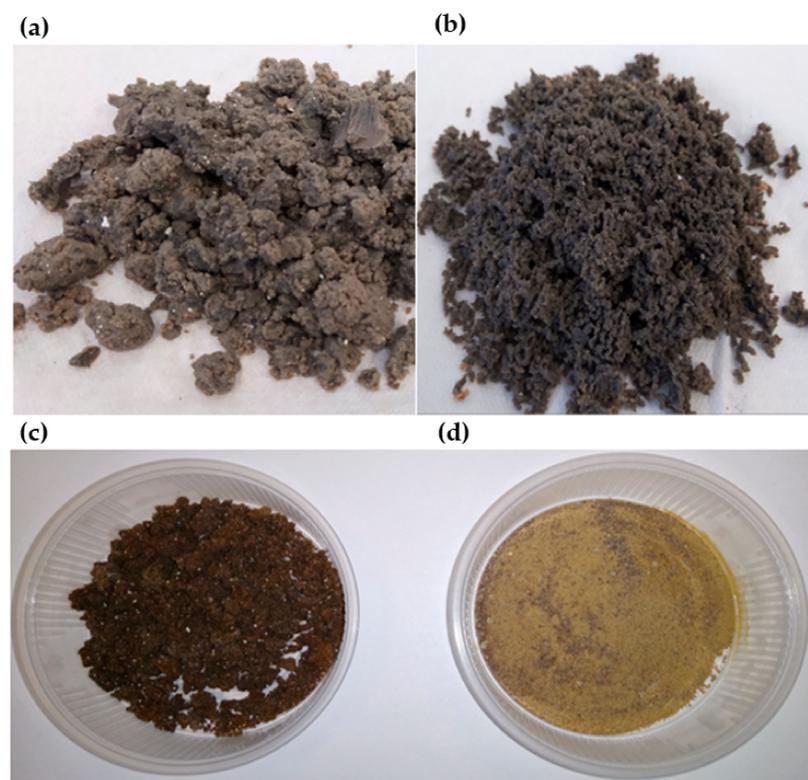


Figure 2. View of the post-floatation dairy sludge (a) unmilled, before drying, (b) milled, before drying, (c) after drying, and (d) after drying and milling.

2.2. Methods

2.2.1. Moisture Content

The determination of the moisture content of the raw materials and mixtures of these raw materials before the pelletization process was performed in accordance with PN-76/R-64752 [27] using an AXIS ASG120 laboratory moisture analyzer. The moisture content was determined five times for each sample: 5 g samples were taken for measurement and dried at 105 °C until the indications of the moisture analyzer during three consecutive readings at 15 s intervals remained unchanged. The mean value of the obtained determinations was taken as the final result of the moisture content determination.

2.2.2. Ultimate Analysis

The content of carbon, nitrogen, hydrogen, and sulfur was determined using the LECO CHN628 analyzer, in accordance with PN-EN 15104 [28], PN-EN ISO 16948 [29], and PN-EN ISO 16994 [30], respectively. The device uses the combustion technique and gives the results as percentages by weight or parts per million (ppm). The analyzer was equipped with software based on the Windows operating system, and an external computer was used for system control and data management.

2.2.3. Particle Size Distribution

The particle size determination of sawdust was carried out using a programmed shaker equipped with a set of sieves, LPz-2e by Multiserv Morek (Poland). During the analysis, a set of 9 sieves with square mesh side dimensions was used: 8.0; 4.0 mm; 2.0 mm; 1.0 mm; 0.5 mm; 0.25 mm; 0.125 mm; and 0.063 mm.

The particle size distribution was determined in accordance with PN-89/R-64798 [31]. The determination principle was based on the manual or mechanical sieving of the sample through a sieve or set of sieves and a weight determination of individual fractions. The clean and dry sieves were stacked on top of each other according to increasing mesh diameter. Then, 100 g of the prepared material was poured onto the upper sieve and covered with a lid, and the sifter was started for 5 min. After the time had elapsed, the sifter was stopped, the sieve frames were lightly tapped on, and then the sifter was started again for 20 s. After the sifter had stopped, each fraction was weighed. The obtained result of weighing was the percentage content of a given fraction. The result was the arithmetic mean of three tests performed. Due to the high moisture content, the analysis was not conducted for sludge.

2.2.4. Bulk Density

A metal cylinder with a volume of 407.5 cm³, a laboratory balance (AX324M by OHAUS), and a steel scraper were used to test the bulk density of the sawdust and produced pellets (PN-EN ISO 17828:2016) [32]. Each time, the cylinder was filled in with the sample until a possibly even plane was obtained.

The bulk density was calculated using the following equation:

$$\rho_{n,p} = \frac{m_p}{V_n} \quad (\text{kg}\cdot\text{m}^{-3}) \quad (1)$$

where m_p —sample mass (kg) and V_n —cylinder volume (m³).

The analysis was performed over three repetitions.

2.2.5. Pelletization Process

The pelletization process of raw material mixtures was carried out on the SS-4 test stand, the main element of which was the P-300 pelletizer fabricated by Protechnika (Poland) equipped with the working system “flat rotary matrix-compacting rollers” [33].

Prior to pelletization, the mixtures were prepared by weighing the appropriate mass of sawdust and sludge and mixing them with the use of an automatic mixer (similar to

the ones used for paint mixing). The mixtures were left for 24 h for the moisture to spread evenly in the mixture volume.

The research was carried out in accordance with the plan presented in Table 1. The SS-4 pelletizer was equipped with a universal meter for measuring the device's power demand and a recorder coupled with a computer. The signals from the sensor were fed to the recorder in the form of binary files, which were further converted and processed with the use of Microsoft Excel (Microsoft, Redmond, WA, USA) and Statistica 13.0PL (Statsoft, Poland) software.

Table 1. Research plan matrix for the pelletization process of sawdust with the addition of post-flotation sewage sludge (z_o).

x_1	$x_1 = z_o$ (%)	$x_2 = Q_m$ ($\text{kg}\cdot\text{h}^{-1}$)
1	10	50
2	15	50
3	20	50

During the tests, the constant values were as follows: moisture content, $w_m = 18\%$; diameter of die holes, $d_o = 6$ mm; feedstock mass flow, $Q_m = 50$ $\text{kg}\cdot\text{h}^{-1}$; compacting roll rotational speed, $n_r = 270$ rpm; and the gap between rollers and die, $h_r = 0.4$ mm.

The set of input quantities (dependent variables) included: the power demand of the pelletizer, N_g ; the kinetic durability of pellets, P_{dx} ; the particle density of pellets, ρ_g ; and the bulk density of pellets, ρ_{ug} .

2.2.6. Kinetic Durability of Pellets

A total of 24 h after the pellets were produced, their kinetic durability was determined using a Holmen tester. The tests were carried out in accordance with the PN-R-64834:1998 [34] and PN-EN 15210-1:2010 [35] standards. During each test, a 100 g sample of pellets was introduced into the tester's chamber. The pellets were put into motion by the air stream and by circulating, hitting the tester's metal perforated walls. The sample remained in the tester's chamber for 60 s. After this time, the remainder of the pellets from the chamber were put on a sieve of 7 mm, sieved, and weighed. The kinetic durability was calculated as the ratio of the weight of the granulate after the test to the mass before the test.

2.2.7. Pellet Particle Density

The determination of the pellets' density was carried out 24 h after leaving the pelletizer. During the measurements, the height and diameter of 15 randomly taken pellets were measured with an accuracy of ± 0.02 mm and their weight was determined using a laboratory balance, AX324M by OHAUS, with an accuracy of ± 0.0001 g. The density of the pellets was calculated as the ratio of their weight to their volume.

2.2.8. Calorific Values of Feedstocks and Pellets

The higher heating value (HHV) of raw materials was tested in accordance with the PN-ISO 1928:2002 standard [36]. The calorimeter KL-12Mn, made by Precyzja-Bit (Poland), was used to determine the HHV. The measuring principle was based on the complete combustion of the fuel sample in a calorimetric bomb in an oxygen atmosphere under increased pressure, and the temperature rises were measured as a thermal effect of sample combustion. The lower heating value (LHV) was calculated as follows:

$$\text{LHV} = \frac{100 - w}{100} \cdot \text{HHV} - \frac{\gamma \cdot w}{1000} \quad (\text{MJ}\cdot\text{kg}^{-1}) \quad (2)$$

where w —moisture content (%); γ —heat of vaporization of water at 20 °C, corresponding to the content of 1% water in the fuel ($\gamma = 24.55$ $\text{kJ}\cdot\text{kg}^{-1}$) ($\text{kJ}\cdot\text{kg}^{-1}$)

2.2.9. Combustion Tests

The combustion of the pellets was carried out with a 25 kW boiler with an automatic fuel-feeding system. The boiler was equipped with a retort burner made of refractory steel with air inlets on several levels, increasing the combustion process's efficiency. Pellets were transported from the hopper to the combusting chamber by means of a screw conveyor. The flue gases produced during combustion were directed through the heat exchanger to the chimney outlet via the chimney draft. The boiler controller automatically selected the amount of fuel supplied for combustion and the amount of air needed for the proper combustion process, based on the results of measurements of the oxygen content in the exhaust gas, which were provided by the lambda probe.

The flue gas composition at the boiler outlet was measured with an MCA10 Dr. Födisch (Germany) analyzer, enabling the measurement of the CO, CO₂, SO₂, NO, NO₂, HCl, and O₂ content in hot exhaust gases. These measurements were carried out using the reference IR method. Combustion tests were carried out under the set operating conditions of the boiler. Before collecting the measurement data, the boiler was heated up for 1 h. The fuel mass flows supplied to the boiler were constant and set at a value of 2.0 kg · h⁻¹ for each of the tested cases, and a constant stream of air dosed to the combustion chamber was established. The boiler temperature was maintained at 65–70 °C in accordance with the accepted principles for the experimental combustion of biomass fuels.

The content of the tested compounds in the exhaust gas included: CO₂, CO, NO, SO₂, and HCl, which were normalized to 10% of the oxygen content according to the formula:

$$X'' = \frac{21 - O_2''}{21 - O_2'} X' \quad (\%, \text{ mg} \cdot \text{Nm}^{-3}) \quad (3)$$

where X''—calculated gas concentration (% · m⁻³), X'—obtained gas concentration (% · m⁻³), O₂'—required oxygen content (%), and O₂''—obtained oxygen content (%).

The excess air coefficient, λ, was calculated as follows:

$$\lambda = \frac{21.5}{21.5 - O_2''} \quad (-) \quad (4)$$

3. Results and Discussion

3.1. Feedstock Characteristics

The post-flotation dairy sludge used was characterized by a moisture content of 83.58% (Table 2); therefore, its pelletization alone would not be possible. The high moisture content was suitable for using the sludge as a binding material in co-pelletization. Water itself acts as a binder due to its ability to create hydrogen bonds during pelletization; thus, a material with a high moisture content can be successfully added to a dry material, perhaps such as the investigated sawdust (ca. 11%), and promote higher-quality pellets (improved mechanical strength). Water's lubricating properties may also decrease the energy consumption of the pelletizing device.

Table 2. Feedstock's physical and chemical properties.

Feedstock Property	Sawdust	Dairy Sludge
Bulk density (a.r.) (kg · m ⁻³)	100.02	n.d.
Moisture content (a.r.) (%wt.)	10.87	83.58
C (d.b.) (%wt.)	47.96	29.20
H (d.b.) (%wt.)	6.73	5.70
N (d.b.) (%wt.)	0.14	5.73
S (d.b.) (%wt.)	0.0007	0.59
Cl (d.b.) (%wt.)	0.004	0.1

wt.—weight, a.r.—as received, d.b.—dry basis, n.d.—no data.

The high protein content in the post-flotation sludge is characterized by a much higher nitrogen content than wood sawdust. From an energetic perspective, this disadvantageous property may have an effect on the increased emissions of nitrogen oxides, NO_x; however, due to the limited share of sludge in the mass of fuel subjected to combustion, this impact can be limited.

Most of the standards for wood pellets (PN-EN 14961-2:2011 [37], DIN 51731 [22], and ÖNORM M7135 [38]) require that the pellets contain less than 0.3% nitrogen. The EN Plus A2 certificate (in accordance with PN-EN 14961-2:2011 [37]) specifies the maximum nitrogen content as 0.5% and the EN Plus B certificate specifies it as 1%. Therefore, the obtained granulate with the addition of up to ca. 15% of post-flotation dairy sludge meets the EN Plus B certificate criterion (according to PN-EN 14961-2:2011 [38]), and with the addition of up to ca. 6% of the sludge, it meets the EN Plus A2 criterion (in accordance with PN-EN 14961-2:2011 [37]).

Another alarming factor detected in the chemical composition of sludge is the high sulfur content of ca. 0.59%. The DIN-Plus certificate and the ÖNORM M7135 [38] standard determine the acceptable sulfur level in wood pellets at a level not exceeding 0.04%. Therefore, pellets with an addition of up to 6% post-flotation dairy sludge meet the criteria of the EN Plus B certificate (in accordance with PN-EN 14961-2:2011 [38]). However, it has to be emphasized that the sulfur content in the sludge is still three times lower than in coal [39]. This perspective has significant importance concerning the replacement of fossil fuels with economically effective green substitutes such as post-flotation dairy sludges. Figure 3 shows the particle size distribution of the raw material (sawdust) supplied for pelletization.

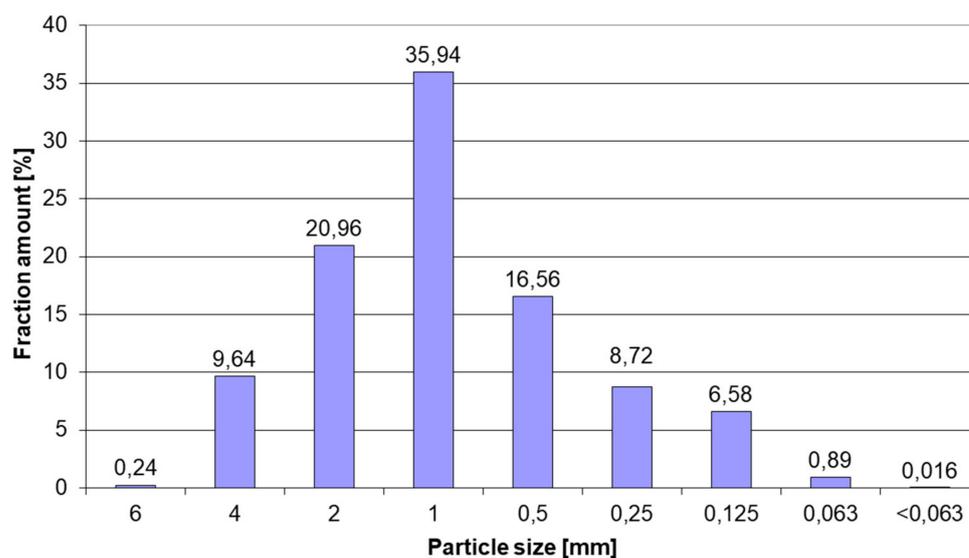


Figure 3. Particle size distribution of raw material (sawdust) used during the research.

The largest percentage of the tested raw material was represented by the 1.00 mm fraction (35.94%). The 2.00 mm fraction (20.96%), the 0.50 mm fraction (16.56%), and the 4.0 mm fraction (9.64%) had slightly smaller shares.

The sawdust contained fine fractions with a particle size of 0.25 mm and smaller, which accounted for ca. 16%wt and which was disadvantageous due to the increased energy consumption of the pelletization process, the lower service life of the working system, and the risk of self-ignition of the dusty fraction. Therefore, adding a material with a high moisture content such as post-flotation sludge can limit the abovementioned obstacles. During mixing, the fine particles will be covered and bound with the sludge, resulting in non-pressure agglomerates. This phenomenon is also proven to reduce the energy consumption of the pelletization process [39].

The raw material used contained ca. 10% of fractions with a particle size of 4 mm and larger. From the point of view of the pelletization process, this too-large fraction may increase the energy consumption of the process and deteriorate the quality of the obtained pellets. Therefore, it is preferred for such fractions to be crushed to a particle size of 1/2 of the pelletizers' die hole diameter.

3.2. Pelletization Process and Pellets' Properties

Figure 4 shows the view of pellets obtained from sawdust and a mixture of sawdust with different contents of post-flotation dairy sludge. The addition of sludge significantly influenced the pellets' color, with a darker shade of the pellets being obtained by increasing the amount of sludge in the mixture. The change in pellet color might be affected by the darker shade of sludge compared to sawdust. On the other hand, the high moisture content in sludge and the high pelletization temperature (ca. 80 °C) may have affected the xylan and pectin (flexible polysaccharides) structures, resulting in darker pellets [40].

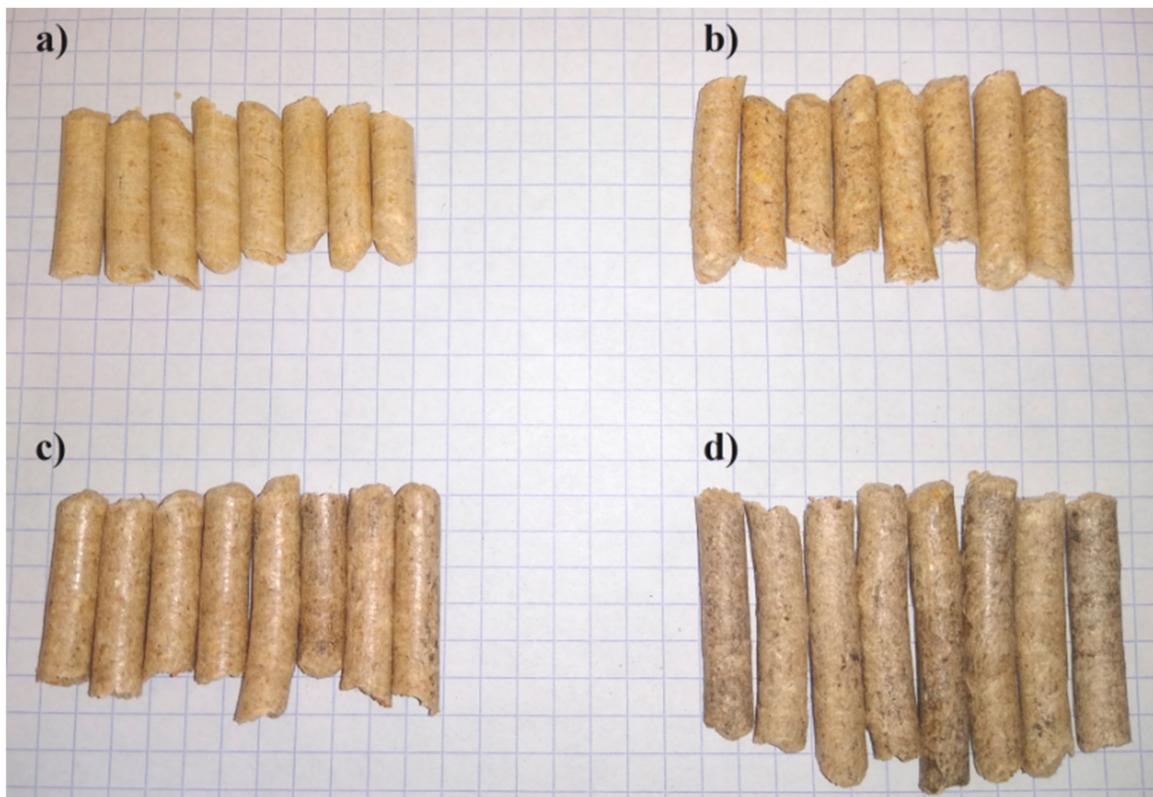


Figure 4. View of the pellets obtained from a mixture of sawdust and different contents of post-flotation dairy sewage sludge: (a) 0%, (b) 10 %, (c) 15%, and (d) 20%.

Moreover, the addition of sludge affected the length of the pellets, which is a common observation for pelletizing mixtures with a higher moisture content. The presence of water supports the creation of physical and chemical bonds such as hydrogen bonds and van der Waals bonds and makes them adhere better to one another, resulting in longer pellets [14].

Figures 5–7 show the results of the influence of the post-flotation dairy sludge content in the mixture with sawdust on the pelletization process (electrical power consumption) and on the kinetic durability, true density, and bulk density of the obtained pellets.

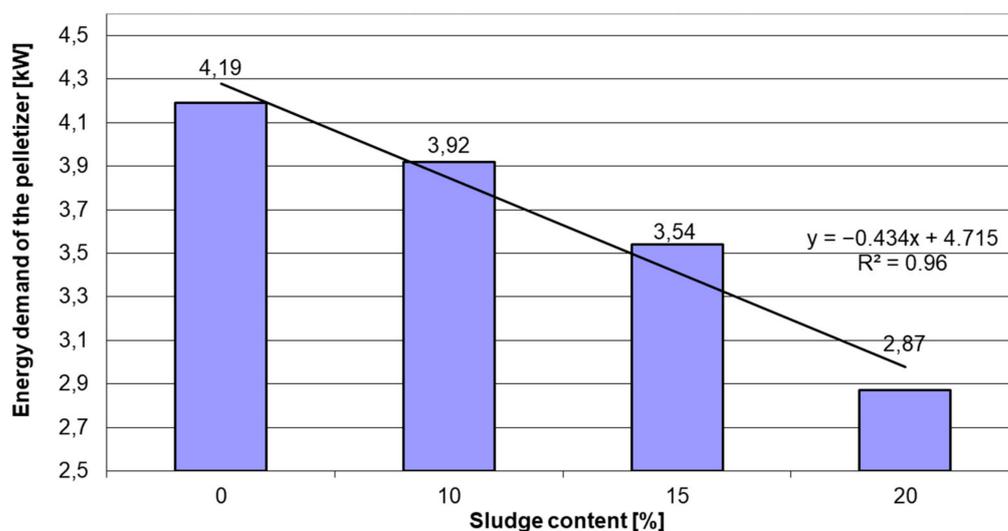


Figure 5. The dependence of the energy demand of the pelletizer on the content of post-flotation dairy sewage sludge in the mixture with sawdust.

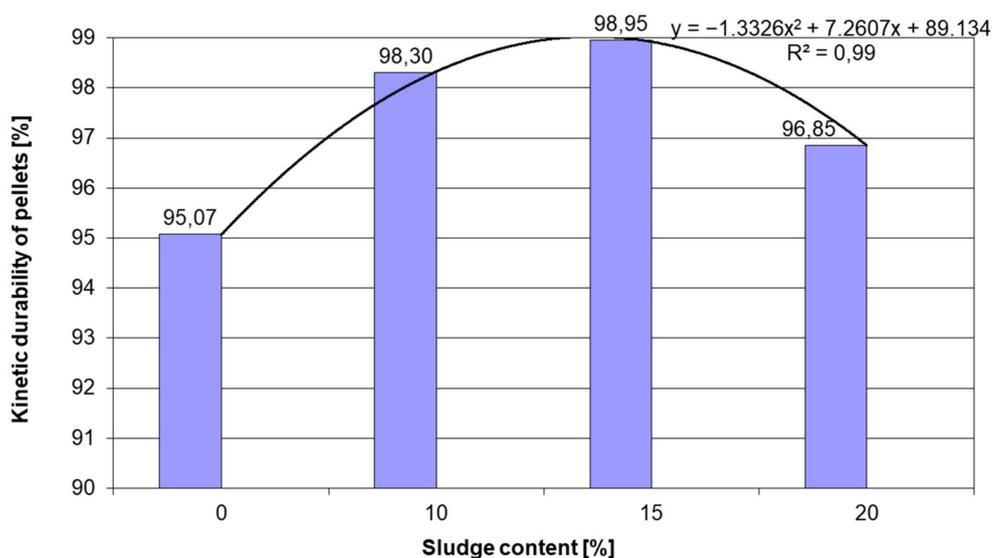


Figure 6. Dependence of the kinetic durability of pellets on the content of post-flotation dairy sewage sludge in the mixture with sawdust.

It was found that the content of post-flotation dairy sludge in the mixture with sawdust had a significant effect on the power demand of the pelletizer (Figure 5). Increasing the content of sludge from 10 to 20% reduced the power demand of the pelletizer by ca. 26.8% (from 3.92 kW to 2.87 kW). The difference between the device energy consumption for a mixture with 20% sludge compared to sawdust alone was 32%; thus, the test confirmed the lubricating properties (reducing friction during pelletization) of the sludge, and therefore, its ability to improve the compaction susceptibility of sawdust.

The moisture content had the highest effect on the energy consumption during pellet production [41]. However, it was observed that an addition of 10% post-flotation dairy sludge had a positive effect on reducing the demand for power, which was ca. 6.5% lower (3.92 kW) than the corresponding demand in the case of granulated sawdust alone with the same moisture content (17%).

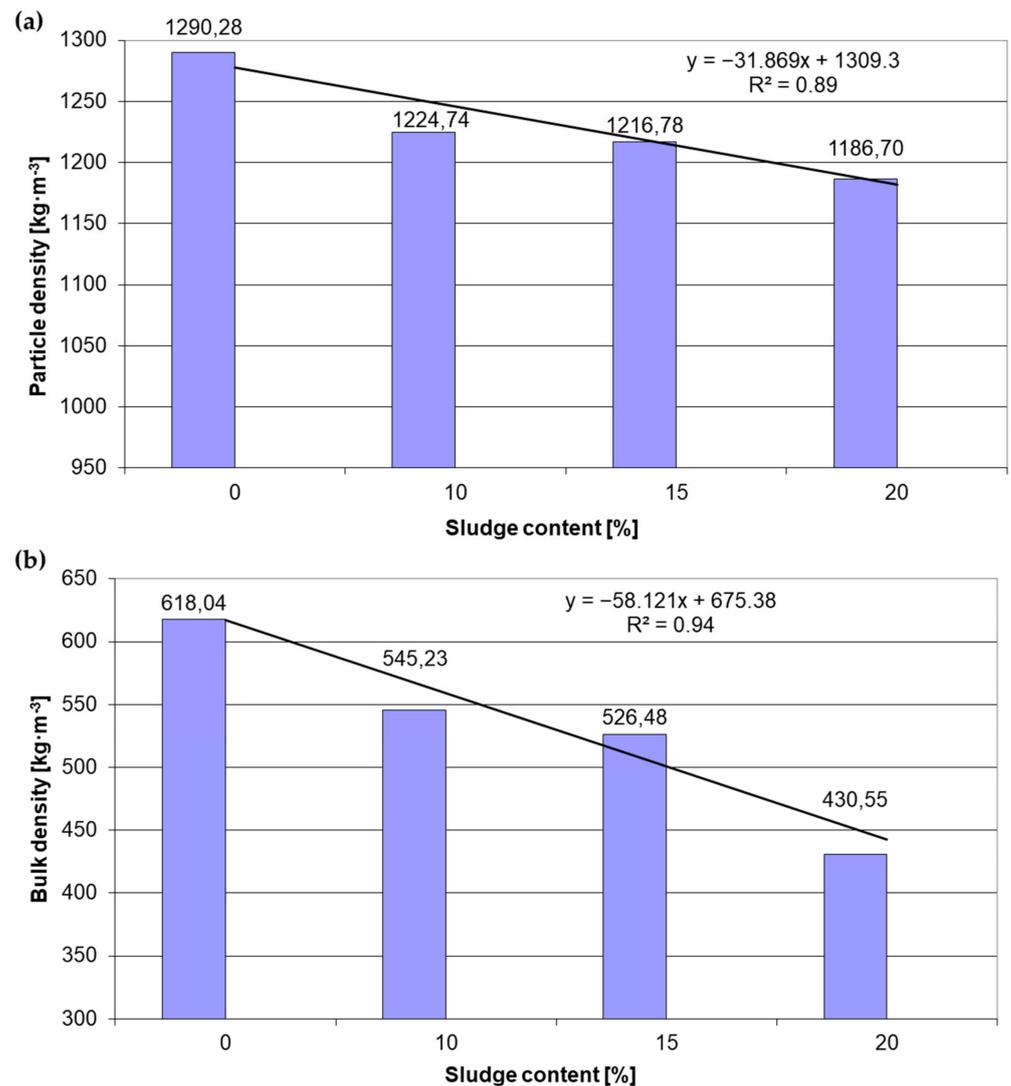


Figure 7. Influence of post-flotation dairy sewage sludge in a mixture with sawdust on: (a) particle density and (b) bulk density.

The obtained research results allowed us to conclude that the conducted pelletization process allowed the creation of fuel pellets from sawdust with the addition of post-flotation dairy sludge that resulted in a much lower energy consumption for their production. Consequently, this highlighted the effective possibility of the utilization of post-flotation sludge for energetic purposes.

Figure 6 shows the influence of the post-flotation dairy sludge content in the pelletized mixture with sawdust on the kinetic durability of the obtained pellets. Kinetic durability is an important factor in pellet quality tests, which illustrates their transportation and feeding properties. The value (%) represents the quantity (mass) of pellets that are impact-resistant during the kinetic test. High values (>96.5%) for this factor are desirable (PN-EN 17225-2) [42].

Increasing the content of post-flotation dairy sludge in the mixture with sawdust from 10 to 15% caused an increase in the kinetic durability of the pellets by about 0.6% (from 98.30% to 98.95%). A further increase in the sludge share from 15 to 20% resulted in a slight decrease in the kinetic durability of the pellets by ca. 2% (from 98.95% to 96.85%). The kinetic durability of pellets with a 15% additive was about 3.3% higher than for pellets obtained from sawdust alone, which was 95.07%.

The results indicate that all varieties of pellets prepared with the addition of sludge were characterized by a higher kinetic durability than pellets made from raw sawdust. Therefore, the presence of sludge provided the beneficial aspects of reduced fine content and lower dust generation during the transportation and handling of the pellets. Moreover, pellets with a bigger length (Figure 4) had higher values of kinetic durability [43], and the trend was also proven for samples with 0 to 15% sludge content. At a content of dairy sludge of 20%, a drop in kinetic durability was observed. The phenomenon was a consequence of a too-high moisture content, resulting in curvy pellets (Figure 4).

Stasiak et al. [44] determined the mechanical properties of pine sawdust pellets mixed in various proportions with wheat and rapeseed straw, and found that pellets obtained from mixed pine sawdust with ground rapeseed straw were characterized by a greater durability and impact resistance. During the tests, they also observed a decrease in strength with an increase in moisture content and an increase in strength with an increase in compaction pressure. Jezerska et al. [45] confirmed the positive effect of a binder addition during sawdust pelletization by compacting sawdust mixtures with the addition of 0, 5, 10, 15, and 20% starch. The obtained pellets had a slightly lower density, but a higher durability, a higher hardness, and resistance to moisture. The addition of starch from 5 to 20% caused a slight decrease in the density of the granules, from $1260 \text{ kg} \cdot \text{m}^{-3}$, compared to the density of the granules obtained from sawdust alone ($1290 \text{ kg} \cdot \text{m}^{-3}$). However, it increased the kinetic durability of the pellets from 80.9 to 99.2%, which was much higher than that of pellets obtained from sawdust alone (70.3%).

Another crucial quality factor in the pellet analysis is the density value. The particle density represents the characteristics of a single pellet particle and is often used for investigating the degree of compaction. Secondly, bulk density describes the storage properties of pellets: the mass of pellets filling a certain volume.

Hereby, the moisture present in post-flotation dairy sludge acted as a pillow-filler by decreasing both the particle and bulk densities. The water particles were dispersed and bonded in-between the solid particles during pelletization. After the process, the pellet particles were left to cool and stabilize for 24 h, and then the measurements were performed. Therefore, by striving to stabilize the partial pressures of water and steam, water particles diffused from the structure of the pellets, leaving empty gaps in their structure and thus lowering the density.

Increasing the content of post-flotation dairy sludge in the mixture with sawdust from 10 to 20% caused a decrease in the particle density of pellets from $1224.74 \text{ kg} \cdot \text{m}^{-3}$ to $1186.70 \text{ kg} \cdot \text{m}^{-3}$. Furthermore, a drop of 17% in the true density was observed as a result of adding sludge to sawdust (comparing the pure sawdust pellets and the pellets with 10% sludge added).

In accordance with the existing standards for wood pellets (DIN 51731—Germany [22], ÖNORM M 7135—Austria [38], SS 18 71 20—Sweden [46], EN 14961—Poland [37], and ISO 17225—European Union [47]), pellets having a particle density above $1000 \text{ kg} \cdot \text{m}^{-3}$ are ranked as high-quality fuels. Therefore, the obtained values for the pellets' particle density confirmed their very good market quality, and with the addition of post-flotation dairy sludge, may constitute a fully-fledged innovative solid fuel.

Pellets produced from a mixture of post-flotation dairy sludge and sawdust can be used as a full-fledged fuel by the professional power industry in high-efficiency boilers (with restrictive parameters for the fuel burned) as well as by individual recipients (users of typical biomass fuel boilers).

The pellet particle density is mainly correlated with its bulk density, and a decrease in the mass accumulated in a single pellet particle result in a lower bulk density [19]. On the other hand, the bulk density can also be affected by the pellet shape, as longer pellets fit less tightly together, causing bigger gaps between them. Therefore, increasing the content of post-flotation dairy sludge in the mixture with sawdust from 10 to 20% caused a decrease in the pellet bulk density from $545.23 \text{ kg} \cdot \text{m}^{-3}$ to $430.55 \text{ kg} \cdot \text{m}^{-3}$. The decrease in the bulk density of pellets was related to the noted increase in their length (Figure 2) with an

increase in the content of post-flotation dairy sludge in the mixture with sawdust. This in turn was related to the increasing content of the binder and the moisture contained in the pelletized mixture [48].

Moreover, the conducted research outlined that a very important factor in the pelletization process of sawdust and post-flotation dairy sludge mixtures is the uniformity of mixing the components. Inaccurate mixing of the components may result in obtaining a heterogeneous pellet (different mechanical and physical properties), and also adversely affects the power demand of the pelletizer. Material containing an insufficient amount of binder, reaching the thickening system, may cause a sudden increase in the current consumption of the device.

The most beneficial additive amount of post-flotation dairy sewage sludge was from 10 to 15%. The produced pellets at these levels had satisfactory quality properties, such as kinetic durability and density, and the energy consumption during its production was meaningfully reduced compared to raw sawdust.

3.3. Calorific Values of Feedstocks and Pellets

The results of the higher heating value and calculations of the lower heating value of sawdust and dried and milled post-flotation dairy sludge are given in Tables 3 and 4, respectively.

Table 3. Test results of higher heating value and lower heating value of sawdust.

No.	Analytical Moisture (%)	Higher Heating Value (MJ · kg ⁻¹)		Lower Heating Value (MJ · kg ⁻¹)	
		For Dry Basis	For Analytical Moisture	For Dry Basis	For Analytical Moisture
1	10.65	23.823	21.234	22.305	19.615
2	10.95	23.786	21.197	22.268	19.578
3	11.02	23.742	21.153	22.224	19.534
4	10.85	23.828	21.238	22.310	19.619
5	10.90	23.656	21.085	22.138	19.466
Average	10.87	23.767	21.181	22.249	19.562

Table 4. Test results of higher heating value and lower heating value of post-flotation dairy sludge.

No.	Analytical Moisture (%)	Higher Heating Value (MJ · kg ⁻¹)		Lower Heating Value (MJ · kg ⁻¹)	
		For Dry Basis	For Analytical Moisture	For Dry Basis	For Analytical Moisture
1	9.65	15.551	14.049	14.198	12.590
2	9.71	15.456	13.963	14.102	12.504
3	9.62	15.552	14.050	14.198	12.591
4	9.69	15.571	14.069	14.218	12.610
5	9.61	15.580	14.078	14.227	12.619
Average	9.66	15.542	14.042	14.189	12.583

Figure 8 shows the values of the HHV and LHV depending on the moisture content of sawdust (Figure 8a) and post-flotation dairy sludge (Figure 8b). The linear regression equations allow for the calculation of the calorific properties of the analyzed feedstock based on the known moisture content (x), which is especially important for calculations performed during industrial pellet production. A high moisture content in solid fuels is undesirable due to heat loss during combustion (energy needed to evaporate the present water) [49]; however, after pelletization, usually pellets are not combusted immediately and have time to stabilize their moisture content during the storage conditions. On the other hand, a too-high moisture limits the compaction abilities of solids by lowering the

friction value and creating water layers in the pellet structure (Christmas tree effect, lower density, and lower kinetic durability) [50].

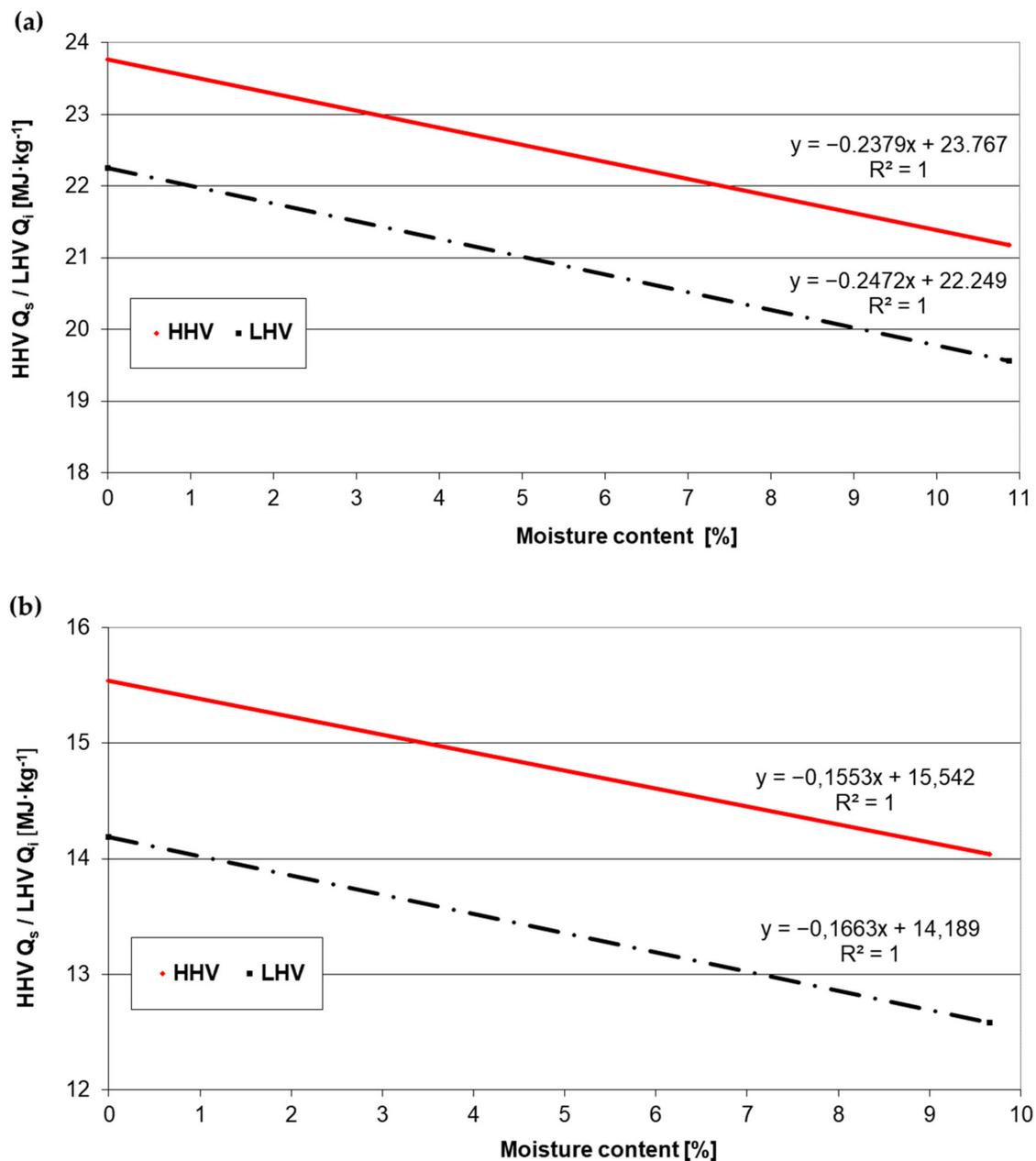


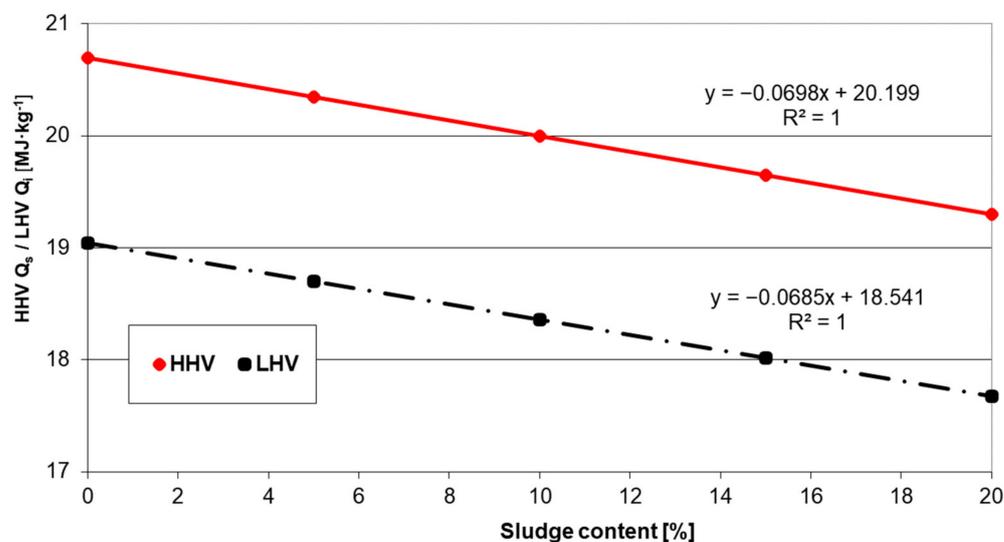
Figure 8. The dependence of the HHV and LHV on the moisture content of (a) sawdust and (b) post-flotation dairy sewage sludge.

Moreover, the calorific values are also affected by the chemical structure of specific fuels. Woo et al. [51] produced fuel pellets from a mixture of waste coffee grounds and pine sawdust. During their research, they found that an increase in the share of coffee grounds in the pellets increased their calorific value due to the high content of C and H in the coffee grounds.

Table 5 and Figure 9 illustrate the influence of the sludge content added to sawdust on the HHV and LHV at a 15%wt moisture content.

Table 5. Influence of post-flotation dairy sewage sludge in a mixture with sawdust.

Post-Flotation Dairy Sludge Content (%)	Higher Heating Value (MJ · kg ⁻¹)	Lower Heating Value (MJ · kg ⁻¹)
	15% Moisture Content	15% Moisture Content
0	20.199	18.541
5	19.850	18.199
10	19.500	17.856
15	19.151	17.514
20	18.802	17.172
25	18.453	16.830

**Figure 9.** Dependencies of the HHV and the LHV of mixtures (at 15% moisture) with sawdust on the content of post-flotation dairy sewage sludge.

The obtained values of the HHV and LHV (Table 5, Figure 9) prove that the addition of post-flotation dairy sludge only slightly reduced the energy values of the produced pellets. For example, the addition of 10% post-flotation dairy sludge to sawdust (total moisture content of 15%) reduced the HHV by ca. 0.698 MJ · kg⁻¹ (3.46%). However, a 10% addition of sludge to sawdust resulted in a reduction of 6.5% in the pelletization energy consumption. Hence, the addition of sludge had a beneficial effect on the pellet quality and production parameters. The importance of using post-flotation dairy sludge also has a positive aspect concerning waste disposal management; rather than directing the sludge to costly purifying facilities, it can be forwarded to pelletization units.

3.4. Exhaust Composition during Combustion

Table 6 presents the combustion effects (exhaust gas composition) obtained during the combustion of sawdust pellets and pellets made of mixtures of sawdust with the addition of post-flotation dairy sludge (10, 15, or 20%).

During the combustion of pellets, high temperatures of ca. 200 °C were observed in the boiler flue. This is a higher temperature than that obtained for the combustion of industrial pellets in the same boiler installation with similar thermal and flow conditions [52]. The above phenomenon proves the favorable influence of the fuel used on the temperature of the boiler flue gases.

Table 6. Composition of exhaust gases from the combustion of pellets made of sawdust and pellets made of mixtures of wood sawdust with the addition of post-flotation dairy sludge.

Parameter	Wood Pellets	Wood Pellets +10% Post-Flotation Dairy Sludge	Wood Pellets +15% Post-Flotation Dairy Sludge	Wood Pellets +20% Post-Flotation Dairy Sludge	Ecodesign
	10% O ₂ in the Exhaust				
CO ₂ (%)	8.68	7.99	7.95	8.05	
CO (mg · Nm ⁻³)	337.21	403.92	407.25	420.36	500
NO (mg · Nm ⁻³)	172.80	198.83	191.13	196.29	200
SO ₂ (mg · Nm ⁻³)	14.76	16.46	21.52	23.91	
HCl (mg · Nm ⁻³)	3.03	16.04	15.87	16.65	
λ (-)	2.43	2.60	2.62	2.58	
Temperature in boiler outlet (°C)	170	200	200	200	

In the case of all analyzed pellets, the emission of carbon monoxide and nitrogen oxide was achieved at a level that is acceptable by the EU Ecodesign Directive, i.e., below 500 and 200 mg · Nm⁻³ of flue gas, respectively [53]. In the tested biomass combustion conditions, the main source of nitrogen oxides was nitrogen contained in the fuel, due to combustion temperatures below 1300 °C. Therefore, it was assumed that the Zeldowicz reaction did not take place and that due to the high λ coefficient, the so-called Prompt NO, i.e., combustion of atmospheric N₂ and hydrocarbons in a rich mixture, did not occur either [54]. The excess air coefficient, λ, for combustion in all cases of pellets with an addition of sludge ranked as 2.58–2.62 and was at a level similar to that for the combustion of sawdust pellets without any additions (2.43). The level obtained was acceptable for the combustion unit where the test was performed (fixed rotary bed boiler, fifth class).

Chlorine contained in biomass is mostly released during biomass combustion in the form of hydrogen chloride, HCl (at temperatures of up to 500 °C), or potassium chloride (at temperatures from 700 to 900 °C) [55,56], which may further react with other components of the exhaust gas, creating dioxins. Due to the subsequent cooling of the exhaust outside the combustion chamber, a large part of the chlorine condensed as salts on the surfaces of the heat exchanger or on the fly ash particles in the flue gas, which may have caused the high-temperature chloride corrosion of the boiler system components. The Ecodesign Directive does not specify the maximum amount of HCl contained in the flue gases from combustion in low-power boilers; however, some German standards limit the accepted value of HCl emissions to below 5 mg · Nm⁻³.

The values of HCl contained in the flue gas from the combustion of sawdust pellets with the addition of post-flotation dairy sludge obtained during the tests ranged from 16.04 mg · Nm⁻³ (with a 10% sludge addition) to 16.65 mg · Nm⁻³ (with a 20% addition of sludge), and proved that these restrictive standards were very slightly exceeded. Trace amounts of HCl in the exhaust did not differ significantly depending on the mixture combusted. Hence, hypothetically, it may have also been related to other elements that were present in the sediments, such as potassium K. The K content has been investigated further to enrich the results' discussion, and it was found to be ca. 0.29%wt., where wood was reported to have an average of 0.05% [57]. High combustion temperatures and high Cl/K ratios may allow for the creation of salts such as KCl and then limit the HCl emission [58], which might explain the stable amount of HCl in the exhaust when increasing the sludge content in the feedstock.

The emission of sulfur oxides depends mainly on the sulfur content of the combusted fuel. Sulfur contained in biomass during combustion, is oxidized mainly to sulfur oxide (IV) SO₂ (and, in small amounts, also to sulfur oxide (VI) SO₃) and forms alkali and sulfates. The importance of sulfur does not arise mainly from SO_x emissions, but from its role in corrosive processes. High concentrations of SO_x in the flue gas contribute to the sulphation

of alkali and alkali metal chlorides, lowering the flue gas temperature, which leads to the release of chlorine [59].

The values of SO₂ contained in the combustion gases from sawdust pellets with the addition of post-flotation dairy sludge ranged from 16.46 mg · m⁻³ (with a 10% sludge addition) to 23.91 mg · m⁻³ (with a 20% addition of sludge). The sulfur content in the sludge was meaningfully higher than in sawdust; therefore, the addition of sludge contributed to the increase in the emissions of SO_x. Due to the low contents (10–20%wt) of sludge in the pelletized mixture, the emission rates were found at low levels.

The addition of post-flotation dairy sludge to sawdust did not adversely affect the combustion effects of the produced pellets. The emission of harmful compounds such as CO, NO, SO₂, and HCl, which were obtained during the sawdust and sludge pellet combustion tests, remained at a level similar to that of the pellet combustion without the addition of sludge. The calculated excess air coefficient was oscillating at a level of 2.6, and therefore was similar to the case of sawdust pellet combustion. This indicated favorable conditions for fuel particle contact with the oxidizing agent (air) in the combustion chamber, and thus confirmed the efficient pellet combustion process.

4. Conclusions

The addition of post-flotation dairy sludge to sawdust had a beneficial influence on the production of fuel pellets by decreasing the pelletization process energy consumption and improving the pellets' kinetic durability. The obtained values of the pellets' density at each level of sludge addition allowed us to conclude that the obtained pellets had good market quality and constituted a full-fledged, innovative solid fuel in accordance with the guidelines of the latest, currently applicable ISO 17225 standard. In addition, the pelletization process of sawdust and post-flotation sludge could be further developed by using a pelletizer with a higher capacity, i.e., the ring-matrix technology and/or by using different constructions of the compacting die and rolls.

The addition of post-flotation dairy sludge only slightly reduced the energy values of the produced pellets and did not have a negative impact on the combustion effects of the pellets produced with its addition. The emission of the harmful compounds CO, NO, SO₂, and HCl released during the pellet combustion tests remained at a level similar to the pellet combustion without the addition of sludge. The excess air coefficient obtained during the tests was also maintained at a level similar to the levels for pellet combustion without the addition of sludge. This proved that the conditions of fuel contact with air in the combustion chamber were favorable, and thus, this confirms the effective pellet combustion process.

An additional positive aspect of using post-flotation dairy sludge as a component of the resulting solid fuel is the possibility of managing large amounts of post-production wastes generated in dairy processing plants. Their management (as used in the production of pellets) on a larger scale is part of the currently promoted EU policy on waste management, which is based on three principles, including: preventing waste generation, recycling and reusing waste, and improving the final disposal and monitoring of waste. The obtained data indicated that dairy plants could improve their circular economy systems by applying post-flotation sludge to the production of fuel pellets, which could be a win–win situation in terms of waste management and decreasing the energy consumption in pellet production.

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