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Toward New Value-Added Products Made from Anaerobic Digestate: Part 1—Study on the Effect of Moisture Content on the Densification of Solid Digestate

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Abstract: Anaerobic digestion (AD) is widely used for the sustainable treatment of biological wastes and the production of biogas. Its byproduct, digestate, is a valuable organic waste and needs appropriate management, which is one of the major concerns with a negative impact on the efficiency of biogas installations. One approach to extend the utilization of digestate as well as improve its handling and storage characteristics is compaction into pellets. This study aimed to evaluate the behavior of digestate during cyclic loading and unloading in a closed matrix. The findings presented here may provide insights into the mechanisms of pellet formation for optimizing the production of pellets and improving their sustainable management. The study can be considered novel as it applied cyclic loading, for the first time, in view of densification modeling and pelleting prediction. A Zwick universal machine was used in the experiments. The moisture content of digestate was found to be 10–22%. Samples were loaded with a constant amplitude of 20 kN for 10 cycles. The distribution of energy inputs, including the total energy, energy of permanent deformations, and energy lost to elastic ones, was thoroughly evaluated. A decrease in the total loading energy was observed in the first cycle, in cycles 2–10, and after all 10 applied cycles due to the rise in the moisture content of digestate. Similar relations were also found for the nonrecoverable energy part. In subsequent cycles of loading/unloading, the values of total energy and permanent deformation energy fell asymptotically. One of the most noteworthy findings of the study was that the absolute values of elastic deformation energy were consistent across all the cycles and moisture levels. However, it was noted that the percentage of energy dissipated to elastic deformation in all cycles significantly increased as the moisture content increased. Loading, which contributed to elastic deformations, was identified as the key factor causing an increase in cumulative energy inputs, and the majority of the energy expended was dissipated. Dissipated energy was the only component that permanently altered the total energy required for compaction. Another important finding, which resulted from the analysis of successive courses of loading and unloading curves, was that the shape of the areas enclosed between the loading/unloading curves was significantly influenced by the moisture content of the digestate.

Keywords: digestate; cyclic loading; biofuel; pelleting; confined compression

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1. Introduction

Biogas plants are important sources of renewable energy. The sequence of fermentation processes taking place in these plants results in the breakdown of biodegradable materials in the absence of oxygen (i.e., anaerobic digestion (AD)). AD is a practical way to treat animal and agricultural wastes, crop plants, municipal organic wastes, sewage sludge released from aerobic wastewater treatment plants, industrial wastes and wastewaters, food wastes, microalgae, and many others.

The operation of a biogas plant requires a large amount of energy. The net energy output can turn negative when substrates are transported along long distances [1]. The most promising areas of research are improving the efficiency of biogas plants, which are based on the following: (a) feedstock optimization—developing methods to improve the quality and consistency of feedstocks, utilizing a wider range of substrates, such as agro-industrial waste, food waste, and algae [2–4]; (b) process optimization—improving the efficiency of AD process and energy recovery, by controlling pH, temperature, and nutrient levels, and developing advanced techniques such as thermophilic digestion [5–7]; (c) biogas upgrade—developing more efficient methods of purification and upgrading biogas to produce biomethane of a higher quality [4,8]; (d) economic and policy analysis—analyzing economic and policy factors that affect the growth and development of the biogas industry, including barriers to investment and adoption which should be identified and addressed [9,10]; and (e) life cycle analysis—conducting more comprehensive life cycle assessments to better understand the environmental impact of biogas production and utilization [11–13].

Digestate, which is the byproduct of AD, is a valuable organic waste and needs proper management as it can affect the efficiency of biogas installations. Some researchers indicate that the problem of energy and material recovery is only partially resolved by AD because the efficiency of organic matter conversion by this process is usually in the range of 13–65% [14]. On the other hand, the demand for digestate-derived goods can boost the financial and net environmental value of AD, and these goods are also a source of money [15,16].

Over the past decade, there has been increasing research on the management of anaerobic digestate. Researchers believe the AD process is more economically viable and digestate can be valorized into new high-value products [17,18]. The possibilities of digestate utilization were recently highlighted in several studies [18–23]. Anaerobic digestate can be potentially used (a) as an organic fertilizer for crops and a soil conditioner as it contains high levels of nitrogen, phosphorus, and potassium [24,25]; (b) as a solid biofuel for heat or electricity generation [1,26]; (c) for nutrient recovery—techniques for recovering nutrients from digestate are being rapidly developed with the aim of improving nutrient management in agriculture and waste treatment systems [27–30]; (d) for conversion into added-value products such as biochar [31,32], activated carbons [33], or composite materials [3,34]; (e) for composting and land application due to its high organic matter content [35]; and (f) as a substrate for the cultivation of special products such as microalgae [36,37].

One approach to extend the utilization of digestate and improve its handling and storage characteristics is compaction into pellets. The production of pellets from digestate has been recognized as an appropriate way to valorize this material and convert it into a valuable source of nutrients [38,39] and energy [1,16,40–43], reducing its environmental impact [44]. The main features that favor the use of digestate for pellet production is its easy availability and sustainability. Digestate pelleting could improve the storability of fertilizers and allow nutrients to be easily mobilized over time, making them a potential source of mineral nutrients for long-term applications [39]. Pulvirenti et al. [44] found that pelleted digestate had a lower pH and a higher content of mineral nutrients. In order to be effective, land application should be performed on time. In this regard, pelletization can enhance the quality of fertilizers, simplify their storage and transportation processes, and lower their costs. Nagy et al. [41] demonstrated that the use of digestate as combustible pellets was more cost-effective than as fertilizer. According to Czekała et al. [16], digestate is a more profitable substrate for producing biofuels compared to sawdust. These authors also demonstrated that digestate retained some energy potential and can be a highly valuable substrate for the production of solid biofuels. The calorific value of solid biofuel derived from digestate is comparable to that of biofuel derived from sawdust or wood, and digestate pellets can be burned using combustion technologies that are already available [26]. Cathcart et al. [1] underline that the availability of substrate can influence production efficiency due to high transportation costs. Nagy et al. [41] emphasize that pellet production should be carried out near a biogas plant in order to make the best use of generated heat for digestate dehydration. Cathcart et al. [1] also point out that, although there are studies evaluating the efficiency of pellet production for energy purposes, many of them are fragmentary.

The possibility of the pelletization of anaerobic digestate for obtaining novel value-added products depends on its susceptibility to densification. The susceptibility of digestate can be determined using direct measurements or experimental or analytical models. In general, researchers and industry practitioners agree that recommendations concerning optimal pelleting conditions are valid only in very limited and specific circumstances. This applies to all biomass resources, and digestate cannot be an exception. One of the parameters that can be optimized is moisture content. Mostafa et al. summarized that the optimal moisture content for obtaining high-quality pellets from woody and biomass materials is 5–28% [45]. This can be related to the high dependency of pelletization process on the properties of processed biomaterials, which are primarily influenced by moisture [46–50]. Thus, the high variability of the physical and chemical properties of anaerobic digestate, which is due to the different substrates used in the AD process and the technologies applied, justifies the need for more universal models. These models should provide a common basis for interpreting and comparing the results for various materials and research conditions, for example, models describing the changes in density under the influence of applied pressure. For developing such a model, research methods that enable the identification and measurement of phenomena occurring during densification are required. Recently, such models were presented for biomass [51,52]. Although the literature on densification modeling dates back to the 1930s, due to the high complexity of the mechanisms occurring during biomass densification, one cannot find a model that is accurate and sensitive enough for interpreting most of the associated phenomena and their translation to practical engineering. There is also no universal method for evaluating the behavior of materials during densification. Therefore, the comprehension and determination of quantitative relationships in the digestate densification process require a profound analysis of the impact of various factors such as the mechanical and flow properties of solid digestate, moisture, and pressure. The need for further research in this field, specifically in the context of digestate or biomass, is highlighted by the limited number of studies.

Uniaxial compression is the most commonly employed procedure to study densification, and researchers mainly focus on the loading phase. Studies describing the behavior of the material during successive phases of loading and unloading are scarce. The concept of cyclic loading is based on the interaction between the rollers of a pelleting machine and the fact that the material being processed can be considered as a series of loading and unloading cycles within the die opening [53,54]. The idea of the application of cyclic loading to biomass densification or pelletization is quite novel and has not been tested in detail. So far, only the study by Sawicki and Świdziński analyzed the application of cyclic loading for biological materials [55]. Additionally, the authors' previous work [56], in which a hysteresis test (one loading and unloading cycle) was utilized to predict the pelletizing process, is the only successful attempt to date.

The objective of the present study was to investigate the influence of the moisture content of digestate on its behavior during cyclic confined compression. This investigation was motivated by the significance of anaerobic digestate management in the context of sustainable resource utilization. The research material was the solid by-product of the AD process. The study tested a novel approach of cyclic loading and unloading, with a focus on the comprehensive analysis of energy and deformation distribution, for more accurate identification and interpretation of the effect of moisture content on digestate densification.

2. Materials and Methods

The scheme of the experiments performed in the study is presented in Figure 1.

2.1. Collection of Solid Anaerobic Digestate

Solid anaerobic digestate was obtained from a biogas facility in Międzyrzec Podlaski owned by BioPower Sp. z o.o. The principal substrate used in the biogas plant was maize silage (70%), with a dry matter (DM) content of about 35%. The liquid substrate (30%) was molasses with 7% DM, obtained from a local distillery.

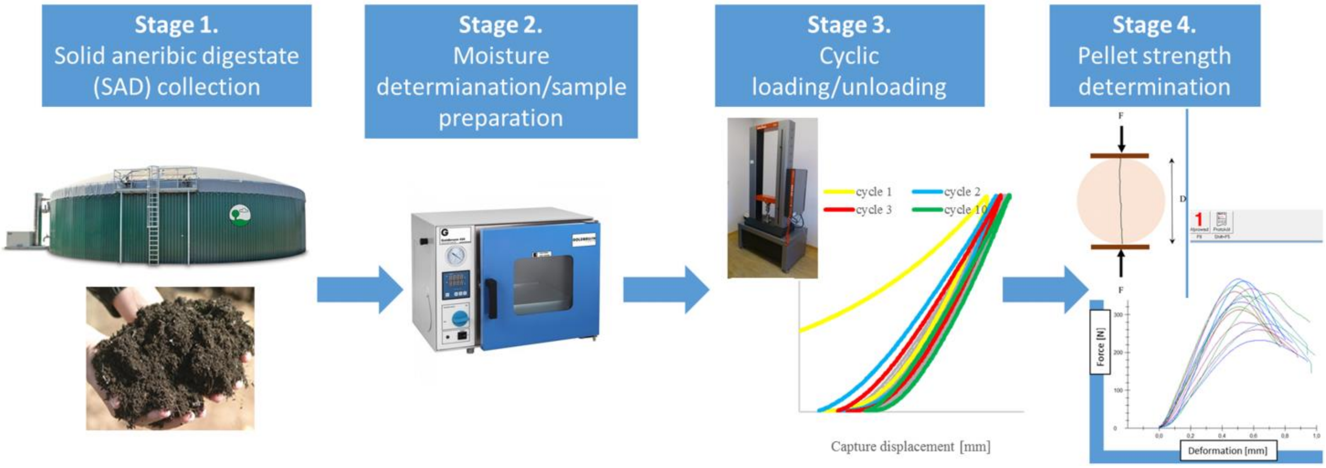

Figure 1. Stages of experiments.

2.2. Moisture Determination/Sample Preparation

The initial moisture of a digestate batch was determined using the air-oven method. Then, three samples, each weighing 5 g, were taken from the batch and dried at 105 °C for 1 h. Then, five samples with different levels of moisture (10, 13, 16, 19, and 22% (wet basis), respectively) were acquired from the batch. The mentioned levels of moisture are ideal for pelleting a variety of biomaterials [45]. The amount of water added/removed was calculated using simple mass balance equations [46]. The samples were cooled for 24 h to equilibrate the water content. The moisture content of the samples was determined again prior to compression experiments.

2.3. Cyclic Loading/Unloading

A Zwick Z020/TN2S universal testing machine (Zwick GmbH, Ulm, Germany) was used in the experiments. Figure 2a presents a scheme of the compression assembly. The diameter of the chamber was 15 mm.

A 2 g sample of digestate was placed in a closed matrix and axially loaded to achieve a constant load of 20 kN. This loading phase was followed by an unloading phase. When the loading force reached 2 N, another loading/unloading cycle began. The cycle number was set to 10. During both loading and unloading, a compression rate of 10 mm min^{-1} was maintained. All the operating parameters were controlled using Zwick's testXpert software. Data were collected from the software at a frequency of 10 Hz. For each moisture content, experiments were carried out in triplicate.

Based on the recorded load–displacement dependencies, the total compressive energy E_t, as well as the values of reversible (elastic) energy E_r and irreversible (permanent) energy $E_{n\text{-}r}$, were calculated (Figure 2b). In addition, the corresponding values of head displacements, including total displacements l_t, elastic displacements l_r, and permanent displacements $l_{n\text{-}r}$, were determined. The relative shares of reversible and irreversible energy and displacements were also estimated ($\%E_r$, $\%E_{n\text{-}r}$, $\%l_r$, and $\%l_{n\text{-}r}$).

The parameters mentioned above were determined for cycle 1, cycles 2–10, and after 10 cycles, and recorded with cycle numbers (e.g., E_{t1}, $E_{t2\text{–}10}$, E_{t10}, l_{t1}, $l_{t2\text{–}10}$, l_{t10}).

For an ideal plastic body, $E_r = 0$, and when $E_{n\text{-}r} = 0$, the material is only elastically deformed. The material is said to be viscoelastic–plastic at intermediate values. The ductility of a material is determined by the relationships between its components [57,58].

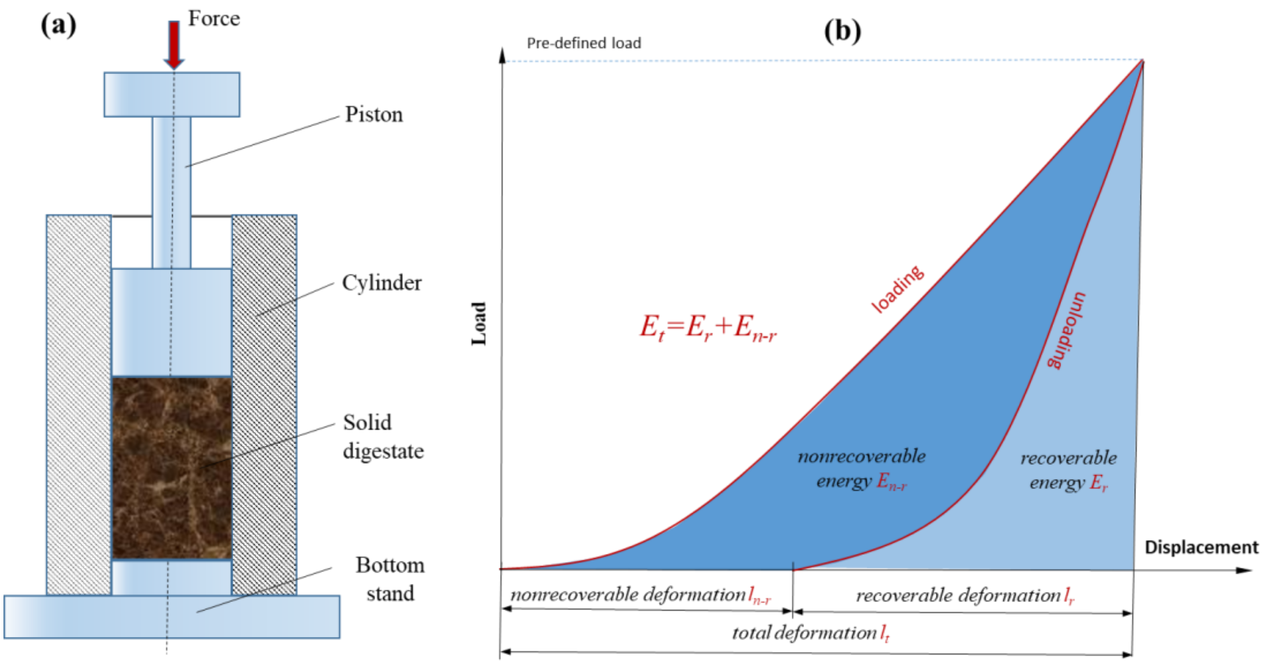

Figure 2. Scheme of the compression assembly (**a**) and a typical strain hysteresis curve for one loading/unloading cycle (**b**).

2.4. Pellet Strength Determination

At the end of loading/unloading experiments, compacted capsules were obtained. After the capsules left the compaction chamber, their dimensions were measured with a digital caliper (accuracy of 0.01 mm). The capsules were then stored for 24 h in a refrigerator. After 24 h, their dimensions were measured again. Subsequently, the strength of capsules was established using the same procedure as the Brazilian indirect tensile test [59,60]. The individual capsule was loaded perpendicularly to its longitudinal axis using the Zwick universal machine. The compression rate was adjusted to 10 mm min^{-1}. The ultimate load related to the capsule length was determined as the index of pellet strength (SI), according to the equation:

$$SI = \frac{2F_{max}}{\pi d l_{24}} \text{ [MPa]} \tag{1}$$

where F_{max} corresponds to the force at the ultimate fracture point, l_{24} is the pellet length after 24 h, and d is the diameter of the capsule [58,59].

Measurements were performed for each capsule representing each individual experiment.

3. Results and Discussion

Figure 3 presents the experimental loading/unloading characteristic of digestate (10 cycles). The capsule compacted in the first cycle was successively compressed in cycles 2–10. As the first loading cycle actually represents the compaction of bulk digestate, it differs markedly from the subsequent cycles. Therefore, the digestate behavior was analyzed separately for the first cycle and the subsequent nine cycles.

3.1. Effect of Moisture on Energies and Deformations—First Cycle (Compaction)

Figure 4a shows the total energy inputs for the first loading phase as well as the proportion of recoverable and nonrecoverable components. With the increase in the moisture content of digestate, the total energy declined. A linear relationship with a strong determination coefficient of 0.992 was found. An analogous tendency was also observed for the nonrecoverable portion of energy (effective compaction work). The rate of decline was almost identical to the total energy inputs (–2.66 vs. –2.63 Nm per unit of moisture), and the correlation was also equally strong (0.993). The elastic energy ranged from 13.18 to

13.59 Nm. No evidence of the influence of moisture was found in this case. The relationship was not statistically significant at α = 0.05, and the determination coefficient was low (0.476). The testing of the average homogeneity hypotheses revealed no discernible effect of moisture content on this parameter.

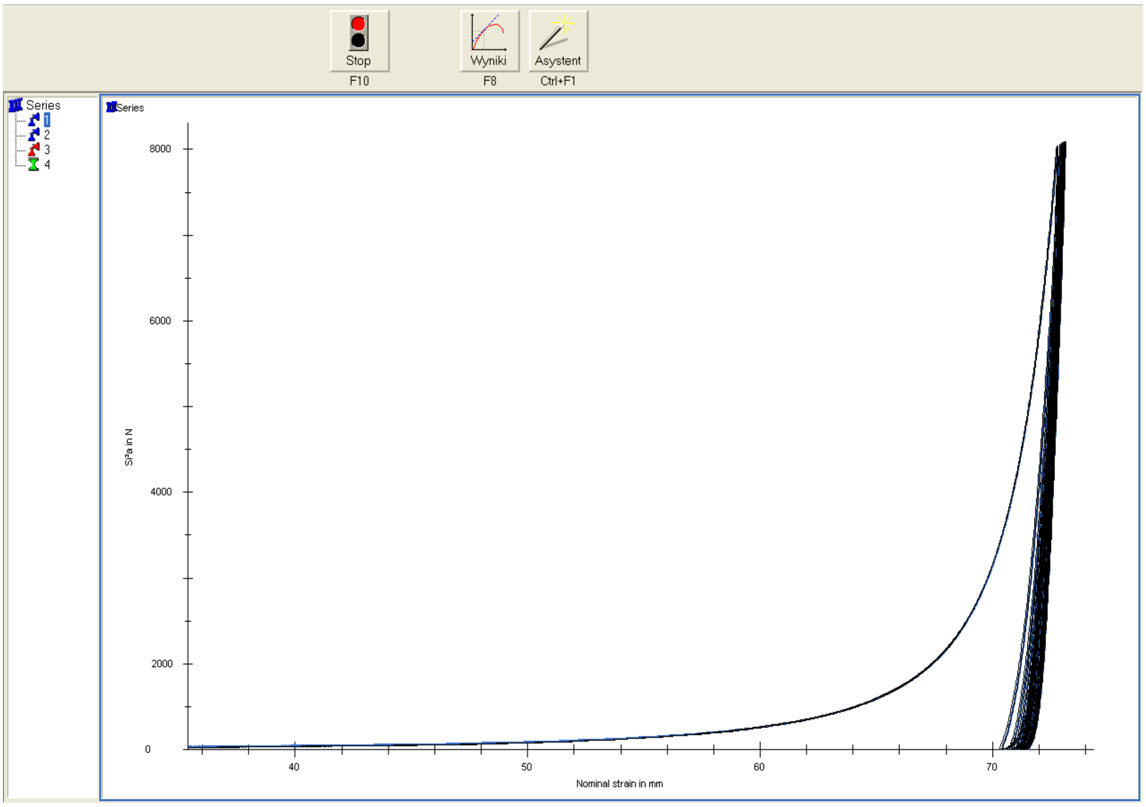

Figure 3. An example of cyclic loading/unloading characteristic of digestate.

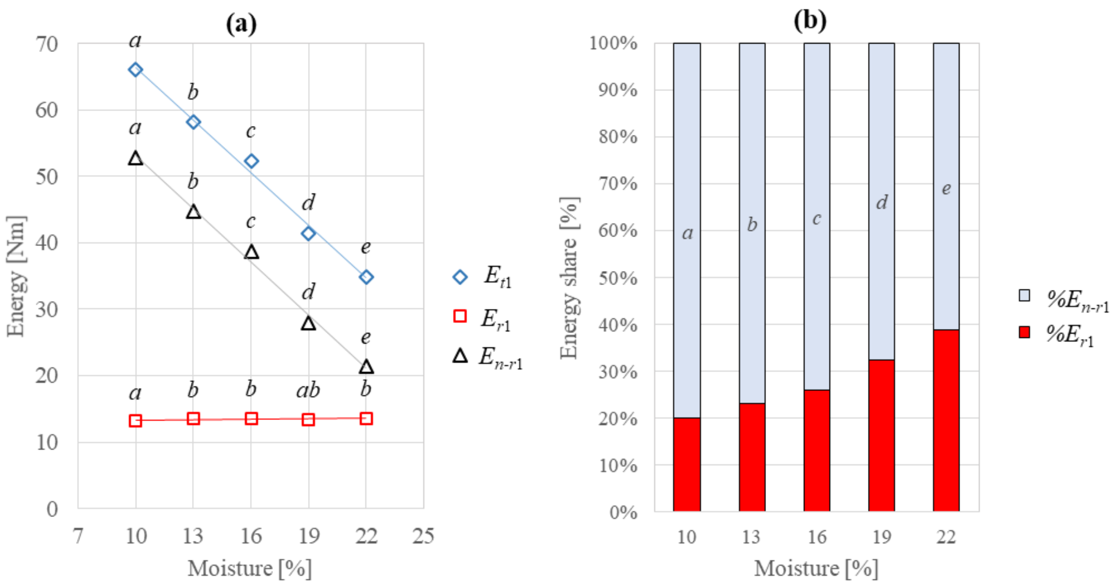

Figure 4. Effect of digestate moisture on energy distribution during the first loading/unloading cycle (**a**) and shares of recoverable end nonrecoverable energies (**b**): *a*–*e* represent homogeneous groups at α = 0.05.

Figure 4b shows the proportion of recoverable and nonrecoverable energy. An increase in the share of the elastic component from roughly 20 to 39% was observed in relation to moisture. This indicates that the permanent energy declined accordingly. As a result, more elasticity was noted in the material with more moisture, and less amount of energy was consumed for capsule formation. Significant differences were found between averages as confirmed by post hoc average comparisons.

In the first cycle, the bulk material was compacted, with large differences in reversible and irreversible displacement values. Due to the complexity of graphical representation, the distribution of displacements for the first cycle is presented as a table (Table 1).

Table 1. Effect of digestate moisture on deformations during the first loading/unloading cycle.

Moisture	l_{t1}	l_{r1}	$l_{n\text{-}r1}$	$\%l_{r1}$	$\%l_{n\text{-}r1}$
%	mm	mm	mm	%	%
10	70.13 ^{a}	2.22 ^{a}	67.90 ^{a}	3.17 ^{a}	96.83 ^{a}
13	63.61 ^{ab}	2.27 ^{ab}	61.34 ^{ab}	3.58 ^{ab}	96.42 ^{ab}
16	62.94 ^{ab}	2.35 ^{bc}	60.59 ^{ab}	3.74 ^{bc}	96.26 ^{bc}
19	58.79 ^{b}	2.40 ^{c}	56.40 ^{b}	4.09 ^{c}	95.91 ^{c}
22	62.14 ^{ab}	2.60 ^{d}	59.54 ^{ab}	4.19 ^{c}	95.81 ^{c}
Average	63.52	2.37	61.15	3.76	96.24

$^{a\text{–}d}$ Homogeneous groups at $\alpha = 0.05$.

The total displacement required to achieve the applied level of load (20 kN) was 63.52 mm, while the reversible and nonreversible deformations were 2.37 and 61.15 mm, respectively. The highest total and nonreversible deformation values were observed for samples with the least moisture content, and the values tended to decrease slightly as the moisture levels increased. Elastic deformations, on the other hand, were higher for samples with more moisture, ranging from about 2.22 mm for 10% moisture to 2.60 mm for 22% moisture. A similar trend was noted for the proportion of elastic and permanent deformations. The share of reversible deformation increased linearly with the increase in moisture from 3.17 to 4.19% ($R^2 = 0.965$). Statistical analyses indicated that moisture had very little effect on total deformations, as can be observed in Figure 5.

When the wet material was compressed, the loading force increased faster with smaller head displacements. The curve's inflection point for wet material was noted at much smaller displacements. As a result, the compression energy inputs (the area under the load–displacement curve) of wet digestate were lower. Frodeson et al. [61] demonstrated that the level of load and chemical composition (material properties) can influence the effect of moisture content. Such an influence was not visible at loads up to 5 kN. However, compression time and, thus, energy input decreased as the moisture content increased. The experiments indicated that an increase in moisture caused a reduction in the deformation of digestate, even at low loads.

Studies investigating the use of various biomaterials for biofuel production [62,63] show that the density of agglomerate in the closed die increases with moisture content. Kulig et al. [64] discovered for pea straw that agglomeration with a lower moisture content was characterized by a higher density and degree of densification. As density is calculated on the basis of deformations, it can be related to capture displacements. A brief comparison of the curves in Figure 5 reveals that the displacement of capture was nearly independent of moisture. Additionally, substantial unpredictability is indicated by the distribution observed for samples with the same content of moisture. These observations indicate that different findings presented by the authors of the cited studies [62–64] are possible. The aforementioned finding is significant in terms of the methodological aspect of compaction experiments and for the development of compaction models. It appears that variations in the early phases of compaction are responsible for the observed differences in deformations. This hypothesis should be validated by further investigation, particularly on smaller deformations.

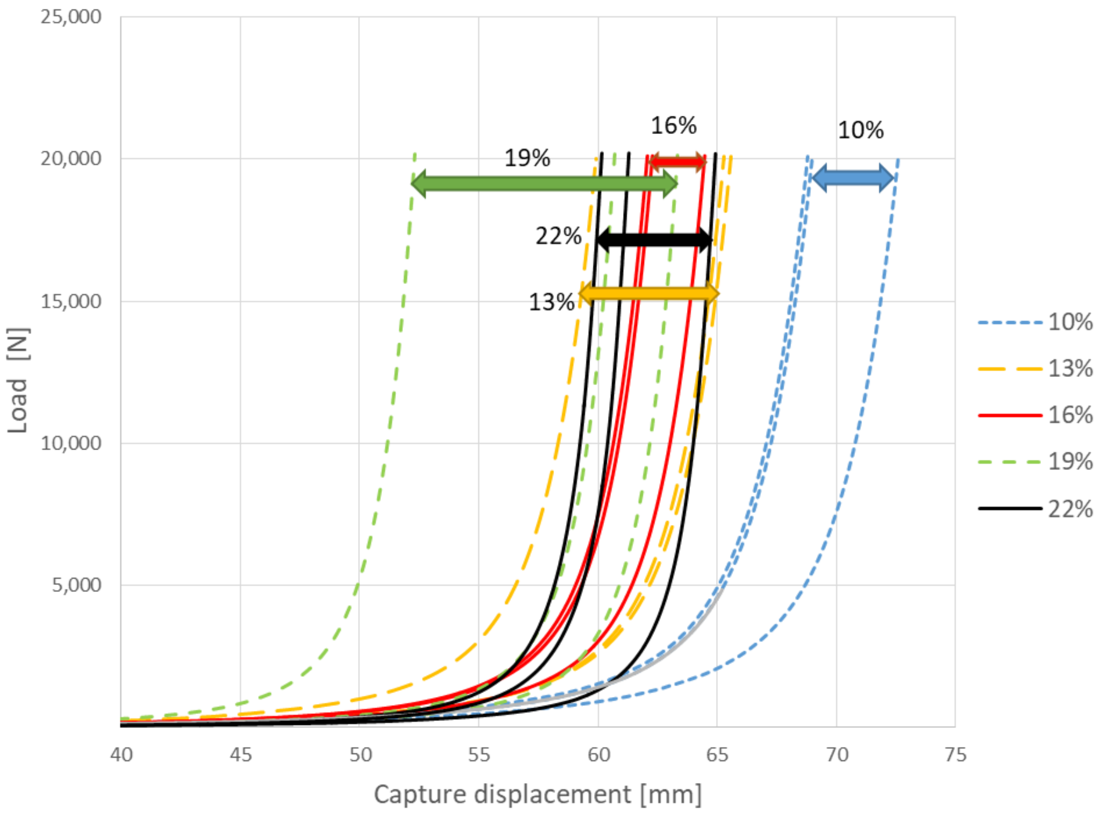

Figure 5. Effect of the moisture content of digestate on its load–displacement characteristics (first cycle, compaction phase).

3.2. Effect of Moisture on Energies and Deformations—Cycles 2–10

Figures 6–8 and Table 2 show the loading/unloading energy inputs and corresponding capture displacements for the successive loading/unloading cycles 2–10.

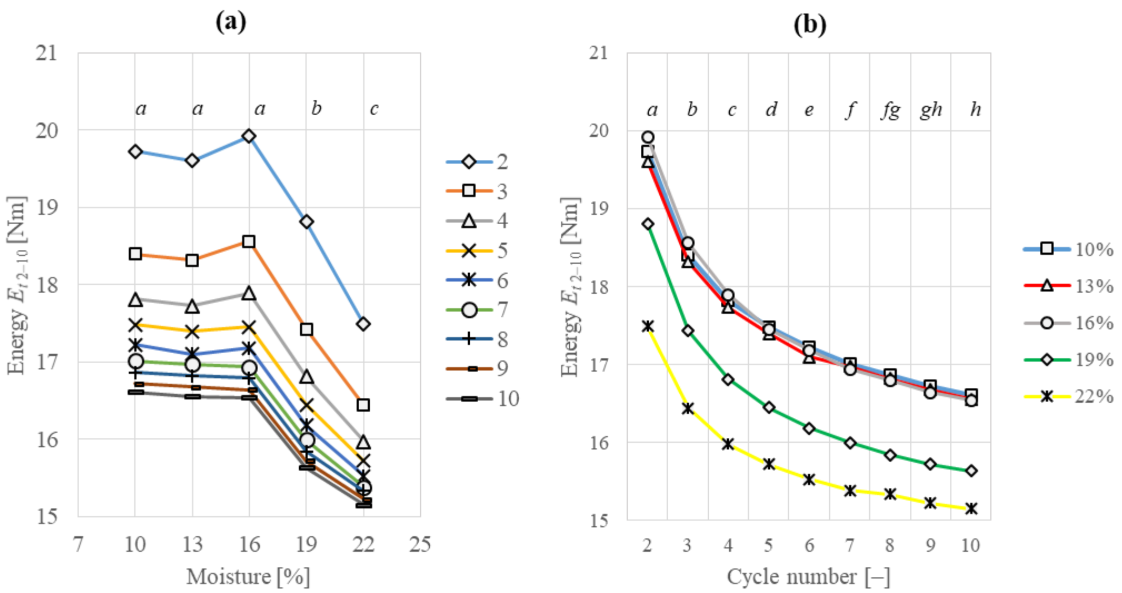

Figure 6. Effect of digestate moisture (**a**) and cycle number (**b**) on loading energy for cycles 2–10: *a*–*h* represent homogeneous groups at α = 0.05.

The total energy loadings did not change significantly for samples with moisture levels of 10, 13, and 16%. However, a clear decrease in energy was noted for samples with higher moisture levels of 19 and 22%. This observation was confirmed by post hoc average comparisons for each of the loading cycles. Three homogeneous groups were distinguished based on the moisture level: 10–16%, 19%, and 22%. Depending on the cycle number, the differences were quite noticeable. The energy consumed in subsequent cycles decreased asymptotically (Figure 6b), which could be associated with the simultaneously decreasing values of head displacements (Table 2). However, there was no decrease in the energy values for moisture levels of 19 and 22%, and the average values of all the analyzed displacements and their percentage shares did not differ significantly at $\alpha = 0.05$.

Table 2 summarizes the effects of moisture and cycle number on the values of capture displacements. Only a slight increase in all three average displacements (total, reversible, and irreversible) was observed with the increase in moisture content. However, for individual cycles, this increase was significantly higher: approximately 12% for the second cycle, 8% for the third, 6% for the fourth, and about 2% for the tenth. The increase in the values of elastic deformation was almost similar to the increase in moisture. Finally, the permanent strain deformations increased more significantly by around 25% in cycle 2 and did not change much after five cycles. For cycles 5–10, no significant effect of moisture on the mean displacement values was observed. On the other hand, the analyzed displacements clearly decreased in the subsequent loading cycles, and the decrease was asymptotic in all cases.

Table 2. Effect of moisture and cycle number on the values of capture displacements.

		Capture Displacement				
		$l_{t2\text{–}10}$	$l_{r2\text{–}10}$	$l_{n\text{-}r2\text{–}10}$	$\%l_{r2\text{–}10}$	$\%l_{n\text{-}r2\text{–}10}$
Moisture (%)	10	2.03 ^{a}	1.96 ^{a}	0.072 ^{a}	96.64 ^{a}	3.36 ^{a}
	13	2.04 ^{a}	1.97 ^{a}	0.069 ^{a}	96.83 ^{a}	3.17 ^{a}
	16	2.06 ^{a}	1.99 ^{a}	0.075 ^{a}	96.64 ^{a}	3.36 ^{a}
	19	2.04 ^{a}	1.96 ^{a}	0.072 ^{a}	96.78 ^{a}	3.22 ^{a}
	22	2.14 ^{a}	2.06 ^{b}	0.081 ^{a}	96.53 ^{a}	3.47 ^{a}
Cycle number (–)	2	2.45 ^{a}	2.17 ^{a}	0.276 ^{a}	88.76 ^{a}	11.24 ^{a}
	3	2.21 ^{b}	2.08 ^{b}	0.125 ^{b}	94.34 ^{b}	5.66 ^{b}
	4	2.10 ^{c}	2.03 ^{c}	0.076 ^{a}	96.38 ^{c}	3.62 ^{c}
	5	2.04 ^{d}	1.9 ^{d}	0.052 ^{d}	97.46 ^{d}	2.54 ^{d}
	6	2.00 ^{de}	1.9 ^{de}	0.039 ^{e}	98.03 ^{e}	1.97 ^{e}
	7	1.97 ^{ef}	1.94 ^{ef}	0.031 ^{e}	98.43 ^{ef}	1.57 ^{ef}
	8	1.94 ^{fg}	1.92 ^{fg}	0.028 ^{ef}	98.58 ^{fg}	1.42 ^{fg}
	9	1.92 ^{g}	1.91 ^{fg}	0.016 ^{f}	99.15 ^{gh}	0.85 ^{gh}
	10	1.91 ^{g}	1.89 ^{g}	0.019 ^{f}	99.02 ^{h}	0.98 ^{h}

$l_{t2\text{–}10}$—total deformation, $l_{r2\text{–}10}$—reversible deformation, $l_{n\text{-}r2\text{–}10}$—nonreversible deformation, $\%l_{r\ 2\text{–}10}$—share of reversible deformation, $\%l_{n\text{-}r2\text{–}10}$—share of nonreversible deformation; $^{a\text{–}h}$ homogeneous groups at $\alpha = 0.05$.

A small decrease in elastic energy with a decrease in moisture content was observed for cycles 2–10. However, the averages only decreased from about 13.95 to 13.35 Nm. This accounts for approximately a 34% decrease in the total energy, with averages decreasing from 17.54 to 15.80. In general, the differences between the means were small, and only the means for 19 and 22% moisture differed statistically from the three others. Similarly, the elastic energy did not change significantly between the cycles.

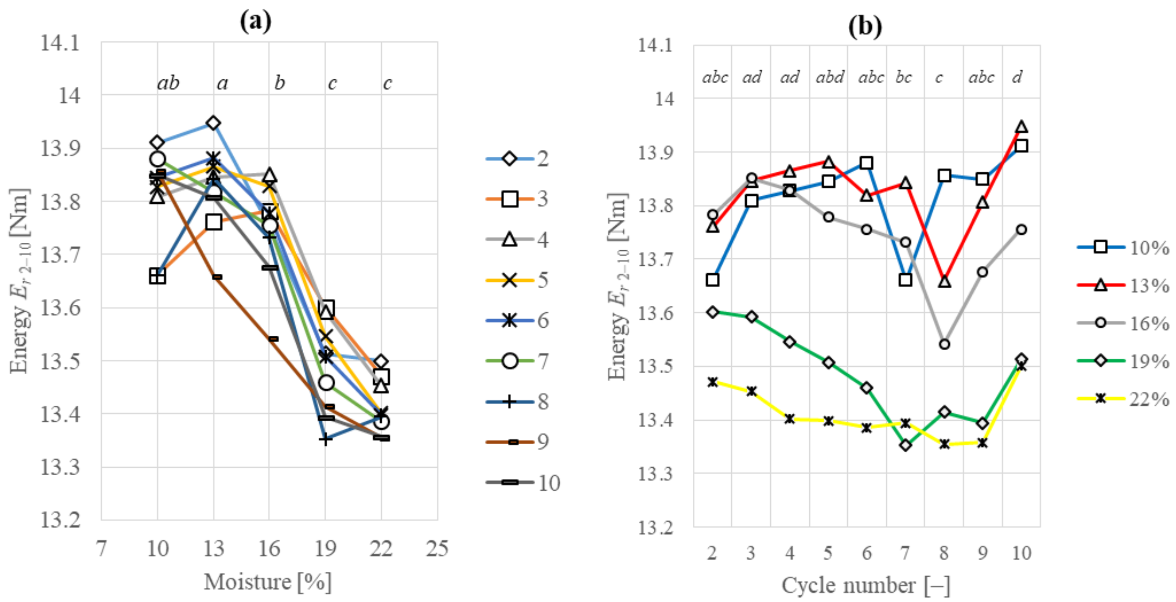

Figure 7. Effect of digestate moisture (**a**) and cycle number (**b**) on recoverable energy for cycles 2–10: *a*–*d* represent homogeneous groups at α = 0.05.

The influence of moisture and cycle number on nonreversible energy inputs in cycles 2–10 was similar to that observed for total energy inputs (Figure 8). The most significant difference was noted for the value order. The average values of this energy were clearly lower than those obtained in cycle 1. Furthermore, this energy was lower in values (unlike in cycle 1) than those of elastic strain energy. The average values decreased from 3.73 to 2.37 Nm in the tested moisture range and from 5.45 to 3.32 Nm in subsequent cycles. It must be noted that the averages were a few times lower than the values observed for the dissipated energy (Figure 7).

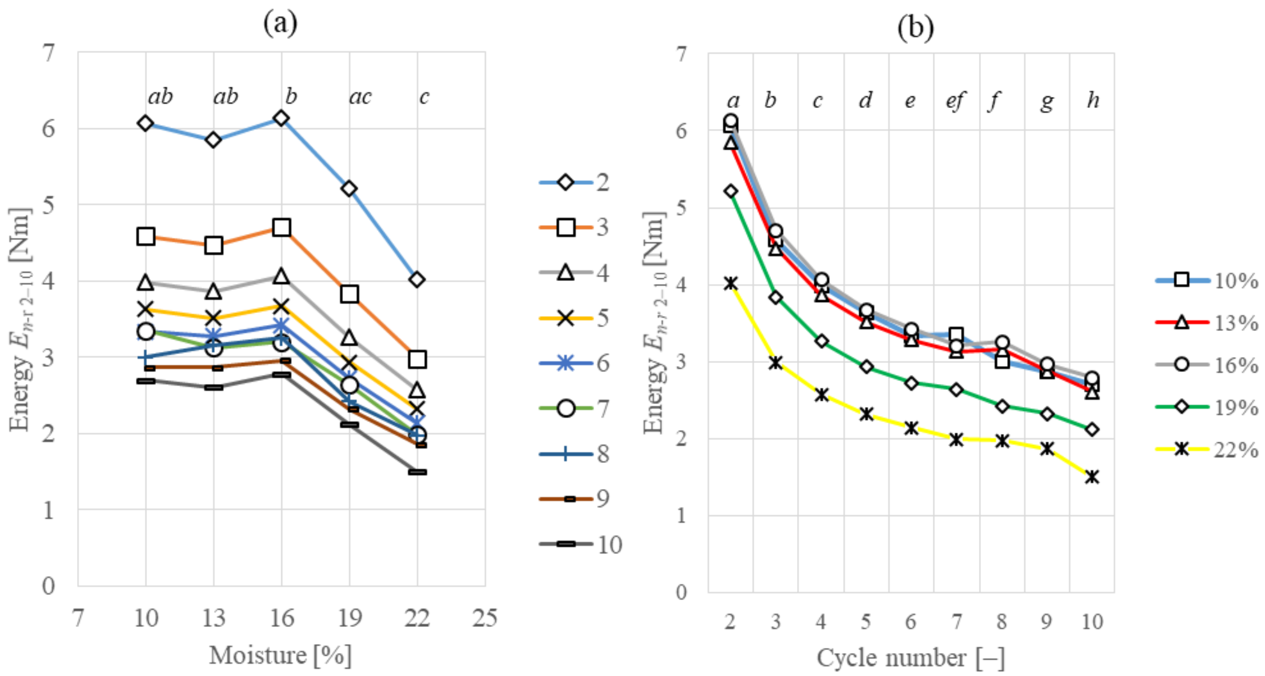

Figure 8. Effect of digestate moisture (**a**) and cycle number (**b**) on nonrecoverable energy for cycles 2–10: *a*–*h* represent homogeneous groups at α = 0.05.

The relationship between the share of reversible and irreversible energy for cycles 2–10 differed from that for cycle 1. A much greater share of irreversible energy was used for compaction in cycle 1, whereas in the subsequent loading cycles, elastic energy constituted the major share of the overall energy (Figure 9).

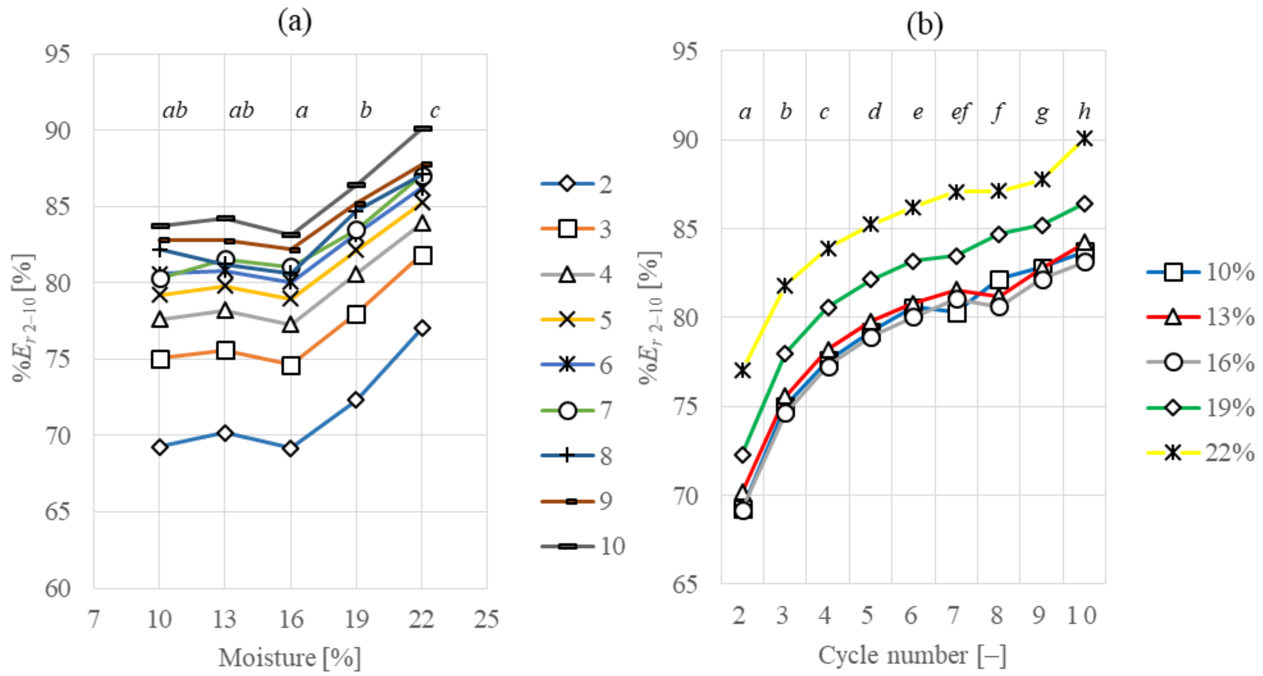

Figure 9. Effect of digestate moisture (**a**) and cycle number (**b**) on the share of recoverable energy for cycles 2–10: *a*–*h* represent homogeneous groups at α = 0.05.

Compared to cycle 1, the influence of moisture content and cycle number on the values of elastic energy obtained was much more evident. The average share of elastic energy was 73% for 10% moisture and increased to 80% for 22% moisture. Depending on the cycle number, the share of this energy ranged from about 71.3 to 85.5%. The share of reversible energy remained stable up to about a 16% moisture level and then increased. This energy also varied significantly depending on the cycle number. For all moisture levels, the increase in this energy with increasing cycle number was clearly asymptotic (logarithmic). This trend was noticeable for all nine loading/unloading cycles. The observed dependence of elastic energy on moisture and the cycle number was obviously opposite in the case of irreversible energy.

Table 2 illustrates the proportion of reversible (elastic) and nonreversible deformations. As can be seen, the ratio of these deformations was not affected by moisture content, while the cycle number had a clear impact. In subsequent cycles, the proportion of reversible displacements increased asymptotically from 88 to 99%. A similar pattern of dependence was noted for each tested moisture.

In summary, the majority of the energy used for sample loading was lost due to elastic deformations during the unloading phase. In succeeding loading/unloading cycles, the percentage share of energy lost increased.

Figure 10 shows examples of the loading/unloading curves for two extreme moisture levels of 10 and 22%. Two differences were the most visible. The first relates to the extent of overall deformation. For the 10% moisture content of digestate, deformation slightly increased in succeeding cycles. On the other hand, displacement remained constant over successive cycles for 22% moisture. The total deformation for cycles 2–10 slightly increased with moisture. The second significant difference was observed in the shape of the area representing permanent deformations. In the region of higher loads, the loading/unloading curves for moist digestate coincided. This implies that the material behaved similarly to an elastic one. The area enclosed by the two curves was clearly distinguishable, while unloading continued below 7–10 kN. However, for dry digestate, the area confined by the loading/unloading curves was more distinctly outlined over the entire unloading range. The energy distribution results suggest that for digestate with the highest moisture level, the values of permanent deformation energy and the percentage share of this energy (the size of the limited field) were the lowest. In cycles 6–10, more than 98% of deformations were reversible, while more than 20% of the energy resulted from permanent deformations. This indicates the existence of the so-called damping capacity, which describes the portion of energy converted to heat and can be defined by the region between the loading/unloading curves. Based on the dependencies observed for energy distribution, it can be concluded that a considerable amount of the energy used in subsequent loading/unloading phases came from elastic deformations. However, a substantial correlation was found between the change in permanent deformation energy and the observed drop in the total energy with rising moisture. This may be related to the surprisingly stable elastic strains that were recorded. The lack of dependence of elastic energy on moisture content and cycle number is an interesting observation that requires confirmation and interpretation. In this sense, additional information can be obtained from an analysis of the effect of load magnitude, which may also verify the above-identified effect of moisture on differences in the course of loading/unloading curves.

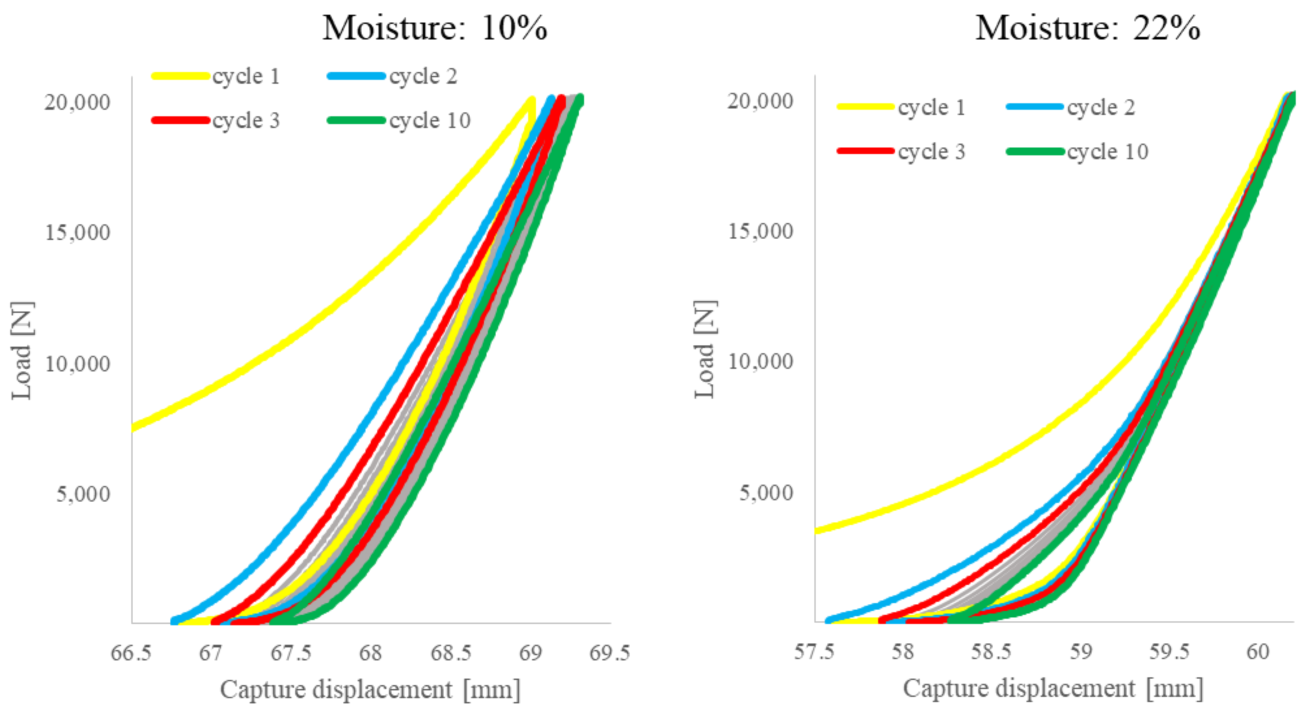

Figure 10. Sequence of loading/unloading cycles for digestate with a moisture content of 10 and 22%.

3.3. Effect of Moisture on Energies and Deformations after 10 Cycles

Figures 11–13 depict the results of the analyses of cumulative energy inputs (sum of all 10 cycles). The total loading energy decreased as moisture content increased. A significant contribution of elastic strain energy was noted. Although the absolute values of this energy did not change significantly with moisture (134.5–137.9 Nm), its relative share of total energy inputs increased from approximately 61% with 10% moisture to 76% with 22% moisture. This may be caused by a lower expenditure of permanent deformation energy, which was observed for digestate with a higher moisture content.

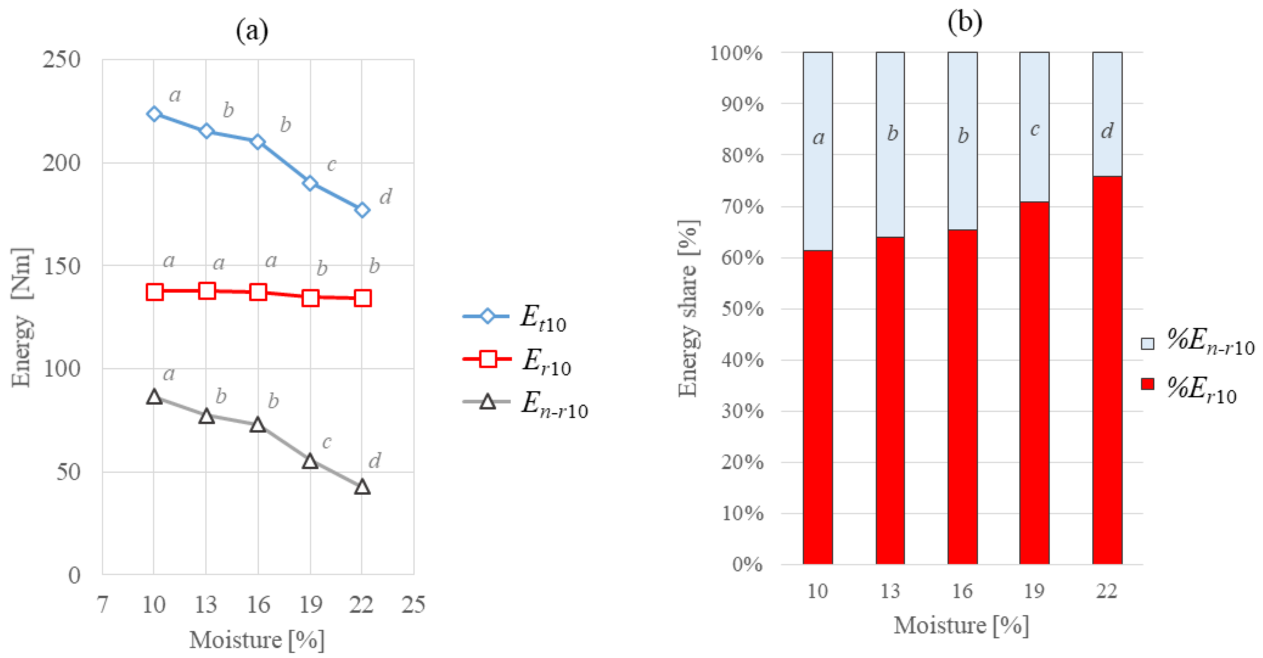

Figure 11. Effect of digestate moisture on energy distribution after 10 loading/unloading cycles (**a**) and shares of recoverable and nonrecoverable energies (**b**): *a*–*d* represent homogeneous groups at α = 0.05.

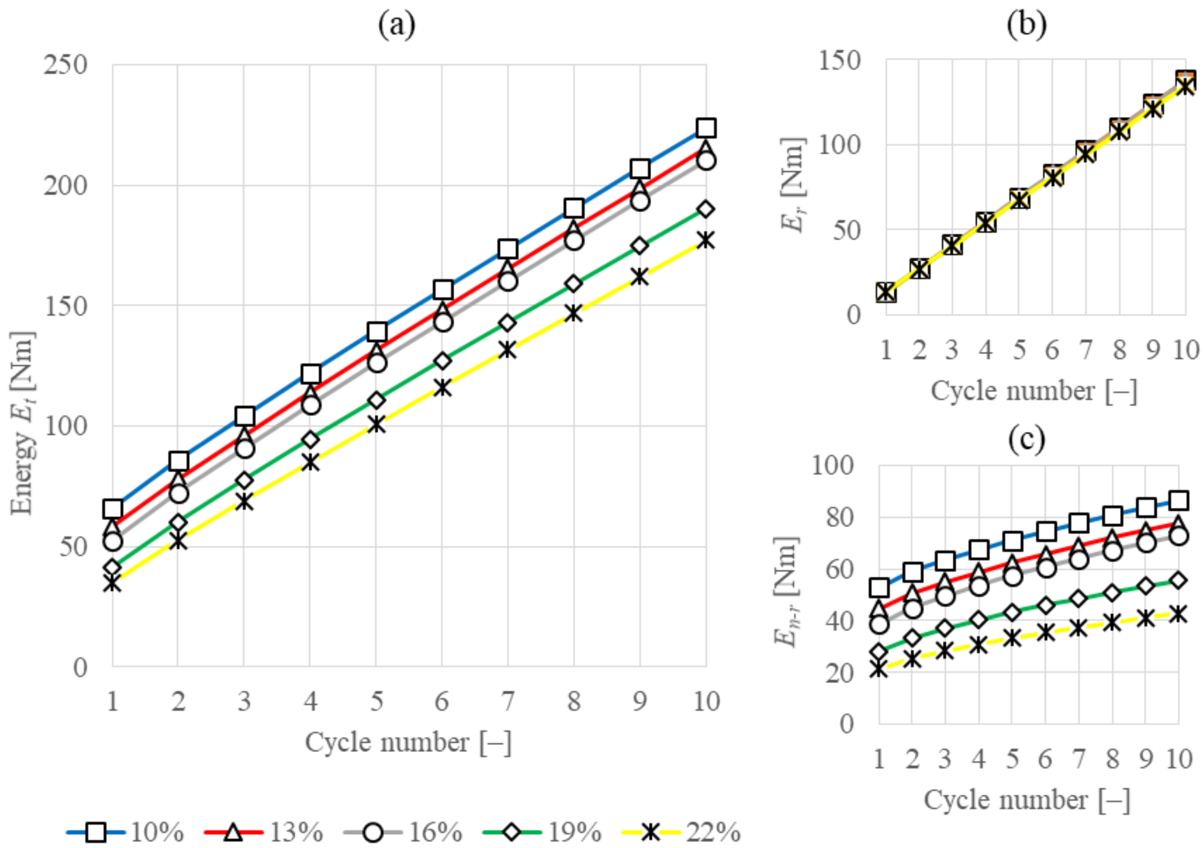

Figure 12. Dependence of cumulative energies on cycle number for various levels of digestate moisture: (**a**) total, (**b**) recoverable, and (**c**) nonrecoverable.

Figure 11 depicts the values of accumulated energy in subsequent loading/unloading cycles. The value of this energy increased linearly with the number of cycles for all levels of moisture. The rate of this increase was almost equal.

Figure 12b,c show the cumulative energies used for elastic and permanent deformations in successive cycles. It was found that moisture had no effect on elastic energy inputs. The curves corresponding to the dependence of elastic energy on the number of cycles nearly coincided. The slope of linear regressions only slightly varied from 13.4 to 13.8. On the other hand, moisture content had a significant influence on the energy of permanent deformations, as the values of this energy clearly decreased when the moisture content of digestate was higher. The values of the coefficient representing the slope of linear regression decreased from around 3.68 for 10% moisture to about 2.30 for 22% moisture. In cases where similar trends were observed, the moisture content did not interact with the number of cycles.

Figure 13 shows the average share of individual energies in the loading/unloading cycle. It should be noted that the proportion of elastic energy in the total energy input increased significantly (around 12 times). The value of this energy was greater than the energy required for agglomerate formation, which began from the third cycle. This implies that pressing energy should be optimized by minimizing the energy lost due to reversible deformation. In practice, this necessitates, among others, the proper selection of load and material residence time in the working space of a pelleting machine.

The curves shown in Figure 13 reveal a significant difference in the rate of increase of permanent and elastic deformation energies. The dependence of both energies on the number of cycles was rectilinear. The average line slope for the five tested moisture levels for permanent deformation energy was approximately five times lower than that for elastic energy (which was lost due to granulate elastic deformation). The coefficient varied between 2.3 for 22% moisture and 3.61 for 10% moisture. However, the increase in elastic energy in subsequent cycles did not significantly differ between samples with different levels of moisture. This demonstrates the significance of the number of cycles, as well as the need for optimizing the operation of granulator rolls, in order to avoid wasting energy on the elastic deformation of the granulated material. On the other hand, further research and analysis of the cyclic loading process with different values of loading force are required (in preparation).

3.4. Effect of Moisture Content on Pellet Strength

The mechanical strength of the obtained agglomerate was found to decrease polynomially as the moisture content of digestate increased. The ultimate load decreased with the increase in deformation (the capsule stiffness decreased). Figure 14 depicts the course of the force related to the agglomerate length unit as a function of moisture content. Simultaneously, a linear relationship was found between the index thus determined and the energy required to complete 10 loading cycles (not shown here).

The most frequently observed effect of increasing moisture content is the deterioration of agglomerate strength. It should be highlighted that the drop of this indicator is fairly more linear than that of some of the other analyzed parameters, which showed a certain shift when the moisture content increased above 16%. As previously demonstrated, the elastic energy input was constant and independent of the digestate moisture content and hence had only little impact on the quality of the final agglomerate. It influenced the total energy used during compression, as has been shown. Thus, the energy of permanent deformation appears to have a significant influence on the strength characteristics of granules. However, for a more accurate interpretation of the results, it is necessary to consider the viscoelastic effects that are present (the damping capacity).

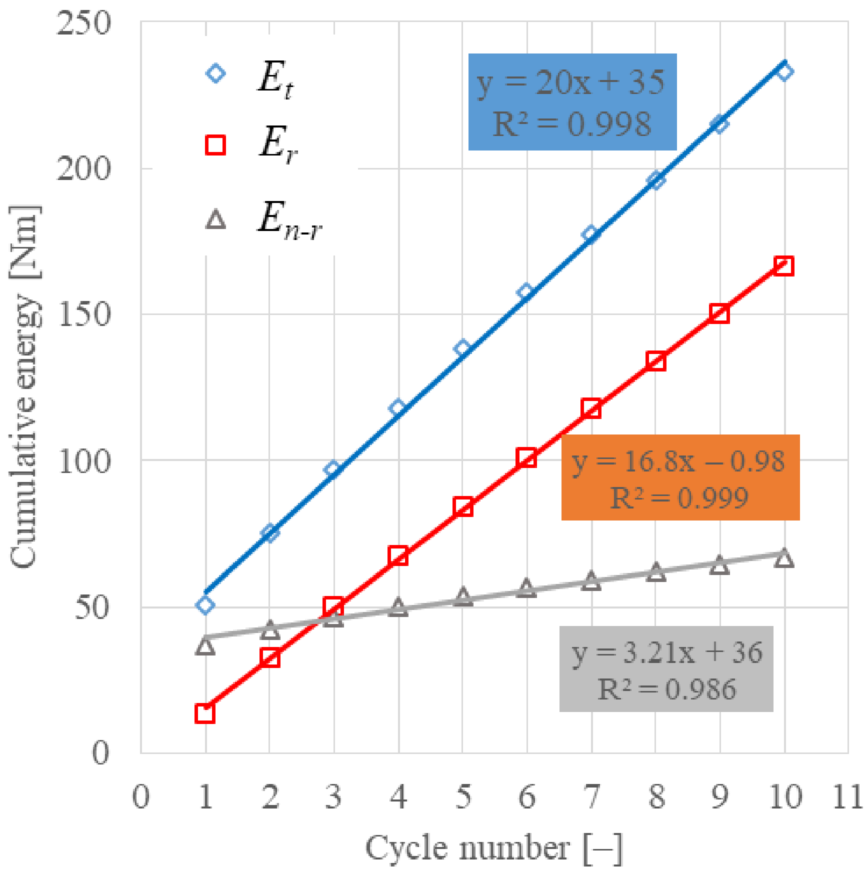

Figure 13. Share of reversible and permanent energy during 10 cycles of loading/unloading of digestate.

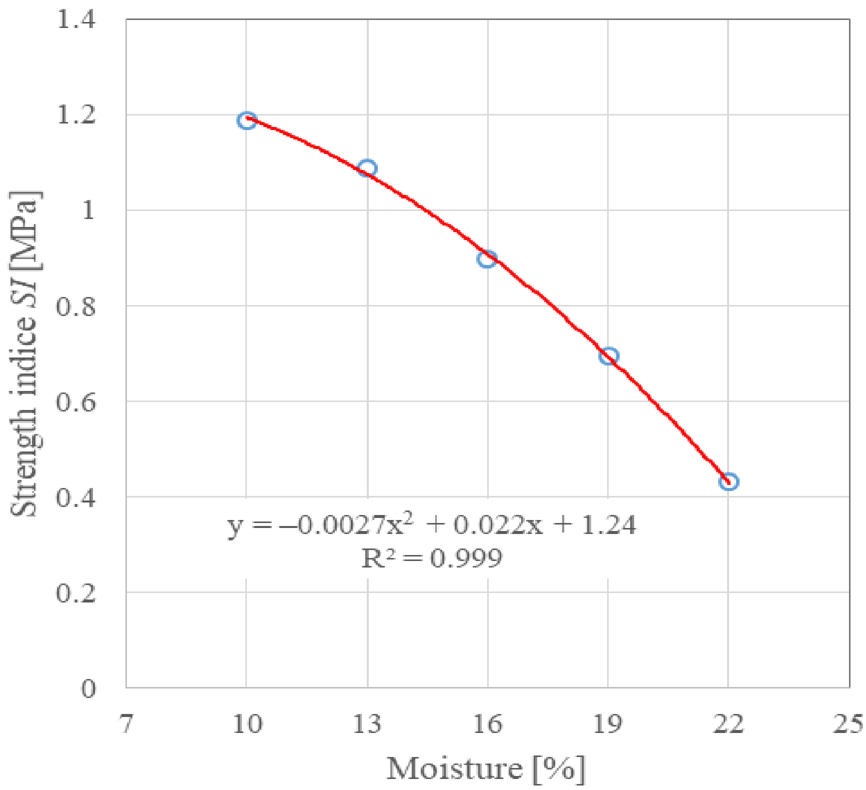

Figure 14. Effect of digestate moisture on the strength of agglomerate.

4. Conclusions

The proposed new approach of analyzing the course of the compaction process using cyclic loading/unloading allowed the determination of the energy distribution of permanent and elastic deformations as well as deformations that are dependent on moisture content and loading cycle. Such findings have not been reported so far. Additionally, the proposed approach allowed certain intriguing phenomena to be recognized, which, from a wider viewpoint, may be helpful for interpreting interactions occurring during digestate compaction and, consequently, for developing a process model. Further research may enable a better understanding of the findings and promote the valorization of anaerobic digestate for its potential application in pellet production.

The study showed that the total energy input decreased with increasing moisture content. The increase in moisture also caused an increase in the share of elastic deformations, which was evident in the first cycle (compaction) as well as successive loading as a result of cycles 2–10. The proportion of elastic deformation energy in the overall energy required in the process increased with the number of compaction cycles. The majority of the energy expended was dissipated through elastic deformations.

The nonobvious increase in the share of elastic deformation energy with the increase in moisture resulted from the lower energy inputs for permanent deformations. Compared to the permanent deformation energy, the proportion of elastic deformation energy increased as the moisture content of digestate increased. One of the noteworthy findings of the study is that the absolute values of elastic energy were largely consistent regardless of digestate moisture and cycle number.

The experiments revealed that moisture content had a significant influence on permanent energy, which could be a reliable indicator of susceptibility to compaction; however, this requires further research.

The strength of the digestate pellets decreased as moisture content increased. Due to the constancy of the elastic deformation energy, the decrease in strength may have resulted from the lower values of nonrecoverable energy, observed at higher moisture levels.

The study also showed that the influence of moisture on the values of deformations was not always unambiguous. Due to the fact that the average share of elastic deformations in the compaction phase was small for cycle 1 (3.76%) and high for cycles 2–10 (96.7%), tests with lower loads may provide additional data. Subsequent works should also consider the observed variability of deformations in the first phase of compaction.

Loading/unloading experiments additionally revealed that both the shape of the space between the loading/unloading curves and the path of these curves were significantly influenced by moisture content. Further investigation across a broad range of load levels will be necessary for confirming and better interpreting this interesting observation.

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