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Article

Helianthus salicifolius as a New Biomass Source for Biogas Production

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Abstract: Renewable energy is becoming a widely discussed topic in the European Union (EU), due to a desire to reduce the negative effects of fossil fuels on climate change and biodiversity. About 60% of the total renewable energy produced in the EU is derived from biomass. Anaerobic digestion (AD) is an important pathway to convert biomass into biogas and then into bioenergy. *Helianthus salicifolius* is a perennial plant, whose biomass can serve as a co-substrate in biogas plants. Biomass composition, in addition to the biomethane and biogas potential, were investigated in raw green biomass and silage obtained from *Helianthus salicifolius* plants grown under different types (mineral and organic) and doses (0, 85, 170 kg N ha^{-1}) of nitrogen fertilization. The biomethane production efficiency from *Helianthus salicifolius* was recorded for 25 days and found to range on average between 169.4 NL kg^{-1} VS for raw biomass and 193.2 NL kg^{-1} VS for silage. It follows from the current study that ensiling increases substrate digestibility and has a positive impact on methane concentration, but the biomethane and biogas production outputs from those substrates did not differ significantly at the end of the process.

Keywords: willow-leaf sunflower; silage; anaerobic digestion; biogas; biomethane; biomass characteristics

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1. Introduction

Reducing energy dependence on non-renewable and unsustainable sources is one of the objectives of the Bioeconomy Strategy for the EU [1]. Currently, the renewable energy sources (RESs) in the EU account for about 20% of all types of energy used [2]. This target had been forecast for 2020 and was achieved by then [3]. An increase in this share is planned, and the EU thus plans to become the first carbon dioxide (CO_2)-neutral continent by 2050. About 60% of all renewable energy in the EU is obtained from biomass [4].

Anaerobic digestion (AD) is a natural biological pathway through which organic biomass/waste can be transformed in the absence of oxygen into biogas and digestate [5]. The former is used for energy needs, and the latter can be processed into biofertilizer. This process was initially employed to remove odor from animal waste, and biogas was then used mostly to provide heat and power to farms and to other users. Later it was applied to other organic waste from food processing [6]. Numerous substrates and raw materials are now processed to obtain biogas. They are used alone or in co-digestion with other types of substrates from different sources. In 2019, there were 18,943 biogas plants in the EU, which produced 167 TWh [7]. Almost 27% (36 Mtoe) of renewable energy in 2016 was generated from agricultural biomass (crops and agricultural by-products) [4], and this percentage is growing. The most economically important crop for biogas production in the EU is maize [8], which prevails over other types of biomass in agriculture, being a benchmark in terms of the volume of biomass per hectare and produced biogas. However, the monoculture of maize (annual crop) leads to the degradation and erosion of soil, the compaction of soils by heavy agricultural machinery needed to carry out agricultural operations, greater severity and higher incidence of crop diseases and pest infestation, and a subsequent loss of biodiversity [9]. Researchers and farmers are looking for crops that can replace maize in the near future in order to reduce some of these adverse effects. There are some perennial crops that are promising in terms of biogas production, in addition to crops that can tolerate better extreme weather conditions, or require lesser technical interventions, including the deployment of heavy equipment, and can be planted in low quality or marginal soils. Therefore, these crops can contribute to a sustainable supply of agricultural biomass in the bioeconomy [7], and a sustainable production of biogas.

Helianthus salicifolius A. Dietr (willow-leaf sunflower) is a semi-woody, perennial herbaceous crop (PHC). It is native to North America, especially dry territories of the central US (Texas and Nebraska) [10,11]. However, other *Helianthus* annual species (*H. annuus*, *H. argophyllus*, *H. bolanderi*, *H. debilis*) are present in Europe, as they were brought here 400 years ago as ornamentals and are therefore better known in the EU. The perennial *Helianthus* spp. are now grown as crops and ornamental plants [12]. The willow-leaf sunflower grows up to 2.5–3.5 m in height, producing numerous shoots. It is light green to purple red in color. It produces small, yellow flowers, similar in their structure to those of common sunflower (*H. annuus*) [10,13], but considerably smaller. Usually, flowers gathered in inflorescences appear at the end of summer and last until the first frost. Single, lanceolate leaves are 15–40 cm long and have a light green color, which makes them similar to willow leaves; hence the species name. In the natural environment, the willow-leaf sunflower produces seeds. However, in the temperate climate conditions of Poland, it is reproduced by dividing rhizomes or seedlings, as the seeds do not have enough time to mature. As a result, this species does not become invasive. In the natural habitat, it can be found more often along roadsides, in wastelands, or on rocky hills. Thus, it has low soil requirements [10] and can grow in alkaline soils [11], which means that a willow-leaf sunflower plantation can be set up on marginal poor soils, and grown without fertilization [14]. *Helianthus salicifolius* is a crop with high biomass potential, and can be used as a green energy source. It is well known for high biomass production [10,15,16], tolerance to harsh environmental conditions, both frost and drought, and resistance to possible infestation by disease, fungi, and pests [10,13,17]. It has been investigated as a source of bioactive substances for pharmaceutical or veterinary purposes [18], and its post-extraction biomass has been tested as a feed for insect rearing [19]. However, there is no information on the possibility of using biomass of this species for the production of biogas as a renewable energy source.

In the research conducted so far, new technological solutions have been searched for, that will improve the efficiency of the biomethane fermentation process of lignocellulosic biomass [20,21]. Commonly used methods are the introduction of a pretreatment stage using physical, chemical, or biological processes, and a combination of these methods [22,23]. In order to increase the degradability of lignocellulose under anaerobic conditions, ultrasonic disintegration [24], a hydrodynamic cavitation generator [25], and hydrothermal depolymerization [26] are used. The use of strong acids and low-temperature conditioning is also promising [27]. Other optimization processes are based on a properly selected substrate composition or by ensuring appropriate technological parameters, including the C/N ratio, temperature, organic load rate, and hydraulic retention time [28,29]. Unconventional methods of influencing the anaerobic bacteria community with physical factors, including a constant magnetic field or microwave radiation, are also used [30,31]. However, little research has so far been focused on assessing the impact of the method of biomass cultivation, including the method and type of fertilization, on its composition, characteristics, and susceptibility to anaerobic degradation, and the final effects related to the efficiency of biogas production. This was the inspiration for the authors to carry out the presented research.

Therefore, the aim of the research was to determine the effect of: (i) a feedstock type of the *Helianthus salicifolius* biomass such as raw biomass and silage, (ii) fertilization type and (iii) N dose, on the biomass characteristics and the effectiveness of AD of this new and unused substrate in biogas production technologies.

2. Materials and Methods

2.1. Experimental Works Design

The research included testing two types of feedstocks as a raw green biomass and silage of *Helianthus salicifolius* (Table 1). The second factor consisted of the type of nitrogen fertilization: mineral, organic, and control without fertilizer. The third factor was composed of three nitrogen doses of fertilizer used: 0, 85, and 170 kg N ha^{-1}, which were applied in May, at the onset of plant vegetative growth. Thus, ten different feedstock variants were obtained.

Table 1. The research design.

Feedstock Type from *Helianthus salicifolius*									
Raw Biomass (R)					**Silage (S)**				
Fertilization Type									
Mineral Fertilizer (M)		**Organic Fertilizer (O)**		**Without Fertilization—Control (C)**	**Mineral Fertilizer (M)**		**Organic Fertilizer (O)**		**Without Fertilization—Control (C)**
Nitrogen dose									
85	170	85	170	0	85	170	85	170	0
Variant									
RM85	RM170	RO85	RO170	RC0	SM85	SM170	SO85	SO170	SC0

RM85—raw feedstock with mineral fertilization 85 kg N ha^{-1}; RM170—raw feedstock with mineral fertilization 170 kg N ha^{-1}; RO85—raw feedstock with organic fertilization 85 kg N ha^{-1}; RO170—raw feedstock with organic fertilization 170 kg N ha^{-1}; RC0—raw feedstock without fertilization; SM85—ensiled feedstock with mineral fertilization 85 kg N ha^{-1}; SM170—ensiled feedstock with mineral fertilization 170 kg N ha^{-1}; SO85—ensiled feedstock with organic fertilization 85 kg N ha^{-1}; SO170—ensiled feedstock with organic fertilization 170 kg N ha^{-1}; SC0—ensiled feedstock without fertilization.

2.2. Feedstock Origin and Silage Preparation

The experiments were conducted on a field at the Research Station of the University of Warmia and Mazury in Olsztyn, Poland (53°99′99.1″ N, 21°15′35.4″ E). The green plants of *Helianthus salicifolius* were harvested as whole plants with a rotary mower at the beginning of September, in two consecutive years: 2019 and 2020 (seventh and eighth year of growth). During the harvest, the whole biomass was chopped with a Viking Ge 220 (München, Germany).

Silage was obtained by ensiling *Helianthus salicifolius* biomass chopped into pieces 1–2 cm in length, and placed in 1000 cm^3 plastic silos immediately after harvesting. Ensiling was performed by manual filling. Tanks were filled to a medium density of 930 kg m^{-3} and subsequently sealed. The silage tanks were kept at a temperature 10–20 °C, for 10 months before the analyses. Ensilage biomass was prepared without preservatives or silage additives in triplicate.

2.3. Analytical Methods

The substrates (silage and raw biomass), digestate, and inoculum were dried and milled in a Retsch SM 200 cutting mill (Haan, Germany) using a 1 mm sieve to prepare the material for analyses. Dry matter (DM) content was determined with the gravimetric method by drying samples in an oven at 105 °C to constant weight (EN ISO 18134−1:2015), using a laboratory dryer. Organic matter (OM) was measured by determining the loss of mass after combusting (ELTRA TGA-THERMOSTEP, Neuss, Germany). The carbon (C) content was specified by burning at 1335 °C (ELTRA CHS−500, Neuss, Germany), where oxygen (O_2) was used to maintain combustion and the carrier gases were passed at a flow of 200 NL/h (PN-EN ISO 16948:2015−07). The nitrogen (N) was analyzed by the Kjeldahl procedure (K−435 mineralizer and B−324 BUCHI distiller, Flawil, Switzerland). Biogas was collected from bioreactors with a 10 mL gas-tight syringe. To analyze the composition, the collected biogas was injected on a gas chromatograph (GC Agilent 7890 A-Agilent Technologies, Santa Clara, CA, USA) with argon (Ar) and helium (He) as the carrier gases at a volume flow of 0.9 NL/h. The injection and detector port temperatures were 150 and 250 °C, respectively. All analyses were performed with three repetitions.

2.4. Fermentation Measurements

The biogas production from silage and raw biomass was performed for a 25-day period under 37 ± 1 °C in AMPTS II (BPC Instruments AB, Lund, Sweden). The measurement system recorded changes in partial pressure coupled with reactors. Glass vessels (500 cm^3) were used as laboratory scale reactors. The amount of biomethane produced in the reactors was detected every 3 h of the incubation. Tests were performed by filling the reactors with approximatively 193 g of the anaerobic sludge and then the biomasses were added. Moreover, 200 g of tested anaerobic sludge was used as a control sample. The initial organic load rate (OLR) was between 4.5 and 5 g VS L^{-1}. Anaerobic sludge (AS; the source of methanogenic bacteria) was used for the fermentation process in bioreactors (which served as the inoculum). The sludge came from a bioreactor operating under mesophilic conditions at 36 ± 1 °C. The parameters of the inoculum of the sludge are presented in Table 2.

Table 2. The parameters of anaerobic sludge inoculum used in respirometers.

Composition Trait	Anaerobic Sludge
DM (%)	6.8 ± 0.2
OM (% d.m.)	73.5 ± 0.9
Ash (% d.m.)	26.5 ± 0.9
C (% d.m.)	41.1 ± 0.8
N (% d.m.)	3.7 ± 0.0
C/N ratio	11.2 ± 0.2

DM—dry matter, OM—organic matter, C—carbon, N—nitrogen, C/N—carbon to nitrogen; ± standard deviation.

Respirometers with sludge and substrate were flushed with compressed (N_2) nitrogen in order to ensure anaerobic conditions inside. The content was mixed at 100 rpm for 30 s every 10 min as the reactors were equipped with automated stirrers, a temperature control, and a stabilizing system. The volume of biomethane generated in the reactors was detected every 3 h during the measurements. Using the bioprocess control software, a gas production report was recorded every day, in already normalized data (standard atmospheric pressure 101.3 kPa, 0 °C and zero moisture content). The endogenous biogas by AS was excluded from the calculations of biomethane production in the tests. Biomethane yields were calculated once a day as the biomethane volume produced over a period of 25 days and expressed as NL kg^{-1} VS. At the end of the process, an aliquot of the remaining biogas in the headspace of the reactors was used to analyze the composition of biogas according to the methodology.

2.5. Statistical Analysis

Anaerobic respirometric tests (AMPTS) and analytical works were performed in triplicate. Statistical data analysis was performed using Statistical 13.3 PL software. Non-linear regression was used to calculate the reaction rate constants. The iterative method was applied to biogas generation rate calculation. ANOVA and Tukey's HSD test were used to examine the significance of the differences between analyzed variables. In all tests, the probability level used was $p < 0.05$. Between the determined factors, the Pearson correlation coefficient was also used.

3. Results and Discussion

3.1. Feedstock Characteristics and Correlation

The results of the analyses suggest that the feedstock type (raw biomass and sludge) had a significant effect on the DM content, C and N contents, and C/N ratio (Table 3). Moreover, the fertilization type significantly affected the OM content, DM content, ash content, N content, and C/N ratio. Finally, the fertilization dose had no significant effect on any of the analyzed parameters. When analyzing the interaction of the principal factors, it was determined that the feedstock type × fertilization type was the only interaction that had influence on the dry matter content.

The characteristics of the silage and raw biomass from *Helianthus salicifolius* are presented in Figure 1. In the studied feedstock, the DM reached values in the range of 32.3–33.7% from silage and 33.4–36.4% from raw material, thus showing significant differences between the feedstock type, fertilization type, and their interaction. The content of DM and the content of N in the feedstock were negatively correlated (−0.87) (Table 4). A negative but rather weak correlation was observed between the contents of DM and C, resulting in a positive correlation (0.83) between the DM content and the C/N ratio. The carbon and nitrogen ratio in feedstock is one of the most important factors that influences biogas production. The results of the present study (Figure 1) showed that the highest C/N ratio (68) occurred in *Helianthus salicifolius* raw biomass from plants treated with an organic fertilization dose of 170 kg N ha^{-1}, whereas the lowest (52) was found in *Helianthus salicifolius* silage biomass obtained without fertilization. In the current study, a higher nitrogen fertilization dose did not correlate with a higher nitrogen content in biomass. However, other authors, who examined Miscanthus, obtained a positive correlation between these two characteristics [32]. The current study showed a negative correlation (−0.43 and −0.35) between the C/N ratio in the feedstocks and the methane and biogas yields, respectively (Table 4). Methane fermentation is a process that is sensitive to environmental conditions due to the interaction between fermenting and methanogenic microorganisms. The C/N ratio is an important factor in the production of methane [33]. The C/N ratio in *Helianthus salicifolius* biomass, regardless of the experimental variant, is significantly above the values considered optimal for efficient AD. The optimal ranges of C/N ratio for an undisturbed course of AD range between 10 and 30 [34–36]. When the C/N ratio is high in the medium, nitrogen is rapidly consumed by the methanogens to meet their protein needs, resulting in low methane production, and at a low C/N ratio, nitrogen will be present as ammonia, which inhibits the metabolism of methanogens due to their toxicity [37,38]. Researchers suggest that the value of this indicator may be within wider limits, especially for the co-fermentation of many substrates, and with the maintenance of stable, close-to- optimum technological parameters [39]. Long-term use of this substrate would inhibit the AD process. Taking into account the characteristics of the biomass, it can be concluded that the *Helianthus salicifolius* biomass should not be used as a monosubstrate in an AD process, but rather as an ingredient of a substrate mixture with a low C/N ratio, e.g., pig slurry, manure, algal biomass.

The characteristics of the mixture of substrate and AS are given in Figure 2. In a recent study, corn sieving waste was used as a co-substrate for AD to control the C/N ratio in order to make the process more efficient, where the highest methane and biogas yields were obtained when the C/N ratio was 15 [40]. In the current study, it was found that the use of *Helianthus salicifolius* biomass as a feedstock for AD can significantly increase the value of the C/N ratio. Due to the high C/N ratio in the *Helianthus salicifolius* biomass, the fermentation process must be effectively prepared, e.g., by adapting and developing the anaerobic bacterial community, introducing nitrogen-rich co-substrates, or using pretreatment processes.

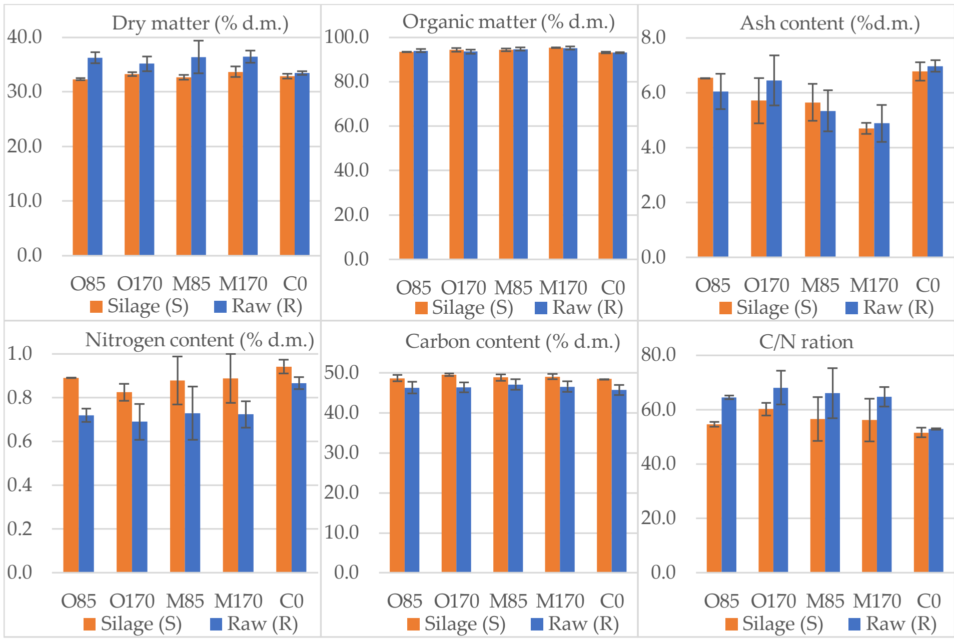


Figure 1. Characteristics of silage and raw material of *Helianthus salicifolius* used for the preparation of feedstock. (C0—without fertilization; M85—mineral fertilization 85 kg N ha^{-1}; M170—mineral fertilization 170 kg N ha^{-1}; O85—organic fertilization 85 kg N ha^{-1}; O170—organic fertilization 170 kg N ha^{-1}).

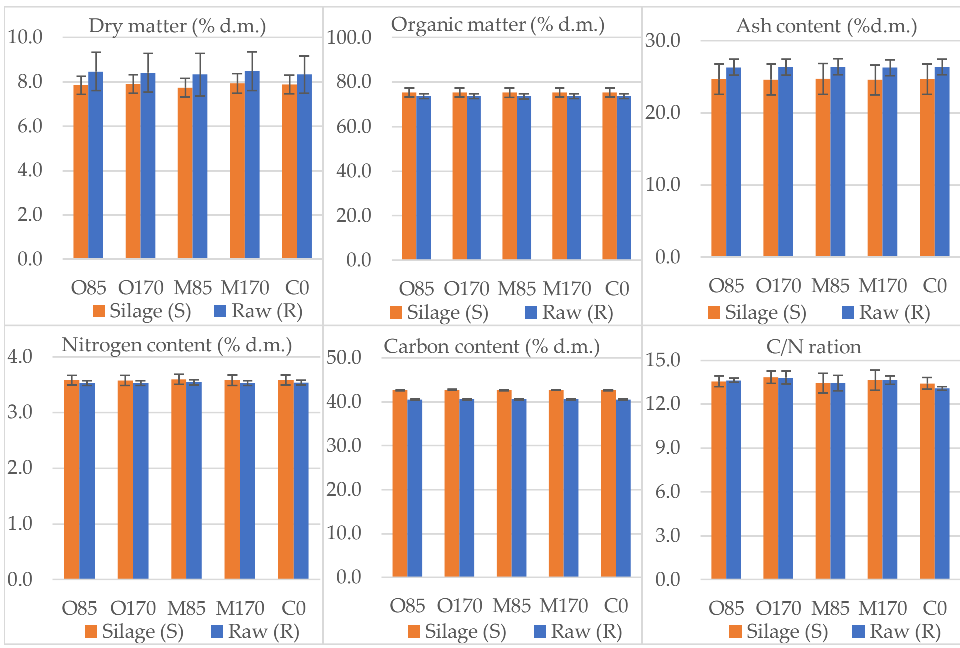


Figure 2. Characteristics of the mixture of substrate silage/raw material/ of *Helianthus salicifolius* and anaerobic sludge used for the anaerobic digestion test (C0—without fertilization; M85—mineral fertilization 85 kg N ha^{-1}; M170—mineral fertilization 170 kg N ha^{-1}; O85—organic fertilization 85 kg N ha^{-1}; O170—organic fertilization 170 kg N ha^{-1}).

Table 3. Analysis of variance (*p*-values) for the analyzed features.

Source of Variation	Dry Matter	Organic Matter	Ash Content	C Content	N Content	C/N Ratio	CO_2	CH_4	Biogas Production	Biomethane Production
Feedstock type	0.000 *	0.657	0.657	0.000 *	0.000 *	0.001 *	0.000 *	0.000 *	0.317	0.097
Fertilization type	0.005 *	0.000 *	0.000 *	0.154	0.000 *	0.000 *	0.005 *	0.016 *	0.632	0.685
Nitrogen (N) dose	0.690	0.115	0.115	0.755	0.511	0.454	0.800	0.561	0.552	0.639
Feedstock type × Fertilization type	0.014 *	0.846	0.846	0.684	0.266	0.101	0.001 *	0.002 *	0.902	0.884
Feedstock type × N dose	0.183	0.140	0.140	0.464	0.875	0.754	0.382	0.235	0.756	0.667
Fertilization type × N dose	0.768	0.308	0.308	0.719	0.610	0.344	0.981	0.818	0.523	0.630
Feedstock type × Fertilization type × N dose	0.532	0.414	0.414	0.871	0.896	0.970	0.123	0.208	0.934	0.953

* Significant values ($p < 0.05$).

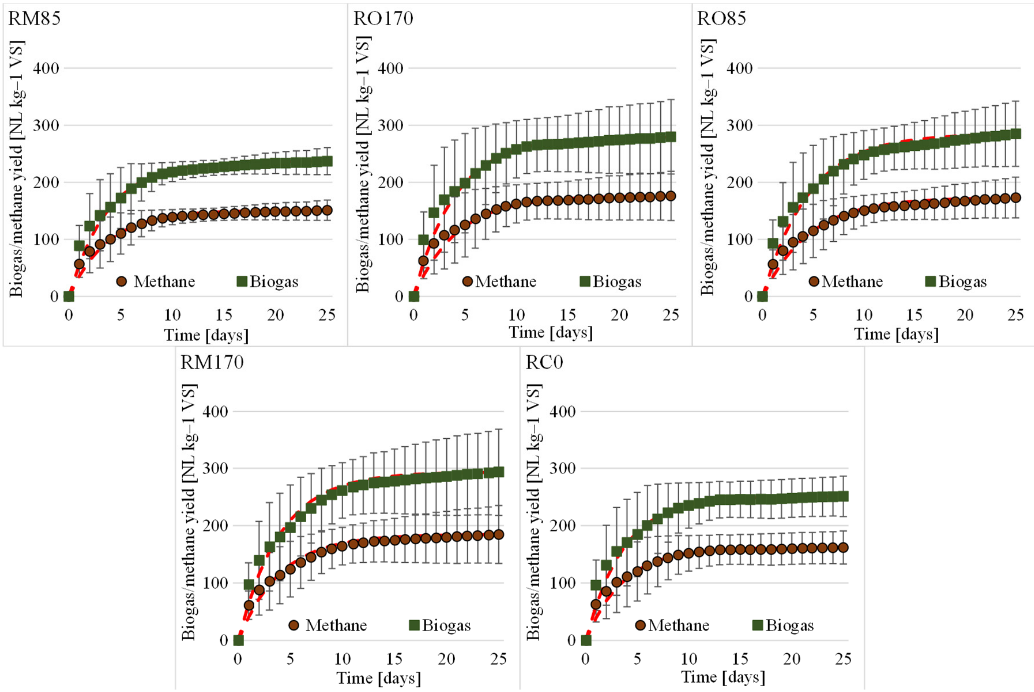
Table 4. The Pearson correlation coefficients for the analyzed traits.

Item	CO_2	CH_4	Biomethane Production	Biogas Production	Organic Matter	Ash Content	Dry Matter Content	C Content	N Content	C/N Ratio
CO_2 %	1.00									
CH_4 %	−0.99 *	1.00								
Biomethane production	−0.63 *	0.64 *	1.00							
Biogas production	−0.45 *	0.47 *	0.98 *	1.00						
Organic matter	−0.11	0.06	−0.22	−0.26	1.00					
Ash content	0.11	−0.06	0.22	0.26	−1.00 *	1.00				
Dry matter content	0.55 *	−0.58 *	−0.38 *	−0.28	0.53 *	−0.53 *	1.00			
C content	−0.60 *	0.59 *	0.47 *	0.39 *	0.09	−0.09	−0.58 *	1.00		
N content	−0.60 *	0.62 *	0.52 *	0.41 *	−0.49 *	0.49 *	−0.87 *	0.48 *	1.00	
C/N ratio	0.47 *	−0.49 *	−0.43 *	−0.35 *	0.59 *	−0.59 *	0.83 *	−0.28	−0.97 *	1.00

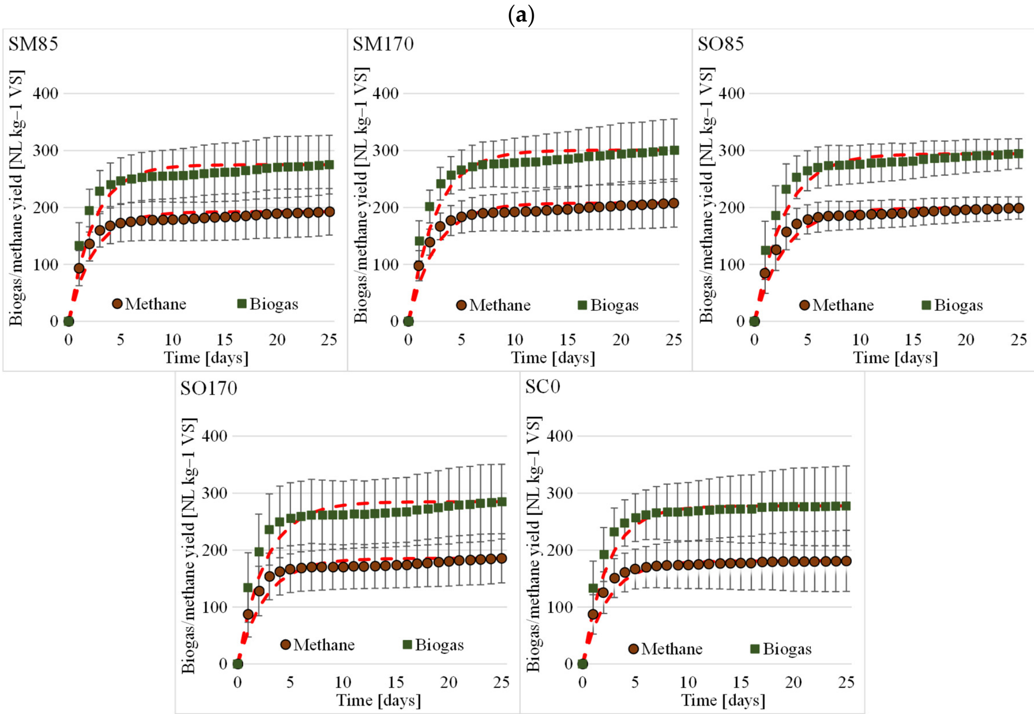
* significant values ($p < 0.05$).

3.2. Biogas and Biomethane Production

For biogas production, energy crops can be used as raw green mass or after certain preconditioning that contributes to the preservation of certain properties of green biomass or helps to improve digestibility before anaerobic digestion. For example, silage may be used when there is a large amount of biomass that cannot be processed immediately [41]. In the current study, ensiling was also used as a pretreatment process. The daily biogas and biomethane production outputs from silages and raw substrates from *Helianthus salicifolius* are summarized in Figure 3. The biogas yield from *Helianthus salicifolius* averaged between 269.29 NL kg^{-1} VS for raw biomass and 286.6 NL kg^{-1} VS for silage (Figure 3). Thus, the methane yield of *Helianthus salicifolius* averaged between 169.4 NL kg^{-1} VS for raw biomass and 193.2 NL kg^{-1} VS for silage. To the best of our knowledge, it is highly probable that the present study is the first and only study that has included biogas tests with *Helianthus salicifolius* substrates. A literature review showed a lack of information regarding biogas production from this plant. However, the data from this research were compared with the results of studies on other perennial crops, such as *Pennisetum purpureum* or *Miscanthus*. Concerning methane yield, *Pennisetum purpureum* harvested at different stages of maturation yielded between 104 and 219 NL kg^{-1} VS [42]. Lower values were found for *Miscanthus*, which was tested for biogas productivity, and produced 84 and 130 NL kg^{-1} VS of biomethane and biogas, respectively, when its biomass was harvested in December [43]. However, later harvest in February had some impact on methane and biogas yields, which rose to 100 and 230 NL kg^{-1} VS, respectively [44]. Higher methane yield from *Miscanthus* (247 NL kg^{-1} VS) on average was achieved in October [32]. Similarly, the highest average methane yield obtained was 356.6 NL kg^{-1} VS, and it was produced from *Miscanthus* ensiled biomass originating from a double-cut harvest system [45]. A study conducted in Poland shows that *Miscanthus* can be used as feedstock in a biogas plant due to it having the highest and most stable yields among energy crops, with maize coming in second [46]. Therefore, the methane and biogas yields obtained in our experiment were lower compared to the methane and biogas yields from other energy crops, such as maize, rye, grass, triticale, sorghum, cardoon, perennial ryegrass, timothy, cocksfoot, tall fescue, cordgrass, switchgrass, and big bluestem, which have been reported by other researchers [47–50]. In the current study, the highest methane and biogas yields, i.e., 207.8 and 300.7 NL kg^{-1} VS respectively, were obtained from silage made from plants treated with the mineral fertilization dose 170 kg N ha^{-1} (Figure 3b); high yields, of 184.6 and 293.4 NL kg^{-1} VS of biomethane and biogas respectively, were also obtained from raw green plants grown in soil supplied the mineral fertilization dose of 170 kg N ha^{-1} (Figure 3a). The CO_2 and CH_4 content in biogas, the biogas production rate, and the reaction rate constant from silage and raw material of *Helianthus salicifolius* are presented in Table 5. The highest effectiveness was achieved with the mineral fertilization dose of 170 kg N ha^{-1} and with silage as a substrate (Figure 3b), where the biogas production rate was r = 117.3 cm^3 day^{-1} and the methane content reached 68.8 ± 1.5% (Table 5). Much lower effectiveness was observed in the case of raw biomass from plants grown at the mineral fertilization dose of 170 kg N ha^{-1} (Figure 3a), where the biogas production rate was r = 73.4 cm^3 day^{-1} and methane content equaled 62.7 ± 1.0 % (Table 5). No statistically significant differences appeared between the analyzed features and between their interactions in terms of biogas and biomethane production (Table 3). However, in other studies with other types of biomass, fertilization and ensiling positively affected biogas yield and methane content [51,52]. Ensiling of other biomasses seems to contribute positively to methane yield, increasing it by up to 11% [53]. However, this is not a rule [54]. Other types of pretreatment have led to a significant increase in methane and biogas yields in the case of *Sida hermafrodita* [55], but not in the methane content [56], which was lower than in our case. Therefore, in the current study, the ensiled *Helianthus salicifolius* substrate achieved higher productivity of biogas and biomethane. However, the results of biomethane and biogas production were not statistically significantly different between the variants. In another study, the methane yield was not found to have increased when the biomass had been ensiled [54]. During storage, ensiling resulted in increasing the nitrogen content but decreased the DM content of silage (Figure 1), which is assumed to be related to the higher amount of protein in the biomass and degradation of the structure of biomass to more easily degradable intermediates. It follows from the current study that silage substrate increases digestibility and has a positive impact on the methane content.



(**a**)



(**b**)

Figure 3. Cumulative biogas and methane yield (NL kg^{-1} VS) of *Helianthus salicifolius* as silage and raw biomass in the 25-day test, depending on the type of fertilization (kg N ha^{-1}): C0—without fertilization, O85—organic fertilization 85, M85—mineral fertilization 85, M170—mineral fertilization 170, O170—organic fertilization 170. (**a**) *Helianthus salicifolius* as raw biomass (R); (**b**) *Helianthus salicifolius* as silage (S).

Table 5. Carbon dioxide (CO_2) and methane (CH_4) content in biogas, methane (r_m), and biogas (r_b) production rate and reaction rate constant ($k_{m,b}$) of the analyzed silage, and raw material of *Helianthus salicifolius*.

Variant	CO_2 (%)	CH_4 (%)	r_m (cm^3 day^{-1})	r_b (cm^3 day^{-1})	$k_{m,b}$ (L day^{-1})
RO85	39.30 ± 0.31	60.70 ± 0.31	38.1	62.8	0.22
RO170	37.51 ± 0.69	62.83 ± 0.76	42.3	69.9	0.25
RM85	36.36 ± 1.12	63.64 ± 1.12	40.8	64.0	0.27
RM170	37.35 ± 1.00	62.65 ± 1.00	46.1	73.4	0.25
RC0	35.83 ± 2.39	64.17 ± 2.39	45.3	70.3	0.28
SO85	32.43 ± 0.69	67.57 ± 0.69	71.7	106.0	0.36
SO170	34.93 ± 0.16	65.08 ± 0.16	70.5	108.3	0.38
SM85	30.37 ± 1.90	69.64 ± 1.90	80.9	115.6	0.42
SM170	29.95 ± 1.21	68.83 ± 1.48	81.0	117.3	0.39
SC0	35.59 ± 3.11	64.41 ± 3.11	76.0	116.6	0.42

± standard deviation.

The feedstock type, fertilization type, and their interactions have a significant influence on the CH_4 and CO_2 concentration (Table 3). It was found that the highest content of CH_4 (69.6%) was obtained from silage with the mineral fertilization dose of 85 kg N ha^{-1} (Table 5). The value of this parameter in raw biomass ranged from 60.7 to 64.2% for organic fertilization with 85 kg N ha^{-1} and for control, respectively. The methane concentration was significantly corelated with the N and C content, at 0.62 and 0.59, respectively (Table 4), whereas it was negatively corelated with the DM content and C/N ratio (−0.58).

Significant differences in the methane production rate (r_m) and biogas production rate (r_b) depending on the experiment variant were found (Table 5). It was found that the *Helianthus salicifolius* silage biodegraded faster and easier in anaerobic conditions, and the fermentation process stabilized after 5 days of using the respirometers. In the case of silage, r_b values varied between 106.0 and 117.3 cm^3 day^{-1}. In the case of raw feedstock, they were almost twice as low, from 62.8 to 73.4 cm^3 day^{-1}. Even if the r_m (methane production rate), r_b (biogas production rate), and $k_{m,b}$ reaction (reaction rate constant) differed between the raw feedstock and ensiled biomass/silage, the methane and biogas yield from these substrates did not differ significantly at the end of the process.

3.3. Digestate Characteristics

Digestate remains after the termination of the biogas generation process. Digestate is rich in various active nutrients that can be extracted or reused on agricultural land as biofertilizer for environmental benefits [52,57,58]. Due to the high C and N content, it contributes positively to soil fertility [57,59].

The digestate composition depends on the feedstock used to produce biogas [60]. The content of OM, DM, ash, C, N, and C/N ratio in the digestate after AD is presented in Table 6. The studied digestate DM had values ranging from 6.1 to 6.3% from silage, and from 6.6 to 6.7% from raw material, depending on the fertilization dose supplied to *Helianthus salicifolius*, and it was slightly lower (5.9%) for the substrate-free digestate (I.A.A.D.) obtained after AD (Table 6). The N and C content in digestate is very important for the C/N ratio. To be used safely as a soil amendment in agriculture (without some pretreatment operations), it is recommended that the C/N ratio be between 15 and 20 [61]. In the present study, this ratio was lower (11.5–12.0 from silage and 12.1–12.5 from raw material). In the current study, the N and C content of the digestate was approximately the same between the variants (Table 6). A recent study has shown that the nitrogen content depends on the feedstock used in AD [58,62].

Table 6. Characteristics of digestate after anaerobic digestion from raw material and ensiled *Helianthus salicifolius*.

Composition Trait	RO85	RO170	RM85	RM170	RC0	SO85	SO170	SM85	SM170	SC0	I.A.A.D.
OM (% d.m.)	72.9 ± 0.3	73.0 ± 0.3	72.6 ± 0.0	73.2 ± 0.3	72.7 ± 0.0	69.5 ± 3.1	69.5 ± 2.3	68.7 ± 2.1	69.4 ± 2.8	70.7 ± 0.2	69.8 ± 1.3
DM (%)	6.7 ± 0.4	6.6 ± 0.5	6.6 ± 0.4	6.6 ± 0.5	6.6 ± 0.6	6.3 ± 0.0	6.2 ± 0.0	6.1 ± 0.1	6.2 ± 0.2	6.2 ± 0.2	5.9 ± 0.1
Ash (% d.m.)	27.1 ± 0.3	27.0 ± 0.3	27.4 ± 0.0	26.8 ± 0.3	27.3 ± 0.0	30.5 ± 3.1	30.5 ± 2.3	31.3 ± 2.1	30.6 ± 2.8	29.3 ± 0.2	30.2 ± 1.3
C (% d.m.)	40.8 ± 0.5	40.6 ± 0.6	40.9 ± 0.4	40.1 ± 0.7	39.8 ± 0.4	39.8 ± 1.6	39.6 ± 0.8	39.2 ± 1.3	39.1 ± 0.6	39.9 ± 0.2	40.3 ± 0.1
N (% d.m.)	3.3 ± 0.2	3.3 ± 0.2	3.4 ± 0.1	3.3 ± 0.2	3.3 ± 0.2	3.3 ± 0.2	3.4 ± 0.2	3.3 ± 0.2	3.4 ± 0.2	3.4 ± 0.3	3.5 ± 0.1
C/N ratio	12.5 ± 0.6	12.2 ± 0.5	12.0 ± 0.4	12.1 ± 0.4	12.2 ± 0.6	12.0 ± 1.1	11.9 ± 0.8	11.8 ± 1.1	11.5 ± 0.7	11.7 ± 0.9	11.4 ±0.2

OM—organic matter; DM—dry mass; C—carbon; N—nitrogen; C/N—carbon to nitrogen ratio; I.A.A.D.—inoculum after anaerobic digestion. ± standard deviation.

4. Conclusions

The perennial crop *Helianthus salicifolius* was investigated in order to determine its characteristics as a new and alternative feedstock for biogas production. During the study, *Helianthus salicifolius* was tested as different feedstock types (raw green biomass and silage), produced from plants grown with different fertilization (organic and mineral) and N doses. It was found that the feedstock type and fertilization type have a significant influence on most of the analyzed features. However, it was found that the feedstock type, fertilization type, and N doses have no significant influence on the biomethane and biogas production. In contrast, *Helianthus salicifolius* silage substrate increases digestibility and has a positive impact on the methane content. Moreover, it was found that significant increases in the methane and biogas production rate were obtained when silage was the feedstock. Silage also biodegraded more easily and faster in anaerobic conditions, and the fermentation process stabilized after 5 days. A higher biogas yield was obtained from silage (286.6 NL kg^{-1} VS) but it was not significantly higher than that from raw biomass. Significant differences in the methane concentration in biogas were found only for the feedstock type, fertilization type, and their interaction. A negative correlation between the C/N ratio and biogas and biomethane production was observed, and a long-term use of this feedstock alone may inhibit the AD process. The lower biogas production from *Helianthus salicifolius* compared to other energy crops would make it less suitable as a monosubstrate in the AD process. Therefore, further biomass investigations could be oriented to evaluating the biogas productivity from *Helianthus salicifolius* biomass with different pretreatment methods, and as a co-substrate rather than a monosubstrate in biogas production.

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