Chemical Composition and Biogas Formation potential of *Sida hermaphrodita* and *Silphium perfoliatum*

Hanna Siwek®, Małgorzata Włodarczyk *, Ewa Moźdźer, Marek Bury® and Teodor Kitczak

Faculty of Environmental Management and Agriculture, West Pomeranian University of Technology in Szczecin, 71-434 Szczecin, Poland; hanna.siwek@zut.edu.pl (H.S.); ewa.mozdzier@zut.edu.pl (E.M.); marek.bury@zut.edu.pl (M.B.); teodor.kitczak@zut.edu.pl (T.K.)

* Correspondence: malgorzata.wlodarczyk@zut.edu.pl; Tel.: +48-9149-6320

Received: 12 August 2019; Accepted: 19 September 2019; Published: 25 September 2019

**Abstract:** Biogas production and use is one of the pillars of the EU strategy for fossil fuels replacement via renewable energies. In Poland, the most commonly used crop for biogas production is maize. There are many factors limiting the cultivation of this crop, which is why alternative plants are sought. The aim of the present paper was to assess the effect of establishing a plantation using seeds, seedlings, and various harvest dates on biogas production from *Silphium perfoliatum* L. (*Silphium*) and from two phenotypes of *Sida hermaphrodita* L. Rusby (*Sida*). Harvesting was conducted in the second (2017) and third year of crop growth (2018). These crops were harvested in June and at the beginning of October as a two-cut strategy. Additionally, Silphium was harvested in early autumn as a one-cut strategy. Specific biogas yield (SBY) and specific biomethane yield (SMY) were estimated using the modified Baserga method. The biogas yield per hectare (BY) was calculated. The crop species, method of establishing a plantation, as well as the date and the number of harvests had a significant effect on the content of the selected chemical components; however, significant differences in terms of SBY were not found for the two-cut strategy. In the case of Silphium, approximately 40% more BY was produced for the two-cut strategy compared to the one-cut strategy. The BY was found to be significantly affected by the biomass yield; markedly higher BY can be obtained from Silphium and the average amount obtained in one year was 8598 m³ ha⁻¹ while 4759 m³ ha⁻¹ was obtained from Sida.

**Keywords:** renewable energy sources; biomass chemical composition; biogas yield; *Silphium perfoliatum* L.; *Sida hermaphrodita* L. Rusby

1. Introduction

Global increases in energy demand and related increased greenhouse gas emission [1], as well as the need to ensure energy security and to further diversify energy sources [2], necessitates taking actions aimed at developing technology for energy production based on the use of biomass. National actions plans drawn up by EU member states envisage that by 2020, the share of energy produced from renewable energy sources should exceed 20% of total energy consumption in the EU. According to the Renewable Energy Directive, the use of biomass for energy production in the EU will provide for half of the renewable target [3]. In comparison with other forms of bioenergy, biogas production is one of the most environmentally friendly technologies [4]. It is estimated that by 2020 in Europe, biogas plant production potential will reach 770 PJ per year [3]. At the end of 2014, there were approximately more than 17,000 active biogas plants in Europe—the largest number were in Germany and Italy [5]. In Poland, only 95 agricultural biogas plants with a total electrical capacity of 101.3 MW operated in
2017 [2]. The structure of Polish agricultural biogas plants is dominated by installations with capacities in the range of 0.5 to 1.5 MW, which together constitute 56% of all biogas plants in Poland [6]. Biogas production potential from agricultural raw materials constitutes the highest share in total estimated biogas potential in Poland [6]. Development of agricultural biogas plants in Poland would result in diversification of the gas supply, the actually available potential of which is 1.7 billion m$^3$ annually, i.e., more than 10% of national consumption [2].

The key success factor of the future role of biogas will be the availability of biomass. All types of biomass can be used as substrates for biogas production as long as they contain carbohydrates, proteins, fats, cellulose, and hemicelluloses as main components [7]. Biogas plants increasingly rely on energy substrates cultivated for this particular reason. For evaluation of energy crops as substrates for biogas production, an easily identifiable criteria are needed [8]. A main factor is the biogas yield per hectare (BY), which is made up of the total dry matter yield (DMY) per area and specific biogas yield (SBY) of this dry mass. The prediction of BY is a very important tool in biogas plant management and bioenergy policies. The content of crude protein, crude fat, crude fiber, and N-free extracts determines the degradability and, thus, the biomethane yield that can be produced through anaerobic digestion [9,10]. The SBY can be estimated under laboratory conditions based on fermentation studies using a continuous method or batch tests, e.g., the Hohenheim biogas yield test [11]. Laboratory tests are very expensive and time-consuming, they are a suboptimal instrument for evaluating short-term breeding progress [12]. Some batch tests allow determination of the specific biomethane yield potential with an acceptable repeatability [8]. Another method is theoretical estimation of biogas yield calculated from the elemental composition using different stoichiometric [9,13] and empirical models developed for individual crops or estimates of SBY by multiple regression of various constituents [10,14,15]. The highest number of empirical models was developed for maize which is considered to be a crop with the highest biogas production potential [10,14,15]. For a practical farmer who plans to establish a biogas plant, the ultimately decisive question is which crop or which variety provides the highest methane yield per hectare, as this has a decisive influence on the profitability of his biogas plant [8,16].

In comparison with laboratory methods, estimating biogas on the basis of the chemical composition of the biomass allows for obtention of results at a lower cost.

When it comes to deciding on crops for energy farming, desired characteristics, local climate, and soil conditions must be considered, such as high yield of dry matter, high calorific value, low energy and low-cost inputs (production), minimal fertilisation, as well as low nutrient requirements [17]. In Poland, the most commonly used crop for biogas production is maize. Currently, the main parameter determining the size of this crop is rainfall. Long-lasting droughts occurring in the spring and summer period, negatively affect its yield [18]. Additionally, the cultivation of maize in monocropping systems has led to several environmental problems like a soil degradation or a loss of biodiversity [19]. Other crops are being sought to replace maize as an energy crop for biogas plants. Two novel perennial crops which are not widely known in Europe and which have been, until now, only under scientific interest and that meet these criteria are (among others) *Sida hermaphrodita* L. Rusby (Sida) and *Silphium perfoliatum* L. (Silphium). These crops are characterised by a wide potential for use, for example, as forage [20,21], energy plants species [22], as well for industrial and medicinal use [21], and have a positive effect on soil quality [22–25]. They are also long flowering plants and a source of nectar and pollen for honeybees [20] and other pollinators, which is a very important aspect of environmental protection. In Poland, research on the biogas production potential of these crops has been conducted within a narrow scope—only for Sida [26].

The aim of the present paper was to assess the effects of our methodology on establishing a plantation and harvest date on biogas production from Silphium and from two phenotypes of Sida.
2. Materials and Methods

2.1. Characteristics of Energy Plants

Sida (*Sida hermaphrodita* L. Rusby) is a perennial crop native to southern regions of North America with no specific requirements in terms of soil and climate. It can be grown for up to 20–30 years and can achieve high biomass yield (20–25 t ha\(^{-1}\)) [20]. Due to the fact of its protein content (i.e., more than 20%), Sida can be a forage crop as well as a pharmaceutical material owing to high mucilage content. The content of cellulose, resin, and wax in its stalks is comparable to that of spruce and pine, therefore Sida can also be used in the pulp and paper industry. Sida holds great potential in combining ecosystem services, such as decreasing soil erosion [22], carbon storage [23], phytoremediation [24], and nitrate leaching, as well as strengthening biodiversity [25]. The most promising application of Sida seems to be for the purpose of biogas production for energy purposes. Its stalks are most suitable for direct combustion and its leaves for biogas production [22]. The calorific value of Sida stalk combustion is comparable to that of beech wood. Furthermore, there have been attempts to carbonize Sida [27].

Silphium (*Silphium perfoliatum* L.) is a large clumping plant from the Asteraceae family. It is a prairie crop originating from North America. Silphium was introduced to Europe in the 18th century owing to its decorative value. It is a medicinal and fodder crop; however, due to the fact of its high content of phenolic acids, its use as animal feed is limited [21,28]. Due to the high content of saponin compounds in its leaves, inflorescence, and rhizomes, Silphium is very useful for the pharmaceutical industry [21]. Silphium is a yellow flowering crop with high ecological value, and which is particularly suitable as an energy plant species owing to its low maintenance requirements and high biomass (15–19 t·ha\(^{-1}\)) and biogas yields. The crop is productive for around 15 years and can be adapted to regions with similar climate conditions, like Europe [23,29]. Silphium has the potential to be a pioneer plant used in the restoration of degraded land [30]. The production of Silphium in the first year is work and cost intensive. After the first year, the care requirements and production costs are low [23].

2.2. Study Site and Experimental Design

The experiment was conducted in the West Pomeranian region of Poland at the Agricultural Experimental Station of the West Pomeranian University of Technology in Szczecin. The study site was located in Lipnik near Stargard (N 53°20’35.8", E 14°98’10.8"). The soil under experiment was a light rust-brown sandy soil of poor rye complex. The average sum of long-term precipitation in the study location is 536 mm per year with an average air temperature of 8.2 °C. During the growing season (April–October), the average long-term precipitation is 359 mm with an average temperature of 13.3 °C. The year 2018 was drier with precipitation of only 253 mm between April and October compared to 615 mm in 2017 during the same period. The average temperature in the growing season of 2017 was 15 °C, whereas in 2018 the temperature was found to be higher and amounted to 16.8 °C.

The experiment was two-factorial: factor A—two phenotypes of *Sida hermaphrodita* (Sida1 and Sida2) and one phenotype of *Silphium perfoliatum*; and factor B—a method of establishing a plantation using seeds (seed) and seedlings (planting). Moreover, in the case of Silphium, the additional factor of the number of harvests in a season was introduced: one or two. The experiment was set-up at the turn of May 2016 in a split-plot design in four replications. The whole experiment had an area of 1250 m\(^2\), and each plot was 12.6 m\(^2\); there were 8 plots for Sida1, 8 for Sida2, and 16 for Silphium. Sida phenotypes were obtained from Germany and were selected from the two outermost habitats in terms of geographical location (the distance was approximately 620 km on the north–south axis) and varied original climatic conditions. Seedlings and seeds of Sida1 were obtained from Baden-Württemberg (MK Jungpflanzen GmbH, Biberach District) and of Sida2 from Lower Saxony (farm enterprise of Dirk Helling-Junghans in the vicinity of Osnabrück). Seedlings and seeds of Silphium were obtained also from Germany (N.L. Chrestensen Erfurter Samen und Pflanzenzucht GmbH (Thüringen)). During the experiment, there were no clear differences in the morphological traits of both phenotypes of Sida,
particularly in reference to the shape and size of leaves, colour, and shape of stalks, and the structure and colour of flowers.

The fore crop on the experiment site was spring barley harvested for grain. After harvest of the fore crop, a traditional method of mechanical cultivation of soil (post-harvest cultivation and pre-winter ploughing) was used. In the spring of 2016, a rotary tiller was used before sowing and planting. Pre-sowing fertilization was applied in the amount of N-100, P-35, and K-110 kg ha\(^{-1}\). The same amount of mineral fertilization was used every year after the start of vegetation.

2.3. Harvesting and Yield

Harvesting was conducted in the second (2017) and the third year (2018) of vegetation. Sida and Silphium plots (each plot of 12.6 m\(^2\)) were harvested in June at the flowering stage which shows the highest potential to produce fodder. The second harvest was at the beginning of October in a period when Sida was found to produce the highest yield [31] as a two-cut strategy (2H). In the case of Silphium, the eight plots were harvested only once per year—at the beginning of October as a one-cut strategy (1H). The fresh mass of harvested material was weighed. The dry matter (DM) was done gravimetrically after drying 2 kg of fresh material at 60 °C for two days and the dry mass yield (DMY) was calculated (Table 1).

| Crop              | 2017     | 2018     |
|-------------------|----------|----------|
|                   | 2H (VI)  | 2H (X)   | 1H (X)   | 2H (VI)  | 2H (X)   | 1H (X)   |
| Sida1 planting    | 13.17    | 1.95     | -        | 4.42     | 2.32     | -        |
| Sida1 seed        | 11.72    | 2.07     | -        | 4.60     | 2.63     | -        |
| Sida2 planting    | 12.52    | 2.24     | -        | 5.74     | 2.79     | -        |
| Sida2 seed        | 7.84     | 1.31     | -        | 2.99     | 1.84     | -        |
| Silphium planting | 21.69    | 4.89     | 13.40    | 9.02     | 3.11     | 20.59    |
| Silphium seed     | 19.36    | 5.34     | 15.66    | 7.74     | 4.53     | 19.07    |

1 2H—two-cut strategy (harvest month); 2 1H—one-cut strategy (harvest month).

2.4. Determination of Chemical Composition

For chemical analysis, 0.5 kg of the air-dried samples was milled to 1 mm and analytical dry matter was done gravimetrically after oven drying 1 g of the air-dried samples at 105 °C. Next, crude protein (XP), crude fibre (XF), crude fat (XL), and crude ash (XA) were determined in triplicate according to standard procedures described in Reference [32]. Nitrogen-free extracts (XX) were mathematically appreciated as the difference between organic matter values and analytically assessed organic compounds [33].

2.5. Calculations to Predict Biogas Yield and Biomethane Yield

The prediction of specific biogas yield (SBY) and specific biomethane yield (SMY) was conducted using the modified Baserga method [8] which presumes the degradation processes of substrates during anaerobic fermentation to be similar to those occurring in the rumen. Correspondingly, SBY and SMY were estimated from the degradability of XF, XP, XL, and XX, for which the digestibility coefficients were: 66, 56, 73, and 70%, respectively [34]. On the basis of DMY and SBY, the biogas yield per hectare (BY) was calculated. Standard conditions for gas volume were applied in the results.

2.6. Data Analysis

All experimental data were calculated using the statistical program Statistica 10.0 for Windows. Statistical analyses were performed using two-way analysis of ANOVA variance. Means were compared
by Tukey’s test and expressed as mean ± standard deviation. Differences were considered to be significant at a level of \( p \leq 0.05 \).

3. Results

Biomass of the analysed crops harvested in two swaths in both the second and third year of vegetation showed significant differences in terms of the content of the analysed components (Figure 1).

![Figure 1. Elemental chemical composition g (kg DM)\(^{-1}\) of the analysed crop biomass with two Sida phenotypes—Sida1 and Sida2—and Silphium.](image)

The biggest differences were found for XP and XL (Figure 1a,b). For the two-cut strategy Silphium contained, on average, 50% more XP in comparison to Sida. The content of XP in Silphium harvested once was, on average, two times lower than that found for Silphium harvested on two dates. Only in the case of Sida was it found that the content of this component increased with the age of the plant. For the two-cut strategy, both analysed crops harvested in June contained, on average, twice the amount of XP as compared with the plants harvested in October. The same tendency was found for XL; however,
neither the age of the plant nor the species had a significant effect on the content of this component—the average content in Sida and Silphium was 2.1% and 1.8% DM, respectively. The XF content in the analysed plants varied—the content determined for Sida was on average 39% DM and for Silphium 28.4% DM (Figure 1c). The content of the XF in the analysed plants was greatly affected by the date of harvest. The XF content in the plants from the second harvest was, on average, 40% higher in the case of Sida and 22% higher for Silphium as compared with the content determined in the plants from the first harvest. The content of XX in Sida and Silphium was comparable and amounted to 47.5 and 52.8% DM, respectively. TheXA content was found to be higher in Silphium than in Sida and was 8.0% and 5.4% of DM, respectively (Figure 1d,e). The method of establishing the plantation and the type of Sida phenotype showed no significant effect on the content of the analysed components.

The number of Silphium harvests per season were shown to have the greatest effect on the content of XP, which, in the case of biomass harvested twice, was on average higher by 100% as compared with biomass harvested only once—the XP content was 8.8% and 4.3% DM, respectively (Figure 1a).

The estimated SBYs from 1 kg DM of biomass samples obtained from individual plants harvested on different dates and different Sida phenotypes were comparable. In the case of Sida, it ranged from 505 to 514 L kg$^{-1}DM$, and for Silphium from 483 to 504 L kg$^{-1}DM$ (Table 2). None of the analysed factors showed a significant effect on the amount of estimated SBY. Similar results were obtained for the share of biomethane in biogas (Table 3), which, for all analysed crops, ranged from 51.0% to 52.5%. The calculated biomethane share in biogas was comparable to that found in biogas obtained from different crop substrates—on average 51% [35].

### Table 2. Mean values of specific biogas yield (SBY) of the analysed crops biomass, L kg$^{-1}DM$.

| Crop           | 2017          | 2018          |
|----------------|---------------|---------------|
|                | 2H (VI)       | 1H (X)        | 2H (VI)       | 2H (X)        | 1H (X)        |
| Sida1 planting | 509.6 ± 9.2 $A$$A$ | -           | 508.1 ± 2.7 $A$$A$ | 512.0 ± 2.7 $A$$A$ | -           |
| Sida1 seed     | 505.2 ± 4.3 $A$$A$ | -           | 504.4 ± 31.5 $A$$A$ | 512.4 ± 0.7 $A$$A$ | -           |
| Sida2 planting | 506.0 ± 46.3 $A$$A$ | -           | 506.8 ± 1.4 $A$$A$ | 508.6 ± 8.3 $A$$A$ | -           |
| Sida2 seed     | 505.0 ± 4.4 $A$$A$ | -           | 507.4 ± 3.1 $A$$A$ | 506.7 ± 2.8 $A$$A$ | -           |
| Silphium planting | 490.8 ± 4.7 $B$$A$ | 490.1 ± 4.8 $B$$A$ | 502.7 ± 2.8 $B$ | 491.8 ± 1.5 $B$$A$ | 499.8 ± 6.3 $A$$A$ | 495.5 ± 1.4 $A$$A$ |
| Silphium seed  | 483.5 ± 2.2 $B$$A$ | 490.1 ± 2.4 $B$$A$ | 504.2 ± 5.9 $B$ | 485.3 ± 2.6 $B$$A$ | 502.1 ± 3.5 $A$$A$ | 501.6 ± 1.3 $A$$A$ |

Data marked with the same letters do not differ statistically according to Tukey’s test at $p \leq 0.05$. Uppercase letters indicate the effect of type of energy crop, lowercase letters indicate the date of harvest.

### Table 3. Mean values of biomethane content in specific biogas yield (SBY) of the analysed crops biomass, %.

| Crop           | 2017          | 2018          |
|----------------|---------------|---------------|
|                | 2H (VI)       | 1H (X)        | 2H (VI)       | 2H (X)        | 1H (X)        |
| Sida1 planting | 51.0 ± 0.9 $A$$A$ | -           | 52.5 ± 0.3 $A$$A$ | 52.2 ± 0.3 $A$$A$ | -           |
| Sida1 seed     | 51.1 ± 0.4 $A$$A$ | -           | 52.5 ± 3.1 $A$$A$ | 51.5 ± 0.1 $A$$A$ | -           |
| Sida2 planting | 51.1 ± 4.6 $A$$A$ | -           | 52.8 ± 0.1 $A$$A$ | 51.9 ± 0.8 $A$$A$ | -           |
| Sida2 seed     | 51.2 ± 0.5 $A$$A$ | -           | 52.8 ± 0.3 $A$$A$ | 51.8 ± 0.3 $A$$A$ | -           |
| Silphium planting | 52.5 ± 0.5 $B$$A$ | 51.6 ± 0.5 $A$$A$ | 51.1 ± 0.3 $B$ | 52.3 ± 0.2 $A$$A$ | 51.5 ± 0.6 $A$$A$ | 51.7 ± 0.1 $A$$A$ |
| Silphium seed  | 52.9 ± 0.2 $B$$A$ | 51.3 ± 0.3 $A$$A$ | 51.3 ± 0.6 $B$ | 52.9 ± 0.1 $A$$A$ | 51.6 ± 0.4 $A$$A$ | 51.4 ± 0.1 $A$$A$ |

Data marked with the same letters do not differ statistically according to Tukey’s test at $p \leq 0.05$. Uppercase letters indicate the effect of the method of establishing the plantation, lowercase letters indicate the date of harvest.

The calculated values of BY from 1 ha showed clear differences among the analysed crops. It was found that the method of establishing the plantation and the date of harvest had significant effects on the aforementioned factor. The experiment revealed that markedly higher BY can be obtained from Silphium (Table 4)—the average amount obtained in one year was 8598 m$^3$ ha$^{-1}$ and 4759 m$^3$ ha$^{-1}$ from Sida. For both crops, the amount of BY from biomass harvested in June was several times higher than that harvested in October—for Sida it was on average 4.0 times higher and for Silphium 3.2 times higher. Higher BY was obtained from Silphium harvested two times per year as compared with the
crop harvested only once—on the means calculated for the results obtained from two years of sowing and planting, the results were 8598 and 7861 m$^3$ ha$^{-1}$, respectively. The method of setting-up the plantation had a significant effect on BY in the case of Sida2, for which it was on average 60% higher in the case of biomass obtained from the plantation set-up by planting, as compared with biomass from the plantation set-up by sowing (for the remaining crops, the calculated differences proved to be insignificant). The BY from biomass Sida1 was on average 16% higher than that obtained from Sida2.

Table 4. Mean values of biogas yield per hectare (BY) of the analysed crops biomass, m$^3$ ha$^{-1}$.

| Crop            | 2017  | 2018  |         |         |         |         |         |         |
|-----------------|-------|-------|---------|---------|---------|---------|---------|---------|
|                 | 2H (VI) | 2H (X) | sum    | 1H      | 2H (VI) | 2H (X) | sum    | 1H      |
| Sida1 planting  | 6334  | 912    | 7246   | -       | 2138    | 1094    | 3232   | -       |
| Sida1 seed      | 5542  | 980    | 6522   | -       | 2203    | 1235    | 3438   | -       |
| Sida2 planting  | 5894  | 1070   | 6964   | -       | 2771    | 1294    | 4064   | -       |
| Sida2 seed      | 3697  | 623    | 4321   | -       | 1437    | 851     | 2288   | -       |
| Silphium planting| 9971 | 2174   | 12,146 | 6130    | 4195    | 1407    | 5602   | 9223    |
| Silphium seed   | 8647  | 2429   | 11,076 | 7262    | 3456    | 2112    | 5568   | 8829    |

Data marked with the same letters do not differ statistically according to Tukey’s test at $p \leq 0.05$. Uppercase letters indicate the effect of the method of establishing the plantation, lowercase letters indicate the date of harvest.

For both analysed crop species harvested twice per year, the amount of BY from biomass produced in 2017 was on average two times higher than that obtained in 2018. This resulted from the atypical meteorological conditions in 2018, i.e., lack of precipitation and prolonged drought (from mid-April to the end of September). Meteorological conditions had no significant effect, only in the case of the Silphium yield harvested only once per year. The means of BY calculated for the results obtained from sowing and planting tests were 6696 in 2017 and 9026 m$^3$ ha$^{-1}$ in 2018.

4. Discussion

The method adopted in the present paper, based on the assumption that biogas production from organic substrates mainly depends on the content of elemental nutrients (crude protein, crude fat, crude fibre, N-free extracts), is commonly used in establishing investment assumptions and planning raw material needs for biogas plants [35]. There are numerous papers which confirm compliance of SBY estimated on the grounds of the content of various components with SBY determined using laboratory methods in digesters. Amon et al. [10] developed a methane energy value model for different energy crops on the basis of the content of nutrients. The SMY of maize and cereals measured in the eudiometer batch digesters were compared with the values estimated with the model. Similar studies were conducted by Speckmaier et al. [36] for grass and maize—when compared with laboratory analysis, the estimated biogas and biomethane yield showed differences of less than 10%. Mittweg et al. [8] compared the SMY from maize, determined by the Hohenheim biogas yield test from SMY estimated using three different models. Two of the tested chemical composition models showed a satisfactory performance compared to the mean of the measured methane values.

The SBY of the analysed crops were very similar; estimated SBY of Sida was on average 2.8% higher than that of Silphium. As shown by Weiland [7], the SBY of the individual substrates varied considerably dependent on their origin, content of organic substance, and substrate composition. Fats provide the highest biogas yield but require a long retention time due to the fact of their poor bioavailability. Carbohydrates and proteins show much faster conversion rates but lower SBY. In our study, the components for which a difference was found among the analysed crops were particularly contents of ash and protein. Mean contents of XA and XP in Silphium were 48% and 49% higher, respectively, than the content of these components in Sida. Ash is not a substrate for biogas production, and protein has a markedly lower digestibility coefficient as compared with the other components included in the model. The XA of the Sida and Silphium were high in comparison with other energy crops, e.g., maize [37]. The XA content in the examined biomass was higher in crops harvested in
June, just as in the study of the chemical properties of perennial energy crops depending on harvest period, which showed that the biomass of the other semi-wood or straw crop species contained less ash on later harvest dates [38]. Due to the relatively high contents of carbohydrates (i.e., Sida 86.4% DM, Silphium 81.3% DM) compared to the other components tested, no significant differences in terms of estimated SBY were found. Similar to the present study, the experiment was conducted by Tîtei [33], who examined the biogas production potential of *Sida hermaphrodita* and *Silphium perfoliatum* harvested twice in June and September, the estimated SBYs were smaller and amounted to 417.8 and 474.8 L kg<sub>DM</sub>⁻¹, respectively, while BY was 7323 m<sup>3</sup> ha⁻¹ and 7013 m<sup>3</sup> ha⁻¹, calculated on the dry matter yield.

Carbohydrates are composed of two fractions, one of which is XF-containing structural carbohydrates, mainly celluloses, lignins, and hemicelluloses, which ferment poorly. The second fraction is XX composed mainly of starch and sugars which are easily fermentable. Laboratory studies conducted for maize show that among the components of the substrate, the highest influence on SMY was found for starch. Correlations calculated for this component were positive and the coefficient of correlation was 0.61—the other components were water soluble carbohydrates (r = 0.41) [39]. Similar results were obtained by Rath et al. [14] who demonstrated that SBY from corn was positively correlated with the contents of starch, XL and XX—correlation coefficients were 0.51, 0.66, and 0.43, respectively [14]. Comparison of the XF and XX contents in the crops under this analysis showed that among the analysed crops, the greatest efficacy of biogas production was found for Silphium harvested twice per year and contained, on average, 38% less XF and 11% more XX as compared with Sida. For Silphium harvested once a year, the content of particular carbohydrate fractions was comparable to that determined in Sida. The study of the effects of harvesting age of Napier grass showed that the different grass harvesting ages promoted different characteristics of biomass feedstock. Longer harvesting age grass contained higher concentrations of the difficult to degrade fraction such as lignin [40].

The most recent studies [14,41–43] on the influence of the chemical composition of energy crops on biogas and methane yield show that the content of XF fractions is suggested as model variables. In their studies on 41 different crops in batch test, Dandikas et al. [41] found that 80% of the sample variation of biogas yield can be explained through lignin which, together with hemicellulose, was suggested as model variable. In numerous cases, batch tests revealed significant negative effects of acid detergent lignin on the biogas yield or methane yield [14]. Cossel et al. [42] conducted a meta-analysis with the use of data from 678 observations of biomass components and SMY from 13 potential biogas crops. They conducted validation of seven published MY estimation models with lignin content as the main variable and established their own models. The studies [14,41–43] confirmed that the prediction of SMY of crop biomass on lignocellulosic components has become a promising tool in biogas plant management and bioenergy policies, and the use of biomass source information can help optimize the precision of SMY prediction. Batch anaerobic digestion tests of SBY and SMY performed for 405 silages from 43 different crop species [43] showed that lignin content of crop biomass was found to be the most significant explanatory variable for SMY. Methane production decreases with increasing lignin content and fibre fractions, while the methane content and methane production rates were mainly affected by the content of XX and neutral detergent fibre, respectively.

The energy potential of Sida and Silphium was predominantly determined by yield of the crops, which for Silphium was, on average, 87% larger than the yield of Sida. The analysed crops showed comparable vulnerability to drought which was recorded in 2018. Unfavourable meteorological conditions most likely resulted in lower yield of the analysed crops—in 2018, it was on average twice as lower than in 2017. A significant effect of dry biomass yield on the amount of obtained biomethane was demonstrated by Oechsner et al. [16], who, on the basis of studies on several varieties of corn, beetroot, sunflower, and sorghum, found a significant positive correlation (R<sup>2</sup> = 0.98) between MY per hectare and dry matter yield.

The number of Silphium harvests had a significant effect on BY; in the case of the two-cut strategy, the effect was greater than in the case of the one-cut strategy for both plantation establishment methods.
Greater differences were identified for the plantation set-up by planting; the BY was higher on average by 51%, as compared to 29% in the case of the plantation set-up by sowing. Similar results were obtained by Jablonowski et al. [31] for Sida. Higher BY was identified for biomass harvested two times per year (in June and October) than that harvested only once (in October)—3778 and 2379 m² ha⁻¹, respectively [31]. Examination of MY of ensiled switchgrass grown in Eastern Canada showed approximately 25% more methane was produced by hectare for the two-cut strategy (2.90–3.44×10⁶ L ha⁻¹ CH₄) compared to the one-cut strategy with a harvest in late summer (2.28–2.77×10⁶ L ha⁻¹ CH₄) [44]. A study focused on the impact of different harvest dates on biogas potential expressed by the MY conducted for four novel perennial crops—cup plant (Silphium perfoliatum), energy dock (Rumex Schavnat), giant knotweed (Falopia sachalinensis var. Igniscum), and Szarvasi (Elymus elongatus ssp. ponticus cv. Szarvasi)—showed that, in most cases, the harvest date significantly influenced the tested crops and parameters [45].

5. Conclusions

The results indicated that for the tested crops, Silphium perfoliatum L. and Sida hermaphrodita L. Rusby, the method of establishing a plantation as well as the date and number of harvests had a significant effect on the content of crude ash and crude protein. However, significant differences in terms of specific biogas yield were not found for the two-cut harvesting strategy. The estimated specific biogas yield from Sida and Silphium biomass indicated the possibility of using these crops as a raw material for biogas production. Meteorological conditions had a significant impact on the biogas yield per hectare; thus, when it comes to deciding on crops for energy farming dry matter yield, crops should be examined under local conditions. The conducted experiment showed that in north-west Poland, markedly higher biogas yields per hectare can be obtained from Silphium—for three year old crops, the average amount obtained in one year was 8598 m³ ha⁻¹, while from Sida it was 4759 m³ ha⁻¹. The research conducted for Silphium confirmed that, for this plant, higher biogas yield can be obtained for the two-cut strategy compared to the one-cut strategy.

Author Contributions: H.S. partly conceived and directed the study, partly performed the experiments, and partly wrote the paper; M.W. partly performed the experiments, partly wrote the paper, and revised the manuscript; M.B. partly conceived and directed the study and partly performed the experiments; E.M. partly performed the experiments; T.K. partly performed the experiments.

Acknowledgments: This research was financially supported by Research project “Novel Pathways of Biomass Production: Assessing the Potential of Sida hermaphrodita and Valuable Timber Trees” (acronym SidaTim) co-financed by The National Centre for Research and Development in Warsaw, Poland (NCBR) within the program ERA-NET CO-FUND FACCE SURPLUS, Agreement No. FACCE SURPLUS/I/SidaTim/03/2016.

Conflicts of Interest: The authors declare no conflicts of interest.

References

1. Faber, A. Greenhouse gas emissions and carbon retention by agriculture. *Fragm. Agron.* **2001**, *4*, 102–117. (In Polish)

2. Gradziuk, P. Potential and forecast for use of agricultural biogas in Poland. *Ann. Pol. Assoc. Agric. Agribus. Econ.* **2017**, *19*, 64–70. (In Polish) [CrossRef]

3. Directive 2009/28/EC of the European Parliament and of the Council of 23 April 2009 on the Promotion of the use of Energy from Renewable Sources. Available online: https://eur-lex.europa.eu/legal-content/EN/ALL/?uri=CELEX%3A32009L0028 (accessed on 23 July 2019).

4. Fehrenbach, H.; Giegrich, J.; Reinhardt, G.; Sayer, U.; Gretz, M.; Lanje, K.; Schmitz, J. Kriterien einer nachhaltigen Bioenergienutzung im globalen Maßstab. *UBA-Forsch.* **2008**, *206*, 41–112.

5. Raboni, M.; Urbini, G. Production and use of biogas in Europe: A survey of current status and perspectives. *Rev. Ambient. Água* **2014**, *9*, 192–202.

6. Gostomczyk, W. State and Prospects for the Development of the Biogas Market in the EU and Poland—Economic Approach. *Sci. J. Wars. Univ. Life Sci.* **2017**, *2*, 48–64.
7. Weiland, P. Biogas production: Current state and perspectives. Appl. Microbiol. Biotechnol. 2010, 85, 849–860. [CrossRef]
8. Mittweg, G.; Oechsner, H.; Hahn, V.; Lemmer, A.; Reinhardt-Hanisch, A. Repeatability of a laboratory batch method to determine the specific biogas and methane yields. Life Sci. 2012, 12, 270–278. [CrossRef]
9. Baserga, U. Co-Digestion in agricultural biogas plants. Digestion of organic residues and energy grass for biogas production. FAT-Ber. 1998, 512, 1–11.
10. Amon, T.; Amon, B.; Kryvoruchko, V.; Machmüller, A.; Hopfner-Sixt, K.; Bodiroza, V.; Hrbek, R.; Friedel, J.; Pötsch, E.; Wagentristl, H.; et al. Methane production through anaerobic digestion of various energy crops grown in sustainable crop rotations. Biore sourc. Technol. 2007, 98, 3204–3212. [CrossRef]
11. Heuwinkel, H.; Oechsner, H. The Hohenheim Biogas Yield Test. Comparison of different laboratory techniques for the digestion of biomass. Agrartech. Forsch. 2003, 3, 148–149.
12. Heuwinkel, H.; Aschmann, A.; Gerlach, R.; Gronauer, A. Die Genauigkeit der Messung des Gasertragspotentials von Substraten mit der Batchmethod. Int. Wiss. Biogas Sci. 2009, 2, 95–103.
13. Weissbach, F. Evaluation of the renewable primary products for biogas production, part I: Gas production potential of fermentable nutrients. Pflanzenbauwissenschaften 2009, 13, 72–85.
14. Rath, J.; Heuwinkel, H.; Herrmann, A. Specific biogas yield of maize can be predicted by the interaction of four biochemical constituents. Bioenergy Res. 2013, 6, 939–952. [CrossRef]
15. Rath, J.; Heuwinkel, H.; Taube, F.; Herrmann, A. Predicting Specific Biogas Yield of Maize-Validation of Different Model Approaches. Bioenergy Res. 2015, 8, 832–842. [CrossRef]
16. Oechsner, H.; Lemmer, A.; Neuberg, C. Crops as a Digestion Substrate in Biogas Plants. Landtechnik 2003, 58, 146–147. [CrossRef]
17. McKendry, P. Energy production from biomass (part 1): Overview of biomass’. Bioresour. Technol. 2002, 83, 37–46. [CrossRef]
18. Podkówska, Z.; Podkówska, L. Use of sugar sorghum (Sorghum Saccharatum) for biogas production. Sci. Nat. Technol. 2016, 10, 1–8. [CrossRef]
19. Altier, M.A. The Ecological Impacts of Large-Scale Agrofuel Monoculture Production Systems in the Americas. BSTS 2009, 2016, 236–244. [CrossRef]
20. Borkowska, H.; Styk, B. Virginia Mallow (Sida Hermaphroditica Rusby) Cultivation and Use; WAR: Lublin, Poland, 2006. (In Polish)
21. Kowalski, R.; Wolski, T. Evaluation of Phenolic Acid Content in Silphium perfoliatum L. Leaves, Inflorescences and Rhizomes. EJP AU 2003, 6. Available online: http://www.ejpau.media.pl/volume6/isssue1/horticulture/article-03.html (accessed on 23 July 2019).
22. Nahm, M.; Morhart, C. Virginia mallow (Sida hermaphrodita (L.) Rusby) as perennial multipurpose crop: Biomass yields, energetic valorization, utilization potentials, and management perspectives. GCB Bioenergy 2018, 10, 393–404. [CrossRef]
23. Gansberger, M.; Montgomery, L.F.R.; Liebhard, P. Botanical characteristics, crop management and potential of Silphium perfoliatum L. as a perennial resource for biogas production: A review. Ind. Crop. Prod. 2015, 63, 362–372. [CrossRef]
24. Antonkiewicz, J.; Kolodziej, B.; Bieliriska, E.J. Phytoextraction of heavy metals from municipal sewage sludge by Rosa multiflora and Sida hermaphrodita. Int. J. Phyt. 2017, 19, 309–318. [CrossRef] [PubMed]
25. Klimont, K. Evaluating the usability of selected plant species to reclamation of the areas degraded by industry and public utilities. Probl. Agric. Eng. 2007, 2, 27–36. (In Polish)
26. Zielinski, M.; Debowski, M.; Kisielewska, M. Effectiveness of Biogas Production from Selected Energy Crops by Anaerobic Methane Digestion Supported by Microwave Radiation. Environ. Pollut. Control 2018, 40, 43–48.
27. Poskart, A.; Szwaja, S.; Musial, D. Torrefaction of Virginia mallow as substitute fuel for domestic boilers. Rynek Energii 2016, 6, 104–108.
28. Pilat, J.; Majtkowski, W.; Majtkowska, G.; Mikolajczak, J.; Goralska, A. The usefulness for ensiling of chosen plant forms of species of Silphium genus. IJCA 2007, 8, 363–368.
29. Weverac, C.; Höller, M.; Beckera, B.; Bierdtumpfeld, A.; Köhlerd, J.; Inghelantde, D.; Westhoffac, P.; Pudebc, R.; Pestsova, E. Towards high-Biomass yielding bioenergy crop Silphium perfoliatum L.: Phenotypic and genotypic evaluation of five cultivated populations. Biomass Bioenergy 2019, 124, 102–113. [CrossRef]
30. Frączek, J.; Mudryk, K.; Wróbel, M. Cup plant *Silphium perfoliatum* L.—Biomass source for biofuels production. *Agric. Eng.* 2011, 6, 21–27.

31. Jablonowski, N.D.; Kollmann, T.; Nabel, M.; Damm, T.; Klose, H.; Mculler, M.; Blaschin, M.; Seibold, S.; Krafft, S.; Kuperjans, I.; et al. Valorization of *Sida* (*Sida hermaphrodita*) biomass for multiple energy purposes. *GCB Bioenergy* 2017, 9, 202–214. [CrossRef]

32. Horwitz, W.; Latimer, G.W.J. *Official Methods of Analysis of the Association of Official Analytical Chemists*; AOAC: Virginia, VA, USA, 2006; pp. 24–44.

33. Tîţei, V. The evaluation of biomass of the *Sida hermaphrodita* and *Silphium perfoliatum* for renewable energy in Moldova. *Sci. Pap. Ser. A Agron.* 2017, 60, 534–540.

34. Podkówwa, W. Agricultural biogas—A renewable energy source. In *Theory, Practical Application*; PWRiL: Warsaw, Poland, 2012.

35. Speckmaier, M.; Schlattmann, M.; Metzner, T.; Gronauer, A. Determining Biogas Yields from Co-Substrates in Semi-Continuously Operated Laboratory-Scale Digesters. *Landtechnik* 2005, 60, 340–341. [CrossRef]

36. Stolarski, M.J.; Krzyzaniak, M.; Sniieq, M.; Slominska, E.; Piorkowski, M.; Filipkowski, R. Thermophysical and chemical properties of perennial energy crops depending on harvest period. *Int. Agrophys.* 2014, 28, 201–211. [CrossRef]

37. Zsubori, Z.T.; Pinter, J.; Spitko, T.; Hegyi, Z.; Marton, C.L. Yield and chemical composition of plant parts of silage maize (*Zea mays*) hybrids and their interest for biogas production. *Maydica* 2013, 58, 34–41.

38. Chanpla, M.; Kullavanijaya, P.; Janejadkarn, A.; Chavalparit, O. Effect of harvesting age and performance evaluation on biogasification from Napier grass in separated stages process. *KSCE J. Civ. Eng.* 2018, 22, 40. [CrossRef]

39. Herrmann, C.; Idler, C.; Heiermann, M. Biogas crops grown in energy crop rotations: Linking chemical composition and methane production characteristics. *Bioresour. Technol.* 2016, 206, 23–35. [CrossRef]

40. Masse, M.; Gilbert, Y.; Savoie, P.; Belanger, G.; Parent, G.; Babineau, D. Methane yield from switchgrass harvested at different stages of development in Eastern Canada. *Bioresour. Technol.* 2010, 101, 9536–9541. [CrossRef]

41. Mast, B.; Lemmer, A.; Oechsner, H.; Reinhardt-Hanisch, A.; Clauepin, H.W.; Graeff-Hönninger, S. Methane yield potential of novel perennial biogas crops influenced by harvest date. *Ind. Crop. Prod.* 2014, 58, 194–203. [CrossRef]