The Analysis of a Prototype Installation for Biogas Production from Chosen Agricultural Substrates

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Abstract: Methane production by fermentation is a complex biochemical process, in which micromolecular organic substances are broken down by anaerobic bacteria into simple stabilized chemicals—mainly methane CH₄ and carbon dioxide CO₂. The organic matter of the slurry consists mainly of fats, proteins and carbohydrates. As a result of biochemical changes in the process of anaerobic decomposition, some of this matter is mineralized to simple chemical compounds. Cattle and pig husbandry offers enormous potential for useable biogas plant substrates. As a result of the constantly increasing amounts of animal husbandry products, and increasingly stringent environmental protection requirements aimed at reusing natural fertilizers, it is necessary to look for alternative processing methods. The need for efficiency in obtaining biogas from substrates (e.g., manure) was met by the laboratory stand presented in this article, for which the Polish patent No. 232200 was obtained. The new technology also allows leaching of the organic liquid, e.g., from manure, and subjecting it to methane fermentation. The solution allows the individual elements of the technological line that determine the fermentation process to be tested under laboratory conditions. It also allows testing of the substrates in terms of fermentation, to determine their physical and chemical characteristics, and then to characterize the fermentation process in terms of the quality and quantity of the resulting biogas and the quality of post-fermentation residues. Compressing biogas for local distribution was also proposed. As part of the research, using a laboratory stand, the organic matter was leached from manure, for the purpose of biogas production. In addition, the biogas yield from manure at varying degrees of maturity was assessed. The best properties in terms of biogas yield forecasting were demonstrated by manure composted for 4–8 weeks.

Keywords: biomass; manure; pocket; biogas yield; methane fermentation; methane production by fermentation

1. Introduction

The growing demand for energy in Europe and the signed climate and energy package, as part of the EU climate and energy policy, aims to almost completely decarbonize the energy sector by 2050, and to reduce carbon dioxide and greenhouse gas emissions by 2030. These changes are resulting in the development of distributed energy, leading to an increase in the share of energy from renewable sources and an improvement in energy efficiency. Currently, biogas plants are highly popular as a form of energy production from renewable energy sources.

A total of 99 million tons of cattle and pig manure are produced annually in Poland, of which 78 million tons is manure. As a result of the legislative changes noted above, the
proper management and usage of raw material is highly important. The location of a biogas plant depends on economic, social, and environmental aspects. It should consider that the investment process is complex and extremely complicated, and difficult to implement in practice. According to a report of the National Center for Agricultural Support (KOWR), presently, only 116 agricultural biogas plants operate in Poland. The Energy Regulatory Office (ERO) announced that, in mid-2020, the installed capacity of all types of biogas plants in Poland was 245.148 MW.

Biogas plants in Poland are designed to use the agricultural waste in the vicinity of the plant to produce biogas. The most commonly used wastes are slaughterhouse waste, waste from sewage treatment plants, maize silage, stillage, and natural fertilizers. Part of the produced energy is locally used for the biogas plant needs, and the remainder is sold. The digestate is used as fertilizer in surrounding fields. Of the many problems related to significant livestock concentrations, the most important is the problem of manure management, which extends beyond the scope of the production process itself. A significant quantity of animal fertilizer provides a clear signal to look for solutions, i.e., rational methods of its processing and use, particular for slurry [1–4].

The production of slurry significantly exceeds the fertilization needs of crops; thus, the excess should be efficiently utilized. One use is to subject the slurry to methane fermentation. Methane production by fermentation is a complex biochemical process, in which micromolecular organic substances are broken down by anaerobic bacteria into simple stabilized chemicals—mainly methane CH$_4$ and carbon dioxide CO$_2$. The organic component of the slurry consists mainly of fats, proteins, and carbohydrates. As a result of biochemical changes in the process of anaerobic decomposition, some of this matter is mineralized to simple chemical compounds. The remaining parts, which are difficult to break down, e.g., cellulose and lignins, remain unchanged and are removed from the fermentation chamber in their original form [5–12].

The main purposes of using methane production by fermentation of natural fertilizers in agriculture are primary utilization and to obtain fuel. Considering the issue as a whole, all of the beneficial aspects should be taken into account: fertilization, energy production and, in particular, ecology [1,13–21].

The aim of this article is to present technology for obtaining biogas mainly from solid manure to increase the efficiency of biogas production. To increase the biogas yield efficiency and to speed up production, the manure can be flushed. An experiment consisting of obtaining biogas by methane fermentation of various substrates before rinsing with distilled water, and after rinsing and filtering them, was performed. This study evaluated the importance of manure washing, e.g., with liquid manure or solid manure.

2. Methane Production by Fermentation from Substrates

Cattle and pig husbandry offers enormous potential for usable biogas plant substrates in biogas installations globally [6]. As a result of the constantly increasing quantity of animal husbandry products, and increasingly stringent environmental protection requirements aimed at reusing natural fertilizers, it is necessary to look for alternative processing methods. Table 1 presents data on the quantity of nutrients in manure and corn silage.

| Substrate       | dw (%) | odw (% dw) | N   | NH$_4$ | P$_2$O$_5$ (% dw) | K$_2$O | Mg       |
|-----------------|--------|------------|-----|--------|-------------------|--------|----------|
| Cattle slurry   | 8–11   | 75–82      | 2.6–6.7 | 1–4 | 0.5–3.3 | 5.5–10 | 0.3–0.7 |
| Pig slurry      | Ca. 7  | 75–86      | 6–18 | 3–17 | 2–10 | 3–7.5 | 0.6–1.5 |
| Cattle manure   | Ca. 25 | 68–76      | 1.1–3.4 | 0.22–2 | 1–1.5 | 2–5 | 1.3 |
| Pig manure      | 20–25 | 75–80      | 2.6–5.2 | 0.9–1.8 | 2.3–2.8 | 2.5–3 | N/A |
| Chicken manure  | Ca. 32 | 63–80      | 5.4 | 0.39 | N/A | N/A | N/A |
| Corn silage     | 20–35 | 85–95      | 1.1–2 | 0.15–0.3 | 0.73 | 1.21 | 0.12 |

where: dw—dry weight. odw—organic dry weight.
Due to the relatively low dry weight content, cattle and pig manure combine well with other substrates (co-substrates). This is not the case with solid manure because, due to its high dry weight content, it usually must be diluted to be pumped; in addition, the solid organic fertilizer must be homogeneous. As a substrate, substrates with high water or energy content are mainly used (distillery slop, fats, etc.), but the most commonly used co-substrate is corn silage [1,23–26].

Storage and processing of cattle and pig slurry is not a major problem, and it can be fed directly to the biogas plant or via a preliminary tank [27].

Analysis of the literature revealed that no comprehensive solution to the above problem has been presented thus far. Most of the publications to date have discussed the problem of manure and slurry management, or the production of biogas.

However, the intensification of agricultural production has contributed to the increase in farming efficiency and reduced prices of agricultural products, and prompted the calls for measures to counteract the negative environmental effects. One of the most effective methods of managing natural fertilizers and waste biomass is anaerobic treatment in a biogas plant. The product of the process is biogas (for energy purposes) and digestate (post-fermentation mass that requires rational management) [28–30].

Based on the literature and our own research, a research problem was formulated as follows: is it possible to increase the efficiency of obtaining biogas from anaerobic fermentation of natural fertilizers under the current legal status?

The need for efficiency of obtaining biogas from substrates (e.g., manure) was met by a laboratory stand (Figure 1) for which the patent No. 232200 of 11 December 2013 was obtained (on 25 January 2019) [31].

In accordance with the patent solution, Figure 1 shows a diagram of a solution that allows testing under laboratory conditions of the individual elements of the technology that comprises the fermentation process. It also allows testing of the substrates in terms of fermentation, to determine their physical and chemical characteristics, and then to characterize the fermentation process according to the quantity of the resulting biogas, and the quality of the biogas and post-fermentation residues. This invention solves the problem of building a test stand for biogas production, mainly from a substrate comprising a mixture of manure, organic waste, and plant mass. The test stand, in accordance with the solution shown in Figure 1, has a substrate fermentation tank (1), cooperating with the preliminary fermentation tank (2), which in turn cooperates with the final fermentation tank (3). The substrate fermentation tank, the preliminary fermentation tank, and the final fermentation tank have an insulating protective layer (5) heated by a heating jacket (5). The biogas produced in these tanks is transported from the tanks via pipes (23) to a main pipe (23'), connected to the biogas tank (24), and then to the cogeneration unit (30). During transport to the tank (24), the biogas is deliquified in a dehydrator (25) and desulphurized in a desulphurizer (26).

Designed for the production of biogas from natural fertilizers and organic residues, the device can enable research on obtaining energy from substrates above 15% of dry weight. The concept of obtaining substrates in a liquid form, as a result of leaching manure at different maturity stages, is presented below. The following analytical results were collected during the successive series of tests.
Figure 1. Diagram of the test stand for the research on obtaining biogas from substrates above 15% of dry weight (patent No. 232200) (own elaboration) 1—cylinder-shaped tank for fermentation and leaching of the solid substrate placed in a mesh basket; 2—cylinder-shaped tank for preliminary fermentation of the leached organic mass; 3—cylinder-shaped tank for final fermentation of the leached organic mass; 4—overflow tank for storing liquid used to leach the solid substrate placed in a mesh basket; 5—external thermal insulation with a heating jacket and thermostat, for tanks no. 1, 2, 3; 6—cover of tank no. 1 with assembly connectors; 7—connector pipe feeding biogas to gas pipe no. 23; 8—connector discharging the leached organic mass (liquid) from the pipeline; 9—valve shutting-off tank no. 1 from no. 2 when loading the basket 35 with new substrate; 10—cover of tank No. 2 with assembly connectors; 11—cover of tank no. 3 with assembly connectors; 12—pipes with valves and pump 13 connecting tanks 2 and 3; 13—mixing pump I; 14—siphon funnel connecting the tanks 3 and 4, maintaining the appropriate gas pressure in the chambers; 15—suction and pressure pump connected to pipeline 36; 16—drainage and mixing pipeline from tank 2 and connected to pump 19; 17—drainage and mixing pipeline from tank 3, connected with pump 19; 18—cover of tank No. 4 with assembly connectors; 19—mixing pump II; 20—anti-outflow and splashing tub; 21—drain pipe of tank no. 4; 22—valves for draining liquid organic mass from tanks No. 2, 3, 4; 23—biogas discharges pipe from fermentation tanks no. 1, 2, 3, 4; 23′—end pipe (main) supplying biogas to the desulphurizer; 24—biogas tank; 25—biogas dehydrator; 26—biogas desulphurizer; 27—flame arrester; 28—biogas meter; 29—thermometers to control the temperature of the charge and the leached organic mass (liquid); 30—cogeneration unit; 31—safety valves to maintain the appropriate biogas pressure in the biogas installation; 32—biogas pressure and flow indicators in a desulphurization unit, made of a U-shaped tube; 33—thermometers to maintain the appropriate temperature of the desulphurizer during its regeneration; 34—valves to sample biogas at various stages of production and desulphurization; 35—mesh basket for solid substrate to leach organic mass; 36—pipeline to transport the leached organic mass from the tank 4 to tank 1; 37—rack; 38—connectors with valves for taking samples from the chambers; 39—inlet connector with a valve, mounted in the cover 18 of the tank 4; 40—liquid level indicator in the cover 18 of tank 4.

3. Materials and Methods

The research work, i.e., the evaluation of the biogas yield during methane production by fermentation, was carried out using the presented installation, in accordance with the procedure of the Institute of Technology and Life Science No. PB-01/LBMPZ-2010/FM standard using the German standard no. DIN 38 414 part 8 method, i.e., in an eudiometric unit with a thermostatic water bath. Additionally, the basic parameters were tested in accordance with I-ZPE/107/ed. 09.10.2008, in accordance with PN-EN 12176: 2004 (regarding the pH measurements), PN-EN 12880: 2004 (regarding the dry matter measurements), and
PN-EN 12879: 2004 (regarding the organic substance research), for ground homogenized samples of the research material. The research schedule is presented in the Table 2. The table concerns the dates of sampling and delivery, in addition to the stages of research and analysis.

Table 2. Schedule of a single study of analytical and experimental parameters for the assessment of organic matter leaching from manure (own elaboration).

| Action                                                                 | Description                                      |
|------------------------------------------------------------------------|--------------------------------------------------|
| Collection and delivery of manure samples: fresh,                       | Daily phase                                      |
| Collection and delivery of manure samples: stucked manure,              | 4–8 week phase                                   |
| Collection and delivery of manure samples: composted manure             | 4–6 month phase                                  |
| Density analysis, pH, sm, smo                                           | 2 samples in 4 replicates                        |
| Attempts to wash out with distilled water and inoculate                 | 4 samples                                        |
| Density analysis, pH, sm, smo, start of biogas fermentation            | 1 cumulative sample in 4 replicates              |
| Completion of biogas fermentation, analysis of density, pH, sm, and smo in the fermentation plant | 1 cumulative sample in 4 replicates |

Fermented separately were:
- the manure before leaching,
- the filtrate,
- the filter residue.

Test conditions:
- the study began on 13 August 2019 and was completed on 23 October 2019,
- K0 control sample—Inoculum of methane fermentation bacteria,
- incubation temperature: 37.0 °C,
- No. of repetitions for 1 sample: 3,
- volume of the fermentation mixture: 400 mL,
- laboratory conditions: temperature 21.6–22.5 °C, humidity 38.9–41.6%, pressure 1007.0–1013.0 hPa,
- total drying time: 60 h,
- total roasting time: 10 h,
- after leaching the samples were ground into chaff 2–3 cm long,
- no stabilizing and buffering additives were used,
- in order to standardize the samples, continuous mixing was applied at a temperature of 20.5 °C.

During the tests, the process conditions of the mixtures were determined as follows:
- pH-H₂O,
- dissolved oxygen (results obtained using a method outside the scope of accreditation),
- LKT/OWN alkaline buffer potential (results obtained using a method outside the scope of accreditation),
- dry weight and organic dry weight load of the fermentation mixture
- days of fermentation,
- biogas yield (the biogas yield is the net amount of gas from the tested sample; the biogas yield from the inoculum was given for guidance purposes).

Symbols used:
- leaching liquid: WD—water, IC—inoculum;
- manure: OS—fresh manure, OP—stacked manure, OD—composted manure;
- material after leaching: PC—filtrate, ST—residue.

The differences in the length of the fermentation time result from the fact that both the slurry and the matter leached from the manure consist of substances that are easily hydrolyzed and therefore undergo biochemical changes during the fermentation cycle. Straw, which is the main component of the manure, is the most difficult material to liquefy.
due to its structure. Thus, the longer the manure is composted in the heap, the shorter the fermentation time.

4. Research Results

Mean results for the evaluation of the process parameters of the tested fermentation substrate mixtures, at a 10% fermentation mixture sample load, are presented in Table 3. Detailed results of the evaluation of the post-process parameters of the tested fermentation substrate mixtures at a given percentage load of the fermentation mixture sample are summarized in Table 4.

Tables 3 and 4 show the mean values for the evaluation of the process parameters of the tested fermentation substrate mixtures, at a 10% fermentation mixture sample load (own elaboration) for the raw material and digestate. The largest CH$_4$ content (approx. 54%) was in fresh manure. The smallest was in stacked manure leached with distilled water with a value of 44%. In the case of digestates, a digestate fresh manure can be listed, which, like the inoculum (control sample), deviated from the other values for organic dry weight by about 10%. The pH-H$_2$O values in both cases (in fresh and digestate composition) remained constant at around 7%.

Figure 2 presents the essential data from Table 3. The highest value of biogas yield was shown by the OP ZTE 15/15—stacked manure sample. The yield was 66.90 ± 5.35 dm$^3$·kg$^{-1}$ dw at 52 d of fermentation. With the same number of fermentation days, fresh manure (OS) obtained a high result of biogas yield—62.49 ± 5.03 dm$^3$·kg$^{-1}$ dw. Samples of PC (OS WD)—fresh manure leached with distilled water and PC (OP WD)—manure leached with distilled water—showed a high biogas yield after 4 d of methane production by fermentation. This was 44.77 ± 3.58 dm$^3$·kg$^{-1}$ dw and 46.06 ± 3.68 dm$^3$·kg$^{-1}$ dw, respectively. The smallest biogas yield at 54 d of fermentation was obtained for the ST (OS WD) sample—after leaching fresh manure with distilled water—of 17.20 ± 1.38 dm$^3$·kg$^{-1}$ dw.

![Figure 2](image_url)
Table 3. Mean results for the evaluation of the process parameters of the tested fermentation substrate mixtures, at a 10% fermentation mixture sample load (own study).

| Process Parameters of the Fermentation Mixture | OS (10% Sample) | PC (OS WD) (10% Sample) | ST (OS WD) (10% Sample) | OP ZTE 15/15 (10% Sample) | PC (OP WD) (10% Sample) | ST (OP WD) (10% Sample) | OD (10% Sample) | PC (OD WD) (10% Sample) | ST (OD WD) (10% Sample) | K0—Inoculum |
|-----------------------------------------------|------------------|--------------------------|--------------------------|-----------------------------|-------------------------|--------------------------|----------------|--------------------------|--------------------------|-------------|
| Initial pH-H2O                                 | 7.28 ± 0.07      | 7.32 ± 0.07              | 7.31 ± 0.07              | 7.16 ± 0.07                  | 7.20 ± 0.07              | 7.20 ± 0.07              | 6.94 ± 0.07 | 6.95 ± 0.08              | 6.94 ± 0.07              | 7.02 ± 0.07 |
| compensation temp. at laboratory temp.: 20.5 °C | 28.2 °C          | 26.1 °C                  | 26.9 °C                  | 25.6 °C                      | 27.8 °C                  | 25.1 °C                  | 24.6 °C      | 26.8 °C                  | 26.1 °C                  | 31.1 °C     |
| Dissolved oxygen O2 *                          | 0.18 mg·L⁻¹      | 0.02 mg·L⁻¹              | 0.06 mg·L⁻¹              | 0.11 mg·L⁻¹                  | 0.02 mg·L⁻¹              | 0.09 mg·L⁻¹              | 0.13 mg·L⁻¹ | 0.02 mg·L⁻¹              | 0.08 mg·L⁻¹              | 0.02 mg·L⁻¹ |
| LKT/OWN alkaline buffer potential *            | 4.16             | 4.68                     | 2.94                     | 4.31                         | 4.53                     | 3.12                     | 4.33          | 4.14                     | 2.06                     | 0.08        |
| Fermentation mixture load ¹                   | 4.54% dw ± 0.14  | 2.60% dw ± 0.05          | 3.57% dw ± 0.07          | 4.51% dw ± 0.14              | 2.53% dw ± 0.05          | 3.47% dw ± 0.10          | 4.20% dw ± 0.13 | 2.49% dw ± 0.05 | 3.34% dw ± 0.07 | 2.68% dw ± 0.04 |
| WITH DRY WEIGHT                                | 68.93% dw ± 2.07 | 69.51% dw ± 2.08         | 67.86% dw ± 2.04         | 68.95% dw ± 2.07             | 69.57% dw ± 2.07         | 68.71% dw ± 2.06         | 69.13% dw ± 2.05 | 67.47% dw ± 2.02 | 69.39% dw ± 2.08 | 67.61% dw ± 1.9 |
| Days of fermentation                          | 52               | 4                        | 54                       | 52                           | 4                        | 56                       | 56            | 4                        | 58                       | 14          |
| Biogas yield, SPFM **                         | 62.49±5.03       | 44.77±5.35               | 17.20±1.38               | 66.90±5.35                   | 46.06±3.68               | 29.84±2.39               | 56.79±4.54   | 20.44±1.64               | 28.34±2.27               | 0.88±0.07   |
| CH₄ content                                   | 53.6%            | 46.8%                    | 51.3%                    | 55.1%                        | 48.8%                    | 50.5%                    | 50.7%         | 43.6%                    | 50.3%                    | 51.6%       |
| NH₃                                           | 23.6 ppm         | 12.3 ppm                 | 20.2 ppm                 | 18.4 ppm                     | 18.5 ppm                 | 12.2 ppm                 | 8.7 ppm       | 4.7 ppm                  | 4.0 ppm                  | 2.56 ppm     |
| H₂S                                           | 128 ppm          | 113 ppm                  | 22 ppm                   | 216 ppm                      | 125 ppm                  | 24 ppm                   | 34 ppm        | 30 ppm                   | 22 ppm                   | 9 ppm        |

¹ The loading of the fermentation mixture with dry mass and dry organic mass in terms of fresh mass and dry mass; * The results for dissolved oxygen and alkaline buffer potential were obtained by a method outside the scope of accreditation; ** The result of biogas yield is the amount of gas from the tested sample, the result of biogas yield from the inoculum is indicative. Symbols: OS—fresh manure; PC (OS WD)—fresh manure leached with distilled water, ST (OS WD)—after leaching fresh manure with distilled water, OP ZTE 15/15—stacked manure, PC (OP WD)—stacked manure leached with distilled water, ST (OP WD)—after leaching stacked manure with distilled water, OD—composted manure, PC (OD WD)—composted manure leached with distilled water, ST (OD WD)—after leaching composted manure with distilled water, K0—inoculum (control sample); †—homogeneous group.
Table 4. Mean results for the evaluation of the process parameters of the tested fermentation substrate mixtures, at a 10% fermentation mixture sample load (own elaboration)—digestate (own study).

| Process Parameters of the Post-Fermentation Mixture | Digestate OS ² | Digestate PC (OS WD) ² | Digestate ST (OS WD) ² | Digestate OP ZTE 15/15 ² | Digestate PC (OP WD) ² | Digestate ST (OP WD) ² | Digestate OD ² | Digestate PC (OD WD) ² | Digestate ST (OD WD) ² | Digestate K0—Inoculum |
|----------------------------------------------------|----------------|------------------------|------------------------|--------------------------|------------------------|------------------------|----------------|------------------------|------------------------|------------------------|
| pH-H2O final compensation temperature at laboratory temperature: 20.3 °C | 7.02 ± 0.07 | 7.18 ± 0.07 | 6.92 ± 0.07 | 7.14 ± 0.08 | 7.55 ± 0.08 | 6.48 ± 0.07 | 7.18 ± 0.07 | 7.52 ± 0.07 | 6.94 ± 0.08 | 7.34 ± 0.07 |
| Dissolved oxygen O2 * | 0.02 mg L⁻¹ | 0.02 mg L⁻¹ | 0.02 mg L⁻¹ | 0.02 mg L⁻¹ | 0.02 mg L⁻¹ | 0.02 mg L⁻¹ | 0.02 mg L⁻¹ | 0.02 mg L⁻¹ | 0.02 mg L⁻¹ | 0.02 mg L⁻¹ |
| Alkaline buffer potential LKT/OWN | 0.68 | 0.05 | 0.83 | 0.89 | 0.08 | 0.98 | 0.76 | 0.04 | 1.12 | 0.028 |
| Residue in the post-fermentation mixture ¹ WITH DRY WEIGHT | 4.4% dw ± 0.1 | 1.5% dw ± 0.1 | 4.1% dw ± 0.1 | 5.3% dw ± 0.1 | 1.8% dw ± 0.1 | 4.0% dw ± 0.1 | 4.6% dw ± 0.1 | 1.2% dw ± 0.1 | 5.1% dw ± 0.1 | 2.10% dw ± 0.0 |
| ORGANIC DRY WEIGHT | 52.2% dw ± 1.6 | 32.0% dw ± 1.3 | 39.8% dw ± 1.3 | 46.3% dw ± 1.4 | 34.2% dw ± 1.3 | 36.8% dw ± 1.3 | 42.8% dw ± 1.4 | 32.4% dw ± 1.3 | 36.1% dw ± 1.3 | 50.4% dw ± 1.8 |

¹ The loading of the fermentation mixture with dry mass and dry organic mass in terms of fresh mass and dry mass; ² 10% sample; * The results for dissolved oxygen and alkaline buffer potential were obtained by a method outside the scope of accreditation. Symbols: Digestate OS—digestate fresh manure; Digestate PC (OS WD)—digestate fresh manure leached with distilled water, Digestate ST (OS WD)—digestate leaching fresh manure with distilled water, Digestate OP ZTE 15/15—digestate stacked manure, Digestate PC (OP WD)—digestate stacked manure leached with distilled water, Digestate ST (OP WD)—digestate after leaching stacked manure with distilled water, Digestate OD—digestateet composted manure, Digestate PC (OD WD)—digestate composted manure leached with distilled water, Digestate ST (OD WD)—digestate after leaching composted manure with distilled water, Digestate K0—inoculum (control sample).
As part of the analysis of the obtained results, the parameters of the fermentation mixture process were analyzed. During the parameter evaluation, the ANOVA method with Duncan’s post-hoc test was used. The method allowed the determination of the influence of the analyzed parameters among themselves. In statistical analysis, the STATISTICA computer program was used. The expected marginal means that characterized the influence of fermented material on the amount of obtained biogas were determined. Statistical analysis confirmed a relationship between the analyzed factors. In a specific case, the ANOVA analysis allowed the influence of individual factors on the initial parameter to be determined by classifying homogeneous groups and mutual relationships of the analyzed parameters. In all cases, the critical level of significance determining the assignment to a particular homogeneous group was below 0.05 (5%). The significance level $p$ showed a lower value than the established level ($p < 0.05$) for the empirical value of the statistics $F(9, 20) = 119.56$. All of the results of the ash statistical analysis are presented in the Table 3. In Table 3, the CH$_4$ content parameter is also shown. The amount of CH$_4$ in the biogas from fresh manure fermentation (OP) was 53.6%. The biogas produced from the residues after washing fresh manure with distilled water had a CH$_4$ content of 51.3%, and in its filtrate it was 46.8%. In the remaining samples, biogas from piled manure and mature manure also had a higher CH$_4$ content than the residue after washing.

However, importantly, after flushing the manure (fresh, stacked, composted), their filtrates had a high yield of biogas at 4 d. Therefore, the share of flushing as a parameter had a large impact on the process of biogas yield, increasing the speed in comparison with unwashed manure. Fresh and stacked unwashed manure was fermented 52 days and composted manure for 56 days. The biogas yield in washed samples was 28.34% to 61.4% lower compared to the manure before washing. In this case, the fermentation time was faster by 48 and 52 days. This influences the efficiency and efficiency of the process and the amount of obtained biogas. From laboratory studies, the following estimates were made, after placing the batch in the digestion chamber every 4 days with fresh manure leached with distilled water (PC (OS WD), stacked manure leached with distilled water (PC (OP WD) and composted manure leached with distilled water) water (PC (OD WD):

- in the case of PC (OS WD)—increased efficiency of biogas yield by approx. 931.36% compared to fresh manure, i.e., approx. $582.01 \pm 3.58 \text{ dm}^3\cdot\text{kg}^{-1}$ (for 52 d);
- in the case of PC (OP WD)—increased efficiency of biogas yield by approx. 895.03% compared to stacked manure, i.e., approx. $598.78 \pm 3.68 \text{ dm}^3\cdot\text{kg}^{-1}$ (for 52 d);
- in the case of PC (OD WD)—increased efficiency of biogas yield by approx. 503.89% compared to the composted manure, i.e., approx. $286.16 \pm 1.64 \text{ dm}^3\cdot\text{kg}^{-1}$ (for 56 d).

The filtrates showed a lower CH$_4$ content. The amount of NH$_3$ tested in the biogas decreased in the filtrates and residues of fresh and mature manure compared to the samples before washing. Only in the manure filtrate washed with water was a slight increase in NH$_3$ content observed. In the case of H$_2$S content, all of the rinsing samples showed a decrease in biogas after fermentation.

After methane production by fermentation, the post-fermentation mixtures presented in Table 4 were tested. It can be noted that the initial pH-H$_2$O of the tested samples (Table 3) slightly changed in relation to the final pH-H$_2$O in the tested post-fermentation mixtures. In all samples after fermentation, the amount of dry organic matter decreased. Additionally, the alkaline buffer potential and dissolved oxygen decreased.

The designed test stand shown in Figure 1, which is the subject of patent No. 232200, allows comprehensive testing of the biogas production efficiency in connection with the quality of the obtained substrate after leaching and fermentation. In particular, it allows:

- analysis of the physico-chemical parameters of manure, the leaching liquid, and the leached mixture and fermentation mixture, including weight, density, pH, humidity, dry weight, organic dry weight, and ash content;
- assessment of the efficiency of leaching of solid particles from batches of manure at various technological stages, i.e., with different degrees of maturity;
• evaluation of biogas profitability of the tested technological phases of manure and the leached mixture to determine the biogas efficiency of the designed technology.

5. Discussion

5.1. Technological Concepts

Based on the above results of fermentation of the organic mass leached from manure, the test stand used, in addition to the technological concepts used by Gicon (Germany), are presented in Figure 3 [32]. This covers the leaching of the liquid organic substrate from the fertilizer mass or corn silage, which is fed into the fermentation chamber as the basic substrate for methane production by fermentation.

The technology for obtaining biogas from leached organic matter, especially manure, was improved in a solution from 2019 (patent application No. P.421062) [33].

The technology designed according to this diagram is shown in Figure 4—vertical section, and in Figure 5—top view.
The patent application presents a system comprising livestock buildings and devices for biogas production, which can be used both in small (family) farms and in agricultural holdings.

The livestock building (1) has a technological connection with the slurry bottom (2), where the manure (3) is collected and then loaded with a loader (4) (connected with a tractor) into the containers (5). During the collection in the manure bottom and loading into the container (5), slurry drips from the manure (3), running off by gravity into a slurry pre-tank (6) situated under the loading area.

The biogas production equipment includes a silo-leaching chamber (7), divided into hermetically closed sectors, in which containers (5) are filled with manure (3) and hermetically closed for a period of about 30 d. The floors in the containers (5) are openwork. An outflow (9) is made in the floor of each of the sectors of the chamber (7). The outflow is connected by the discharge pipe (10) directed to the organic fraction tank (11). In this tank, the organic fraction is collected after rinsing the manure (3) contained in the containers (5) in the chamber (7).

At the top, in each sector of the chamber (7), sprinkler nozzles (12) are mounted, across which the slurry for leaching the manure (3) is supplied. The organic fraction leached from the manure (3) together with the solid elements are transferred through the discharge pipe (10) to the tank (11).

The organic fraction tank (11) is connected by a pipe (14) to the fermentation chamber of the biogas plant (13). At the end of the pipe (14), a pump (15) is installed in the tank (11). The pump (15) is responsible for forcing the organic fraction into the fermentation chamber (13), where the methane fermentation takes place.

During the methane production by fermentation, biogas is produced which is discharged into the biogas tank via a pipe. Biogas is also produced when the manure (3) is stored in the chamber (7). Biogas is discharged from each sector via pipe (8). Using overpressure, it is pumped into the tank after dehydration and desulphurization [34].

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Figure 4. Technological scheme of biogas production from natural fertilizers—vertical section (own elaboration). 1—livestock building, 2—slurry, 3—manure, 4—loader, 5—container, 6—slurry tank, 7—set of hermetic chambers for the containers, 8—gas pipeline, 9—organic liquid fraction outflow, 10—liquid organic fraction discharge pipeline, 11—liquid organic fraction tank, 12—sprinklers, 13—fermentation chamber (biogas plant), 14—pipeline supplying the liquid organic fraction, 15—pump, 17—container for expanded clay with methane bacteria, 18—solar collector, 19—heat exchanger, 20—fermented substrate outflow pipeline, 21—equalization tank; 22—separator supply pipeline, 23—separator, 24—slab for the separated solid mass, 25—liquid tank for the separated fermented substrate, 26—sprinkler supply pipeline, 27—control valve “I”, 28—control valve “II” of the main storage tank, 29—pipeline feeding the processed liquid to the storage tank, 31—agitator, 32—screw conveyor feeding the substrate into the biogas chamber, 35—pipeline for transporting the slurry.
by gravity into a final tank (25), which is connected to the separator (23). Sprinkler nozzles (12) are installed in the silo-leaching chamber (7), in the upper walls, for flushing out the manure (3) accumulated in the containers (5). The sprinklers are supplied with manure liquid from the pump (26) to the separated liquid from the final container via the pipe (27). The control valve (28) is installed on the liquid manure pipe (27), enabling the liquid manure flow to be switched to the appropriate sector (8). The surplus liquid manure is routed through pipe (29) to a storage tank (30).

An axial agitator (31) is installed in the fermentation chamber (13), which ensures homogenization of the mass and allows uniform fermentation. A further piece of the unit is the screw conveyor (32) by means of which the energy crop substrate is added to the fermentation chamber (13). Generally, this is shredded maize in raw or silage form, or grass. The biogas resulting from methane fermentation should be subjected to dehydrated and desulphurized. After this treatment, the biogas is then transported to the storage tank. The next step is to transfer the biogas (biomethane) via a pipeline (32) to the cogeneration unit (33). In this unit, the biogas is converted into heat and electricity [34].

Expanded clay was used to stimulate the fermentation phenomenon. This is located in the tubular containers (17), which are positioned in the side wall of the fermentation chamber (13). An important part of the chamber (13) equipment is a solar panel (18), supplying heaters (19), installed inside the fermentation chamber (13). From the side wall of the chamber (13) there is a drainage pipe (20) directed to the equalization tank (21) is provided. This enables the chamber (13) to gravitationally discharge the digested substrate and maintain a constant level of this substrate in the chamber (13). The fermented substrate accumulated in the equalization tank (21), by means of the pump (15) installed therein, is fed through the separator pipe (22) to the separator (23). The separator (23) separates the solid fractions from the liquid fractions. The solid fraction in the form of a separated mass is discharged to a heap in a composting slab (24). It is the used for animal bedding or compost, and the liquid fraction, in the form of a slurry liquid, is discharged by gravity into a final tank (25), which is connected to the separator (23). Sprinkler nozzles (12) are installed in the silo-leaching chamber (7), in the upper walls, for flushing out the manure (3) accumulated in the containers (5). The sprinklers are supplied with manure liquid from the pump (26) to the separated liquid from the final container via the pipe (27). The control valve (28) is installed on the liquid manure pipe (27), enabling the liquid

**Figure 5.** Technological scheme of biogas production from natural fertilizers—horizontal view (own elaboration). 1—livestock building, 2—slurry, 4—loader, 5—container, 6—slurry tank, 7—set of hermetic chambers for containers, 8—gas pipeline, 11—liquid organic fraction tank, 13—fermentation chamber (biogas plant), 16—biogas tank, 18—solar collector, 23—separator, 24—slab for separated solid mass, 29—pipeline feeding the processed liquid to the storage tank, 30—processed liquid storage tank, 31—agitator, 33—cogeneration unit, 34—slab for leached mass, 35—pipeline for transporting the slurry.
manure flow to be switched to the appropriate sector (8). The surplus liquid manure is routed through pipe (29) to a storage tank (30).

An axial agitator (31) is installed in the fermentation chamber (13), which ensures homogenization of the mass and allows uniform fermentation. A further piece of the unit is the screw conveyor (32) by means of which the energy crop substrate is added to the fermentation chamber (13). Generally, this is shredded maize in raw or silage form, or grass. The biogas resulting from methane fermentation should be subjected to dehydrated and desulphurized. After this treatment, the biogas is then transported to the storage tank. The next step is to transfer the biogas (biomethane) via a pipeline (32) to the cogeneration unit (33). In this unit, the biogas is converted into heat and electricity [34].

The selection of individual parameters of the above-described concept of a substrate-based biogas installation for methane production by fermentation is presented in Table 5.

Table 5. Biogas yield and methane content in natural fertilizers; own study according to Schattauer, Weiland [22].

| Substrate         | Biogas Yield (m³·t⁻¹ dw) | Biogas Yield (m³·t⁻¹ odw) | CH₄ Content (% Vol.) |
|-------------------|--------------------------|---------------------------|---------------------|
| Cattle slurry     | 20–30                    | 200–500                   | 60                  |
| Pig slurry        | 20–35                    | 300–700                   | 60–70               |
| Cattle manure     | 40–50                    | 210–300                   | 60                  |
| Pig manure        | 55–65                    | 270–450                   | 60                  |
| Chicken manure    | 70–90                    | 250–450                   | 60                  |
| Corn silage       | 170–200                  | 450–700                   | 50–55               |

where: dw—dry weight; odw—organic dry weight.

Biogas yield from cattle slurry with a yield of 20 to 30 m³·vol⁻¹ of substrate is slightly lower than that of pig slurry. In addition, the gas from cattle slurry has a significantly lower average methane content compared to the gas from pig slurry. This is because the cow’s stomach works in a similar way to a biogas plant, thus, the slurry is pre-fermented.

5.2. Local Transport and Distribution of Biogas

The obtained biogas is usually transported from the source to the place of use via pipelines, in a compressed form. In cases in which the construction of a pipeline is economically ineffective or impossible, for example for geographic reasons, gas can be transported in appropriate liquefied or gas tanks under high pressure. Compressed gas may be transported occasionally in small dedicated high-pressure cylinders, or as part of special transport systems. The composition of raw biogas varies widely, depending on the raw material and fermentation technology, and generally consists of 55–75% methane, 25–45% carbon dioxide, 1–5% hydrogen, 0–0.3% nitrogen, 0.1–0.5% oxygen and 0.1–3.0% hydrogen sulfide. This gas is saturated with water vapor and may also contain a number of other impurities, such as ammonia, carbon monoxide, and volatile siloxanes, and even droplets of liquid and mechanical impurities entrained from the fermenter. Biogas is produced in a dispersed system and in small amounts, which complicates its use and often necessitates transport of the biogas to the place of its use [6].

The core of the invention (patent No. 232201 of 13 June 2017) is a system for transporting small amounts of biogas. This consists of a compression unit and a high-pressure container mounted on the platform, from which the gas is transferred to the storage and expansion unit located at the site where the biogas is used. A biogas treating module installed at the biogas inlet, before the compression station, removes at least mechanical impurities and suspended liquid drops, water vapor to dew point level under post-compression conditions, and hydrogen sulfide and siloxanes. Usually, adsorption treatment is used for small amounts of biogas. Preferably, two detachably connected high-pressure containers are placed on the platform trailer [35].
The invention is demonstrated in Figure 6, which schematically shows a system for transporting small amounts of biogas. The raw biogas is supplied to the treatment unit (1), which uses a type 3A zeolite-filled adsorber for treatment. The treated biogas is then sent to the compression station (6), where it is pressurized to 25 mPa with a piston compressor, and then forced into a 140 dm$^3$ high-pressure container (3) mounted on a platform trailer via a flexible hose. It is preferable to use two detachable containers (3). The trailer with high-pressure containers (3) is transported to the place where biogas is used. There, the high-pressure containers (3) are connected to the storage and expansion module (4) with a flexible hose. The biogas from the containers (3) is pumped into the module (4), from where it is directed for use via the reduction valve (5).

This system was used in Jaworze, in the Experimental Station of National Research Institute of Animal Production (in Grodziec Śląski).

The installation was designed and then implemented as a prototype under the BIOS-TRATEG1 program by a team of the Institute of Technology and Life Sciences in cooperation with NGV “Autogas Sp. z o.o”. The concept of the presented system consists of the following technological units: a module for cleaning biogas fed from the biogas tank, a compressor, high-pressure containers/cylinders for storing the compressed biogas mounted on a transport trailer, a station for expanding and collecting biogas for energetic needs of animal production, and a reduction valve allowing to supply energy receivers with the transported biogas.

Detailed diagrams of cylinder filling and biogas transporting units, and installations for unloading compressed biogas in livestock buildings, are shown in Figures 6 and 7.
The view of the biogas refueling and compression station with brief technical characteristics of the compressor is shown in Figure 8. A view of the connected cylinders with the regulator to supply biogas consumers is shown in Figure 9. In addition, a transport trailer with cylinders filled with biogas is shown in Figure 10.

5.3. Advantages of Prototype Installation

The presented prototype of the installation for laboratory testing of the efficiency of biogas extraction (Figure 1), patent No. 233300, allows for the selection of the composition and characteristics of the substrates subjected to organic matter leaching and further methane fermentation.

During the laboratory tests, the characteristics of the energy potential of the substrate subjected to the initial treatment were determined. The presented solution can be used entirely for biogas treatment and production, or it can be an element of biogas installation equipment, e.g., above 75 kW with a stocking density of more than 200 LU with annual production of manure of 1200 m³ and slurry of 1800 m³. Installations according to the discussed parameters for the needs of a specialized farm above 200 LU are presented in Figures 4 and 5.

Figure 7. Diagram of the installation assembly at the livestock building for biogas unloading: 1—connector for refueling from the trailer; 2—pressure gauge; 3—stationary cylinders with a capacity of 140 L H2O (140 m³ × 5 pcs. = 700 m³ gas) located next to the livestock building; 4—shut-off valves (manual); 5—reducer 250 bar/2 bar; 6—reducer 2 bar/2 kPa; 7—biogas reception; 8—earthing; E—earthing wire; GSW—main equalizing beam; CC—equalizing cables (own study).

Figure 8. Gas compression station—view of the gas installation. Gas compressor with a maximum power input of 2.5 kW and a nominal capacity of 5 m³/h (at 15 °C, 200 bar, at 0.1 bar gas pressure at the inlet) (resources of the Institute of Technology and Life Sciences).
trailer with cylinders filled with biogas is shown in Figure 10. In addition, a transport equipment, e.g., above 75 kW with a stocking density of more than 200 LU with annual production of manure of 1200 m³ and slurry of 1800 m³. Installations according to the discussed parameters for the needs of a specialized farm above 200 LU are presented in Figures 4 and 5.

5.3. Advantages of Prototype Installation

The solutions allowing biogas to be obtained from substrates above 15% of dry matter (manure and energy crops) presented above allow for the adaptation of the presented technologies in farms conducting, for example, bedding livestock and production of energy crops (e.g., corn). The prototype installation for obtaining biogas is of significant importance in terms of obtaining renewable energy and is a universal solution that allows supplementation of an operating biogas plant or it can be a stand-alone installation. This allows the transition from conventional energy sources to renewable energy sources.

The presented prototype installation increases the efficiency of the process by flushing the manure with distilled water. In real conditions, slurry and liquid manure can be used for flushing. As a result, the production efficiency is increased. After rinsing, the tested samples ferment in 4 d, rather than 52 or 54 d, and a satisfactory biogas yield is obtained more quickly. As a result, the fermentation chamber can be used more quickly for the next batch, which allows an increase in biogas production. Additionally, with such a
rate of biogas production, a smaller fermentation chamber can be built, which lowers the installation costs.

According to the assessment of a traditional biogas installation, the real cost of electricity production expressed in EUR·MWh$^{-1}$ is 27.02. The real cost of thermal energy is EUR 49.26·MWh$^{-1}$. According to the estimated parameters, the cost of the 400 m$^3$ installation may be 45% lower than the cost of a traditional installation.

6. Conclusions

In Poland, the barn breeding system is dominant, producing approx. 50 million tonnes of manure per year. The presented technological solutions for obtaining biogas from substrates above 15% of dry weight enable the biogas yield to be increased during the fermentation process. The presented technologies also make it possible to obtain biogas from liquid mixtures leached from the solid substrates in biogas chambers, in line with the needs of agricultural producers and with ecological requirements.

- the obtained analytical details from standard (laboratory) tests allow the energy efficiency of the substrates subjected to anaerobic digestion to be assessed, to supply installations in farms above 100 LU;
- based on the research results, the presented technologies allow a 15–20% greater methane content in biogas to be obtained than in the case of conventional installations;
- the gas can be obtained from leached manure or silage substrate, which is then used for the production of compost or mulch;
- the obtained substrate in the form of post-fermentation liquid can then be separated and immediately used as a valuable natural fertilizer or as fertilizer granules;
- the obtained biogas can also be transported with the use of small gas cylinders when the construction of the pipeline is economically ineffective or impossible.

The results for the evaluation of the process parameters of the tested fermentation substrate mixtures (10%) of the fermentation mixture sample lead to the following conclusions:

- Among the substrates used in the experiment, i.e., three types of manure, the best properties in terms of biogas yield forecasting were demonstrated by stacked manure, before fresh manure and composted manure.
- For the OS substrate (fresh manure), the yield of biogas was $62.49 \pm 5.03$ dm$^3$·kg$^{-1}$ dw and for OP (stacked manure) the yield was higher by 7.05%, whereas for OD (composted manure) the yield was lower by 9.12%.
- For filtrates (PC) from fresh manure (leached with distilled water) the yield was $44.77 \pm 3.58$ dm$^3$·kg$^{-1}$ dw, and for composted manure leached with distilled water the yield was $46.06 \pm 3.68$ dm$^3$·kg$^{-1}$ dw.
- For the residue (ST), after leaching fresh manure (with distilled water), the yield was $17.20 \pm 1.38$ dm$^3$·kg$^{-1}$ dw.
- The derived statistics in the form of an analysis of variance were used to investigate the influence of material parameters (fermented material) on the process (biogas yield). The analysis in some cases confirmed the statistical relationship, e.g., fresh manure (OS) and composted manure (OD) belonged to completely different homogeneous groups than manure influenced by distilled water, ranging from 57 to 63 dm$^3$·kg$^{-1}$ dw.
- Methane fermentation of washed manure (fresh, stacked, composted) required less time (4 d), rather than 52 and 54 d (in the case of manure before flushing).
- Washing the manure increases the production efficiency, because after 4 days a high yield of biogas is obtained, therefore, after the end of fermentation, a new batch can be introduced into the fermentation chamber more often and subjected to methane fermentation.
- The estimated efficiency of the biogas yield from washed samples is higher by approx. 503.89% to 931.36% compared to samples before washing.

The washed substrate samples with distilled water enables the biogas yield to be increased, with short fermentation time. In addition, the washing residue can also be
subJECTED TO METHANE PRODUCTION BY FERMENTATION IN ORDER TO BE ABLE TO USE IT LATER, E.G., IN ARABLE FIELDS. FURTHER RESEARCH ON THIS INSTALLATION IS BEING CARRIED OUT AS PART OF DOCTORAL STUDIES ON THE ENERGY AND FERTILIZATION EFFECTS OF METHANE PRODUCTION BY FERMENTATION (ANAEROBIC FERMENTATION FROM THE OBTAINED LIQUID FRACTION FROM MANURE AND ENERGY CROPS) AND OTHER STUDIES. THE RESEARCH PRESENTED IN THIS PAPER ALLOWS FOR A COMPREHENSIVE STUDY OF THE EFFICIENCY OF OBTAINING BIOMETHANE IN CONNECTION WITH THE QUALITY OF THE OBTAINED SUBSTRATE AFTER WASHING AND FERMENTATION, IN PARTICULAR:

- Analysis of physico-chemical parameters of manure, washing liquid, leached mixture and fermentation mixture, including weight, density, pH, humidity, dry matter, organic dry matter, and ash content;
- Assessment of the efficiency of leaching of solid particles from a batch of manure at various technological stages, i.e., with a different degree of maturity;
- Evaluation of the quality of the fermentation substrate of energy;
- Evaluation of the biogas yield of the tested technological phases of manure and the leached mixture to determine the biogas efficiency of the designed technology.

In addition, the obtained results of this research will allow the selection of technological and technical parameters to be carried out on farms or installations operating for the needs of heat and electricity. The limitations of the presented research may be the inability to conduct the research in the biogas facility. Therefore, the research facility can operate a number of biogas installations or can be used to develop recommended feedstock compositions in target installations.

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