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Study of Methane Fermentation of Cattle Manure in the Mesophilic Regime with the Addition of Crude Glycerine

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Abstract: The urgency of the study is due to the need to increase the productivity of biogas plants by intensifying the process of methane fermentation of cattle manure in mesophilic mode by adding to it the waste from biodiesel production: crude glycerine. To substantiate the rational amount of crude glycerine in the substrate, the following tasks were performed: determination of dry matter, dry organic matter, and moisture of the substrate from cattle manure with the addition of crude glycerine; conducting experimental studies on biogas yield during fermentation of cattle manure with the addition of crude glycerine with periodic loading of the substrate; and development of a biogas yield model and determination of the rational composition of crude glycerine with its gradual loading into biogas plants with cattle manure. The article presents the results of research on fermentation of substrates in a laboratory biogas plant with a useful volume of 30 L, which fermented different proportions of crude glycerine with cattle manure at a temperature of 30 °C, 35 °C, and 40 °C. The scientific novelty of the work is to determine the patterns of intensification of the process of methane fermentation of cattle manure with the addition of different portions of crude glycerine. A rapid increase in biogas yield is observed when the glycerol content is up to 0.75%. With the addition of more glycerine, the growth of biogas yield slows down. The digester of the biogas plant, where experimental studies were conducted on the fermentation of substrates based on cattle manure with the addition of co-substrates, is suitable for periodic loading of the substrate. As a rule, existing biogas plants use a gradual mode of loading the digester. Conducting experimental studies on biogas yield during fermentation of cattle manure with the addition of crude glycerine with periodic loading of the substrate makes it possible to build a mathematical model of biogas yield and determine the rational composition (up to 0.75%) of crude glycerine with its gradual loading in biogas plants. Adding 0.75% of crude glycerine to the substrate at a fermentation temperature of 30 °C allows to increase the biogas yield by 2.5 times and proportionally increase the production of heat and electricity. The practical application of this knowledge allows the design of an appropriate capacity of the biogas storage tank (gasholder).

Keywords: biogas plant; substrate; crude glycerine; cattle manure; biosludge

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Citation: Romaniuk, W.; Rogovskii, I.; Polishchuk, V.; Titova, L.; Borek, K.; Wardal, W.J.; Shvorov, S.; Dvornyk, Y.; Sivak, I.; Drahniev, S.; et al. Study of Methane Fermentation of Cattle Manure in the Mesophilic Regime with the Addition of Crude Glycerine. *Energies* **2022**, *15*, 3439. https://doi.org/10.3390/en15093439

Academic Editor: Attilio Converti

Received: 11 April 2022
Accepted: 6 May 2022
Published: 8 May 2022

1. Introduction

The lack of energy resources in the world determines the intensification of activities in the field of adaptation and operation of existing or new technologies for the production of clean and cheap energy from renewable sources [1,2]. Biofuels are used to replace petroleum fuels: biomass (wood, agricultural and processing waste, energy crops), fuel pellets, and biomass briquettes [3], including torrefied fuel pellets and briquettes [4], biodiesel, and biohydrogen. Biogas technologies are being actively introduced into production. The urgency of the study is due to the need to increase the productivity of biogas plants by intensifying the process of methane fermentation of cattle manure. Such manure is characterized by rather low productivity of biogas output, due to which modern biogas plants have low profitability [5]. In order to increase the yield of biogas from cattle manure, it is advisable to use as co-substrates—waste from agricultural and processing production, at the same time eliminating the need for their disposal. Such co-substrates include sediments of vegetable oils [6], soapstock [7], substandard flour [8], and extruded straw [9,10]. One of such co-substrates is crude glycerine, which is formed as a by-product of biodiesel production [11,12].

In [13–21], the compatible methane fermentation of cattle manure with crude glycerine was studied, while in [22–34] also the fermentation of other substrates (sewage, maize silage, algae, etc.) with crude glycerine. The papers note that the use of crude glycerine as a co-substrate generally increases the yield of biogas. At the same time, the rate of biogas yield increased with the addition of 2% of crude glycerine [35], while the addition of 10% of crude glycerine to the substrate inhibited the rate of biogas yield [35,36].

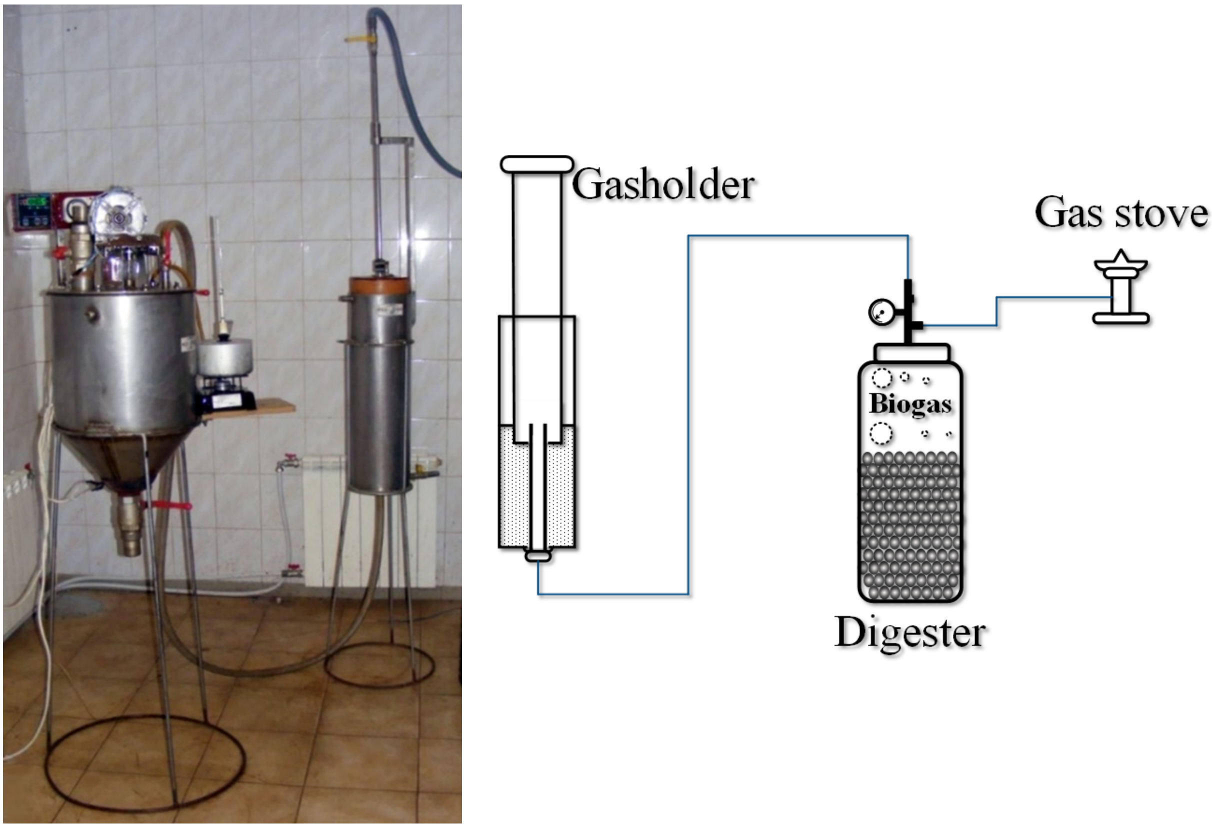
Thus, there is a need for further research to substantiate the concentration of crude glycerine in the substrate based on cattle manure. The aim of this study is to increase the yield of biogas from cattle manure by adding crude glycerine in mesophilic mode.

2. Materials and Methods

An experimental study of the methods and regimes of cattle manure methane fermentation with the addition of crude glycerine for biogas production was carried out at a lab-scale biogas plant (Figure 1a) in the educational scientific laboratory of bioconversion in the agro-industrial complex of the National University of Life and Environmental Sciences of Ukraine. The biogas plant includes a digester with a usable volume of 30 L and a gasholder (Figure 1b).

Biogas from the methane tank through the pipeline enters the gasholder, where it is stored. This installation uses a “wet” gasholder, which consists of two hollow cylindrical containers (the body and a cylinder-level gauge) as well as a guide. The body of the gasholder is filled with water, in which the hollow cylinder-level gauge floats like a boat. Biogas enters the inner cavity of the cylinder-level gauge, which rises above the body of the gasholder along with the guide that allows determining the presence and volume of gas in the gasholder. From the gasholder, biogas enters the gas stove by squeezing the mass of the cylinder-level gauge.

The digester (fermentation chamber) works with periodic substrate loading. To the substrate that consisted of 3.5 kg of cattle manure and 5 kg of water was added crude glycerine weighing 50, 100, 200, and 300 mL, or 0.6%, 1.2%, 2.3%, and 3.4% of the substrate mass, respectively. The 30 L digester was half loaded with the substrate (loading factor 0.5, loaded volume 15 L). When adding a new portion of the substrate, the fermented substrate was changed by half (emptying coefficient −0.5). The temperature regime of the digester during the study was 30 °C, 35 °C, and 40 °C (mesophilic mode). For control, experiments were performed without the addition of crude glycerine.



(**a**) (**b**)

Figure 1. Lab-scale biogas plant: (**a**) general view and (**b**) principal scheme.

The output of biogas was recorded once a day after raising the cylinder-level meter of the gasholder (on the guide of the cylinder-level-meter-attached scale, calibrated in cubic centimeters).

The volume of biogas generated in the digester in the interval between readings of the gasholder–cylinder lifting is determined by the formula:

$$V_b = \frac{\pi \cdot D_g}{4} \cdot H_g \quad (1)$$

where V_b is the volume of biogas generated in the digester in the interval between the readings of the gasholder–cylinder lifting (cm^3), D_g is the inner diameter of the cylinder-level gauge gasholder (cm), and H_g is the height of the cylinder-level gauge of the gasholder (cm).

The productivity of the digester on biogas is calculated by the formula:

$$Q_b = \frac{V_b}{\Delta t} \quad (2)$$

where Q_b is the productivity of the biogas (cm^3/h) and Δt is the time interval between readings of raising the cylinder-level gauge of the gasholder (h).

The time interval Δt between readings of raising the cylinder-level gauge of the gasholder is determined by the formula:

$$\Delta t = 24 - t_2 + t_1 \quad (3)$$

where t_2 is the reading time of the previous indicator of the cylinder-level gauge (h) and t_1 is the reading time of the current level of the cylinder-level gauge (h).

The total amount of biogas obtained during the experiment was specified by the formula:

$$V_{Genb} = \sum V_b \tag{4}$$

where V_{Genb} is the total amount of biogas obtained during the experiment (cm^3).

The average yield of biogas during the experiment was calculated by the formula:

$$V_{Av\,b} = \frac{V_{Gen\,b}}{t_{Gen}} \tag{5}$$

where V_{Avb} is the average biogas yield during the experiment (cm^3/day) and t_{Gen} is the total time of the experiment (days).

In most literature sources, the yield of biogas from the substrate is estimated in L/kg COP. Therefore, to be able to compare the obtained results with the data from the literature, we converted the obtained results into the dimension of L/(h·kg COP). To do this, we determined the dry weight of the substrate, which is loaded into the digester, according to the formula:

$$M_{DMs} = \frac{M_s \cdot DM}{100} \tag{6}$$

where M_{DMs} is the dry matter mass of the substrate (kg), M_s is the substrate mass (kg), and DM is the dry matter content in the substrate (%).

After that, the mass of dry organic matter of the substrate, which is loaded into the digester, is determined by the formula:

$$M_{DOMs} = \frac{M_{DMs} \cdot DOM_{DM}}{100} \tag{7}$$

where M_{DOMs} is the mass of dry organic matter of the substrate (kg) and DOM_{DM} is the dry organic matter content in the dry matter of the substrate (%).

If the percentage of dry organic matter in the substrate is known, the mass of dry organic matter of the substrate, which is loaded into the digester, is determined by the formula:

$$M_{DOMs} = \frac{M_{DMs} \cdot DOM}{100} \tag{8}$$

where M_{DOMs} is the mass of dry organic matter of the substrate (kg) and DOM is the content of dry organic matter in the substrate (%).

The hourly productivity of the biogas, referred to as the dry organic matter content in the substrate, is determined by the formula:

$$Q_{b/DOMhr} = 10^{-3} \cdot \frac{Q_b}{M_{DOMs}} \tag{9}$$

where $Q_{b/DOMhr}$ is the hourly productivity of the biogas, referred to as the content in the substrate of dry organic matter (L/(h·kg DOM)) and Q_b is biogas productivity of the digester (cm^3/h).

If the hourly productivity of the biogas, related to the dry organic matter content in the substrate, is multiplied by the time between readings of the cylinder-level gauge of the gasholder, the daily biogas productivity of the digester, related to the dry organic matter content in the substrate, is determined by the equation:

$$Q_{b/DOMd} = Q_{b/DOMhr} \cdot \Delta t \tag{10}$$

where $Q_{b/DOMhr}$ is the daily biogas productivity of the methane tank, referred to as the dry organic matter content in the substrate (L/(h·kg DOM)) and Δt is the time interval between readings of raising the cylinder-level gauge of the gasholder (h).

The accumulated biogas yield is determined by the formula:

$$Q_{b\,ac} = \sum_{Q=1}^{n} Q_{b/DOMhr} \tag{11}$$

where $Q_{b\,ac}$ is the accumulated biogas yield (L/(kg DOM)), i is summation index, and n is upper summation limit (days).

3. Results and Discussion

3.1. Experimental Study of Biogas Yield during Fermentation of Cattle Manure with the Addition of Crude Glycerine

The results of the experimental study of the dynamics of the rate of biogas output over time are shown in Figure 2.

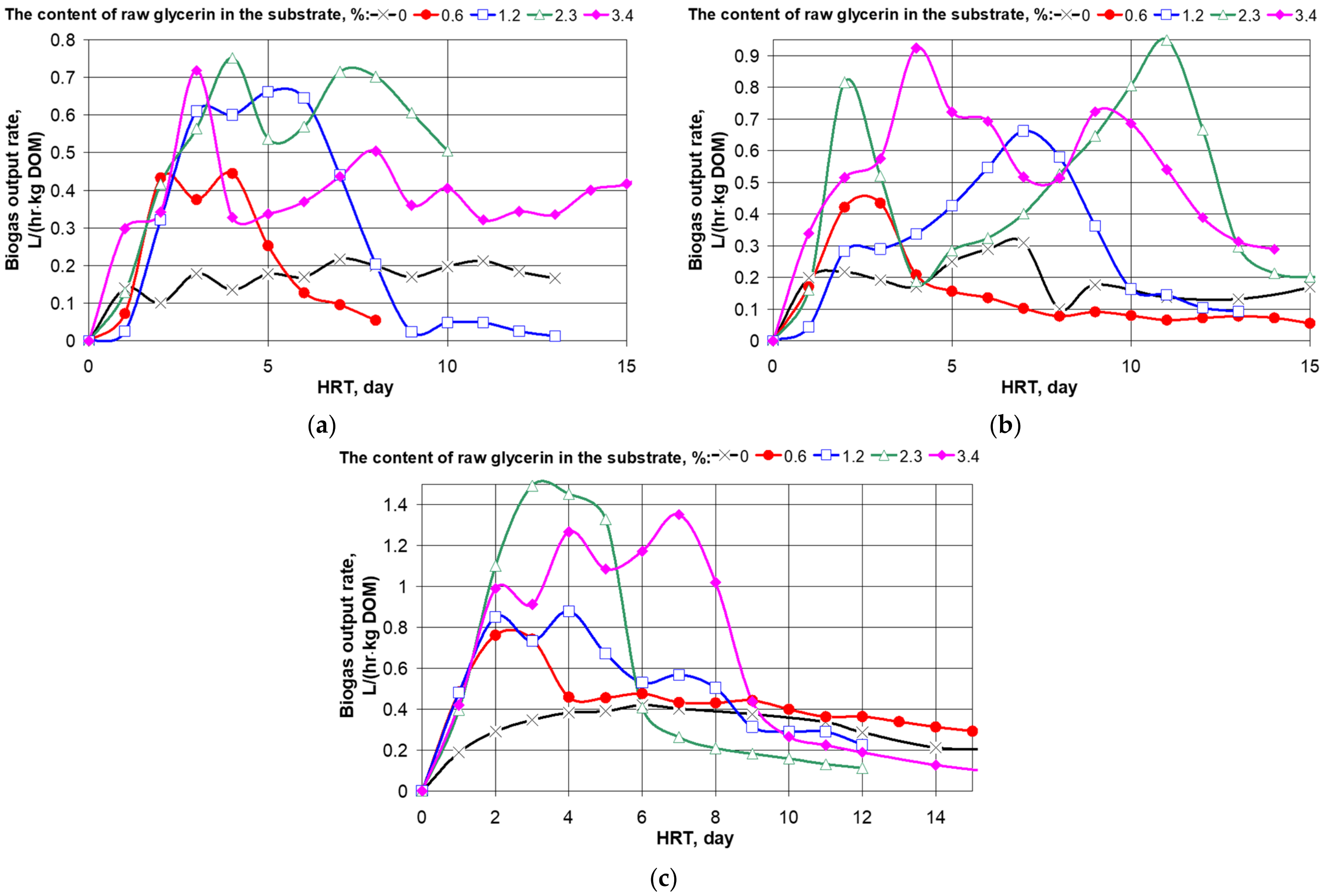


Figure 2. The rate of biogas yield during fermentation of cattle manure with the addition of crude glycerine at the fermentation temperature: (**a**) 30 °C, (**b**) 35 °C, and (**c**) 40 °C.

A characteristic feature of fermentation of cattle manure with the addition of crude glycerine is a short fermentation period, which is 6–12 days. Moreover, the lower the fermentation temperature, the shorter the fermentation period. If the volume of glycerine is higher in the substrate, the fermentation is more intense. There have been cases of clogging of the digester exhaust pipe with manure due to intense foaming. To avoid this phenomenon, the digester was only half filled with the substrate. Diauxia is also a characteristic phenomenon in the fermentation of cattle manure with the addition of crude glycerine.

The values of the maximum yield of biogas of cattle manure fermentation with the addition of crude glycerine in the periodic system of the digester loading are given in Table 1. The conversion factor of biogas yield from the dimension L/(h·kg DOM) to the dimension L/(h·kg) when adding 0.6% of glycerol to the substrate was 17.032 kg/(kg DOM); 1.2% glycerol, 15.867 kg/(kg DOM); 2.3% glycerol, 13.987 kg/(kg DOM); and 3.4% glycerol, 12.536 kg/(kg DOM).

Table 1. Maximum biogas yield of cattle manure fermentation with the addition of crude glycerine for the periodic loading system (L/(h·kg DOM)).

Glycerine Content in the Substrate, %	Maximum Biogas Yield, L/(h·kg DOM), at the Methane Fermentation Temperature		
	30 °C	35 °C	40 °C
0	0.217	0.311	0.402
0.6	0.445	0.604	0.761
1.2	0.662	0.792	0.879
2.3	0.752	0.949	1.494
3.4	0.931	1.200	1.352

The accumulated biogas yield of cattle manure fermentation with the addition of glycerine is shown in Figure 3.

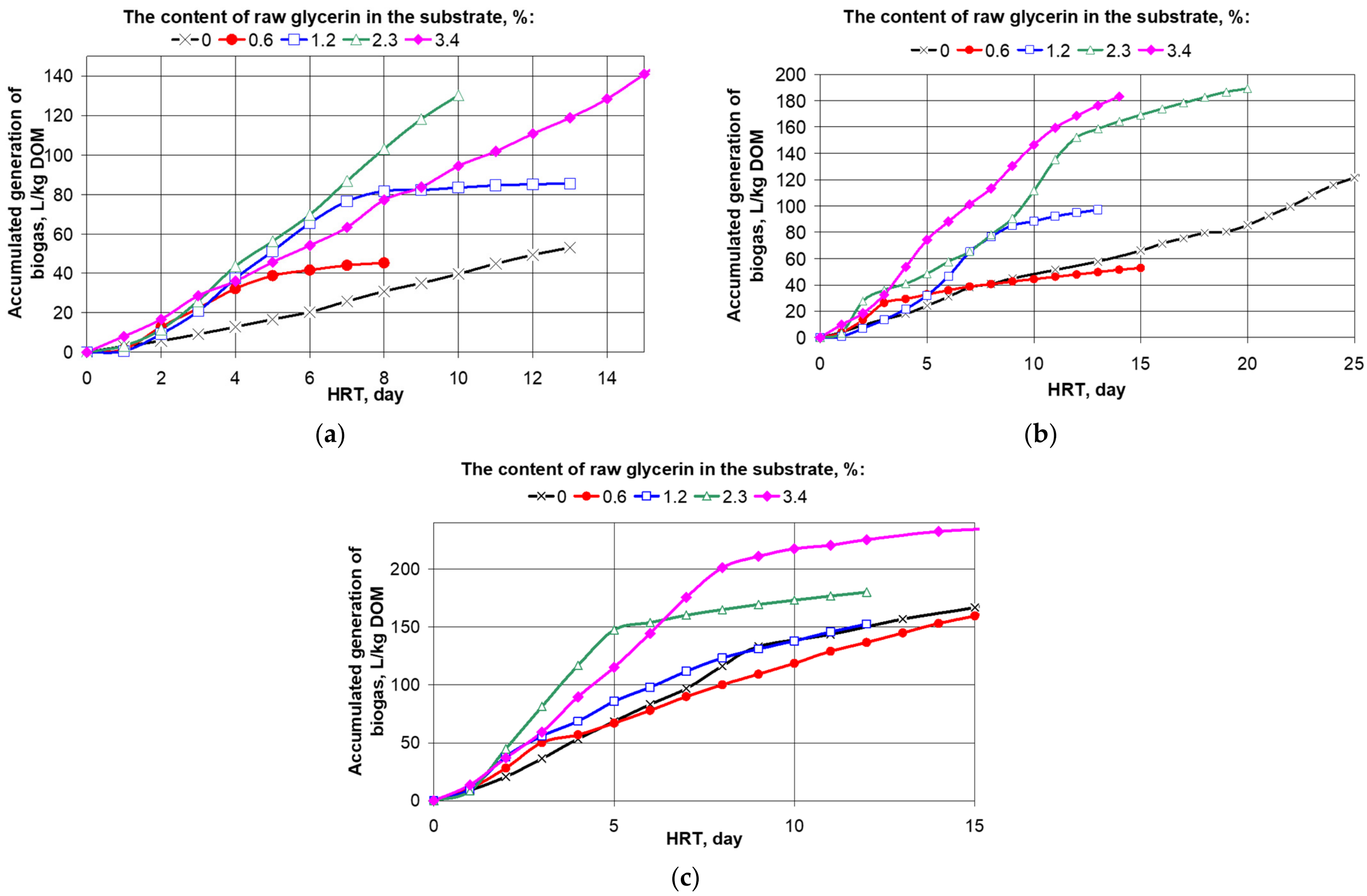


Figure 3. Accumulated biogas yield of cattle manure fermentation with the addition of crude glycerine at the fermentation temperature: (**a**) 30 °C, (**b**) 35 °C, and (**c**) 40 °C.

At low temperatures, the accumulated biogas yield of cattle manure fermentation with the addition of glycerol was higher in comparison with the accumulated biogas yield during cattle manure fermentation only. Thus, at a fermentation temperature of 30 °C, the accumulated biogas yield was about 47 L/(kg DOM) during 10 days of cattle manure fermentation with the addition of 8% DOM glycerol, it was 83.4 L/(kg DOM) in the case of 14.8% DOM glycerol, it was 130.2 L/(kg DOM) with 25.7% DOM glycerol, it was 94.3 L/(kg DOM) with 34.2% DOM glycerol, and it was 39.8 L/(kg DOM) with fermentation of cattle manure alone. However, as the fermentation temperature increased, the accumulated biogas yield of cattle manure fermentation only gradually exceeded the accumulated biogas yield of the fermentation of cattle manure with the addition of glycerol. Thus, at a fermentation temperature of 35 °C, the accumulated biogas yield was about 44.4 L/(kg DOM) during 10 days of cattle manure fermentation with the addition of 8% DOM glycerol, it was 88.3 L/(kg DOM) with 14.8% DOM glycerol, it was 111.7 L/(kg DOM) with 25.7% DOM glycerol, it was 146.5 L/(kg DOM) with 34.2% DOM glycerol, it was 85.2 L/(kg DOM) with 51% DOM glycerol, and it was about 50 L/(kg DOM) when fermenting only cattle manure. At a fermentation temperature of 40 °C, the accumulated biogas yield was 118.5 L/(kg DOM) during 10 days of cattle manure fermentation with the addition of 8% DOM glycerol, it was 137.9 L/(kg DOM) with 14.8% DOM glycerol, it was 173.2 L/(kg DOM) with 25.7% DOM glycerol, it was 217.4 L/(kg DOM) with 34.2% DOM glycerol, and it was about 140 L/(kg DOM) when fermenting only cattle manure. This phenomenon is explained not by the best biogas yield of cattle manure fermentation, but by the fact that this yield is stretched for a long time, and during cattle manure fermentation with glycerol, there is an intensive biogas yield for 3–5 days, after which it quickly stops.

The accumulated yield of biogas during the fermentation of cattle manure with the addition of crude glycerine is approximated by a polynomial.

$$Q_{\text{accumul}} = b_n \cdot t^n + b_{n-1} \cdot t^{n-1} + b_1 \cdot t + b_0 \tag{12}$$

where Q_{accumul} is the accumulated biogas yield (L/(kg DOM)), t is the fermentation time (days), and n is the degree of the polynomial.

The coefficients of the polynomial (Equation (12)) are given in Table 2.

Coefficients of determination of the approximated curves (Equation (12)) are given with the coefficients of the polynomial in Table 2. They are approaching 1, which suggests that the obtained regression equations fairly accurately reflect the experimental data.

When tested by Fisher's test [37] (p. 201 (8.52)), the significance of the coefficients of determination was established. When tested by Student's criterion [37] (p. 201 (8.55)), it was established that all coefficients b_1 and b_2 were significant. The coefficients b_3 and b_4 were unreliable (except for coefficient b_3 for the regression equation, which describes the accumulated biogas yield at a fermentation temperature of 35 °C for the glycerol content in the substrate of 1.2%, which was significant). However, the results obtained by regression equation (Equation (12)), Table 2, without these unreliable coefficients, differed significantly from the experimental data, so these coefficients must be present in the regression equations. As a result of checking the significance of the coefficients of determination of the approximated curves (Equation (12)), Table 2, it was determined that they were significant.

Biogas obtained by fermentation of cattle manure with the addition of crude glycerine in the first few days of fermentation did not burn. Moreover, at lower fermentation temperature, increased the time in which no biogas combustion was observed. Thus, at a fermentation temperature of 30–35 °C, the absence of combustion in most cases was observed for 2–3 days, and at a temperature of 40 °C, it was for 1 day. In all cases, the next day after the start of combustion, biogas burned poorly, emitting less heat. Subsequently, the combustion of biogas stabilized with a combustion heat of 18–20 MJ/m^3 for a fermentation temperature of 30–35 °C and 14–18 MJ/m^3 for a fermentation temperature of 40 °C.

Table 2. Polynomial coefficients describing the accumulated biogas yield during the fermentation of cattle manure with the addition of crude glycerine.

No.	Glycerine Content in the Substrate, %	Polynomial Coefficients					R^2
		b_4	b_3	b_2	b_1	b_0	
1	**2**	**3**	**4**	**5**	**6**	**7**	**8**
Fermentation temperature 30 °C							
1	0	-	-	0.08	3.2	−0.62	0.9982
2	0.6	-	-	−0.57	11	−5	0.9759
3	1.2	-	-	−0.67	16.4	−13	0.9693
4	2.3	-	-	0.36	10.3	−5	0.9955
5	3.4	-	-	0.1	8.9	−4	0.9857
Fermentation temperature 35 °C							
6	0	-	-	-	5.77	−14.5	0.861
7	0.6	-	-	−0.26	7.2	1.3	0.9625
8	1.2	-	−0.128	2.32	−1.26	−0.12	0.9982
9	2.3	-	−0.043	1.1	4.36	4.7	0.988
10	3.4	-	−0.06	1.04	10.4	2	0.9978
11	6.6	-	-	0.09	9.4	−0.6	0.974
Fermentation temperature 40 °C							
12	0	-	−0.026	0.33	14.3	−7.7	0.9991
13	0.6	-	-	−0.26	14.5	0.9	0.9981
14	1.2	-	-	−0.69	21.2	−3.6	0.997
15	2.3	-	0.034	−2.5	41	−15	0.9721
16	3.4	-	-	−1.2	35	−20	0.9835

3.2. Simulation of Biogas Yield with Gradual Loading of the Digester Based on the Results of Experimental Studies of Biogas Yield with Periodic Loading

The digester of the biogas plant, where experimental studies were carried out on the fermentation of substrates based on cattle manure with the addition of co-substrates, is suitable for periodic loading of the substrate. The mode of the substrate's gradual loading is quite difficult to realize. However, in practice, on existing biogas plants, the periodic loading mode of the digester is rarely used; more often, a gradual loading is used, when the substrate is loaded into the digester in small portions after a certain time (usually about 1 h). The biogas yield reaches the maximum value that can be achieved with a periodic loading system, and it is maintained at this level throughout the operation of the biogas plant. Therefore, based on experiments with the periodic loading system of the digester, it is possible to model the biogas yield with a gradual loading system. The biogas yield in a gradual loading system is close to the maximum biogas yield in a periodic loading system.

The simulated yield of biogas during the fermentation of cattle manure with the addition of crude glycerine for the gradual loading of the digester is shown in Figure 4, and the values of the maximum biogas yield that were used to model the process are presented in Table 3.

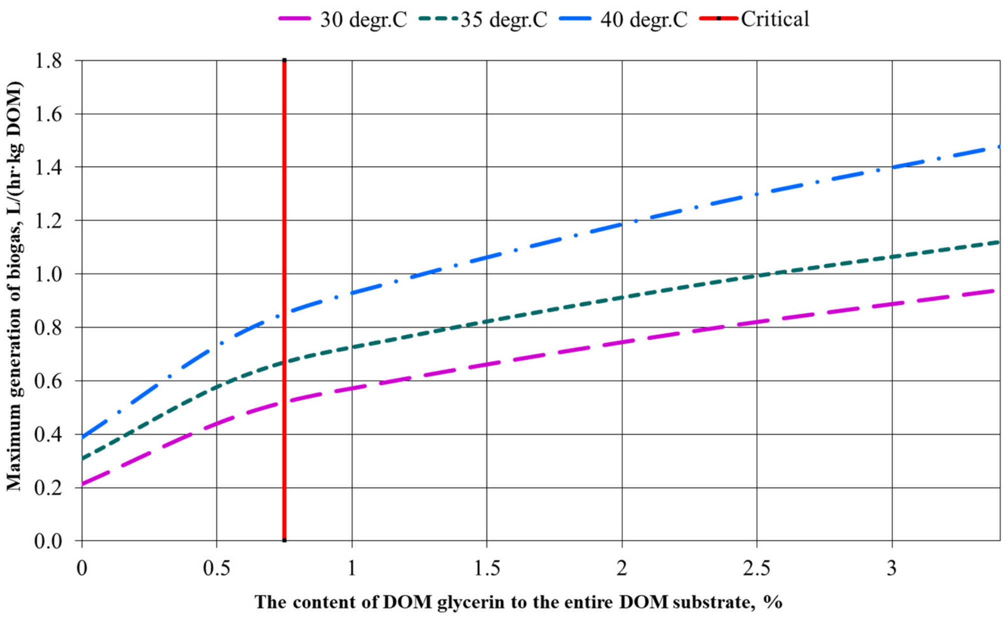


Figure 4. Simulated biogas yield during fermentation of cattle manure with the addition of crude glycerine for gradual loading of the digester.

Table 3. Power function coefficients (Equation (13)) describing the simulated biogas yield during the fermentation of cattle manure with the addition of crude glycerine for gradual loading of the digester.

Fermentation Temperature, °C	Power Function Coefficients			R^2
	b_2	b_1	b_0	
30	0.354	0.568	0.214	0.9826
35	0.412	0.553	0.308	0.9802
40	0.535	0.581	0.388	0.8821
45	0.377	0.59	0.859	0.8575
50	0.504	0.336	0.976	0.9316

The simulated yield of biogas during the fermentation of cattle manure with the addition of crude glycerine for the gradual loading of the digester is approximated by the power function:

$$Q_{b\,\mathrm{mod}} = b_2 \cdot G^{b_1} + b_0 \quad (13)$$

where $Q_{b\mathrm{mod}}$ is simulated biogas output for the gradual loading of the digester (L/(kg DOM)), G is the content of glycerol in the substrate (%), and b_0, b_1, and b_2 are coefficients of the power function.

The coefficients of the power function (Equation (13)) are given in Table 3.

Coefficients of determination of approximate functions (Equation (13)) describing the simulated biogas yield during the fermentation of cattle manure with the addition of crude glycerine for the gradual loading of the digester at all investigated fermentation temperatures approached 1, which suggests that the obtained regression equations fairly accurately reflect the experimental data. When tested by Fisher's test [37] (p. 201 (8.52)), the significance of the coefficients of determination was established for α = 5% for all investigated regimes and also for α = 1% for temperature regimes of the digester at 30 °C, 35 °C, and 40 °C. Student's test [37] (p. 201 (8.55)) showed that the coefficients of the power function are significant.

Since we considered the simulated biogas yield of cattle manure fermentation with the addition of crude glycerine for gradual loading of the digester depending on two variables, namely the glycerol content in the substrate, G, and the fermentation temperature

T, Equation (13) with the coefficients, given in Table 3, can be approximated by the function of two variables:

$$Q_{b\,\mathrm{mod}} = \left[6.08 \times 10^{-5} \cdot G^4 - 9.58 \times 10^{-3} \cdot G^3 + 0.55894 \cdot G^2 - 14.29677 \cdot G + 135.659\right] \times G^{\left(-1.21\times10^{-5}\cdot G^4 + 1.737\times10^{-3} G^3 - 9.208\times10^{-2} G^2 + 2.14187\cdot G - 17.899\right)} + 4.15 \times 10^{-6} \cdot G^{3.1649} \quad (14)$$

where $Q_{b\mathrm{mod}}$ is the simulated biogas output for gradual loading of the digester (L/(h·kg DOM)), G is the glycerol content in the substrate (%), and G is the fermentation temperature (°C).

The coefficient of determination was R^2 = 0.8654 of the regression equation, determined by Equation (14). It is not so close to unity to claim that the obtained regression equation accurately reflects the experimental data for the fermentation temperature of 30–40 °C and the glycerol content in the substrate is not more than 3.4%. However, greater accuracy was not achieved. When it was tested by Fisher's test [37] (p. 201 (8.52)), the significance of the coefficient of determination was established. Student's test [37] (p. 201 (8.55)) showed that all the coefficients of the approximation (Equation (15)) were significant, except for the coefficients 1.121×10^{-5} and 4.15×10^{-6}, which were unreliable, but their removal can cause a significant difference between the experimental and calculated values. The response surface, built according to the regression equation (Equation (14)), is shown in Figure 5.

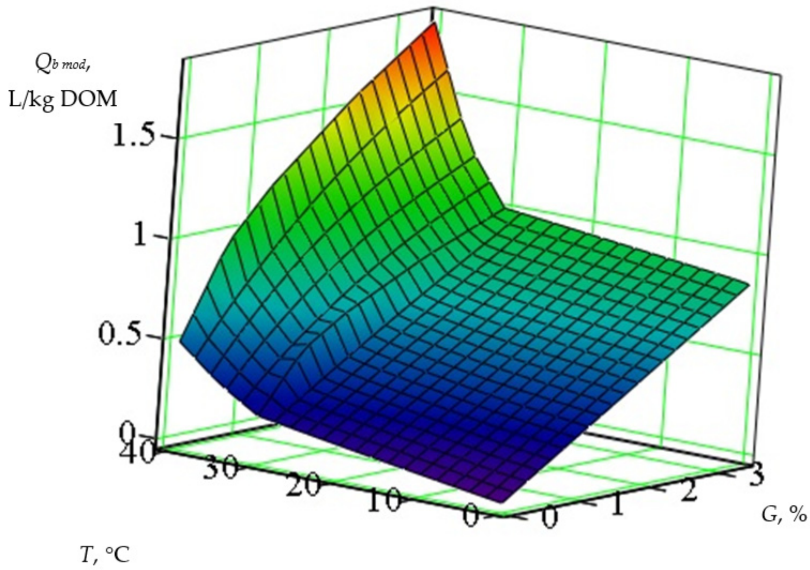


Figure 5. Response surface describing the simulated biogas yield for gradual loading of the digester during fermentation of cattle manure with the addition of crude glycerine.

As can be seen from Figures 4 and 5, with the addition of more crude glycerine, the biogas yield increased. However, it grew unevenly. When up to 0.75% of glycerine was added to the substrate, there was an intensive increase in biogas yield. With the addition of more glycerine, the growth of biogas yield slowed down. Thus, if at a fermentation temperature of 30 °C, an increase in the content of crude glycerine in the substrate from 0% to 0.75% causes an increase in biogas yield by 2.5 times, the increase in the content of crude glycerine in the substrate from 0.75% to 3.4%, i.e., by 25%, is only by 1.7 times. In addition, if the increase in crude glycerine in the substrate from 0% to 0.75% at a fermentation temperature of 40 °C causes an increase in biogas yield by 1.5 times, the increase in crude glycerine in the substrate from 0.75% to 3.4%, that is, by 25%, is only by 1.2 times.

Crude glycerine is a waste from biodiesel production. Currently, for several reasons, biodiesel production in Ukraine is developing at a slow pace. Therefore, to reduce the cost of valuable co-substrates, biogas producers, who have access to crude glycerine as a waste from biodiesel production, the resources of which are limited, are recommended to prepare a substrate with a content of not more than 0.75% of crude glycerine.

4. Conclusions

For increasing the efficiency of biogas production in the mesophilic regime, it is advisable to use crude glycerine, which is the waste from biodiesel production, as a co-substrate. Recycling crude glycerine causes problems for the biodiesel industry.

The scientific novelty of the work is the determination of the intensification patterns of the methane fermentation process of cattle manure based on the addition of different proportions of crude glycerine. An intensive increase in the biogas yield is observed if the glycerin content is 0.75% of the substrate. With the addition of more glycerine, the biogas yield decreases. Thus, at a fermentation temperature of 30 °C, the increase in the crude glycerine content in the substrate from 0% to 0.75% causes the biogas yield to increase by 2.5 times, while the increase in the crude glycerine content in the substrate from 0.75% to 3.4% causes the biogas yield to increase by only 1.7 times.

The increase in the crude glycerine content in the substrate from 0% to 0.75% at a fermentation temperature of 40 °C causes an increase in biogas yield by 1.5 times. In the case of the increase in the crude glycerine content in the substrate from 0.75% to 3.4%, the biogas yield grows by only 1.2 times.

Author Contributions: Conceptualization, W.R., I.R., V.P., L.T., K.B., W.J.W., S.S., Y.D., I.S., S.D., D.D. and K.R.; methodology, W.R., I.R., V.P., L.T., K.B., W.J.W., S.S., Y.D., I.S., S.D., D.D. and K.R.; software, W.R., I.R., V.P., L.T., K.B., I.S., S.D., D.D. and K.R.; validation, W.R., I.R., V.P., L.T., K.B., W.J.W., S.S., Y.D., I.S., S.D., D.D. and K.R.; formal analysis, W.R., I.R., V.P., L.T., K.B., W.J.W., S.S., Y.D., I.S., S.D., D.D. and K.R.; investigation, W.R., I.R., V.P., L.T., K.B., W.J.W., S.S., Y.D., I.S., S.D., D.D. and K.R.; resources, W.R., I.R., V.P., L.T., K.B., W.J.W., S.S., Y.D., I.S., S.D., D.D. and K.R.; data curation, W.R., I.R., V.P., L.T., K.B., I.S., S.D., D.D. and K.R.; writing—original draft preparation, W.R., I.R., V.P., L.T., K.B., W.J.W., S.S., Y.D., I.S., S.D., D.D. and K.R.; writing—review and editing, W.R., I.R., V.P., L.T., K.B., W.J.W., S.S., Y.D., I.S., S.D., D.D. and K.R.; visualization, W.R., I.R., V.P., L.T., K.B., W.J.W., S.S., Y.D., I.S., S.D., D.D. and K.R.; supervision, W.R., I.R., K.B., W.J.W., S.S., S.D., D.D. and K.R.; project administration, W.R., I.R., V.P., L.T., K.B., W.J.W., S.S., Y.D., I.S., S.D., D.D. and K.R.; funding acquisition, W.R., I.R., V.P., L.T., K.B., W.J.W., S.S., Y.D., I.S., S.D., D.D. and K.R. All authors have read and agreed to the published version of the manuscript.

Funding: This research received no external funding.

Institutional Review Board Statement: Not applicable.

Informed Consent Statement: Not applicable.

Data Availability Statement: Not applicable.

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

Abbreviations

COI	chemical oxygen index
DOM	dry organic matter
VS	volatile solids
DM	dry matter
HRT	hydraulic retention time

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