



# Article Valorization of Dairy By-Products: Efficiency of Energy Production from Biogas Obtained in Anaerobic Digestion of Ultrafiltration Permeates

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**Abstract:** This study aimed at evaluating the methane potential of two ultrafiltration (UFP) and two diafiltration (DFP) permeates generated during milk protein concentration. The permeates were characterized by a different chemical oxygen demand (COD) ranging from 7610 mg  $O_2/L$  to 57,020 mg  $O_2/L$ . The CH<sub>4</sub> production efficiency was recorded for 20 days and ranged from 149 to 181 NL/kg COD<sub>added</sub>. Moreover, the possibilities of the use of UFP/DFP to produce electricity and heat with a combined heat and power (CHP) unit was analyzed to underline the impact of the implementation of anaerobic digestion on the electric and thermal energy requirements of a dairy plant. It was concluded that the application of anaerobic digestion to UFP and DFP treatments generates the energy required to cover all the large-scale dairy plant energy demands and produce extra income. The amount of permeates generated annually in the analyzed dairy plant will enable the production of approx. 22,699 MWh of electricity and 85,516 GJ of heat. This would require a biogas plant with a 3 MW yield. Additionally, the lactose production from UFP/DFP was considered as an alternative or parallel solution for its management. The study confirmed that the biogas and lactose production from UFP/DFP enables plant owners to adjust a plant's management towards one of these two solutions.

Keywords: UF permeate; lactose; dairy wastes; anaerobic digestion; biogas

# 1. Introduction

The significant growth of the global population in the last decades has resulted in the development of the industrialization of the food production and processing sectors. The dairy industry is a part of the food sector that fulfills the demand for products such as milk, cheese, milk powder, etc. [1]. The consumption of dairy products has experienced a significant rise in many countries, resulting in the establishment of large-scale production factories [2].

While serving the global demand for milk-based products, the dairy industry generates a significant amount of pollution due to its use of water and its emission of effluents into the environment. In Europe, nearly 29 million tons of dairy products end up as waste each year. Within this waste, there are processing effluents as well as spoiled and mishandled products [1,3].

The cheese sector produces high quantities of whey and milk permeate that represent a major disposal problem [4]. Ultrafiltration (UF) and diafiltration (DF) permeates are the effluents generated during the concentration of milk or whey proteins (Figure 1). The diafiltration process is a variation of a membrane process, in which fresh solvent is added to the retentate produced during the first stage of the filtration process to replenish the filtered volume; consequently, the small molecules such as salts are washed away from the retained



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**Copyright:** © 2022 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (https:// creativecommons.org/licenses/by/ 4.0/). macromolecules. The purpose of applying a DF stage is to obtain a retentate of a higher purity [5–9]. The effluents generated during milk and whey processing are characterized by an extreme chemical oxygen demand (COD) up to approximately 70,000 mg  $O_2/L$  [10]. Based on a simple mass balance, it is estimated that for 100 kg of raw milk between 80 to 87.5 kg of UF permeate is produced, while only 12.5-20 kg is transformed into the final product—milk protein concentrate (MPC) in liquid form [11]. The concentration of whey or milk serum (the permeate obtained after the microfiltration of skim milk, with a chemical composition similar to whey) generates even higher amounts of UF/DF permeate due to the higher volume reduction factor achieved during protein concentration. The UF/DF permeate contains primarily water, lactose, minerals, and a small number of nitrogencontaining compounds. Most of the nitrogen content of the UF/DF permeate from milk consists of nonprotein nitrogen compounds normally found in milk [12]. Generally, the UF permeates generated in the dairy industry are considered a potential source of lactose production in large-scale dairy plants (Figure 1). The primary feed material for lactose production is the UF permeate of sweet/cheese whey generated during the production of whey protein concentrates (WPC) and whey protein isolates (WPI). About 4.2 million tons of whey protein powders were produced worldwide in 2018 [13]. A less valuable source of feed for lactose isolation is the production of MPC and milk protein isolates (MPI). Their annual production reached approximately 190 thousand tons globally in 2017 [14]. The industrial-scale producers prefer lactose sources that demand the least amount of processing and create minimal waste [15]. Thus, the management of UF permeates from MPC and MPI production is still a challenge for the dairy industry.



**Figure 1.** Flow diagram for concentration of proteins and lactose production in dairy industry. The final products are marked in italics.

Anaerobic digestion (AD) can be employed as the first biological treatment process necessary for the partial stabilization of food-processing wastes (such as dairy wastewater) prior to their utilization or disposal, as well as for the production of biogas [16–18]. In addition to reducing the pollutive potential of these wastes, the biogas produced from AD could be used for space and water heating in food-processing plants. AD has been used as a means for dairy waste neutralization [19,20]. The AD of dairy wastewater (mainly cheese whey) has been widely studied using single- and two-stage systems [21–23]. However, several authors [24–29] reported a low biogas productivity and methane yield associated with the low pH of the fermented whey or its high COD. Differently constructed reactors have been used to enhance the treatment of dairy wastewater [4]. Nevertheless, the vast majority of published research is focused on the management of whey, whose components can be successfully recovered in a dairy factory, whereas little is known about exploiting the potential of UF permeates containing a relatively high concentration of lactose.

The biochemical methane potential (BMP) test is often applied to assess the anaerobic biodegradability of a tested substrate, whereby its specific methane yield may be evaluated [30]. BMP tests can indicate the potential for biogas production from various substrates [31].

Due to the microbial role in the AD process, kinetic methods are commonly used to simulate anaerobic biodegradation. Hydrolysis is often assumed to be the rate-limiting step in AD, and the researchers often model batch BMP data using a first-order kinetic model. A first order kinetic model has been successfully applied to AD using different kinds of reactors [32,33].

Moreover, AD was recently recognized as an environmentally friendly technology for producing renewable energy as biogas [34,35]. Therefore, biogas production allows for the increase in the share of renewable sources in the national energy scheme, which is the EU's goal as set out in the renewable energy directive (RED II) [36]. Due to the still-growing energy demand in European countries, it is predicted that there will be a rapid development of biogas plants [37]. These installations enable the production of biogas (under controlled conditions), which is a mixture of methane (45–74%), carbon dioxide (25–54%), and trace amounts of other components, i.e., hydrogen sulfide, ammonia, hydrogen, nitrogen, and oxygen, in a concentration up to 1–2% of the total biogas volume. The methane content in the produced gas is mainly affected by the components found in the fermentation substrate [37].

Additionally, the production of biogas in agricultural areas may provide extra income from agricultural activities, which creates an opportunity to develop the local economy in rural areas and promote circular economic principles in local communities [35,38–40]. An on-site biogas plant can be a significant source of additional income for a plant specialized in dairy production [20]. Moreover, local farmers may benefit from either selling the wastes generated from agricultural production as a co-substrate used in a biogas plant or from receiving high-quality organic fertilizer left over from biogas production [41]. New biogas plants will also offer job opportunities.

The aim of this study is to analyze the possibilities of using the waste from dairy processing to produce electricity and heat from the biomethane generated in the process of anaerobic digestion. The analysis is based on the effluents produced by a large-scale dairy factory. BMP tests were performed using four different substrates, namely, UF and DF permeates obtained after the filtration of milk and milk serum. Their energy potentials were calculated and a simplified financial analysis concerning the profits generated via energy vs. lactose sale was conducted.

The key novelty of this study is the assessment of the possibility of valorizing the permeates generated during DF by using them in biogas production and comparing their effects with typical UF. Attempts were made to estimate the relationship between the degree of lactose recovery and the amount of biogas obtained. A case study was proposed for a dairy plant using membrane techniques.

#### 2. Materials and Methods

# 2.1. Materials

The ultrafiltration permeates (UFP—wastes from dairy processing) used in research experiments were obtained from Dairy Research and Development Center located at University of Warmia and Mazury in Olsztyn. These permeates were produced from ultrafiltration (UF) and diafiltration (DF) processes of two different dairy products (100 L of milk and 170 L of milk serum, Figure 2). The UF and DF processes were carried out using UF module (Koch Membrane Systems, Wilmington, MA, USA) fitted with 3838-HFK-131 membrane (spiral wound element; semipermeable polyethersulfone layer on a polyester backing material) with a membrane area of 6.7 m<sup>2</sup>, molecular weight cut-off value of 10,000 Da, and a feed spacer model N 31 mil (used in accordance with manufacturer's specifications). The unit was operated under a constant transmembrane pressure of 4 bar at 50 °C for either UF or DF processes. Milk was ultra-filtrated to achieve a concentration factor of approximately 4.6×. The UF retentate was diluted with reverse osmosis water at 50 °C to bring the weight back to the original total weight of the starting feed for DF. Then, the diluted UF retentate was diafiltrated to achieve a concentration factor of  $6.6 \times$ . Diafiltration was continued until the protein content of the retentate, measured by MilkoScan™ FT2 (Foss, Hillerød, Denmark), was 13.5%. Milk serum was UF to achieve a concentration factor of approximately  $5.5 \times$ . The UF retentate was diluted with reverse osmosis water at 50 °C to bring the weight back to the original total weight of the starting feed for DF. Then, the diluted UF retentate was diafiltrated to achieve a concentration factor of  $8.5 \times$ . Diafiltration was continued until the protein content of the retentate, measured by MilkoScan™ FT2 (Foss, Hillerød, Denmark), was 2.2% of protein in the retentate. The permeates were sampled in a representative manner, and then stored at 4 °C.

# **Dairy waste production**



**Figure 2.** Production of dairy wastes (ultrafiltration permeates = fermentation substrates): UFP-M = milk ultrafiltration permeate; DFP-M = milk diafiltration permeate; UFP-MS = milk serum ultrafiltration permeate; DFP-MS = milk serum diafiltration permeate; UF = ultrafiltration; DF = diafiltration; MF = microfiltration; RO = reverse osmosis.

The fermentative inoculum (anaerobic sludge—the source of methanogenic bacteria) came from a bioreactor operating under mesophilic conditions at  $36 \pm 1$  °C. The parameters of the sludge are reported in Table 1.

Table 1. The parameters of anaerobic sludge used as inoculum.

Trait	Anaerobic Sludge
	$6.8\pm0.2$
VS [% TS]	$73.5\pm0.9$
Ash [% TS]	$26.5\pm0.9$
C [% TS]	$41.1\pm0.8$
N [% TS]	$3.7\pm0.0$
C/N ratio	$11.2\pm0.2$

TS—total solids; VS—volatile solids; C—carbon; N—nitrogen; C/N—carbon to nitrogen; ±standard deviation.

#### 2.2. Analysis of Physicochemical Parameters

The pH of the substrates was measured using a pH meter (CP-401, Elmetron, Zabrze, Poland) equipped with an electrode IJ-44C IONODE (Elmetron). The permeate samples were analyzed to obtain content of total solids (TS) and volatile solids (VS) using a gravimetric method. The samples were dried at  $105 \,^{\circ}$ C [42]. The total nitrogen (TN) content was determined using Kjeldahl method (AOAC International, Gaithersburg, MD, USA, 2007; method 991.20; 33.2.11). The total calcium content was analyzed using atomic absorption spectrometry. The measurements were carried out in accordance with ISO 8070-2007 IDF 119:2007(E) (ISO/IDF, 2007) using Thermo Scientific iCE 3000 Series AA Spectrometer (Thermo Fisher Scientific, Franklin, MA, USA) and at the wavelength of 422.7 nm. The amount of lactose was estimated using Milko-Scan<sup>™</sup> FT2 (Foss, Hillerød, Denmark). The analysis of ash was performed using the muffle furnace technique at 530 °C (PN-A-86364:1996). The chemical oxygen demand (COD) of the samples was determined with the aid of cuvette test LCK 914 (HACH LANGE GmbH, Berlin, Germany). The composition and percentage of individual components of the biogas outflow  $(CH_4, CO_2)$  were analyzed using a gas chromatograph (GC, 7890A Agilent, Santa Clara, CA, USA) equipped with a thermoconductometric detector (TCD). Biogas was collected from the bioreactors with a 10 mL gas-tight syringe. The carrier gases used were argon (Ar) and helium (He) at a volume flow of 0.9 NL/h. The injection and detector port temperatures were 150 and 250 °C, respectively.

#### 2.3. Biogas Efficiency Research

The methane yield of tested substrates was determined using a volumetric gas production method, namely, the Automated Methane Potential Test System II (AMPTS II, BPC Instruments AB, Lund, Sweden). The fermentation measurements were performed at  $37 \pm 1$  °C. The measurement system recorded changes in partial pressure coupled with reactors. Each AMPTS reactor (glass vessel of 500 mL total volume) was equipped with an individual automated stirrer (100 rpm for 30 s every 10 min). There were 15 AMPTS bottles: [four substrates and one control]  $\times$  triplicate replication, where the substrates were UFP-M, DFP-M, UFP-MS, and DFP-MS, and control was inoculum only. Tests were performed by filling the reactors with approximately 200 g of the anaerobic sludge and then the fermentation substrates were added. The initial organic load rate (OLR) was approximately 5 g COD/L. Anaerobic conditions in the reactors were achieved by flushing pure nitrogen through the mixture of sludge and substrate. Using the bioprocess control software, a gas production report was recorded every day. This program generates results of the volume of gas already normalized (standard atmospheric pressure 101.3 kPa, at 0 °C, and with zero moisture content). The measurement was carried out until the available organic compounds were fully decomposed. Subsequent measurements of the amount of biogas were automatically compared with each other, and when ten consecutive gas volume measurements did not differ by more than 1%, the measurement was completed. The endogenous biogas generated by anaerobic sludge was excluded from the calculations

## 2.4. Kinetic Analysis

Nonlinear regression was used to obtain the rate constants of methane production from plots of volume of methane per mass of  $COD_{added}$  versus time. According to the approach reported by Llabres-Luengo and Mata-Alvarez [43], the first order linear regression is defined as:

$$Y(t) = -Y_{max}(e^{-kt} - 1)$$
 (1)

where Y—methane yield (NL CH<sub>4</sub>/kg  $COD_{added}$ ), Y<sub>max</sub>—maximal methane yield (NL CH<sub>4</sub>/kg  $COD_{added}$ ), k—kinetic constant (1/day), and t—time (day). The methane production rate was calculated as the product of the k and the maximal volume of methane per mass of  $COD_{added}$  obtained from the first order kinetics.

To evaluate the model results, Statistica software (version 13.1, 1984–2016, StatSoft, Inc., Tulsa, OK, USA) was used for calculations of the determination coefficient R<sup>2</sup>.

#### 2.5. Energetic Calculations

The calculations of the energy potential of substrates (UFP-M, DFP-M, UFP-MS, and DFP-MS) were based on the conducted fermentation tests and methodology described by Cieślik et al. [44] and Kozłowski et al. [20]. The amount of electricity and heat produced from combined heat and power (CHP) unit were determined using Equations (2) and (3):

$$E_{E} = V_{CH_{4}} \times Re_{CH_{4}} \times \eta_{e}$$
<sup>(2)</sup>

where  $E_E$ —amount of produced energy [MWh/kg COD<sub>added</sub>], V<sub>CH4</sub>—volume of produced methane [m<sup>3</sup>/kg COD<sub>added</sub>], Re<sub>CH4</sub>—energy efficiency ratio of methane [0.00917 MWh/m<sup>3</sup>], and  $\eta_e$ —electrical efficiency of CHP. For the purpose of these calculations, the efficiency of 43% was assumed for the unit offered by PAKTOMA (Łódź, Poland), a Polish manufacturer of modern co-generation units for biogas plants).

Heat produced in CHP unit was determined from Equation (3):

$$E_{\rm H} = V_{\rm CH_4} \times {\rm Re}_{\rm CH_4} \times \eta_t \tag{3}$$

where  $E_H$ —amount of produced heat [MWh/kg COD<sub>added</sub>], V<sub>CH4</sub>—volume of produced methane [m<sup>3</sup>/kg COD<sub>added</sub>], Re<sub>CH4</sub>—energy efficiency ratio of methane [0.00917 MWh/m<sup>3</sup>], and  $\eta_t$ —heat efficiency of CHP. For the purpose of these calculations, the efficiency of 45% was assumed for the unit offered by PAKTOMA, a Polish manufacturer of modern cogeneration units for biogas plants).

#### 2.6. Case Study

This paper describes potential heat and electric power produced from biogas generated by anaerobic digestion of liquid by-products obtained from protein concentration in a largescale dairy plant. The data regarding daily milk and whey throughput along with the usage of electric energy come from the reports published by the company and the community council. Waste yield was calculated from raw milk and whey throughput and processing using membrane technology.

The assumption is that the dairy plant process daily about 1200 t of raw milk, which is solely dedicated for the production of milk concentrates. The whole milk is first supplied to the separator and consequently discharged as two flows, skim milk and cream, of which the cream normally represents about 10% of the total throughput [45]. The total amount of liquid discharged as skim milk will then be 1080 t. In a typical UF process, pasteurized skim milk is concentrated to a final volume of one-fifth to one-eighth of the volume of the original skim milk to produce milk protein concentrate (MPC) that has 65 to 75% content

of protein relative to total solids. For the production of MPC with higher protein content, ultrafiltration is used in conjunction with the DF process [46]. Considering the amount of skim milk processed in the analyzed dairy plant, the filtration process characterized by volume concentration factor of 5 will generate about 864 t of permeate at each stage of filtration.

Another side stream processed by the factory is whey (800 t/day). Composition of whey is similar to that of milk serum, which is why its processing is easily comparable with processing of milk serum. In a typical UF process, pasteurized whey is concentrated to a final volume of between one-sixth to one-ninth of the volume of the original whey (based on the research performed previously) to produce serum protein concentrate (SPC60 or WPC60) that contains 60% content of protein. For the production of SPC with higher protein content, UF is used in conjunction with the DF process. Considering the amount of whey processed in analyzed dairy plant, the filtration process characterized by volume concentration factor of 8.5 will generate about 706 t of permeate at each stage of filtration.

The daily demand for electric power in this particular dairy plant equals 11 MWh. This covers the demand of all processing departments. Apart from manufacturing milk and whey concentrates, the factory also produces cheese, yogurts, and buttermilk.

#### 2.7. Statistical Analysis

The statistical analysis was performed using the Statistica software (version 13.1, 1984–2016, StatSoft, Inc., Tulsa, OK, USA). Statistical evaluation of data was conducted using one-way ANOVA (p < 0.05) followed by Tukey's post hoc test.

## 3. Results and Discussion

## 3.1. Analysis of Methane Production

To calculate the energetic potential of the substrates, it is essential to assess their physical and chemical properties. The basic parameters—such as the TS content, VS content, COD, and pH—characterizing the substrates used for the fermentation process performed in this study are presented in Table 2.

Table 2.	The	physicoche	emical parar	neters of th	he fermenta	ation substrates.
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Davamatar	Substrate						
r alaineter	UFP-M	DFP-M	UFP-MS	DFP-MS			
TS [%]	$5.60^{a} \pm 0.06$	$1.81 \ ^{\rm c} \pm 0.05$	$3.8^{b} \pm 0.02$	$0.86 \ ^{ m d} \pm 0.09$			
VS [%]	$5.13~^{\rm a}\pm0.06$	$1.65~^{ m a,b}\pm 0.08$	$3.46 \ ^{\mathrm{b}} \pm 0.07$	$0.79~^{\rm a,b}\pm 0.03$			
Ash [%]	$0.45~^{\rm a}\pm0.01$	$0.15~^{\rm c}\pm0.01$	$0.332 \ ^{\mathrm{b}} \pm 0.004$	$0.07~^{ m d} \pm 0.01$			
Ca [mg/L]	185.5 a $\pm$ 9.6	34.8 $^{\rm c}\pm2.0$	54.4 $^{\mathrm{b}}\pm2.1$	$3.6^{\rm d} \pm 0.1$			
TN [%]	$0.18~^{\rm a}\pm0.03$	0.06 $^{\rm c}\pm 0.01$	$0.097 \ ^{\mathrm{b}} \pm 0.003$	$0.021 \ ^{ m d} \pm 0.002$			
Lactose [%]	$4.97~^{\rm a}\pm0.02$	$1.59~^{\rm c}\pm0.18$	$3.37 \ ^{\mathrm{b}} \pm 0.02$	$0.76~^{ m d} \pm 0.01$			
pН	$6.55^{\text{ b}} \pm 0.18$	$6.84~^{\rm c}\pm0.10$	$6.84~^{\rm c}\pm0.04$	7.03 $^{\rm a}\pm 0.06$			
$COD [mg O_2/L]$	57,020 $^{\mathrm{a}}\pm510$	19,150 $^{\rm c}\pm1500$	38,600 $^{ m b}$ $\pm$ 780	7610 $^{ m d}$ $\pm$ 430			

TS—total solids; Ca—calcium; TN—total nitrogen calculated as total N × 6.38; COD—chemical oxygen demand; UFP-M = milk ultrafiltration permeate; DFP-M = milk diafiltration permeate; UFP-MS = milk serum ultrafiltration permeate; DFP-MS = milk serum diafiltration permeate; Data are expressed as mean (n = 6)  $\pm$  standard deviation; <sup>a–d</sup> Means within a row with different superscripts are different (p < 0.05).

The results concerning the TS content indicated the significant (p < 0.05) difference between the four tested materials. The permeates obtained in the DF stage, during either the milk or milk serum UF process, contained mostly water (over 98%) and a very low number of solids (Table 2). An about three to four times greater volume of DF permeate— for DFP-M and DFP-MS, respectively—is required to obtain the same organic load as in the permeates obtained after first stage of processing for the UF/concentration (UFP-M and UFP-MS, respectively).

The respective streams obtained either during UF (UFP-M vs. UFP-MS) or DF (DFP-M vs. DFP-MS) were characterized by a higher TS content when milk was subjected to

filtration in comparison with milk serum. That is why, from a biogas plant exploitation point of view, the effluents generated during the filtration of the milk (UFP-M and DFP-M) used as substrates may result in a much higher profitability when compared to the streams obtained after milk serum processing (UFP-MS and DFP-MS, respectively).

The concentrations of organic matter expressed as the COD in the permeates after the UF process were 38,600 and 57,020 mg O<sub>2</sub>/L for UFP-MS and UFP-M, respectively. Whereas the COD values of the permeates obtained in the DF process were equal to 7610 and 19,150 mg O<sub>2</sub>/L for DFP-MS and DFP-M, respectively (Table 2). The permeates generated after the UF process in the dairy industry contribute a high COD as they are rich in lactose [12]. Lactose contributes around 90% of the COD. High COD values indicate that the dairy wastes produced are strong and fluctuating types [10]. In general, the COD value of the dairy effluents depends on the protein, fat, and carbohydrate content, which was confirmed by the Person's correlation coefficient (r > 0.97, *p* < 0.05) obtained in the current study (Table 3). The waste load equivalents of these constituents are 1 kg of fat, which contributes 3 kg of COD; 1 kg of protein, which contributes 1.36 kg of COD; and 1 kg of lactose, which contributes 1.13 kg of COD [10,47].

Table 3. The Pearson's correlation coefficients for the analyzed traits.

Trait	COD	VS	Ash	Ca	TN	Lactose	pН	TS	CH <sub>4</sub> Production
COD	1.00								
VS	0.11	1.00							
Ash	1.00 *	0.05	1.00						
Ca	0.93 *	0.30	0.90 *	1.00					
TN	0.97 *	0.23	0.95 *	0.98 *	1.00				
Lactose	1.00 *	0.10	1.00 *	0.93 *	0.97 *	1.00			
pН	-0.87 *	-0.34	-0.84 *	-0.94 *	-0.94 *	-0.87 *	1.00		
TS	1.00 *	0.11	1.00 *	0.93 *	0.97 *	1.00 *	-0.87 *	1.00	
CH <sub>4</sub> production	-0.08	0.06	-0.06	-0.17	-0.15	-0.07	0.32	-0.07	1.00

\* significant values (p < 0.05).

The biochemical methane potential value obtained in the current experiment is expressed by the volume of methane produced per kilogram of added organic matter, which is expressed in COD, as the permeate is a liquid. The methane potential of the UF/DF permeates, noted at the end of the experiments, ranged from 149 to 181 NL CH<sub>4</sub> per kg of added COD (Table 4). There were no significant (p > 0.05) differences detected between the methane yields generated from the four tested dairy waste samples at the end of the experiment (20 days). Moreover, the methane content in the biogas did not differ significantly between the four tested samples and reached about 69% (Table 5).

**Table 4.** Methane yield, methane production rate, and reaction rate constant (k) obtained in conducted experiments.

Variant	Methane Yield [NL/kg COD <sub>added</sub> ]	Methane Production Rate [NL/kg COD/d]	k [1/d]	R <sup>2</sup>
UFP-M	150 $^{\rm a}\pm32$	52	0.37 <sup>a</sup>	0.91
DFP-M	$149~^{ m a}\pm13$	51	0.37 <sup>a</sup>	0.87
UFP-MS	181 $^{\rm a}\pm3$	46	0.28 <sup>a</sup>	0.91
DFP-MS	$169~^{a}\pm28$	48	0.29 <sup>a</sup>	0.99

K—the rate constant of methane production; UFP-M = milk ultrafiltration permeate; DFP-M = milk diafiltration permeate; UFP-MS = milk serum ultrafiltration permeate; DFP-MS = milk serum diafiltration permeate; Data are expressed as mean (n = 3)  $\pm$  standard deviation; <sup>a</sup>—Means within a column sharing the same superscript are not significantly different from each other (p > 0.05).

Variant	CH4 [%]	CO <sub>2</sub> [%]	
UFP-M	69.39 <sup>a</sup>	30.61 <sup>a</sup>	
DFP-M	69.23 <sup>a</sup>	30.77 <sup>a</sup>	
UFP-MS	68.82 <sup>a</sup>	31.18 <sup>a</sup>	
DFP-MS	69.29 <sup>a</sup>	30.71 <sup>a</sup>	

Table 5. Methane (CH<sub>4</sub>) and carbon dioxide (CO<sub>2</sub>) content in produced biogas.

<sup>a</sup>—Means within a column sharing the same superscript are not significantly different from each other (p > 0.05).

The efficiency of biomethane production from dairy waste varies across publications and depends on the applied parameters. For instance, in the research performed by Fernández et al. [48], a yield of 314 L CH<sub>4</sub>/kg COD<sub>added</sub> was demonstrated during single-stage anaerobic digestion of whey under thermophilic conditions, with an OLR up to 1.5 kg  $O_2/m^3$ day. In another study, described by Kisielewska et al. [49], who evaluated the digestion of a solution of dried ultra-filtrated whey permeate in a mesophilic system (UASB methanogenic reactor; 35 °C) at OLR rates from 4.6 to 6.9 kg COD/m<sup>3</sup>, the obtained methane yield reached about 120 L CH<sub>4</sub>/kg COD<sub>added</sub>.

The fermentation charts for all the tested substrates are presented in Figure 3. The amount of generated methane increased during the first 10 days of measurements in all the cases of the tested substrates. After this time, the methane yield remained at a constant level. This indicated a fast hydrolysis process and a good adaptation of the anaerobic sludge to the substrate.



**Figure 3.** Comparison between measured data (points) and data calculated with the first order regression (red line) for specific methane production from ultrafiltration permeates in the 20-day test: UFP-M = milk ultrafiltration permeate; DFP-M = milk diafiltration permeate; UFP-MS = milk serum ultrafiltration permeate; DFP-MS = milk serum diafiltration permeate. The gas from the inoculum has been subtracted and the data have been expressed as volume of  $CH_4$  per kg of  $COD_{added}$ .

In the current experiment, the registration of the volume of produced CH<sub>4</sub> was carried out until the available organic compounds were fully decomposed. The methane potential

test was terminated when the difference between the subsequent ten reads was equal to or less than 1%. In all cases of the tested permeates, the measurement did not last less than 20 days. For comparison, the time of decomposition obtained in the study performed by Kozłowski et al. [20] (with batch culture technology, and mesophilic conditions of 39 °C  $\pm$  1 °C), who used whey as a fermentation substrate, was seven days shorter than that noted in the current experiment (13 days vs. 20 days). The decomposition time is a crucial parameter to consider at the planning stage for future biogas plants. It enables the estimation of the size of the reactor.

## 3.2. Kinetic Analysis

The comparison between the measured data and the data calculated with the first order regression is presented in Figure 3. The coefficients for the tested model are presented in Table 4. The results from the first order regression accurately fitted the measured data. The determination coefficient for the four tested waste types is between 0.87 and 0.99. Regarding the kinetic constant, the first order regression yielded values ranging from 0.29 to 0.37/day. However, the difference between the mean values for the four tested permeates was nonsignificant (p > 0.05). The kinetic constant obtained in this experiment is comparable with the kinetic constants reported in the literature for dairy liquid wastes such as cheese whey [33] (with a hydrolysis constant k between 0.36–0.55/day; a COD of substrates between 50,000–78,000 mg/L; an inoculum/substrate ratio of 6; and a temp. of 35 °C). Mainardis et al. [33] obtained a good correlation ( $R^2 > 0.97$ ) between the predicted and measured methane potential values using a first-order kinetic model.

Moreover, the CH<sub>4</sub> flux analysis conducted in the current study revealed one peak on the first day of digestion (35–81 NL CH<sub>4</sub>/kg  $COD_{added}$ /day), followed by a less intense successive peak (<6 NL CH<sub>4</sub>/kg  $COD_{added}$ /day) on the fourth day. According to Mainardis et al. [33], this outcome can be ascribed to the different molecules present in the substrate, which are consecutively degraded. A significant fraction of the permeates was easily biodegradable by the used biomass.

## 3.3. Energy Analysis

The results of the simplified economic and energy calculations are presented in Table 6. The maximum generated electric power was estimated at 10,632 MWh/a for UFP-M and a minimum of 1374 MWh/a for DFP-MS. In general, the higher the COD of the substrates, the higher the electric power produced and thus the higher profitability of the biogas plant. In the work by Kozłowski et al. [20], energy data from a dairy factory, producing 400 t of whey per day (two times lower than in the case of the dairy plant analyzed in this study), were reported: they claimed a biomethane yield of 3,508,487 m<sup>3</sup> per annum, which is a significantly greater amount than the summarized methane yield noted for UFP-MS and DFP-MS in this study  $(2,167,430 \text{ m}^3/\text{a})$ . It is also important to highlight that in the research conducted by Kozłowski et al. [20], whey was used as a fermentation substrate, and its amount was almost half of that of UFP-MS. Whey contains more TS than UF/DF permeate, which is why the amount of biogas generated is expected to be higher than that from milk serum permeates. Therefore, in the future research, the co-digestion of UF/DF permeates in combination with other wastes should also be considered. The results presented by Comino et al. [50] indicate that the co-digestion of a high volume of whey (up to 65% in volume) and cattle slurry is possible without the use of chemicals for pH correction, but also that this kind of mix has a similar energetic potential for anaerobic digestion as energy crops such as maize. Anaerobic co-digestion can overcome most of the limitations of anaerobic mono-digestion by producing more  $CH_4$  due to the synergy of the substrates and the supplementation of the missing nutrients by the co-substrates [51, 52].

As calculated, the dairy plant can generate approximately 23,000 MWh of electricity and about 85,500 GJ of heat on an annual basis while using all the generated permeates considered in this study. The total electric energy output value corresponds to approximately 2.8 MW of the gross nominal capacity, which not only covers the annual demand of the whole factory (0.5 MW, calculated based on the daily demand of 11 MWh/d) but also enables the sale of remaining energy. In the case of an electric power calculation expressed in MW, the working time of the CHP during the whole year of exploitation was assumed to be equal to 8100 h/a. It should be mentioned that the feasible working time in a typical biogas plant is between 7200 and 8100 h/a [35]. This is much lower than the 8760 h/a value corresponding to the entire year's 24/7 operating time. This is related to many exploitation issues and breaks in biogas generation due to, i.e., mixing system failures and other mechanical damages.

Based on the data reported by Kozłowski et al. [20], it is assumed that a 3 MW biogas plant is capable of generating 24,000 MWh of electric power, which means that in the analyzed case there is still an approx. 4% power reserve for a potential increase in waste processing. The reserve may be used for an anaerobic digestion of wastes obtained by other dairy plants of the same owner, which are much smaller and do not have appropriate equipment for the further utilization of the obtained permeate. However, the transportation cost should then be considered. In the analyzed case, the manufacturer has an advantage in that they do not have to acquire a substrate for biogas production, because they can use their own effluents.

Apart from serving as potential substrates for anaerobic digestion, the UF permeates may also be considered a profitable source material for lactose production. This is especially relevant in the case of large-scale dairy factories possessing appropriate equipment since the manufacture of lactose powder requires additional investment and operation costs for equipment dedicated to the further processing of the permeate [53]. A large amount of crystalline lactose is produced globally. The major source for lactose production is the UF permeate of sweet whey. However, any other sources rich in lactose such as the UF permeates of milk may be used [15]. Therefore, the potential income of the dairy factory from the sale of lactose powder is also presented in Table 6. The degree of lactose production was estimated based on an annual generation of UF/DF permeates calculated in the case study analyzed in the current research. It should be noted that the operating costs were not included in Table 6. According to the analysis conducted by Silva et al. [54], for the manufacture of 1 kg of lactose, an approximately EUR 0.20 expenditure is required.

Parameter	Unit	UFP-M	DFP-M	UFP-MS	DFP-MS
Daily consumption of raw milk	t/d	1200	-	-	-
Daily consumption of milk serum/whey	t/d	-	-	800	-
		Bio	omethane product	ion	
Daily generated permeate	t/d	864	864	706	706
Annually generated permeate	t/a	315,360	315,360	257,690	257,690
Dairy OLR (COD)	$kgO_2/L$	0.057	0.019	0.039	0.008
Total yearly OLR (COD)	$kgO_2/a$	17,975,520	5,991,840	10,049,910	2,061,520
Methane efficiency	Nm <sup>3</sup> /kg COD	0.150	0.149	0.181	0.169
Methane production	m <sup>3</sup> /a	2,696,328	892,784	1,819,033	348,397
Annual electric energy	MWh/a	10,632	3520	7173	1374
Daily electric energy	MWh/d	29.1	9.6	19.7	3.8
Heat	GJ/a	40,055	13,263	27,022	5176
Electric power	MW	1.3	0.4	0.9	0.2
Electric power price <sup>1</sup>	EUR/MWh	96.50			
Heat price <sup>2</sup>	EUR/GJ	8.77			
Electric power value	EUR/a	1,025,988	339,680	692,195	132,591
Heat value	EUR/a	351,282	116,317	236,983	45,390
Total electric power value	EUR/a	2,190,454			
Total heat value	EUR/a	749,972			

**Table 6.** Simplified possible electricity and heat production from dairy waste in comparison to lactose production.

	0. 60.00				
Parameter	Unit	UFP-M	DFP-M	UFP-MS	DFP-MS
	Lac	tose powder produ	ction (edible; non-	pharmaceutical ra	nge)
Yearly production <sup>3</sup>	t/a	10,971	3532	6083	1387
Lactose powder price <sup>4</sup>	EUR/t		8	99	
Lactose powder value Total lactose value	EUR/a EUR/a	9,862,929 19,753,727	3,175,268	5,468,617	1,246,913

Table 6. Cont.

UFP-M = milk ultrafiltration permeate; DFP-M = milk diafiltration permeate; UFP-MS = milk serum ultrafiltration permeate; DFP-MS = milk serum diafiltration permeate; UF = ultrafiltration; OLR—organic load rate; COD—chemical oxygen demand; <sup>1</sup>—price estimated according to Energy Regulatory Office [55]; <sup>2</sup>—the price of heat produced from Poland's most popular energy carrier, i.e., hard coal, was used; <sup>3</sup>—amount of powder estimated based on production yield of 70% [53]; <sup>4</sup>—price estimated based on the report published by CLAL.it [56].

Thus, the total lactose value from Table 6 should be reduced by the cost of production, resulting in an approximately EUR 15.4 MM profit generated from lactose sale. In purely financial terms, food-grade lactose production exceeds the profitability of biogas production in the on-site biogas plant. However, other aspects such as the renewable energy demand and the geopolitical situation should also be considered by the plant owners. It is worth noting that one of the current objectives of the Bioeconomy Strategy for the EU is a reduction in energy dependence on non-renewable and unsustainable sources [57].

In summary, considering that the UF/DF permeates obtained during milk serum production are not primary sources of lactose [15] and considering the promising results of biomethane potential analysis conducted in this study, as well as the global growing demand for renewable energy, these effluents should be used as substrates in anaerobic digestion rather than lactose production. Additionally, the biomethane generated solely from UF/DF permeates of milk serum in a biogas plant can still provide an energy supply in an amount exceeding the needs of a large-scale dairy factory (1.1 MW vs. 0.5 MW) and simultaneously enabling the generation of additional profits from the sale of energy excess. Such a situation (the surplus of UF/DF permeates) enables plant owners to adjust UF/DF permeate management towards one of two analyzed solutions: lactose versus biogas production. Additionally, the rationale for the plant owner to opt for a less profitable form of gas production may stem from the fact that the development of a biogas plant requires fewer investment costs than a technological line dedicated to lactose production [20,54]. It is estimated that the return on investment in the construction of a 1 MW biogas plant is 3 years [58].

#### 4. Conclusions

The methane generation potential of four UF/DF permeates with different lactose concentrations (0.76–4.97%) was tested. A higher lactose content contributed to a higher COD of the tested effluents. The methane yield obtained in this study ( $\sim 0.16 \text{ Nm}^3/\text{kg}$ COD<sub>added</sub>) was comparable with that of the previously reported data on anaerobic digestion of UF whey permeates. This indicated the potential of using UF/DF permeates as substrates for anaerobic digestion, which may be another efficient means for its utilization. The economic analysis of the simulated electric and heat energy production with the CHP unit showed that the implementation of anaerobic digestion at the dairy plant level could provide a level of electricity and heat exceeding the amount necessary for the functioning of a large-scale factory. This could contribute to the improvement of a plant's energy balance and to a reduction in transport and management costs. When selling the energy, the profits should also be considered. Moreover, the estimated profit from lactose production, considered as alternative permeate-processing method, showed that it is an even more profitable approach. However, different aspects such as the geopolitical situation and energy market prices should also be considered while making decisions regarding the management of UF/DF permeates. It is very likely that the energy prices will rise in the near future due to the limited availability of fossil fuels. The proven efficiency of biogas

and lactose production through ultrafiltration permeates enables plant owners to adjust their management towards one of these two solutions.

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