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BIOTECHNOLOGY

Evaluation of biogas production from residues of the dietary supplement industry

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ABSTRACT. The increase in the industrial generation of dietary residues negatively affects the planet. Biogas, a result from anaerobic digestion, is produced by the degradation of organic compost converted into energy. Consequently, this energy can be used as an alternative source and contribute to managing industrial residues. Thus, this study aimed to evaluate biogas production from industrial residues in a dietary supplement industry located in the municipality of Estrela, state of Rio Grande do Sul. The methodology is based on the Automated Biogas Measurement System (ABMS), described by Konrad, Hasan, Marder, Zulian, and Guerini Filho (2021), using triplicate samples, and carried out at the Energy and Sustainable Technologies Research Center (ESTRC), located in the Scientific and Technological Park inside the Taquari Valley University - Univates (Tecnovates). The analyzed samples had different characteristics, such as solid and liquid residue. Results showed that the oily, liquid residue presents a higher potential for biogas production, and, in the same way, other substrates also showed positive results for potential biogas and methane production (BBP and BMP). The highest methane percentage reached by the samples was 63.94, 71.83; 70.55; 71.24; and 67.38% (for SR, GLR, OLR, Mix 50/50 RL and Mix S/L 96/4, respectively). Residues from dietary supplements showed positive potential for the production of biogas and methane. Furthermore, the effluent of anaerobic digestion can be used as a fertilizer, since it is rich in vitamins and minerals.

Keywords: biomethane; bioenergy; dietary residues; reuse; fuel.

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Introduction

Human activities, along with natural resources, must be responsible and sustainable for the preservation of nature and life (Obaideen et al., 2022). The use of petroleum as a source of energy has been increasing constantly; our reservoirs, on the contrary, diminish. Also, natural gas and coal are similar in terms of availability. As a consequence, there is the exploitation of new energy sources as alternatives, not only as a strategy to reduce pollution but also to be used economically, since fossil fuels are ending. This change in energy sources occurs gradually, while less polluting energy potentials are being explored. (Vieira, Nadaleti, & Sarto, 2021).

Waste is a material without viable use from a technical or economic point of view (Bellote et al., 2018). A number of different wastes, such as effluents, residual oil, and residual food, are produced by daily industrial practices. Due to their heterogeneous nature, proper management becomes a challenge and more complex technologies must be applied (Mishra et al., 2021). Due to this, there is a difficulty in generating the use of these elements, however, the by-product of a process can be transformed into raw material for another. Thus, in this cycle, all resources are fully used, and jobs can still be generated and the environmental impact reduced (Bellote et al., 2018).

The exponential increase in food waste generation negatively impacts public health and the ecosystem. There is a need for managing such material through administrative standards and scientific practices. The waste consists of proteins, lipids, and carbohydrates which will become less-complex products, such as glucose, amino acids, and fatty acids. For that reason, anaerobic digestion and the use of biogas for energy production are alternatives for managing these by-products (Mishra et al., 2021).

Biogas is a product of anaerobic digestion, whose main composite is methane (CH_4), as well as carbon dioxide (CO_2). Gas is generated by decomposition of organic compounds, by microorganisms under anaerobic conditions, and turned into bioenergy. The energy produced in this process can be used as an alternative energy source, which, in addition to effectively managing urban and industrial waste, also reduces the emission of greenhouse gases, helping in economic and environmental sustainability; especially if followed by efficient public policies. Anaerobic digestion waste residues originate from numerous human, social and. Also, biogas generators can be designed on smaller scales, a macroscale, embracing cities (Obaideen et al., 2022; Wang, Xia, Wang, & Xu, 2022).

The burning of biogas, as well as producing energy, can reduce the emission of greenhouse gases. CH_4 is twenty-eight times more harmful to global warming than CO_2, but by using it, fossil fuels are replaced and the amount of CO_2 released into the atmosphere lessens (Hernandez, Paula, Possetti, Cantão, & Aisse, 2021). Brazil has the advantage of being one of the cleanest energy matrices worldwide, with most of this energy coming from hydropower plants. Biogas represents only 1.22% of the country's energy matrix; which demonstrates the enormous potential to be explored (Souza, Rocha, & Brianezi, 2021).

Dietary supplements are defined as sources of substances with physiological or nutritional effect, aiming to complement the diet of a healthy human being. The main target audience for the aforesaid products are athletes or physically active people (Neves & Caldas, 2015). Moreover, the search for dietary supplements has been growing throughout the world, with the market being valued at US$ 140,1 billion in 2018, and expected to reach the value of US$216,3 billion by 2026 (Laamanen, Desjardins, Senhorinho, & Scott, 2021).

Food residue can be used in anaerobic digestion, resulting in biogas and biofertilizer, while also reducing emission or contamination, usually generated through conventional disposal of waste. (Westerholm, Liu, & Schnürer, 2020). Conventionally, more than ninety-five percent of food waste produced worldwide is disposed into landfills. In this way, the energy potential is lost, and harmful gases which contribute to the greenhouse effect are generated. (Clercq et al., 2017).

The evaluation of biogas production through waste from the dietary supplement industry is not studied enough, even though it is a growing market. The analysis of the material regarding its potential for biogas generation and energy conversion is an important scientific advance that seeks to fill an existing gap, as there is not much data available in the literature regarding the potential for biogas production from this type of waste.

From data obtained by studies, it is possible to evaluate the technical and economical viability of installing a biodigester for biogas generation and conversion to electric energy; Furthermore, it also opens up the opportunity for partnerships with companies aiming at anaerobic co-digestion with different substrates, which presents positive results. (Hasan et al., 2018; Matinc, Tonetto, Hasan, & Konrad, 2017; Hasan et al., 2017).

In view of this, the goal of this study was to check the possibility of using biogas energy to establish a circular economy process in the sector and develop a biogas production datase from the industrial residue of dietary supplements, bringing information regarding the scientific-technical development linked to the theme.

Methodology

The experiment was conducted in the Center of Research in Energy and Sustainable Technologies (CPETS) located in the Scientific and Technological Park of Universidade do Vale do Taquari - Univates (Tecnovates). The waste residue was collected from a dietary supplement company in the municipality of Estrela, state of Rio Grande do Sul.

Waste generation survey

The waste residue analyzed in this work comes from the production of solid and liquid dietary supplements. Storage at the company takes place in 20 L plastic drums for liquids, and in 200 L containers for solid waste.

Waste Transport Manifests (WTMs) are documents used by the supervisory body to control the disposal of industrial waste. From the WTMs made available at each collection on *Fepam* (State Foundation for Environmental Protection) website, a survey was carried out on the generation of organic waste in the company over six months. These quantities are listed in Table 1.

Table 1. Survey of organic waste generation.

Date	Solid Waste (kg)	Liquid Waste (kg)
Month 1	800.00	0.00
Month 2	555.00	55.00
Month 3	555.00	0.10
Month 4	1,450.00	140.20
Month 5	200.00	0.00
Month 6	780.00	60.00
Total (kg)	4,340.00	255.30

From the data gathered, a ratio of 94/6 (m/m) of solid/liquid waste produced by the company was obtained. The characterization of these residues is: solid residue, formed by powder and capsules, composed of vitamins, minerals, bioactive substances, proteins, amino acids, fibers, and carbohydrates. The liquid residue has two characteristics: vegetable oils that have not undergone thermal processing with vitamins; or water and glycerin with other nutrients, where vitamins and minerals may be present.

Biochemical biogas potential and biochemical methane potential

Assessments of the Biochemical Methane and Biogas Potential (BMP and BBP) are carried out to check the degradation of a residue and its capacity to produce biogas and methane. The CH_4 performance indicates the biodegradability of the subtract, that is, the fraction of solid waste added or degraded during anaerobic digestion. (Sánchez-Reyes et al., 2016).

BMP and BBP were determined by Equation 1. The result is expressed in liters of biogas or methane per kilogram of volatile solids. (L kg_{VS}^{-1}) (Verein Deutscher Ingenieure [VDI], 2006).

$$BMP\ or\ BBP\ = \frac{Vs - Vi(\frac{mSVis}{mSVi})}{mSVs} \qquad (1)$$

V_S: cumulative biogas or methane volume in the inoculant + co-digestion treatment (L);
V_i : cumulative biogas or methane volume in the treatment with inoculant (L);
mSV_{is}: volatile solids mass in the inoculant + biomass treatment (kg);
mSV_i: volatile solids mass in the inoculant treatment (kg);
mSV_s: volatile solids mass in the co-digestion treatment (kg).

Total solids

Total Solids (TS) represent the solids that remain after water removal. Volatile Solids (VS) are the biodegradable organic fraction subjected to degradation (Akunna, 2019).

For TS analysis, samples were homogenized and weighed (10 g or 20 mL), in previously calcined porcelain crucibles, and taken to an oven at 105°C for 24h, using the dry heat technique. After drying, samples were cooled in a desiccator and weighed again. Triplicates were used as per Standard Methods 2540 (American Public Health Association [APHA], American Water Works Association [AWWA], & Water Environment Federation [WEF], 1999). The calculation of total solids was performed according to Equation 2.

$$TS(\%) = \frac{(A-B)\ .100}{(C-B)} \qquad (2)$$

In which:
A: post-oven weight (g);
B: glassware weight (g);
C: glassware weight + sample (g).

Volatile solids and fixed solids

Analysis for VS and Fixed Solids (FS) were carried out from the dry material obtained in the TS analysis. This material was sent to a muffle furnace at 550°C for approximately 2h. Later, samples were cooled in a desiccator for final weighing. Triplicates were used as per Standard Methods 2540 (APHA et al., 1999). TS and FS calculations were performed according to Equations 3 and 4.

$$VS(\%) = \frac{(A-D)\ .100}{A-B} \qquad (3)$$

$$TS(\%) = \frac{(D-B)\ .100}{A-B} \qquad (4)$$

In which:
A: post-oven weight (g);
B: glassware weight (g);
C: glassware weight + sample (g);
D: post muffle weight (g).

Anaerobic digestion assay

The anaerobic digestion experiment was carried out on a laboratory scale in batches, adding at once all the residue. The procedure was performed as described by Konrad et al. (2021). The proportion of residue and inoculum is defined based on the VDI 4630 standard, which establishes that the total solids content inside the reactor must not exceed 10% (VDI, 2006).

The proportions stipulated for the digestion test were:

1. 100% solid residue (SR);
2. 100% glycerinated liquid residue (GLR);
3. 100% oily liquid residue (OLR);
4. 50/50 liquid residue (Mix 50/50 LR.);
5. 96/4 solid residue/liquid residue (Mix S/L 96/4).

To start the experiment, the residue was added with the inoculum into 1 L glass reactors, in the stipulated proportions, not exceeding 10% total solids inside the reactor. Reactors were filled with 500 g (inoculum + residue). The inoculum used was a digestate provided by the laboratory. To validate the anaerobic digestion analysis, a control with microcrystalline cellulose (MC) was performed. Cellulose has an optimal biogas conversion and should reach 80% capacity. At this stage, the pH was measured again, which should be between 6 and 8 for the experiment to be successful (VDI, 2006). In addition, reactors containing only the inoculum were also incubated, which served as the blank. Table 2 presents the inoculum/substrate ratio added to each reactor at the beginning of the anaerobic digestion.

Table 2. Inoculum/substrate ratio added to each reactor at the beginning of anaerobic digestion.

Sample		Inoculum Mass (g)	Inoculum gVS	Sample Mass (g)	Sample gVS
Triplicate I	Inoculum	500,00	9,74	-	-
Triplicate II	Cellulose Microcrystalline	494,91	9,64	5,09	4,82
Triplicate III	Solid Residue	495,17	9,42	6,79	4,81
Triplicate IV	Glycerinated Liquid Residue	483,37	9,42	16,63	4,71
Triplicate V	Oily Liquid Residue	495,17	9,65	4,83	4,82
Triplicate VI	Mix 50/50 R. L.	494,53	9,64	5,47	4,82
Triplicate VII	Mix S/L 96/4	493,42	9,61	6,58	4,81

Reactors were placed in adapted incubators and maintained at a controlled mesophilic temperature (35°C).

The volume of biogas was estimated by sensors allocated to U-shaped tubes, connected to the reactors contained an acidic solution. Inside the tube, there was a polystyrene sphere, which moves as the gas is produced and, at a certain point, obstructs the passage of light between two sensors. This triggers an event, which is recorded through a control center and transcribed into excel spreadsheets (Konrad et al., 2021).

To complete the experiment and remove the incubation reactors, the VDI 4630 (VDI, 2006) standard establishes that the daily volume of biogas produced must be less than 1% total cumulative, for three consecutive days.

Potential of hydrogen

Potential of hydrogen (pH) of the sample was measured before the onset of the experiment, with the analysis of solids. For this, a digital pH meter from Digimed was used.

Methane quantification

The methane content in biogas was monitored three times a week, on Mondays, Wednesdays, and Fridays, by a specific sensor, called Advanced Gasmitter, produced by the company PRONOVA *Analysentechnik GmbH&Co*. On the days between measurements, as well as on weekends, an average value was calculated. This sensor detects the methane content in biogas and expresses the result as a percentage (%).

Results and discussion

The pH is an important variable in the anaerobic digestion process. The methanogenic activity of bacteria is significantly reduced at a pH below 5.5 as their activity is more satisfactory at a pH above 6.5 (Latif, Mehta, & Batstone, 2017). Table 3 presents the pH of the reactors at the beginning and at the end of the experiment.

Table 3. pH before and after incubation.

Sample	Pre-incubation pH	Initial pH (incubation)	Final pH (after incubation)
Inoculum		7.93 ± 0.04	7.82 ± 0.04
Microcrystalline Cellulose		7.94 ± 0.10	7.57 ± 0.04
Solid residue	3.81 ± 0.07	7.74 ± 0.06	7.81 ± 0.06
Glycerinated Liquid Residue	2.84 ± 0.05	7.59 ± 0.15	7.86 ± 0.05
Oily Liquid Residue	4.12 ± 0.10	8.07 ± 0.16	7.70 ± 0.12
Mix 50/50 LR	3.00 ± 0.10	7.84 ± 0.08	7.77 ± 0.02
Mix S/L 96/4	3.92 ± 0.19	7.71 ± 0.06	7.80 ± 0.03

Rincón-Pérez et al. (2021) found shorter reaction times at neutral pH (7.5) compared to alkaline pH (9). Despite the acidic pH of the samples, the amount added to the reactors was low, not affecting the pH of the mixture (substrate + inoculum). Therefore, the anaerobic digestion test was carried out in the best pH range, keeping close to the values at the end of the anaerobic digestion.

Hydraulic retention time (HRT) is an important parameter considering the anaerobic digestion performance. Changes in this time lead to an imbalance in the microbial culture (Bi et al., 2020). Also, Bi et al. (2020) conducted a study involving food residues and cattle manure, which demonstrated that an HRT of 15 to 25 days results in a high methane yield (236-257 mL/gSV) in addition to contributing to solid removal. Even not being combined with bovine manure, the HRT of anaerobic digestion in the present study was 27 days, very close to that reported by Bi et al. (2020).

Tables 4 and 5 were divided for better data visualization and they show the parameters evaluated in the anaerobic digestion test. The results confirm high methane yield for all substrates. Highlight for the BBPs found in the OLR, Mix 50/50 R.L. and Mix S/L 96/4, which showed values greater than the MC, demonstrating substrate assimilation by microorganisms, converting organic matter into biogas. The SR and GLR, on the other hand, presented lower values, but were very close to the MC, also showing a good conversion of organic matter. Choong, Norli, Abdullah, and Yhaya (2016) argue that trace elements are necessary to increase the performance of anaerobic digestion, including advantages such as digester stability, low levels of volatile fatty acids, greater degradation of organic matter, and greater production of biogas, which may be an indication of the optimal values of BBP, BMP and yields of the evaluated substrates, as they contain vitamins and minerals in their composition.

Table 4. Parameters evaluated in the anaerobic digestion test in the inoculum, MC, SR and GLR.

Parameters	Inoculum	MC	SR	GLR
Biogas Volume (L_{Biogas})	209.01 ± 72.66	3906.34 ± 230.03	3887.09 ± 642.19	3343.47 ± 378.92
Methane Volume ($L_{Methane}$)	18.16 ± 6.45	1611.29 ± 142.81	1788.46 ± 321.17	1306.20 ± 169.44
Maximum CH_4 (%)	20.36 ± 0.32	54.42 ± 9.40	63.94 ± 2.06	71.83 ± 2.52
BBP ($L_{Biogas}.kg_{VS}^{-1}$)	21.45^{d} ± 7.46	766.98^{c} ± 47.84	753.84^{c} ± 131.10	641.61^{c} ± 77.58
BMP ($L_{Methane}.kg_{VS}^{-1}$)	1.86^{c} ± 0.66	330.33^{b} ± 29.58	362.60^{b} ± 65.57	263.20^{b} ± 34.69
Biogas yield ($L_{Biogas}.kg_{Biomass}^{-1}$)	0.42 ± 0.15	727.29 ± 45.36	541.31 ± 94.14	188.64 ± 22.81
Methane yield ($L_{Methane}.kg_{Biomass}^{-1}$)	0.04 ± 0.01	313.23 ± 28.05	260.37 ± 47.09	77.38 ± 10.20

Table 5. Parameters evaluated in the anaerobic digestion test on residues OLR, Mix 50/50 LR and Mix S/L 96/4.

Parameters	OLR	Mix 50/50 LR	Mix S/L 96/4
Biogas Volume (L_{Biogas})	5,809.74 ± 413.78	4,594.42 ± 588.20	3,641.33 ± 230.36
Methane Volume ($L_{Methane}$)	3,460.54 ± 311.10	2,650.43 ± 355.59	1,733.53 ± 105.13
Maximum CH_4 (%)	70.55 ± 3.39	71.24 ± 1.51	67.38 ± 0.26
BBP ($L_{Biogas}.kg_{VS}^{-1}$)	$1,160.95^{a}$ ± 86.15	993.68^{b} ± 131.50	712.63^{c} ± 47.48
BMP ($L_{Methane}.kg_{VS}^{-1}$)	713.32^{a} ± 64.44	596.17^{a} ± 79.49	355.91^{b} ± 21.50
Biogas yield ($L_{Biogas}.kg_{Biomass}^{-1}$)	1,158.42 ± 85.96	800.56 ± 105.94	520.45 ± 34.68
Methane yield ($L_{Methane}.kg_{Biomass}^{-1}$)	711.76 ± 64.30	480.31 ± 64.04	259.93 ± 15.70

A study carried out by Alves, Mahler, Oliveira, Reis, and Bassin (2022) found BBP and BMP values for anaerobic digestion with food waste of 305.0 $L_{Biogas}.kg_{VS}^{-1}$ and 236.1 $L_{Methane}.kg_{VS}^{-1}$, and 692.6 $L_{Biogas}.kg_{VS}^{-1}$ and 525.7 $L_{Methane}.kg_{VS}^{-1}$ for digestion of food waste added with 3% glycerin. In the present study, BBP and BMP values were 753.84 $L_{Biogas}.kg_{VS}^{-1}$ and 362.60 $L_{Methane}.kg_{VS}^{-1}$ for SR, higher than reported by Alves et al. (2022), as well as 641.61 $L_{Biogas}.kg_{VS}^{-1}$ and 263.20 $L_{Methane}.kg_{VS}^{-1}$ for GLR, with a BBP close to that obtained by the author, and a lower BMP. When analyzed Mix 96/4, with values of 712.63 $L_{Biogas}.kg_{VS}^{-1}$ and 355.91 $L_{Methane}.kg_{VS}^{-1}$, for BBP and BMP, respectively, an increase in BMP was observed with the combination of solid waste with the liquid (about 2% glycerin), demonstrating that glycerin, when added to the process, can increase biogas production.

In Figure 1, the most prominent substrate was OLR, with a BBP of 1,160.95 $L_{Biogas}.kg_{VS}^{-1}$ and a BMP of 713.32 $L_{Methane}.kg_{VS}^{-1}$. Compared to Mix S/L 96/ 4, with a BBP of 712.63 $L_{Biogas}.kg_{VS}^{-1}$ and a BMP of 355.91 $L_{Methane}.kg_{VS}^{-1}$, the Oily Liquid Residue had values 1.6 and 2.0 times higher (BBP and BMP). In studies carried out by Li, Jin, Borrion, and Li (2018) on food waste and vegetable oil, material containing a higher lipid content (53%) showed a higher BBP, with up to 1,015 $L_{Biogas}.kg_{VS}^{-1}$, values similar to those found in the present study, with the OLR. The sample with the lowest BBP (480 $L_{Biogas}.kg_{VS}^{-1}$) was, consequently, the one containing a smaller amount of oil in its composition (33%).

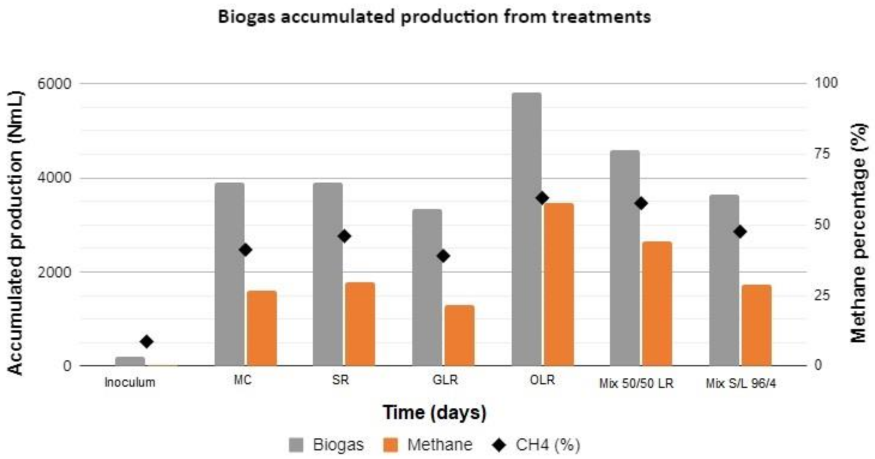

Figure 1. Biochemical potential of biogas and methane from substrates.

The average of methane was 46.01, 39.02, 59.52, 57.66, and 47.63% for substrates SR, GLR, OLR, Mix 50/50 LR and Mix S/L 96/4, respectively. The maximum percentage reached for the samples was 63.94, 71.83, 70.55, 71.24 and 67.38% (for SR, GLR, OLR, Mix 50/50 LR and Mix S/L 96/4, respectively). This demonstrates that the OLR obtained a higher average percentage of methane, but the GLR reached a higher peak of methane during anaerobic digestion. The energy capacity of a substrate can be evaluated from the maximum percentage of methane produced, therefore, the higher the methane content, the greater the calorific value (Hasan et al., 2018; Dalpaz et al., 2020). All samples presented satisfactory percentages of methane during the analysis, proving to be an interesting alternative for energy use.

When critically analyzing the results obtained, it is possible to discuss the viability of implementing a biodigester. The yield of Mix S/L 96/4 was 520.45 $L.kg_{Biomass}^{-1}$, indicating that within one month, biogas production is approximately 400 m^3 and can serve as a source of energy in the industry. One factor to be considered is seasonality, as the solid/liquid ratio of waste produced may vary according to factory production and greater sales of certain products. Despite this variation, there is no reason for a significant loss in biodigester yield, since all substrates showed excellent BBP and BMP results. Therefore, there are substrates with high production potential that should be properly exploited.

The performance of the microcrystalline cellulose BBP has to reach 80%, for validation of the anaerobic digestion assay, that is, 600 $mL_N.g_{SV}^{-1}$ of its potential (Silva, Konrad, Callado, Marder, & Araujo, 2020). The BBP value reached for the MC was 766.98 $L_{Biogas}.kg_{VS}^{-1}$ (Table 4), validating the experiment.

Statistical analysis

Results of BBP and BMP of the samples were tested by analysis of variance (ANOVA), followed by Tukey's test at a significance level of 5% for comparison between groups, using the *software* Bioestat.

Conclusion

Our results were positive for all substrates, with satisfactory production of biogas and methane, as well as BBP and BMP. As for biogas production, substrates showing higher values were those with a higher content of volatile solids (OLR and Mix 50/50). The one that represents the real proportion of the industry, Mix S/L 96/4, reached a maximum percentage of methane of 67.38%, indicating a good potential for energy use, in addition to BBP and BMP similar to microcrystalline cellulose, which demonstrates an optimal biogas conversion.

The data analysis carried out in the present study pointed out that industrial residues from dietary supplements have the potential to produce biogas and methane. The residues, when used alone or in combination, presented satisfactory results. A biodigester project for energy generation can be thought, being only necessary to evaluate the best way of use. In addition, the effluent from anaerobic digestion is rich in nutrients, and can be used as fertilizer, carrying out the exploitation cycle, with a focus on the circular economy.

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