Waste Management 169 (2023) 310–318

Contents lists available at ScienceDirect

Waste Management

journal homepage: www.elsevier.com/locate/wasman

Valorization of strawberry extrudate waste: Recovery of phenolic compounds by direct-hydrothermal treatment and subsequent methane production by mesophilic semi-continuous anaerobic digestion

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ARTICLE INFO

Keywords:
Agro-waste
Biorefinery
Economic assessment
Hydrothermal pre-treatment
Phenolic compounds recovery

ABSTRACT

Strawberry extrudate (SE) is an underused by-product from strawberry industry. Recovery of the phenolic compounds present in SE would represent a very interesting valorisation option. Two main challenges need to be solved, firstly, the solubilisation and recovery of the phenolic compounds contained in SE, and, after that, the stabilisation of the resulted de-phenolized SE. The present research evaluates the potential of a biorefinery process combining a hydrothermal pre-treatment, followed by a phenolic extraction process and, finally, the anaerobic digestion of the remaining SE for producing energy that will contribute to compensate the energy requirements of the whole system. Following the hydrothermal pre-treatment at 170 °C for 60 min, an extraction of 0.6 ± 0.1 g of gallic acid per kilogram of SE was achieved using an adsorbent resin, representing a recovery rate of 64 %. Long-term semi-continuous anaerobic digestion of de-phenolized SE was evaluated at different organic loading rates to evaluate the stability of the process. The anaerobic digestion of pre-treated SE achieved a stable methane production value of 243 ± 34 mL CH_4·g volatile solids^{-1}·d^{-1} at an organic loading rate (ORL) of 1.25 g volatile solids·L^{-1}·d^{-1}. During the operation at this ORL, the control parameters including pH, alkalinity, soluble chemical organic demand (sCOD), and volatile fatty acid (VFA) remained stable and consistently constant. Specifically, the VFA in the reactor during this stable period achieved a value of 102 ± 128 mg O2/L. Also, an economic balance showed that the minimal price of the generated phenolic extract for having benefited from the proposed biorefinery system was 0.812 €·(g of gallic acid equivalents)$^{-1}$, a price within the range of phenolic compounds used in the food industry.

1. Introduction

Strawberries contains a wide range of phenolic compounds, high fiber content, ascorbic acid among others compounds (Cubero-Cardoso et al., 2020b; Giampieri et al., 2012). This health-supporting properties have led to the substantial economic role of the strawberry, with a global strawberry market value exceeding US$ 23,600 million per year (FAO, 2022; Warner et al., 2021). In the last ten years, world strawberry production has increased from 6 to almost 9 million tons (FAO, 2022). This increase in strawberry production leads to an increase in the wastes generated by the strawberry industry, mainly those generated in the manufacture of by-products such as jams, yogurts and/or ice creams,

Abbreviations: BMP, Biochemical Methane Potential; C2, Acetic acid; C3, Propionic acid; COD, Total Chemical Oxygen Demand; DLP, de-phenolized Liquid Phase; HTR, Hydraulic Retention Time; i-C4, *iso*-Butyric acid; i-C5, *iso*-Valeric acid; LP, Liquid Phase; MS, Mineral Solids; n-C4, n-Butyric acid; n-C5, n-Valeric acid; OLR, Organic Loading Rate; sCOD, Soluble Chemical Oxygen Demand; SE, Strawberry Extrudate; SP, Solid Phase; SS, Synthetic Solution; TS, Total solids; VFA, Volatile Fatty Acids; VS, Volatile solids.

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https://doi.org/10.1016/j.wasman.2023.07.016
Received 3 April 2023; Received in revised form 7 June 2023; Accepted 12 July 2023
Available online 25 July 2023

called strawberry extrudate (SE) (Cubero-Cardoso et al., 2020b). The increase in the generation of SE is negatively associated with the cost derived from the need for an adequate treatment, in order to avoid the environmental impacts of this easily putrefiable substrate (Massé et al., 2016). However, SE, similar to strawberries, contains different high-value compounds that could be interesting in being extracted (Cubero-Cardoso et al., 2020b).

Different hydrothermal treatments have been proposed and applied for SE to facilitate the extraction of high-added value compounds from the liquid phase after centrifugation (Trujillo-Reyes et al., 2019). The lignocellulosic material contained in the SE possesses a large amount of phenolic compounds and a large amount of soluble sugars, as was recently reported in previous studies (Millati et al., 2020; Rodríguez-Gutiérrez et al., 2019). After hydrothermal treatment, the recovery of such phenolic compounds has been reported to be done by adsorption with an adsorbent resin (Chavan and Gaikwad, 2021; Li et al., 2021; Neggad et al., 2021). For example, resin Amberlite XAD 16 extracted around 50 % of phenolic compounds contained in the SE hydrothermal pre-treated (Cubero-Cardoso et al., 2020d).

The application of hydrothermal treatments can lead to the degradation of only thermosensitive phenolic compounds, thereby facilitating the production of recalcitrant compounds that are difficult to biodegrade. However, these treatments do not promote the degradation of most of the phenolic compounds that later are extracted in the liquid phase (Cubero-Cardoso et al., 2020c; Struck et al., 2016). The extraction of the phenolic compounds contained in SE, which are well-known microbial inhibitors, could be beneficial for subsequent biological management of the remaining biomass (Rashama et al., 2021). The anaerobic digestion process of agro-waste is a well-established biotechnology commonly used to obtain energy from the produced methane (Yan et al., 2017). Several studies of anaerobic digestion of diverse agro-wastes have been carried out, observing how the pre-treatment proposed in this study improved the methane production obtained from these residues (Ahmed et al., 2021; Serrano et al., 2019a, 2019b; Trujillo-Reyes et al., 2019). Methane production is usually increased due to the solubilized lignocellulosic biomass through hydrothermal pre-treatments carried out at temperatures above 120–180 °C (Bhatia et al., 2021; Serrano et al., 2019b). The hydrothermal pre-treatment of the SE at these temperatures should favor its subsequent anaerobic digestion due to breaking bonds between cellulose, hemicellulose, and lignin. In addition, pre-treatment improves the accessibility of hydrolytic enzymes to the released polysaccharides (Yadav et al., 2019). However, severe conditions during the hydrothermal treatments should be avoided due to the formation of refractory compounds for biogas production, as was observed in the other studies (Ghimire et al., 2021; Liao et al., 2021; Serrano et al., 2019b; Sheng et al., 2023). In a study carried out by Cubero-Cardoso et al. (2020d), was observed that implementing hydrothermal pre-treatments at temperatures of 170 °C for 60 min or 220 °C for 5 min, followed by the extraction of phenolic compounds, resulted in a considerable increase in the methane production rate during biochemical methane potential (BMP) tests when compared to untreated SE. In another recent study, the same substrate was used to perform a semi-continuous anaerobic digestion test after the waste pre-treatment at 220 °C for 5 min and subsequent extraction of phenolic compounds, observing that the reactors were unstable, despite what was observed in the BMP tests. This study reported a low methane production of 135 ± 11 mL CH_4 ·(g volatile solids (VS))$^{-1}$·d^{-1} at an organic loading rate (OLR) of 0.50 g VS·L^{-1}·d^{-1}, observing that the pH and alkalinity were not stable and decreased with increasing OLR (Cubero-Cardoso et al., 2020a). This indicates that long-term operation is influenced by certain factors, such as the accumulation of inhibitors, which were not detected in a short time study, such as the BMP test (Cubero-Cardoso et al., 2020d). Therefore, it is necessary to evaluate pre-treatment conditions less severe than 220 °C to see if the methane production is stable at the long-time operation.

Therefore, the objective of this work was to evaluate the valorization of SE through a combination of phenolic compounds extraction and a semi-continuous anaerobic digestion process. To achieve this objective, it was carried out a hydrothermal pre-treatment of SE at 170 °C for 60 min, recovering phenolic compounds with the adsorbent resin Amberlite XAD 16. The remaining biomass was subjected to a mesophilic semi-continuous anaerobic digestion to maximized organic loading rates. Finally, an economic assessment of the valorization process was done.

2. Materials and methods

2.1. Pre-treatment of strawberry extrudate: Direct-hydrothermal treatment and extraction of phenolic compounds

The strawberry extrudate (SE) used in this study was described in detail in (Cubero-Cardoso et al., 2020d). Hydrothermal treatments have been carried out at a temperature of 170 °C and 8.7 kg (cm^2)$^{-1}$ pressure for 60 min using a direct steam treatment reactor described in a previous work (Cubero-Cardoso et al., 2020d). The treatment of the SE was carried out twice throughout the experimental time. The first time, 10.06 kg of SE were treated with a steam consumption of 1.03 kg steam·(kg SE)$^{-1}$. The second time, 4.93 kg of SE were treated, and 2.22 kg steam·(kg SE)$^{-1}$ were consumed. After each treatment, and to promote the recovery of phenolic compounds, the treated SE was centrifuged at 4700 g/1450 rpm (Centrifuge Comteifa, S. L., Barcelona, Spain) for separating a liquid phase (LP) and a solid phase (SP). To extract phenolic compounds, a 140 cm tall column with a diameter of 4.5 cm was packed with 100 mL of Amberlite XAD16 adsorbent resin. The resin was preconditioned by adding 3 L of water and allowing it to settle. Next, the resin was activated with 0.5 L of 50 % ethanol and 10 L of LP was passed through the column to extract the phenolic compounds. This process resulted in the extraction of the desired phenolic compounds, which were collected, while the DLP was further used in the subsequent anaerobic digestion process. The phenolic compounds were then diluted by adding 2 L of 80 % ethanol and 1 L of 96 % ethanol. The extraction efficiency of phenolic compounds from the LP was determined to be 55 % and 64 % for each respective hydrothermal treatment conducted, resulting in similar concentrations of phenolic compounds in the DLP. The extraction efficiency of phenolic compounds from the LP was determined to be 55 % and 64 % for each respective hydrothermal treatment conducted, resulting in similar concentrations of phenolic compounds in the DLP. The physicochemical characterization of different samples resulting from hydrothermal treatments, phenolic compounds extraction, and SE without treatment are shown in Table 1.

2.2. Experimental procedure of mesophilic semi-continuous anaerobic digestion

The anaerobic flocculent inoculum used in this study was obtained in the “Copero” wastewater treatment plant in Sevilla, Spain. The main chemical characteristics of the anaerobic inoculum were: pH = 7.1 ± 0.1; Alkalinity = 4500 ± 100 mg $CaCO_3$·L^{-1}; Total Solids (TS) = 21100 ± 100 mg·L^{-1}; Volatile Solids (VS) = 13300 ± 200 mg·L^{-1}; Total COD (COD) = 18900 ± 600 mg O_2·L^{-1}; Soluble COD (CODs) = 520 ± 10 mg O_2·L^{-1}; Total Phenolic compounds = 100 ± 1 mg gallic acid eq.·L^{-1}; and Total Sugars = 20 ± 1 mg glucose eq.·L^{-1}. Three glass reactors of 2.0 L total volume were operated in anaerobic conditions as triplicates, with a final working volume of 1.7 L for each of them. The reactors were inoculated with anaerobic inoculum at a 10 g VS·L^{-1} concentration. The reactors were continuously stirred through a cylindrical magnetic bar. Every day samples of the reactors were taken and fed with a syringe of 100 mL capacity. The volume of produced methane was measured after CO_2 removal with a NaOH solution (3 N) in tight bubblers. Methane production was expressed at standard temperature and pressure conditions (25 °C and 1 atm).

During the first 45 days, the three reactors were conditioned and

Table 1
Physicochemical characterization of different phases resulting from untreated and pre-treated strawberry extrudate (SE).

		First hydrothermal treatment			Second hydrothermal treatment		
	SE	**SP**	**LP**	**DLP**	**SP**	**LP**	**DLP**
pH	3.7 ± 0.1	4.1 ± 0.1	3.9 ± 0.1	4.0 ± 0.1	3.9 ± 0.1	3.9 ± 0.1	4.0 ± 0.1
TS (mg·(kg SE)$^{-1}$)	142000 ± 1000	122000 ± 3000	35800 ± 200	27000 ± 1000	82000 ± 1600	48000 ± 400	46200 ± 200
VS (mg·(kg SE)$^{-1}$)	137000 ± 1000	117000 ± 3000	33400 ± 200	27000 ± 1000	79000 ± 1500	45000 ± 1400	42000 ± 300
COD (mg O_2·(kg SE)$^{-1}$)	200000 ± 6000	173000 ± 700	49200 ± 900	35600 ± 200	108000 ± 2000	56800 ± 500	46000 ± 1000
sCOD (mg O_2·(kg SE)$^{-1}$)	47000 ± 400	27500 ± 300	45200 ± 800	38300 ± 300	2500 ± 80	56000 ± 1000	47000 ± 1000
Total phenolic compounds (mg gallic acid eq.·(kg SE)$^{-1}$)	1410 ± 5	2200 ± 50	1020 ± 10	460 ± 20	1430 ± 30	2560 ± 70	930 ± 20
Total sugars (mg glucose·(kg SE)$^{-1}$)	2085 ± 70	9010 ± 10	18700 ± 100	16045 ± 170	3900 ± 10	21550 ± 450	18070 ± 500

TS, total solids; VS, volatile solids; COD, chemical oxygen demand; sCOD, soluble chemical oxygen demand; SP, solid phase; LP, liquid phase; DLP dephenolised liquid phase.

acclimated with a mixture of a synthetic solution (SS) with glucose (50 g·L^{-1}) and sodium acetate (25.2 g·L^{-1}) and untreated SE, as described in Table 2. After this adaption, the reactors were operated for 273 days being fed with a mixture of SP and DLP in the same relation in VS after hydrothermal treatment, using different OLRs (Table 2) All through the assay, hydraulic retention time (HTR) was 21 days. The SP and DLP substrate obtained in the second pre-treatment was used from 226 days to the end of the experimental time (day 273).

The volatile solids (VS) removal was calculated by dividing the grams of VS added each day between the grams of VS inside the reactor (**Equation (1)**).

$$VSremoval(\%) = \frac{g_{reactor}VS}{g_{added}VS} x100 \quad (1)$$

2.2.1. Chemical analyses

For the water-soluble compounds measured in the SE and the solid phase (SP), 160 g of distilled water were added to 20 g of sample and keeping it stirred for 24 h (Thompson et al., 2002). Soluble compounds from samples were centrifuged and microfiltered with 0.45 μm nylon microfilters. To extract the total phenolic compounds from all samples (SP, SE, and digestate), a mixture of 20 mL methanol and water (v/v, 80/20) was added to 10 g of each sample. The samples were then incubated in a water bath at 70 °C for 1 h. Subsequently, microfiltration was performed using 0.45 μm nylon microfilters.

pH, TS total solids (TS), volatile solids (VS), alkalinity, and soluble chemical oxygen demand (sCOD) were measured in the samples according to the Standard Methods (APHA, 2017). Chemical oxygen demand (COD), total phenolic compounds, and total sugars were measured by colorimetric methods, Folin Ciocalteu and Antrone, respectively, in different samples as described in the previous work (Rodríguez-Gutiérrez et al., 2019). Volatile fatty acids (VFA) (C2-C5) were determined using a Shimadzu GC-2010 gas chromatograph equipped with a column of 100 % ethylene glycol composition of 0.25 mm × 25 m and a flame ionization detector (FID). The oven temperature gradually increases from 100 to 170 °C at a rate of 5 °C·min^{-1}. Nitrogen (30 mL·min^{-1}), hydrogen (40 mL·min^{-1}), and air (399.8 mL·min^{-1}) were used as carrier gas at a flow rate of 40.1 mL·min^{-1} at 456 kPa. Individual VFA concentrations were determined as COD using the stoichiometric COD factor of 1.0667, 1.512, 1.813, 1.813, 2.036, and 2.036 for acetic, propionic, butyric, *iso*-butyric, valeric, and *iso*-valeric, respectively (Paulose and Kaparaju, 2021). All measurements were made in triplicate to obtain mean and standard deviation values.

Table 2
Anaerobic digestion set-up and experimental design.

Experimental Stage	Duration	Feeded Substrate	OLR (g VS·L^{-1}·d^{-1})
Bio-stimulation	16 days	SS	1.00
Adaptation	4 days	SS:SE 75:25, in VS	1.00
	13 days	SS:SE 50:50, in VS	1.00
	12 days	SE 100, in VS	1.00
Stage 1	78 days (0–77)	SP + DLP	1.00
Stage 2	21 days (78–98)	SP + DLP	1.50
Stage 3	76 days (99–174)	SP + DLP	1.00
Stage 4	73 days (175–247)	SP + DLP	1.25
Stage 5	26 days (248–273)	SP + DLP	1.50

SS, Synthetic solution with glucose (50 g·L^{-1}) and sodium acetate (25.2 g·L^{-1}); OLR, organic loading rate; VS, volatile solids; SE, strawberry extrudate; SP, solid phase; DLP, dephenolised liquid phase.

2.3. Economic assessment methodology

An economic evaluation was carried out to predict the feasibility of the valuation of SE proposed in this study. A good option for a scenario includes the pre-treatment of the SE with the high-temperature hydrothermal treatment followed by the extraction of phenolic compounds and an anaerobic digestion process with the most stable OLR achieved in the experimental procedure. The economic assessment took into account various factors such as methane production, hydrothermal treatment, extraction of phenolic compounds, and the cost of electricity.

The energy production in the anaerobic digester was based on the experimental methane production values obtained at ORL = 1.25 g VS/L·d (Table 2), while the lower calorific power of methane was 35,793 J/L (Serrano et al., 2015). The energy efficiency obtained through a cogeneration biogas engine was considered as 33 % and 55 % for electricity and thermal energy, respectively (Cano et al., 2014). To increase the waste temperature from 20 to 35 °C (required operating temperature) while accounting for thermal loss of 10 %, a specific heat of 4.18 kJ/kg·°C was used to determine the thermal energy requirement. The electricity consumption was estimated based on the values of 1.8·103 kJ/m^3 and 3.0·102 kJ/m^3 of reactor for pumping and stirring, respectively (Ferrer et al., 2009). For hydrothermal pretreatment, enthalpy values of 104.9 kJ/kg (water at 20 °C and 1 kg/cm^2) and 2664.2 kJ/kg (steam at 170 °C and 8.7 kg/cm^2) were used to calculate the thermal energy requirement. The amount of steam was determined experimentally as 1.03 kg of steam/kg of SE, and when the energy requirement exceeded the thermal energy generated in the anaerobic digester, a methane supply was used to produce the amount of consumed steam. A heat recovery system was also included with a thermal efficiency of 80 % (Cano et al., 2014). Regarding phenolic compound extraction, the electricity consumption was estimated based on the electricity employed

for pumping, and a phenol extraction efficiency of 43 % was obtained for the recovery of gallic acid observed during the experiments in the liquid phase after the extraction process. The cost of phenolic compound extraction was 0.50 €/kg of SE (Trujillo-Reyes et al., 2019). In terms of prices, the cost of electricity was 0.125 €/kWh (Eurostat, 2017), while the cost of methane was 0.04 €/kW (Fermoso et al., 2018). The net benefit of the studied SE valorization option was determined by assessing the economic balance between operational costs and revenue generated from sales. The minimum sales price for the extracted phenol (expressed as €/g gallic acid equivalents) was calculated by assuming a net benefit value of zero.

3. Results and discussion

3.1. Influence of organic loading rate on methane production

The mixture SP + DLP obtained after hydrothermal treatment and subsequent extraction of phenolic compounds from SE was evaluated by monitoring the variation of the daily methane production rate with time (Fig. 1). In the first stage, i.e., 0–77 days and OLR of 1.00 g VS·L^{-1}·d^{-1}, the methane production rate presented a mean value of 216 ± 26 mL CH_4·(g VS)$^{-1}$·d^{-1}, and VS removal around 70 % (Table 2) during 3 HRTs. This methane production rate value at OLR of 1.00 g VS·L^{-1}·d^{-1} was 21 % higher than that achieved for an olive mill solid waste treated with the same hydrothermal treatment, and a similar phenolic compounds extraction method (Serrano et al., 2019b). When the OLR was increased to 1.50 g VS·L^{-1}·d^{-1} on day 78, inhibition in the methane production process was observed (Fig. 1), which after 1 HRT presented an average value of around 66 ± 26 mL CH_4·(g VS)$^{-1}$·d^{-1} and VS removal of 61 %. An increase in solid concentrations inside the reactor (TS and VS) was also observed due to the decrease in degradation percentage (Table 3). According to Siles et al. (2013), the anaerobic digestion of untreated strawberry extrudate, gives a the methane production of 230 mL of CH_4/g VS at an OLRs = 1 g of VS/(L·d). Another study found that untreated strawberry residue obtained from rejections provided by a supermarket produced a methane production of 231 mL of CH_4/g of VS at an OLR of 4.4 g of VS/(L·d) (Arhoun et al., 2017).In a previous work by Cubero-Cardoso et al.,(2022), a methane production of 344 ± 21 L CH_4/kg VS was achieved when a feed SE at 2 g VS/L was provided once a week. Furthermore, other previous studies examining the biomethanization of SE through BMP test have reported an increase in methane production yield when different hydrothermal treatments were applied (Cubero-Cardoso et al., 2020d; Trujillo-Reyes et al., 2019).

Table 3
The average values of the measured parameters were obtained from samples taken from the reactors at different OLRs during the anaerobic digestion process.

	Stage 1	Stage 2	Stage 3	Stage 4	Stage 5
OLR (g VS/(L·d)	**1.00**	**1.50**	**1.00**	**1.25**	**1.50**
Days	0–77	78–98	99–174	175–247	248–273
pH	6.8 ± 0.3	5.8 ± 0.2	6.8 ± 0.2	6.9 ± 0.2	5.2 ± 0.7
Alkalinity (mg $CaCO_3$·L^{-1})	1920 ± 670	1200 ± 500	2300 ± 200	2200 ± 600	900 ± 400
TS (mg TS·L^{-1})	12693 ± 2627	21145 ± 3382	19046 ± 3028	20346 ± 3895	28699 ± 1719
VS (mg VS·L^{-1})	10250 ± 2875	18423 ± 3015	16412 ± 2844	17969 ± 3920	26272 ± 1521
sCOD (mg O_2·L^{-1})	891 ± 432	5368 ± 979	2780 ± 1522	1296 ± 188	6320 ± 2748
Total VFA (mg O_2·L^{-1})	240 ± 331	3842 ± 1042	1313 ± 1295	102 ± 128	5068 ± 3265
Ratio VFA/CODs (%)	27	72	47	8	80
Total phenolic compounds (mg gallic acid eq.·L^{-1})	186 ± 77	423 ± 28	372 ± 78	363 ± 57	430 ± 48
Methane production (mL CH_4·(g VS)$^{-1}$ d^{-1})	216 ± 26	66 ± 26	253 ± 26	243 ± 34	71 ± 81
VS removal (%)	70 ± 2	61 ± 8	67 ± 15	69 ± 9	58 ± 7

OLR, organic loading rate; TS, total solids; VS, volatile solids; sCOD, soluble chemical oxygen demand; VFA, volatile fatty acids.

In the third stage, between days 99–174, the OLR was reduced to 1.00 g VS·L^{-1}·d^{-1}. The methane production rate was recovered, achieving a mean value of 253 ± 26 mL CH_4·(g VS)$^{-1}$·d^{-1} and VS removal of around 67 %. The increase of the OLR to 1.25 g VS·L^{-1}·d^{-1} from day 175, showed a similar methane production rate and VS removal to OLR of 1.00 g VS·L^{-1}·d^{-1} (Table 2), which presented an average value of around 243 ± 34 mL CH_4·(g VS)$^{-1}$·d^{-1} and 69 %, respectively.

Finally, in stage 5, the OLR of reactors was increased on days 248 from 1.25 to 1.5 g VS·L^{-1}·d^{-1} and operated till the end of the experimental time on day 273. Inhibition was observed in the methane

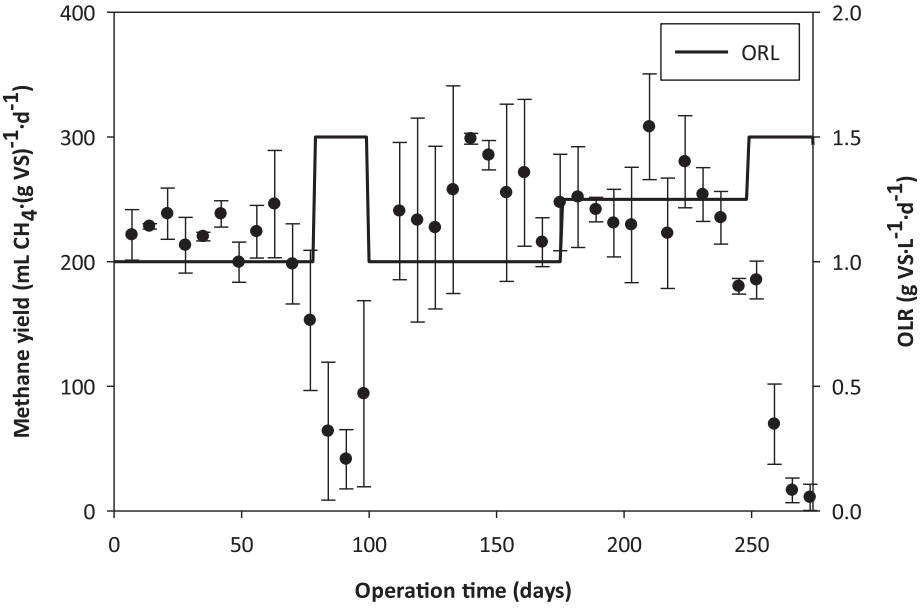

Fig. 1. Variation of the methane yield and organic loading rates (OLR) throughout the experimental time.

production (Fig. 1), i.e., methane production rate rapidly dropped, which presented values less than 20 mL CH_4·(g VS)$^{-1}$·d^{-1} and VS removal of around 58 % up to the complete inhibition of the reactor at day 273. This increase in OLR was performed to evaluate whether the extended operating time could induce the anaerobic biomass in the digester to adapt to the substrate so that the digester can operate at 1.50 g VS·L^{-1}·d^{-1}. In the study carried out by Cubero-Cardoso et al.(2020a) it was observed a 54 % reduction in methane production at an OLR of 0.75 g VS·L^{-1}·d^{-1}, with the same substrate pre-treated hydrothermally with the steam explosion at 220 °C for 5 min.

3.2. Variation of organic matter concentration and stability of anaerobic process

In order to control the degradation of the organic matter fed and to monitor the stability of the anaerobic digestion process with the variation in the OLR, the following parameters were measured daily: pH, alkalinity, sCOD, and VFA (Fig. 2**A and B**). In the first 77 days with OLR of 1.00 g VS·L^{-1}·d^{-1}, the sCOD presented an average value of around 891 ± 432 mg O_2·L^{-1}·d^{-1}, being the ratio of VFA/sCOD of 27 %. There was also a decrease in pH and alkalinity values around 6.4 and 1200 mg $CaCO_3$·L^{-1}, respectively. These decreases were also observed for the SE after a steam explosion, at 220 °C for 5 min, and phenolic compounds recovery treatments and the same OLR at 1.00 g VS·L^{-1}·d^{-1} in the study reported by Cubero-Cardoso et al. (Cubero-Cardoso et al., 2020a). Therefore, with the change in OLR, sodium bicarbonate was added to the reactors to buffer the drop in pH to achieve values between 6.5 and 7.5, the optimal range for a stable anaerobic process (Mao et al., 2015; Wheatley, 1990). When the OLR was increased to 1.50 g VS·L^{-1}·d^{-1}, between days 78–98, a marked increase in sCOD was observed, which presented an average value of 5368 ± 979 mg O_2·L^{-1} with a ratio of VFA/sCOD of 72 %, related to inhibition in methane production due to

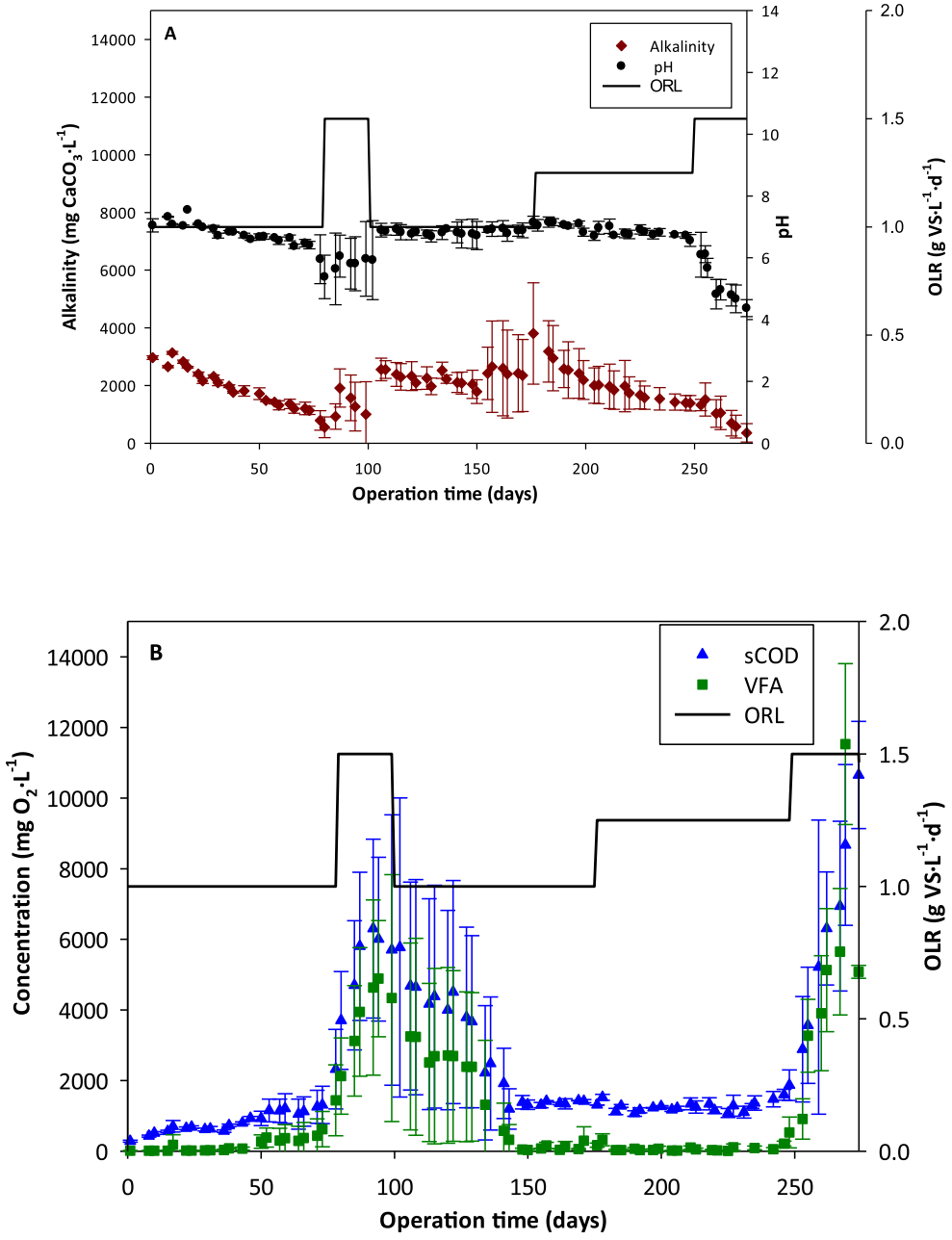

Fig. 2. Variation of (**A**) pH (●) and alkalinity (♦) and (**B**) concentration of soluble chemical oxygen demand (sCOD) (▲) and volatile fatty acids (VFA) (■) throughout the experimental time respect to the different assayed organic loading rates (OLR).

the acidification of the reactors.

In stage 3, operating again with OLR of 1.00 g VS·L^{-1}·d^{-1}, between days 99–174, stabilization of the reactors was observed after the inhibition process, achieving a decrease in the accumulation of sCOD, which presented a mean value of 2780 ± 1522 mg O_2·L^{-1} and a ratio of VFA/sCOD of 47 %. Furthermore, stabilization of the reactors was also observed in the almost neutral pH values, i.e., 6.8 ± 0.2, and an average alkalinity value of 2300 ± 200 mg $CaCO_3$·L^{-1}. In the stage 4, increased in OLR to 1.25 g VS·L^{-1}·d^{-1}, between days 175–247. The sCOD presented a stable mean value of around 1296 ± 188 mg O_2·L^{-1} and a ratio of VFA/sCOD of 8 %. Moreover, pH was stabilized with a 6.9 ± 0.2 and an average alkalinity value of 2200 ± 600 mg $CaCO_3$·L^{-1}.

Finally, more significant inhibition was observed in stage 5, when the OLR of reactors was increased on days 248 from 1.25 to 1.5 g VS·L^{-1}·d^{-1} and operated till the end of the experimental time on day 273. Accumulation of sCOD was also found with an average value of 6320 ± 2748 mg O_2·L^{-1} and VFA/sCOD ratio of 80 %, completely inhibiting the reactor. During stage 5, alkalinity decreased until 900 ± 400 mg $CaCO_3$·L^{-1}, causing an acidification of the reactors with average pH values of 5.2 ± 0.7 (Table 3). When the reactors were operated at an OLR of 1.50 g VS·L^{-1}·d^{-1} in stages 2 and 5 alkalinity values were below the ideal conditions for anaerobic digestion, achieving amounts between 2000 and 4000 mg $CaCO_3$·L^{-1} (Casallas-Ojeda et al., 2021; Fernández-Rodríguez et al., 2019). The alkalinity dropped sharply, which may be due to low pH values in the feeder substrate in the reactors (Table 1) and high ORL loads, similar to the study by Arhoun et al. (2017).

Individual VFAs, between C2-C5, were measured throughout the experiment and expressed in mg O_2·L^{-1} (Fig. 3). In stage 1, individual VFA concentrations were less than 50 mg O_2·L·, except for propionic acid, for which its concentration increased to values of 350 mg O_2·L·. In stage 2, a considerable concentration of VFA was produced due to instability of the process at high ORL, showing C2 and C3 mean values of around 1300 mg O_2·L·, and i-C4, n-C4, i-C5 and n-C5 concentrations of less of 1000 mg O_2·L^{-1}. At the end of stage 3, between days 99–174, the reactors were effectively recovered from the previous inhibition episode. The VFA accumulation was solved, presenting mean values for each VFA at the end of stage 3 below 50 mg O_2·L^{-1}, the same to stage 1. Continuing this decreasing trend and remaining constant until 247 day during stage 4 with OLR of 1.25 g VS·L^{-1}·d^{-1}. Finally, when the OLR increased again up to 1.50 g VS·L^{-1}·d^{-1} in stage 5, between days 248–273, a higher inhibition was observed, reaching concentrations greater than 1000 mg O_2·L^{-1} for each acid studied. An increase in the ORL results in observable instability in the reactor, reflected by fluctuations in control parameters (Trujillo-Reyes et al., 2022). pH begins to decrease due to elevated levels of VFA and reduced alkalinity. The presence of acetic and propionic acid, as observed in previous studies, contributes to this instability. Acetate has been described as the least toxic fatty acid, propionate the most inhibitory acid at concentrations of approximately 3000 mg·L^{-1} (Montañés et al., 2013). Another study related that acetic acid at concentrations higher than 800 mg·L^{-1} could trigger a failure in the anaerobic digestion process (Casallas-Ojeda et al., 2021).However, the presence of longer-chain VFAs such as butyric and valeric acids suggests that the hydrolytic stage is functioning effectively, leading to alkalinity consumption (Rubio et al., 2022). It also caused a simultaneous reduction in the microbial activity of the methanogenesis stage, as was observed in the decrease in methane production (Fig. 1) (Mao et al., 2015).

3.3. Variation of phenolic compounds with the organic loading rate

Phenolic compounds, expressed as mg gallic acid equivalent·L^{-1}, were carefully monitored because of their high inhibitory potential of the anaerobic digestion (Fig. 4) (Battista et al., 2015). During stages 1 and 2, despite the variation OLR, the phenolic compound concentration gradually increased until reaching an average value of 423 ± 28 mg gallic acid·L^{-1} at the end of stage 2. During stages 3 and 4, it is observed the concentration of phenolic compounds remained with only slight variations at values around 350 mg gallic acid·L^{-1}. When the reactors operated under unstable conditions due to the increase of OLRL at 1.50 g VS·L^{-1}·d^{-1} in stage 5, the phenolic compound concentration increased up to a value of 599 ± 64 mg gallic acid·L^{-1} (Fig. 4). This increase in phenolic compounds was similar to the trend reported by Serrano et al., (2019b) for the anaerobic digestion of olive mill solid waste subjected to a hydrothermal pretreatment at 170 °C during 60 min. However, the

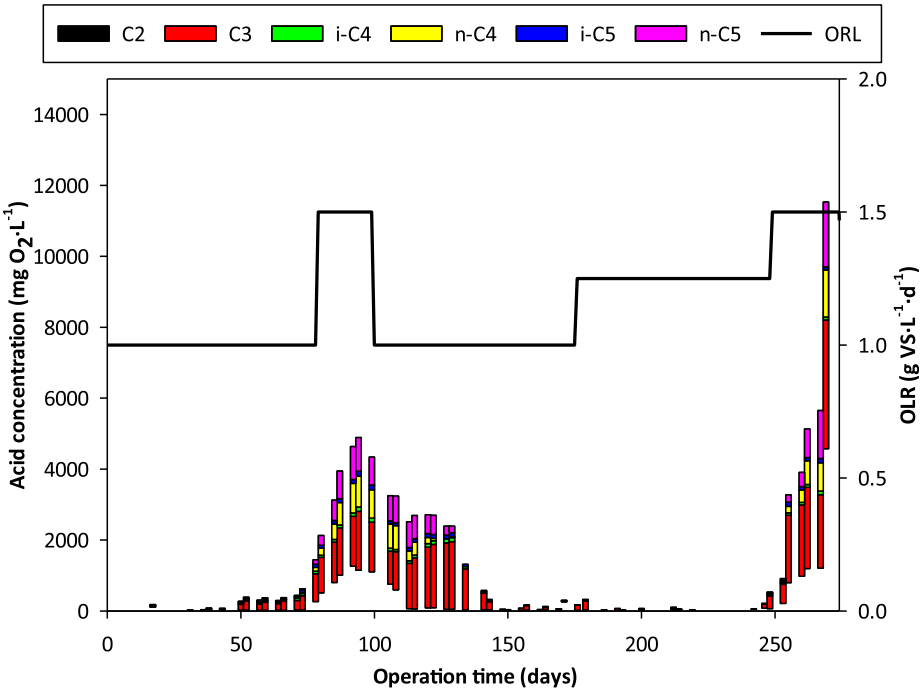

Fig. 3. Variation of the concentration of acetic acid (C2), propionic acid (C3) *iso*-butiric acid (i-C4), n-butiric acid (n-C4), *iso*-valeric acid (i-C5) and n-valeric acid (n-C5) respect tothe different organic loading rates (OLR) throughout the experimental time.

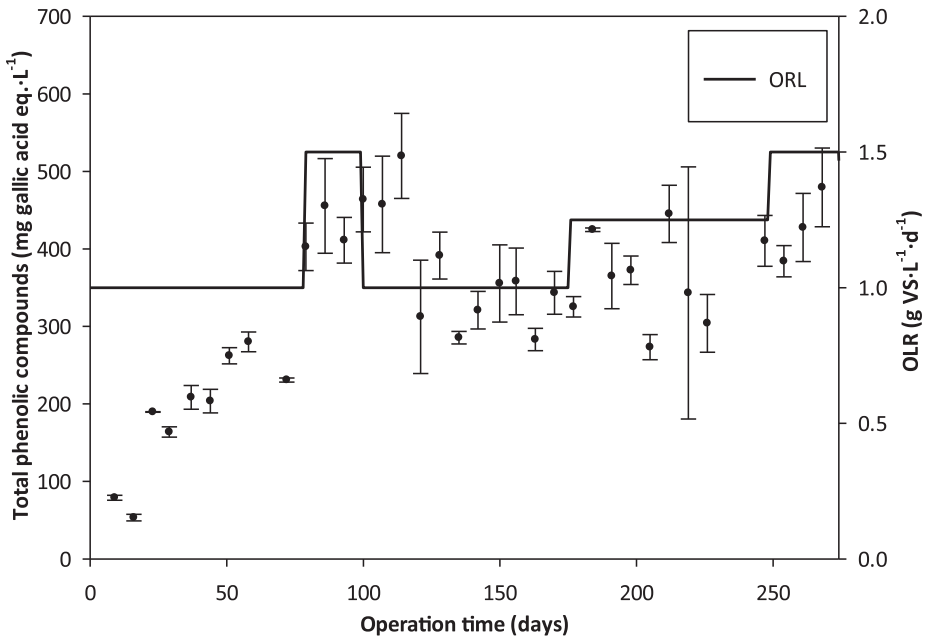

Fig. 4. Variation of the concentration of total phenolic compound (expressed as mg equivalents of gallic acid per litre)with respect to the organic loading rate (OLR) throughout the experimental time.

total phenolic compound concentrations in the present research were lower than those found in the study carried out with olive mill solid waste, probably due to the initial high phenolic content in the raw olive mill solid waste(Serrano et al., 2019b). Despite the inhibitory potential of the phenolic compounds (Battista et al., 2015; Borja et al., 1996; Caroca et al., 2021)the concentration determined in these tests were consistently lower than those considered to have an inhibitory effect on the anaerobic digestion process, i.e., 2000 mg·L^{-1} (Calabrò et al., 2018). Therefore, the instability must not be only due to the accumulation phenolic compounds, but probably to several factors in which the concentration of phenolic compounds may has contributed. In this sense, it is known that heat treatments of lignocellulosic material can entail the formation of different compounds such as furans or neutral and acidic oligosaccharides with or without phenolic residues that may have antimicrobial activity (Lama-Muñoz et al., 2012).

3.4. Economic assessment of valorization process

The minimum sales price of the phenolic compounds extract was calculated for a zero net profit. According to this calculation, sales prices higher than 0.812 €·(g of gallic acid equivalents)$^{-1}$ would ensure the economic viability or the proposed process at an OLR of 1.25 g VS·L^{-1}·d^{-1} (Fig. 5). In the study carried out by Cubero-Cardoso et al. (2020a), in which SE was hydrothermally treated at 220 °C for 5 min, the price for phenolic compounds extract for zero net profit was 0.610 €·(g of gallic acid equivalents)$^{-1}$. The lower minimum price for zero net profit in Cubero-Cardoso et al. (2020a) can be explained by the higher phenolic compound recovery reported by these authors, i.e. 0.90 g of gallic acid·(kg SE)$^{-1}$ versus 0.6 g of gallic acid·(kg SE)$^{-1}$ in the present study. Despite this higher phenolic compound obtaining, the use of more severe conditions in Cubero-Cardoso et al. (2020a) during the hydrothermal pretreatment hindered the stability of the subsequent anaerobic digestion process, reporting an stable operation only at an OLR of 0.50 g VS·L^{-1}·d^{-1}. On another hand, the steam consumed in the hydrothermal treatment of SE at 170 °C of SE, 1.03 kg of steam·(kg SE)$^{-1}$, was much less than the values reported in the study of Cubero-Cardoso et al. (2020a), i.e., 6.71 kg of steam·(kg SE)$^{-1}$. This difference allows ensuring that steam consumption in the present research would be fully covered by the energy produced from the methane production yield at an OLR of 1.25 g VS·L^{-1}·d^{-1}. When compared to other similar studies involving agri-food residues, such as thermally pre-treated olive mill solid waste with a value of 0.052 (Serrano et al., 2019b) and 0.042 (Serrano et al.,

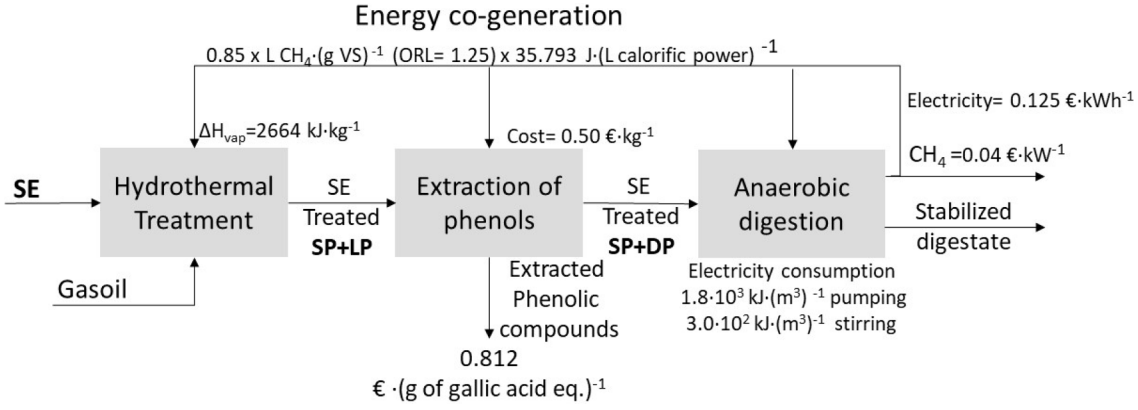

Fig. 5. Block diagram of the valorization from strawberry extrudate (SE) based on Trujillo-Reyes et al., (2019), where SP, solid phase; LP, liquid phase; DP, dephenolised phase; VS, volatile solids; OLR, organic loading rate.

2019a) €·(g of gallic acid equivalents)$^{-1}$, the valorization of SE yielded a significantly higher sales price.

4. Conclusions

This study applies a biorefinery approach to valorise strawberry extrudate (SE) to obtain phenolic compounds and biomethane. A hydrothermal treatment was successfully applied to SE at a high temperature to more readily get a high concentration of phenolic compounds from a liquid fraction. Long-time operation anaerobic digestion from the mixture of the desphenolized liquid and the solid fraction after the pretreatment improved methane production concerning other studies and achieved stability at an ORL of 1.25 g VS·L^{-1}·d^{-1}. The economic analysis demonstrated that the valorization option of SE yielded a comparable sales price for the phenol extract, specifically EUR 0.812 per gram of gallic acid equivalents, indicating a positive economic assessment. The study showed that the proposed biorefinery process for SE might improve the strawberry sector valorising this underused by-product.

Funding

This research was funded by the Spanish Ministry of Economy, Industry, and Competitiveness through Project CTM2017-83870-R. The authors are grateful to the Regional Government of Andalusia, Junta de Andalucía, Consejería de Economía y Conocimiento (Project UHU-1257728), for providing additional financial support.

CRediT authorship contribution statement

Juan Cubero-Cardoso: Conceptualization, Methodology, Formal analysis, Writing – original draft. **Elena Jiménez-Páez:** Conceptualization, Methodology, Writing – original draft. **Ángeles Trujillo-Reyes:** Conceptualization, Methodology. **Antonio Serrano:** Conceptualization, Formal analysis, Writing – review & editing. **Juan Urbano:** Writing – review & editing, Funding acquisition. **Guillermo Rodríguez-Gutiérrez:** Conceptualization, Methodology. **Rafael Borja:** Resources, Funding acquisition. **Fernando G. Fermoso:** Conceptualization, Resources, Writing – review & editing, Supervision, Funding acquisition.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

Data will be made available on request.

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