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Full Length Article

Anaerobic digestion of food waste coupled with biogas upgrading in an outdoors algal-bacterial photobioreactor at pilot scale

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ABSTRACT

This work aimed at integrating the anaerobic digestion of food waste (FW) with photosynthetic biogas upgrading at pilot scale in order to obtain a high quality biomethane and a nutrient-laden algal biomass as the main byproducts from FW treatment. The performance of a 100 L anaerobic digester treating food waste integrated via raw biogas and digestate injection with a 1.2 m^2 outdoors high-rate algal pond (HRAP) was evaluated. Biogas production in the digester averaged 790 ± 89 mL g VS_{in}^{-1} (68 ± 8 L d^{-1}) (35 °C, 1 bar) at a loading rate of 0.86 g VS L^{-1} d^{-1} and a steady state chemical oxygen demand removal efficiency of 83 ± 7%. The biogas produced (60% CH_4 / 39% CO_2) was upgraded in a 2.5 L absorption column interconnected with the HRAP via culture broth recirculation at a liquid to biogas ratio of 2, resulting in a maximum CO_2 removal efficiency of 90% and a maximum CH_4 content of 93.9%. The HRAP, supplied with the centrifuged liquid digestate supplemented with synthetic wastewater (5.0 ± 1.1 L d^{-1}, Total nitrogen (TN) = 793 ± 110 mg N L^{-1}, P-PO_4^{3-} = 39 ± 19 mg P L^{-1}), supported TN and total phosphorus maximum removal efficiencies of 100% in both cases. *Pseudoanabaena* sp. and *Chlorella vulgaris* were identified as the dominant species.

1. Introduction

Food waste (FW) represents nowadays a major contributor to environmental pollution, which causes also severe economic and ethical reputation losses in the European Union (EU). The Food and Agriculture Organization estimated that one third of the food produced annually in the World is lost or wasted [1,2]. In the EU, 138 million tons of FW are generated annually, and this number is expected to continue increasing due to EU population growth [3]. In this context, FW valorization into bioenergy and biofertilizers represents a promising strategy to mitigate both the environmental and economic issues caused by uncontrolled FW disposal.

Anaerobic digestion (AD) for biogas production is one of the most popular technologies used for FW valorisation due to its high potential for energy production, nutrients recovery and greenhouse emissions reduction [3–6]. AD it is a biological process where organic feedstock are converted through the biocatalytic action of multiple anaerobic microorganisms into an energy gas vector mainly composed of CH_4 and CO_2. Throughout this technology, AD converts FW into biogas with a methane yield ranging from 0.46 to 0.53 m^3 CH_4 per kg of volatile solids (VS) of FW [7,8]. It has been reported that the use of FW as fermentation substrate supports high growth rates of acidogens at the acidogenesis stage compared with the methanogens at the methanogenesis stage [9,10], which could lead to the inhibition of methane production process. In this context, the redution of FW loading rate to the digester or co-digestion with other substrates to increase C/N ratio could overcome this drawback. Additionally, conventionals FW treatments are able to remove organic matter, however the nitrogen and phosphorous present in these wastes is not completely eliminated during AD [11,12]. The use of FW as fermentation substrate supports high growth rates of acidogens at the acidogenesis stage compared with the methanogens at the methanogenesis stage [9,10]. Nutrient removal from digestates needs to be carried out via additional post-treatment processes based on bacterial or microalgae [13]. The biomass produced during digestate treatment can be even digested to generate more biogas [14–17]. The biogas typically produced during the AD of FW has a composition of 60–70% CH_4, 30–40% CO_2 and trace levels of other gases such as N_2, O_2 and H_2S

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Nomenclature

List of Acronyms

AC	Absorption column
AD	Anaerobic Digestion
COD	Chemical oxygen demand
DO	Dissolved oxygen
EU	European Union
FW	Food waste
HRAP	High rate algal pond
HRT	Hydraulic retention time
IC	Inorganic carbon
L/G	Liquid to biogas ratio
OLR	Organic loading rate
PAR	Photosynthetic active radiation
SWW	Synthetic wastewater
TN	Total Nitrogen
TOC	Total organic carbon
TP	Total phosphorus
TS	Total solid
VFA	Volatile fatty acids
VS	Volatile solid
VSS	Volatile suspended solids

[3]. This biogas can partially substitute fossil fuels for the generation of heat and power and for transportation. However, biogas upgrading is required in order to obtain biomethane to power industrial or commercial vehicles ($CH_4 \geq 85\%$, $CO_2 \leq 10\%$, $O_2 \leq 2\%$ and minor levels of H_2S) [18] or injected into natural gas grids ($CO_2 \leq 2\%$, $O_2 \leq 1\%$). Similarly, a nutrient rich liquid effluent, namely digestate, is obtained from the AD of FW.

Recently, photosynthetic processes have emerged as an ecofriendly and profitable technology for biogas upgrading able to simultaneously remove CO_2 and H_2S from biogas and capturing nutrients from digestate. In this context, algae-bacteria systems are based on the ability of microalgae to fix CO_2 from biogas and release O_2 using solar radiation energy and of sulfur oxidizing bacteria to aerobically oxidize H_2S into SO_4^{2-} using the available dissolved oxygen in the cultivation broth of the photobioreactor due to photosynthetic activity [19–23]. This technology has been previously optimized and proved outdoors in multiple photobioreactor configurations. In this regard, the potential of a high rate algal pond (HRAP) (shallow raceway ponds where the circulation of the algal broth occurs via a low-power paddle wheel) of 180 L coupled to an absoprtion column (AC) in order to boost the mass transfer of the CO_2 from the biogas to the microalgae aqueous broth for biogas upgrading was validated for the first time in a summer period [24]. Later, taking advantage of the microalgal capacity for nutrient recovery from waste effluents, the performance of such a system to remove organic matter and nutrients from sewage sludge and to upgrade biogas was studied outdoors across the different seasons of the year, considering the high dependence of microalgal activity and growth on temperatures and ligth intensities [25]. At larger scales, the liquid to biogas (L/G) flowrate ratio in the absorption column, the influence of the type of wastewater and the hydraulic retention time (HRT) were evaluated in a 9.6 m^3 HRAP [26]. Apart from HRAPs, horizontal hybrid tubular photobioreactors (11.7 m^3) have been also studied at a demo scale [27] and the effect of alkalinity and the abovemention L/G ratio on biomethane quality were assessed. Most of these research works either used synthetic biogas or digestates, or when using real biogas and digestate, these effluents never originated from the same anaerobic digester.

This work aimed at producing a high quality biomethane and a nutrient-laden algal biomass as the main byproducts from the integration of the anaerobic digestion of FW coupled with photosynthetic biogas upgrading in an outdoors pilot scale HRAP. The effect of air-aided CO_2 stripping from the HRAP, L/G ratio and use of greenhouse on the performance of biogas upgrading and nutrient recovery from centrifuged digestate were assessed during summer, autumn and winter conditions. In previous works, only synthetic biogas was used. In this work, a real biogas was obtained from the anaerobic digestion of food waste. This anaerobic digestor was coupled directly to the HRAP system in order to utilize both the biogas and the digestate produced.

2. Materials and methods

2.1. Food waste and synthetic wastewater

Food waste was obtained from a local restaurant (Santovenia de Pisuerga, Spain). Impurities such as animal bones, fish bones and recalcitrant fruit skins were removed, and the FW was grinded (900 W, 15 min) with a hand blender to achieve a homogeneous composition and frozen at −4 °C. Hydrolysis of FW during grinding and freezing was considered negligible since these wastes were previously cooked but grinding likely improved microbial hydrolysis when fed into the anaerobic digester. Dilution of FW was carried out prior feeding to the digester to maintain a constant VS concentration of 60 g kg^{-1}. FW exhibited a composition of 62 g L^{-1} of chemical oxygen demand (COD), 100.1 g Kg^{-1} of total solids (TS) and 61 g Kg^{-1} of VS.

The composition (per liter of distilled water) of the synthetic wastewater (SWW) used to provide enough nutrients and alkalinity to the HRAP to capture the CO_2 present in the biogas was: 5.0 g $NaHCO_3$, 0.85 g $C_8H_5KO_4$, 0.73 g Casein peptone, 1.7 g NH_4Cl, 0.09 g CH_4N_2O, 0.224 g K_2HPO_4, 0.0175 g NaCl, 0.01 g $CaCl_2$ and 0.005 g $MgSO_4$. These chemicals were added in order to simulate the liquid fraction of a FW digestate. This composition resulted in a concentration of total organic carbon (TOC) of 436 ± 38 mg L^{-1}, of inorganic carbon (IC) of 673 ± 38 mg L^{-1} and of total nitrogen (TN) of 614 ± 41 mg L^{-1}.

2.2. Experimental set-up

The open-air experimental pilot plant was established at the Institute of Sustainable Processes of Valladolid University (Valladolid, Spain). It was composed of an anaerobic digester of 170 L (height = 1800 mm; internal diameter = 350 mm) with a working volume of 100 L coupled to an automated 2.0 L column for biogas flowrate monitoring. The reactor operated under mesophilic conditions (35 °C) at 1 bar of pressure and constant mixing provided by an internal liquid recirculation pump (~30 L h^{-1}) (Bredel SPX15, Watson-Marlow). The anaerobic digester was interconnected to an outdoors HRAP of 180 L with an illuminated surface area of 1.2 m^2 (length = 1700 mm; depth = 150 mm; width = 820 mm) and a rate of cultivation broth recirculation of 200 mm s^{-1}. Additional details about the building characteristics of the HRAP can be found elsewhere [28]. Biogas was sparged at the bottom of a 2.5 L bubble column (internal diameter = 44 mm; height = 1650 mm) through a metallic diffuser of 2 μm pore size (Fig. 1).

2.3. Operating conditions and sampling procedures

Process operation was carried out under continuous mode in one experimental set-up from August the 5th 2020 to December the 30th 2020. Five different phases (namely I, II, III, IV and V) (Table 1) were stablished as a function of the environmental and operational conditions, primarily due to environmental temperature and photosynthetic active radiation (PAR). The anaerobic digester was seeded with 100 L of anaerobic sludge obtained from full-scale anaerobic digesters of sludge in the wastewater treatment plant of Valladolid (Spain). This anaerobic inoculum exhibited a VS/TS ratio of ≈ 60%. FW was fed daily to the anaerobic digester at a rate of 1.4 L d^{-1} (HRT of 60 d) and an organic loading rate (OLR) of 0.86 g VS L^{-1} d^{-1}. The volume of biogas and digestate produced in the anaerobic digester were measured daily. The

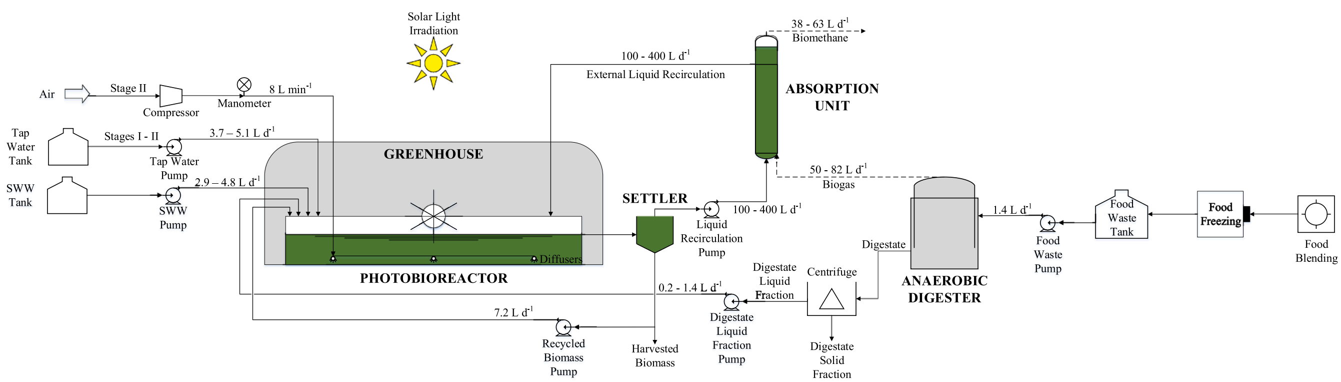

Fig. 1. Schematic diagram of the outdoors experimental algal-bacterial photobioreactor coupled to the anaerobic digester treating food waste.

Table 1
Environmental and operational parameters during the five operational stages in the integrated FW digester-HRAP system.

Stage					
Parameter	**I**	**II**	**III**	**IV**	**V**
Date	05-Aug – 30-Aug	31-Aug – 13-Sep	14-Sep – 30-Sep	01-Oct – 11-Nov	12-Nov – 31-Dec
Use of Greenhouse	No	No	No	Yes	Yes
L/G ratio	2.0	2.0	5.0	2.0	2.0
Air Supply (L min^{-1})	0.0	8.0	0.0	0.0	0.0
Make up water (L d^{-1})	4.6 ± 0.4	4.4 ± 0.1	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0
Morning Average DO (mg L^{-1})	2.4 ± 1.1	8.1 ± 1.5	2.3 ± 1.0	2.7 ± 1.8	6.1 ± 2.3
Afternoon Average DO (mg L^{-1})	2.4 ± 1.2	5.8 ± 1.5	2.3 ± 0.8	2.1 ± 1.5	5.3 ± 1.9
Average Evaporation Rate (L m^{-2} d^{-1})	7.9 ± 1.6	8.0 ± 0.9	5.1 ± 1.8	2.8 ± 1.1	2.9 ± 1.4
Biomass productivity (g m^{-2} d^{-1})	22.5	22.5	22.5	15.0	0.0

digester was insulated, and the walls heated with an electric resistance to maintain a temperature of 35 °C. The main indicators of anaerobic digester performance were the methane yield, the OLR, removal efficiency (RE) of soluble COD and total COD and removal efficiency of VS. The methane yield, the organic loading rate and the REs were calculated according to Eq. (1), Eq. (2) and Eq. (3):

$$MethaneYield = \frac{Q_{biogas} \times C_{CH_4}}{Q_{in}FW \times C_{totalCOD}} \quad (1)$$

$$OLR = \frac{VS_i \times Q}{V} \quad (2)$$

$$RE = \frac{(Q_{in} \times C_{in}) - (Q_{out} \times C_{out})}{(Q_{in} \times C_{in})} \times 100 \quad (3)$$

where VS_i is the volatile solids concentration in FW, V is the volume of the reactor, Q_{in} and Q_{out} represent the inlet and outlet flow rates (L d^{-1}), and C_{in} and C_{out} are the inlet and outlet concentrations of the target parameter, respectively.

The HRAP was inoculated with a microalgal culture previously grown in an outdoors photobioreactor consisting of *Chlorella vulgaris (2%)* and *Pseudoanabaena* sp. *(98%)* (percentages expressed in number of cells) at a concentration of 550 mg volatile suspended solids (VSS) L^{-1}. Liquid digestate from the anaerobic digester (centrifuged for 10 min at 10000 rpm) and SWW were mixed before feeding the HRAP in order to ensure the necessary amount of nutrients and alkalinity that enables the effective capture of CO_2. These streams were supplied at a flowrate of 0.2–3.0 L d^{-1} and 2.9–4.8 L d^{-1}, respectively, with a mix SWW/liquid digestate ratio of 3.1 ± 1.2. The solid phase of digestate after centrifugation was not used. The biogas obtained from the anaerobic digestion of FW was injected into the AC at flow rates of 50–82 L d^{-1} set in co-current flow and operated at L/G ratios of 2.0 (stages I, II, IV and V) and 5.0 (stage III). The compositions of the biomethane obtained in the AC (%CH_4, %CO_2, %O_2, and %N_2) and the removal efficiency of CO_2 (Eq. (3)) where the main indicators of the biogas upgrading performance. A greenhouse covered the HRAP in order to improve the performance of the system for the period of autumn and winter (stages IV and V). Air supply was introduced via porous gas diffusers in 3 different positions into the HRAP at a flowrate of 8.0 L d^{-1} to support a direct CO_2 stripping during stage II. Tap water was added to offset the losses of water by evaporation during stages I and II at a flow rate of 0–5.1 L d^{-1}. Biomass productivity was fixed via wasting of a fraction of settled biomass from the settler in order to support the growth of microalgae in stages I to III (22.5 g m^{-2} d^{-1}), IV (15.0 g m^{-2} d^{-1}) and V (0.0 g m^{-2} d^{-1}) (Table 1). The recirculation rate of biomass from the settler to the HRAP was set at 7.2 L d^{-1}. During stages IV and V, Na_2CO_3 was added directly into the HRAP in order to increase the IC concentration to 1500 and 2000 mg L^{-1}, respectively.

The pH in the cultivation broth of the HRAP and in the digestate of the anaerobic digester were daily measured at 9:00 am. PAR and ambient temperature were measured daily both inside and outside of the greenhouse. Dissolved Oxygen (DO) concentration and temperature in the cultivation broth were measured twice per day at (9 a.m. and 4p.m.).

The concentrations of CH_4, CO_2, N_2 and O_2 in the raw biogas and upgraded biomethane were determined in duplicate by sampling 100 μL of gas stream at 10 a.m. twice per week. In order to determine IC, TOC, TN, N-NH_4^+, N-NO_3^-, N-NO_2^-, P-PO_4^{3-} and S-SO_4^{2-} concentrations, a volume of 100 mL from the SWW, centrifuged digestate and cultivation broth were drawn twice a week. Similarly, 100 mL of FW and digestate were drawn twice a week in order to determine the COD, VS and the volatile fatty acids (VFA) concentrations. Experimental determinations of TOC, IC, TN and COD were performed in duplicate. Once a month, a sample of the cultivation broth was analyzed to determine microalgae population structure.

2.4. Analytical procedures

The pH, PAR, temperature, DO, biogas and biomethane composition were recorded according to Marín et al. [25]. N-NO_3^-, N-NO_2^-, P-PO_4^{3-} and

S-SO_4^{2-} concentrations were quantified by HPLC-IC by high performance liquid chromatography coupled with a detector based on ion conductivity (HPLC-IC) (Waters 432, conductivity detector, USA).

Gas concentrations of CH_4, CO_2, N_2 and O_2 in the raw biogas and upgraded biomethane were determined using a Varian CP-3800 GC-TCD (Palo Alto, USA) according to Posadas et al. [22]. TOC, IC and TN concentrations were analyzed according to Posadas et al. [24]. VFA concentrations were analyzed in an Agilent 7820A GC-FID (Agilent Technologies, Santa Clara, USA) according to López et al. [29]. COD, VSS and VS analyses were carried out according to APHA [30]. N-NH_4^+ concentration was determined with an ammonium specific electrode Orion Dual Star (Thermo Scientific, The Netherlands). Finally, the structure of microalgae population was monitored by morphological characterization according to Marín et al. [20].

2.5. Statistical analysis

The results here presented were provided as the average values along with their standard deviation under steady state conditions. An analysis of variance (ANOVA) was performed to determine the influence of the environmental conditions on the quality of the upgraded biogas.

3. Results and discussion

3.1. Environmental parameters in the HRAP

Significant changes in environmental parameters were recorded as a result of the outdoors operation of the system. The ambient PAR observed in phases I to V ranged from 19 to 1289, 38 to 1160, 17 to 1142, 25 to 981 and 8 to 744 μmol m^{-2} s^{-1}, respectively (Fig. A.1). Similarly, the PAR observed inside the greenhouse during stages IV and V ranged from 12 to 724 and 4 to 358 μmol m^{-2} s^{-1}, respectively (Fig. A.1). In general, the use of the greenhouse entailed a 40% reduction of the ambient PAR impinging into the HRAP. This value was in agreement with the previously reported by Marín et al. [20], who recorded an ambient PAR reduction of 36% inside the greenhouse.

The ambient temperature decreased along the experiment due to the seasonal environmental changes since the experiment started in summer and ended at the beginning of winter. The ambient temperatures recorded in stages I to V in the morning and afternoon ranged from 14 to 37, 11 to 28, 7 to 28, 1 to 24 and −3 to 16 °C, respectively (Fig A.2). The gradual decrease in ambient temperature during autumn required the use of a greenhouse to safeguard microalgal metabolic activity. The temperature inside the greenhouse during stage IV ranged from 4 to 51 °C and during stage V from 1 to 30 °C (Fig A.2). The latter values were in agreement with those recorded by Marín et al. [20] during the equivalent period (-4 to 26 °C). The use of the greenhouse prevented the freezing of the culture broth. Indeed, the temperature in the HRAP culture broth during stages I to V ranged from 13.1 to 34.8, 8.9 to 30.1, 8.0 to 28.1, 6.7 to 28.1 and 2.2 to 20.7 °C, respectively (Fig A.2).

PAR and temperature were the two most important environmental parameters that governed the behavior of other environmental and operating parameters such as the evaporation rate, DO concentration and biomass productivity. The average evaporation rates from the HRAP recorded in stages I to V were 7.9 ± 1.6, 8.0 ± 0.9, 5.1 ± 1.8, 2.8 ± 1.1 and 2.9 ± 1.4 L m^{-2} d^{-1}, respectively (Table 1). The DO concentrations in stages I to V in the morning averaged 2.4 ± 1.1, 8.1 ± 1.5, 2.3 ± 1.0, 2.7 ± 1.8 and 6.1 ± 2.3 mg L^{-1}, respectively, while in the afternoon accounted for 2.4 ± 1.2, 5.8 ± 1.5, 2.3 ± 0.8, 2.1 ± 1.5 and 5.3 ± 1.9 mg L^{-1}, respectively (Table 1). Biomass productivity was maintained constant during stages I to III at a value of 22.5 g m^{-2} d^{-1} and decreased in stage IV to 15.0 g m^{-2} d^{-1}. The low temperatures prevailing in the culture broth during stage V supported process operation without biomass wastage (0.0 g m^{-2} d^{-1}) (Table 1). It is important to highlight that even when working without biomass production during stage V, the DO concentration was higher as a consequence of the lower temperature, since lower temperatures increase gas solubility in liquids and decrease the activity of oxygen demanding microorganisms and processes (heterotrophs, nitrifiers, algal endogeneous respiration). The values here recorded for biomass productivity were in agreement with Marín et al. [20,25] and Posadas et al. [24], who registered a maximum productivity of biomass of 22.5 g m^{-2} d^{-1} in a similar outdoors HRAP during summer.

3.2. Anaerobic digestion of food waste

The production and yield of biogas in the pilot anaerobic digester treating FW fluctuated in the range 50–82 L d^{-1} (Fig. 2a) and 581–954 mL g VS_{in}^{-1}, respectively, with an average methane production yield of 790 ± 89 mL CH_4 g VS_{in}^{-1}. The composition of the biogas remained constant at 60.0 ± 1.2% CH_4, 38.7 ± 1.3% CO_2, 0.3 ± 0.1% O_2, 1.0 ± 0.4% N_2. Interestingly, the presence of H_2S in the biogas was not detected. This fact could be explained by the low amount of sulfur present in the FW (mostly composed by potatoes, lettuce and tomato). Similar results were reported by Yue et al., [31], who reached a methane yield of 450 mL CH_4 gVS^{-1} with non-pre-treated FW in a batch anaerobic reactor and 587 ± 11 mL CH_4 gVS^{-1} when using FW pre-treated with ultrasound. Similarly, Wang et al., [32] reported a methane yield of 490 ± 40 mL CH_4 gVS^{-1} during single-phase co-digestion of fruit and vegetables waste and kitchen waste at an OLR of 1 g VS L^{-1} d^{-1}. Babaee and Shayegan [33] also recorded a biogas yield of 400 mL gVS^{-1} (64% CH_4) at a OLR of 1.4 g VS L^{-1} d^{-1} in a semi continuous anaerobic digester fed with vegetable wastes.

The total COD of the FW ranged from 58 to 73 g O_2 L^{-1} and the soluble COD from 13 to 23 g O_2 L^{-1} (Fig. 2b). These variations were due to the different composition of the food remains from the local restaurant that supplied the FW here used. The composition of FW typically changes depending on geographical and seasonal conditions [34,35]. Despite these variations, the total and soluble COD in the digestate remained relatively constant, exhibiting a composition of 11.1 ± 5.8 and 1.3 ± 1.4 g O_2 L^{-1}, respectively. This resulted in total and soluble COD removals of 82 ± 7% and 92 ± 6%, respectively. In contrast, the VS concentrations of the anaerobic broth in the digester gradually decreased from 9.2 to 4.7 g L^{-1} during the first 70 days of experiment and stabilized at 5.9 ± 0.5 g L^{-1} from day 100 onwards (Fig. 2c). It is important to highlight that the decrease in VS concentration was a consequence of the stabilization of the anaerobic digester with the substrate used, because at the beginning it was inoculated with anaerobic digestion sludge obtained from the full-scale anaerobic digesters of sludge at the wastewater treatment plant of Valladolid (Spain). The digester exhibited average VS removals of 88 ± 3%, which were in accordance to those observed by Babaee and Shayegan [33]. These authors reported a VS removal of 88% at an OLR of 1.4 g VS L^{-1} d^{-1} and an HRT of 25 days and observed a decrease in VS removal efficiencies when increasing the OLR. The results here obtained showed that the organic matter contained in the FW was readily available for the anaerobic microbial consortium. FW feedstock typically exhibits a high biodegradability and a low pH and C/N ratio (C/N of 19 and 12 in fruit-vegetable waste and kitchen waste, respectively) [36], which requires digester operation at long HRTs or low-moderate OLR to avoid process failures [34].

In this context, a rapid biodegradation of the organic matter present in the FW may lead VFAs accumulation during the acidogenesis and acetogenesis phases, resulting in the inhibition of methane production. Acetic acid was the most abundant VFA in the digestate followed by butyric acid (maximum concentration of 96 mg L^{-1}), and to a lesser extent by propionic acid. The concentration of acetic acid gradually decreased from 1052 mg L^{-1} at the start of the experiment to 259 mg L^{-1} by day 60 and increased again up to 555 mg L^{-1} from day 110 onwards but supported a stable operation of the digester. Likewise, Nagao et al., [37] reported a low VFA concentration in a digester treating FW at 8 days of HRT and an OLR of 3.7 g VS L^{-1} d^{-1}, which increased up to 8149

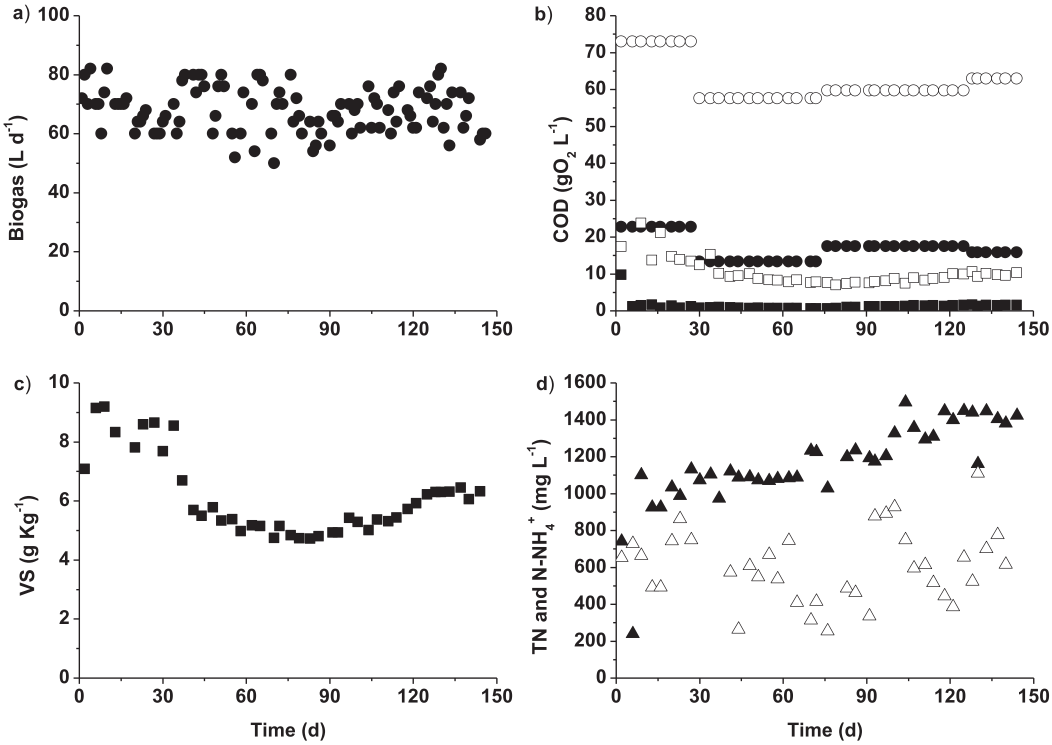

Fig. 2. Time course of (a) biogas production, (b) total chemical oxygen demand (empty symbols) and soluble chemical oxygen demand (solid symbols) in the food waste (circles) and digestate (squares), (c) volatile solid concentration in the digestate and (d) total nitrogen (▲) and N-NH_4^+ (Δ) in the digestate of the 100 L anaerobic digester.

mg L^{-1} when the OLR was increased to 5.5 g VS L^{-1} d^{-1} with a subsequent reduction in biogas production and process failure. Finally, the pH remained stable at 8.2 ± 0.2.

FW is typically characterized by a low C/N ratio, where the high organic nitrogen content may result, via ammonification, in ammonium and free ammonia accumulation in the reactor. These high ammonia concentrations can inhibit the AD process since free NH_3 can diffuse throughout cell membrane and hinder cell functioning. The range of critical concentrations leading to process failure for NH_4^+ and NH_3 is typically set at 1500–1700 mg L^{-1} and 150 mg L^{-1}, respectively [38,39].

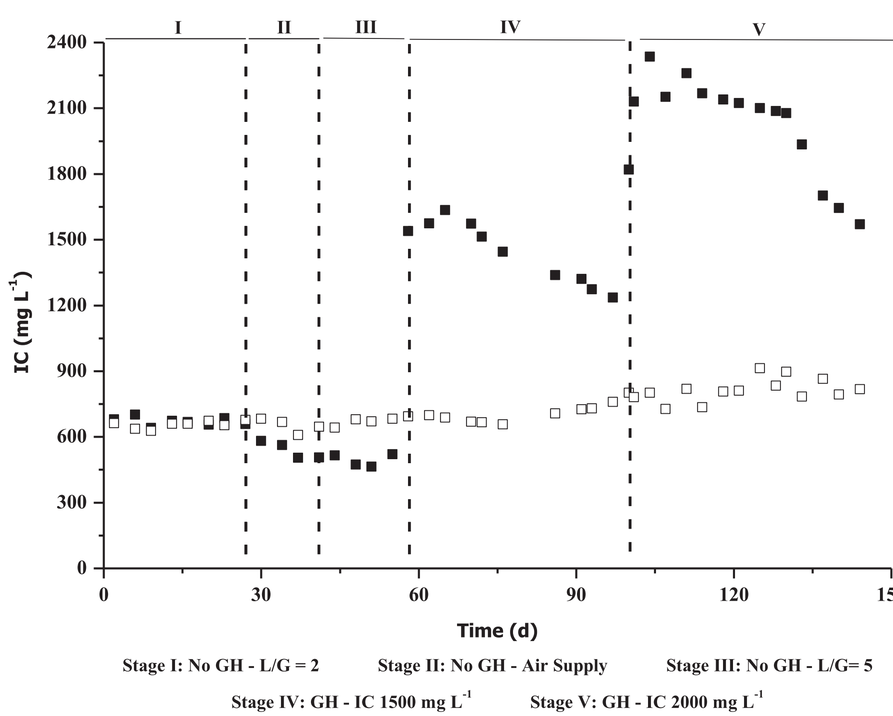

Fig. 3. Time course of the inorganic carbon concentration in the influent (□) and in the cultivation broth of the HRAP (■).

Yenigün and Demirel [40] reported AD collapse at a total ammonia nitrogen (free ammonia + ammonium) concentration of 1.7–1.8 g L^{-1}. In this study, the dilution of FW prior feeding the digester reduced the relevance of the potential inhibition caused by a low C/N ratio and ammonification. Indeed, ammonium and ammonia concentrations accounted for 778 ± 250 and 120 ± 73 mg L^{-1}, respectively. The dissociation of ammonium into ammonia is governed by the pH and temperature, which accounted for 778 ± 250 and 120 ± 73 mg L^{-1}, respectively, in the pilot digester of this study. These values were lower than those typically reported in literature for anaerobically digested FW effluents [41,42]. For instance, Cheng et al. [43] observed a concentration of 2200 mg TN L^{-1} and 2120 mg N-NH_3 L^{-1} in an anaerobic fermenter of food waste operated at a HRT of 25 d, while Nwoba et al. [44] reported an anaerobically FW digestate containing 5226 ± 116 mg N-NH_3 L^{-1}.

3.3. HRAP performance

The concentration of IC found in the HRAP influent (digestate + SWW) stayed constant at 725 ± 78 mg L^{-1} throughout the entire experimental period (Fig. 3). Similarly, the concentration found in the microalgae broth of the HRAP was unaltered during stage I at 672 ± 20 mg L^{-1} and a pH of 8.6 ± 0.1 (Fig. A.3). A decrease in the IC concentration from 658 to 521 mg L^{-1} was registered during stage II and III probably due to CO_2 stripping and algal uptake. This reduction in IC concentration occurred concomitantly with a decrease in pH value during stage II and III from 8.9 to 8.4. At the beginning of stage IV, Na_2CO_3 was added directly into the HRAP in order to increase the IC concentration to 1500 mg L^{-1} and pH up to 9.6, and ultimately support a more effective CO_2 capture in the biogas absorption column. A gradual decrease was recorded in the IC concentration to 1236 mg L^{-1} and pH value to 8.7 at the end of stage IV. It is worth to mention that many microalgal strains have the ability to utilize bicarbonate and carbonate species, the prevailing forms of IC at the pH observed in the cultivation broth of the HRAP, as a carbon source [45]. In stage V, an additional injection of Na_2CO_3 was added to the HRAP in order to further increase the IC concentration to 2100 mg L^{-1} and pH up to 9.5. During this stage a high decrease in the IC to 1570 mg L^{-1} and in the pH value to 8.5 were recorded along with a deterioration in algal growth despite the absence of biomass wastage, which suggested that stripping was the main IC removal mechanisms (Fig. 3).

TN concentration in the influent increased at the beginning of stage I till the end of stage V from 592 to 950 mg L^{-1} as a consequence of the rise of TN concentration in the digestate (Fig. 4a). However, the TN concentration in the culture broth remained constant throughout the total experimental period and averaged 689 ± 51 mg L^{-1} (Fig. 4a). TN biomass recovery was calculated according to Eq. (4):

$$Recovery = \frac{Areal\ Biomass\ Productivity \times HRAP \times i_{content}}{Q_{in} \times C_{in}} \times 100 \quad (4)$$

where Q_{in} represent the inlet digestate flow rates (L d^{-1}), C_{in} the inlet concentrations of the target nutrient (N or P), and $i_{content}$ is the content of the target nutrient in the biomass. According with this equation the TN biomass recovery accounted for 60.3 ± 4.7, 52.2 ± 4.1, 49.7 ± 8.6, 51.1 ± 16.0 and 0% in stages I, II, III, IV and V, respectively. Considering

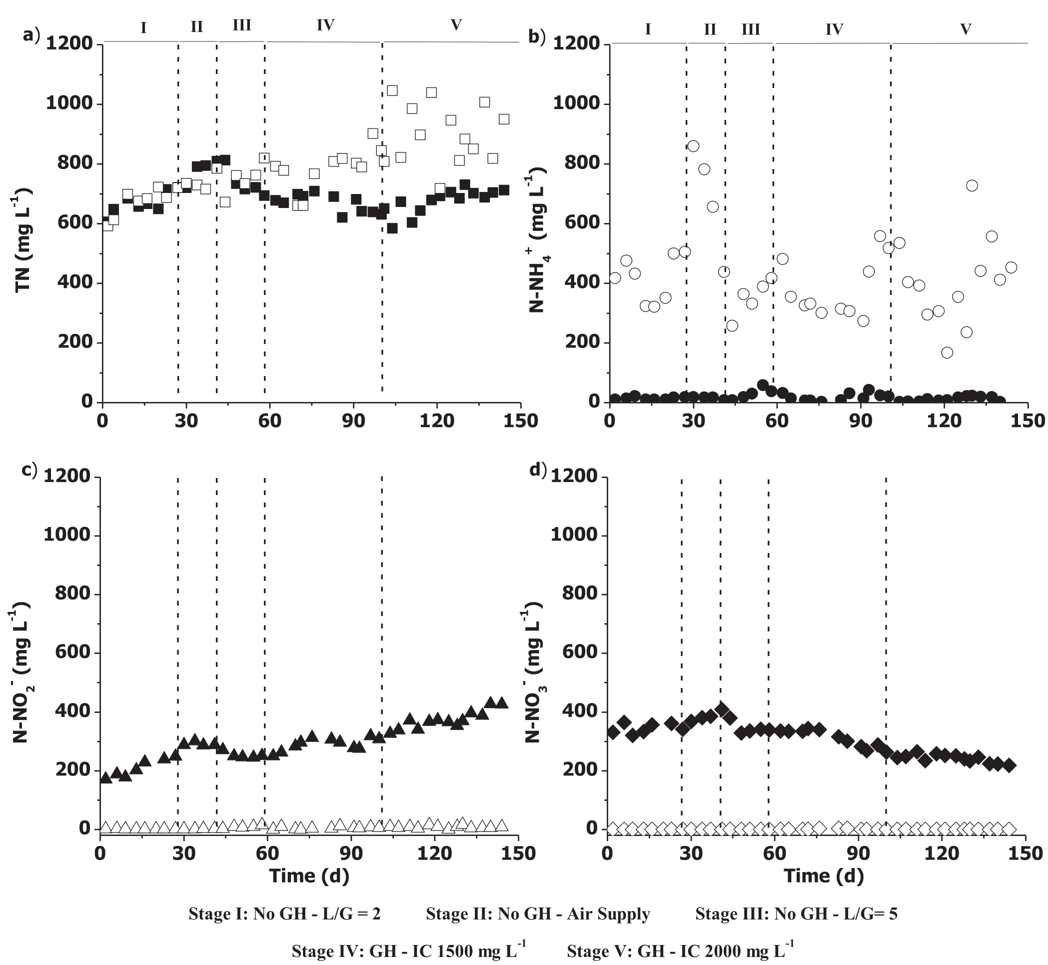

Fig. 4. Time course of the concentration of (a) total nitrogen, (b) N-NH_4^+ (c) N-NO_2^- and (d) N-NO_3^- in the influent (empty symbols) and cultivation broth of the HRAP (solid symbols).

the absence of liquid effluent in the HRAP, extraction of biomass from the settler and ammonia stripping were the only nitrogen outlets from the system. Since TN concentration remained stable throughout the experimental period, a 100% of TN-REs was considered in view of the fact that no accumulation or depletion was produced. N-NH_4^+ concentrations observed in the influent presented significant fluctuations all over the experimental time, with average values of 423 ± 143 mg L^{-1} (Fig. 4b). However, the N-NH_4^+ concentration in the HRAP was almost negligible (average values of 16 ± 12 mg L^{-1}) (Fig. 4b). This low value of N-NH_4^+ concentration in the HRAP compared with the influent was likely due to the active assimilation of NH_4^+ as a nitrogen source by microalgae [45], to the bacterial oxidation of N-NH_4^+ into N-NO_2^-, and to stripping at the high pH prevailing in the culture both. Free ammonia concentrations of 229 mg L^{-1} were present in the liquid fraction of the digestate at 25 °C and pH of 8.9 [46]. Under these circumstances, the concentration of N-NO_2^- in the HRAP gradually rose from 171 mg L^{-1} at the beginning of the experimental time, up to 427 mg L^{-1} at the end of phase V (Fig. 4c). Interestingly, the high pH of the cultivation broth along with the high biomass concentration likely mitigated the inhibitory effect of the free nitrous acid [47]. In contrast, concentration of N-NO_3^- in the HRAP reduced from 331 mg L^{-1} at the start of stage I, down to 218 mg L^{-1} at the end of experiment (Fig. 4d), thus suggesting that the activity of N-NO_2^- oxidizers was partially inhibited. Similar findings were reported by Marin et al., [25], who attributed the inhibition of the NO_2^- oxidizers to the increased salinity in the culture broth.

The observed P-PO_4^{3-} concentration in the influent of the HRAP was constant in phases I to III at 22 ± 6 mg L^{-1}. However, this concentration increased during stage IV from 31 to 76 mg L^{-1}, followed by a gradual decrease from 78 to 43 mg L^{-1} in stage V. The dynamics of P-PO_4^{3-} concentration during phases IV and V were a consequence of variations in P-PO_4^{3-} concentration in the digestate. However, a gradual decrease on P-PO_4^{3-} concentration in the cultivation broth of the HRAP was observed, from 141 to 94 mg L^{-1} at the beginning of stage I till the end of stage IV and kept constant during stage V at 89 ± 12 mg L^{-1}. A preliminary mass balance estimation revealed that this decrease in P-PO_4^{3-} levels in the HRAP in phases I to IV was the result of the recovery of P in the

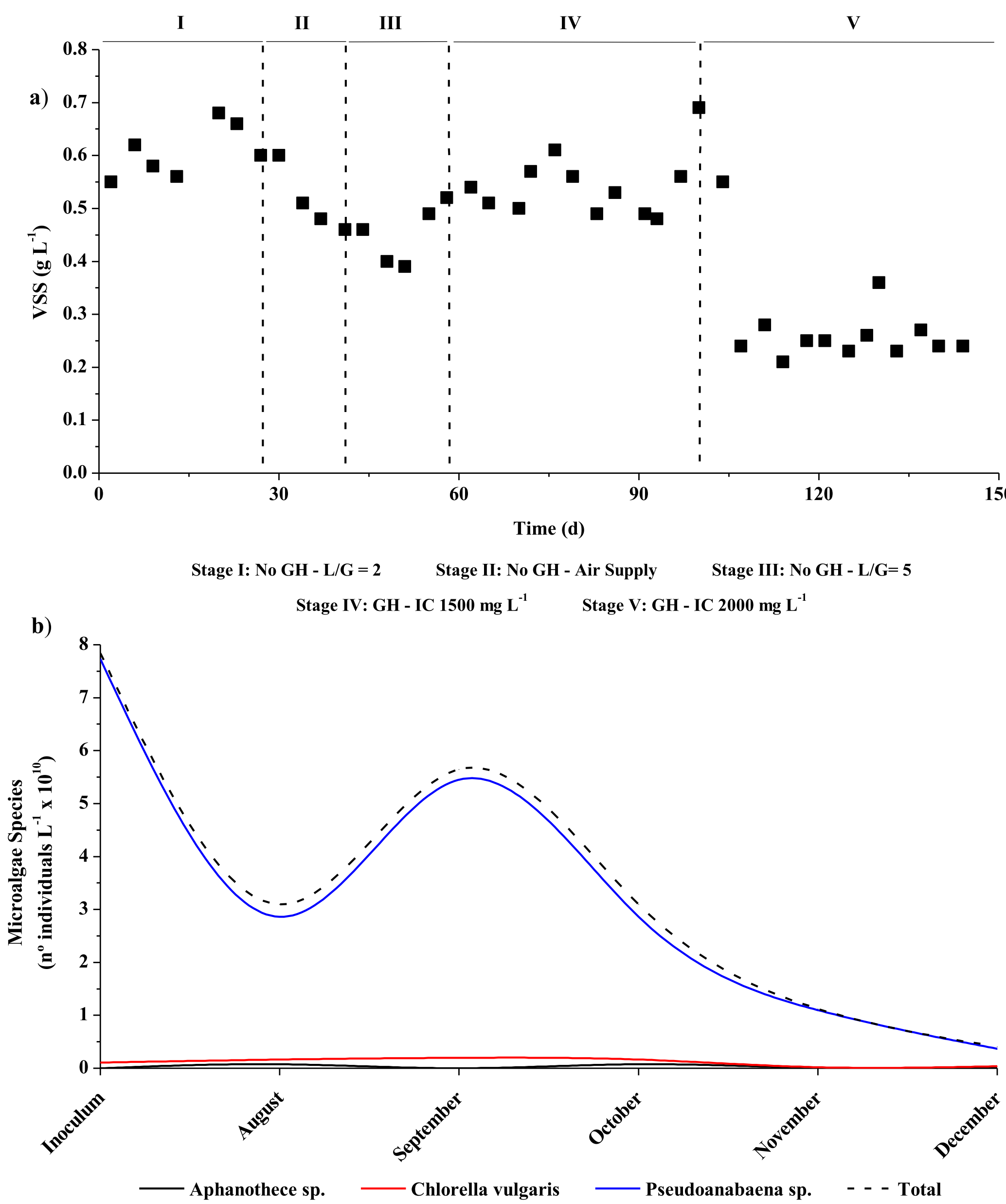

Fig. 5. Time course of the (a) concentration of volatile suspended solids in the HRAP and (b) structure of microalgae population in the HRAP.

harvested biomass (according to Eq. (4)), which accounted for 100 ± 0% in stages I, II and III, 91.7 ± 13 and 0% in stages IV and V, respectively, based on an experimental P algal-bacterial biomass content of 1.3 ± 0.1% and the determination of soluble PO_4^{3-} concentration by HPLC-IC.

The VSS concentration in the HRAP fluctuated during stage I between 0.55 and 0.68 g L^{-1} (Fig. 5a). In stage II, a gradual decrease in VSS concentration was recorded from 0.60 to 0.48 g L^{-1} by the end of stage II. During stage III, biomass concentration fluctuated between 0.39 g L^{-1} and 0.49 g L^{-1} and remained constant at 0.53 ± 0.04 g L^{-1} during stage IV as a result of the reduction in biomass productivity. Finally, a significant decrease in VSS concentration to 0.31 ± 0.14 g L^{-1} in the absence of biomass withdrawal was recorded in stage V likely due to the harsh environmental conditions (high pH, high alkalinity, low temperatures and irradiances) (Fig. 5a) [48]. At this point it should be also highlighted that the use of the greenhouse successfully prevented the freezing of the culture broth during winter conditions.

Pseudoanabaena sp. was the dominant algal specie in the algal-bacterial consortium throughout the experiment (Fig. 5b). A reduction in the concentration of *Pseudoanabaena* sp. from 7.7×10^{10} to 2.9×10^{10} n° of individual L^{-1} was recorded during August. An increase in the concentration of *Pseudoanabaena* sp. up to 5.4×10^{10} n° of individual L^{-1} was observed along September. Finally, a progressive decrease in the concentration *Pseudoanabaena* sp. occurred from the end of September onwards, with a concentration 0.4×10^{10} n° of individual L^{-1} by the end of the experiment (Fig. 5b). These variations may be related to the optimal temperature of this cyanobacterium (20–30 °C) [49]. *Chlorella vulgaris* and *Aphanothece* sp. were also present in the algal-bacterial consortium, but their occurrence was negligible compared to *Pseudoanabaena* sp., reaching maximum concentrations of 0.2×10^{10} and 0.1×10^{10} n° of individual L^{-1}, respectively (Fig. 5b).

Average values of carbon and nitrogen contents of 42.4 ± 1.4% and 8.9 ± 0.4% were recorded in the harvested biomass regardless of the months and phases of operation. This carbon and nitrogen content remained within the typical range of values reported in previous works. For example, Bi and He [50], Toledo-Cervantes et al [23], Posadas et al [24], Marín et al [28] and Marín et al [20] reported values of carbon and nitrogen content of 58.0 and 6.8%, 46.5 and 7.2%, 41.1 and 6.7%, 43.1 and 8.0% and 43.2 and 5.9%, respectively.

3.4. Biogas upgrading performance

CO_2 concentration in the raw biogas remained relatively constant throughout the experimental time and averaged a percentage of 38.7 ± 1.3 (Fig. 6a). An increase in biomethane CO_2 concentration from 13.5 to

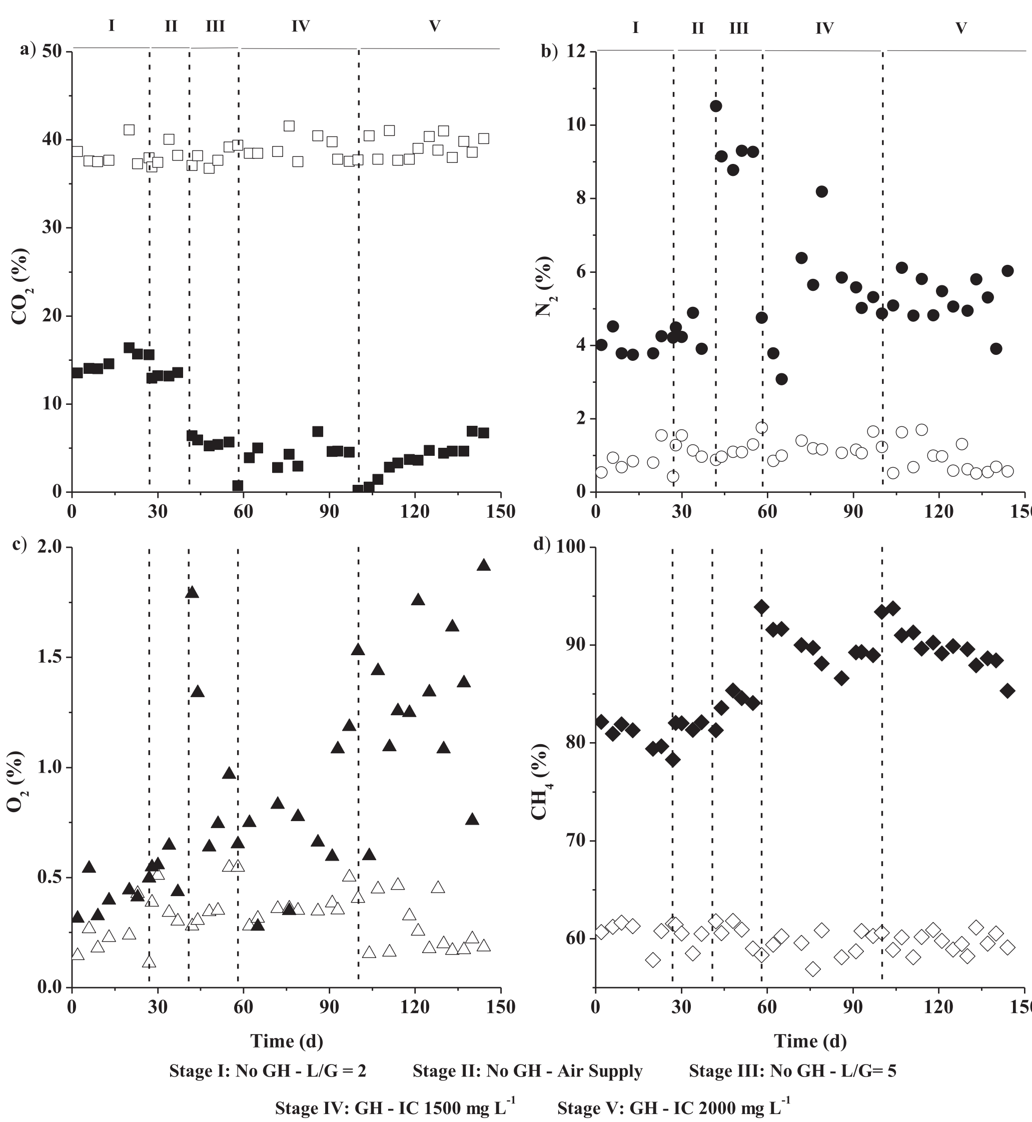

Fig. 6. Time course of the concentration of (a) CO_2, (b) N_2, (c) O_2 and (d) CH_4 in the raw biogas (empty symbols) and biomethane (solid symbols).

16.4 % was observed during stage I (Fig. 6a). In stage II, the concentration of CO_2 decreased to 13.7 ± 1.1% and remained constant during all the stage aided by CO_2 stripping from the HRAP induced by air injection. During stage III, the increase applied in L/G ratio from 2.0 to 5.0 entailed a reduction in the CO_2 concentration of the biomethane to 5.7 ± 0.5%, which remained constant during this stage. The increase in pH and IC concentration in the algal broth up to 1500 mg L^{-1} at stage IV entailed a severe reduction in the CO_2 concentration to 0.7%. However, a progressive increase in CO_2 concentration up to 4.5% was recorded by the end of stage IV. Similarly, CO_2 concentration decreased to 0.2% as result of the increase in IC concentration of the HRAP at the start of stage V. Nevertheless, the gradual reduction in IC and pH in the culture broth mediated the increase in biomethane CO_2 concentration up to 6.9% at the end of the final stage (Fig. 6a). Biomethane CO_2 concentration in stages IV and V was directly correlated with the buffer capacity and pH found in the culture broth, which governed CO_2 absorption in AC. Despite the biomethane CO_2 concentrations achieved in this work were higher than those reported in previous outdoors studies conducted by Marín et al., [20] (0.3–7.9%) using a biogas composed of 70% CH_4 and 29.5% CO_2, the CO_2 removal efficiencies here achieved in stages III to V (85.2, 90.0 and 90.5%, respectively) were high due to the high concentrations of CO_2 in the raw biogas. The CO_2 concentrations achieved during stages III to V fulfilled with current biomethane standard for use as vehicle fuel ($CO_2 \leq 10\%$) [18].

N_2 concentration in the raw biogas remained constant throughout the experimental time at an average value of 1.0 ± 0.4% (Fig. 6b). Similarly, N_2 concentration in biomethane during stages I and II averaged 4.2 ± 0.4%. However, the increase in the L/G ratio in stage III mediated an increase in N_2 concentration in biomethane up to 9.4 ± 0.7% as a result of the enhanced dissolved N_2 stripping at higher liquid flowrates [51]. Finally, the resumption of the L/G ratio to 2 in combination with the increase in IC concentration of the culture broth and pH through stages IV and V entailed a decrease in N_2 concentration in the biomethane to average values of 5.3 ± 1.0% (Fig. 6b).

In stages I and II, the O_2 concentration recorded in biomethane remained constant at 0.5 ± 0.1% (Fig. 6c). In stage III, an increase in the O_2 concentration of biomethane up to 1.1 ± 0.5% was measured mediated by the increase in the L/G ratio. Finally, despite the resumption in the L/G ratio, high O_2 concentrations ranging from 0.7% at the beginning of stage IV to 1.9% at the end of stage V were recorded as a result of the higher dissolved O_2 concentration at the low temperatures prevailing in the cultivation broth. These low temperatures mediated a higher O_2 solubility and a reduced microbial O_2 respiration, the latter also marginal due to the low biomass concentrations in stage V (Fig. 6c).

Finally, the CH_4 concentration recorded in the raw biogas remained constant at 60.0 ± 1.2% (Fig. 6d). The CH_4 concentration observed in the biomethane throughout stages I and II was 80.9 ± 1.1 and 81.2 ± 1.6%, respectively. During stage III, a slight increase of CH_4 concentration in biomethane up to 83.8 ± 1.5% was observed. The raise in IC concentration to 1500 mg L^{-1} and pH up to 9.6 in stage IV entailed an initial increase in the CH_4 concentration of biomethane up to a value of 93.9%. However, a gradual decrease until the end of the stage was recorded (89.0%). Similarly, CH_4 concentration increased to 93.4% as a result of the increase in IC concentration and pH of the HRAP in stage V, and progressively decreased during stage V to 85.3% (Fig. 6d). In spite of the fact that the biogas achieved from the anaerobic digestion of FW exhibited lower CH_4 content compared with the raw synthetic biogas typically used in previous works, a high CH_4 content (>85%) was achieved in the biomethane [23,24,52,53]. Indeed, the CH_4 concentrations achieved during stages III to V fulfilled with current European regulations on the use of biogas as vehicle fuel ($CH_4 \geq 85\%$) [18].

3.5. Energy study

The energy demand E (kW-h) required per cubic meter of biogas treated was calculated according to Mendoza et al. [54] and Toledo-Cervantes et al [55]. Power consumption for biogas sparging in the AC was calculated according to Eq. (5), the power required for the liquid recirculation between the settler and the AC was calculated according to Eq. (6), the power requirements for pumping the FW to the anaerobic digester, the liquid digestate to the HRAP, the SWW to the HRAP and part of the settled biomass from the settler to the HRAP were calculated according to Eq. (7), the power requirement to circulate liquid in the HRAP was calculated according to Eq. (8) and the energy needed to heat the FW was calculated according to Eq. (9).

$$E_{gas} = \frac{Q_{gas} \times \Delta P}{0.7} \tag{5}$$

$$E_{liq-rec} = \frac{Q_{liq-rec} \times \rho \times g \times H}{0.7} \tag{6}$$

$$E_{liq} = Q_{liq} \times \rho \times g \times H_f \tag{7}$$

$$E_{HRAP} = \frac{Q \times \rho \times g \times n^2 \times v^2 \times L}{R^{4/3}} \tag{8}$$

$$E_{HeatFW} = \dot{m} \times C_P \times \Delta T \times 0.80 \times 0.95 \tag{9}$$

where Q_{gas} is the flowrate of biogas, ΔP is the pressure drop in the biogas absorption column, $Q_{liq\text{-}rec}$ is the flowrate of liquid from settler to AC, H is water column height, Q_{liq} stands for the flowrate of FW, liquid digestate, SWW or settled biomass, H_f is the pressure drop calculated according to the Darcy–Weisbach equation, Q is the volumetric flow rate of the HRAP, ρ is the water density, g is the Earth gravity constant, n is the Manning friction factor, v is the internal liquid velocity in HRAP, L is the total length of the HRAP, R is the hydraulic radius, $\dot{m}$ is the mass flow rate, C_p is the specific heat and ΔT is the temperature rise required to heat the FW.

In this context, the energy demand in the system represented 0.41 kW-h m^{-3} of biogas treated. It is important to highlight that in a real scale system the injection of the FW in the anaerobic digester is done in solid form and not in diluted form as in laboratory scale. With this consideration, the energy demand in the system would be reduced to 0.21 kW-h m^{-3} of biogas treated.

4. Conclusions

The present proof-of-concept study in an outdoors pilot plant showing an integral FW anaerobic digestion system coupled to photosynthetic biogas upgrading and nutrient recovery from digestate supplemented with synthetic wastewater as microalgae biomass. The alkalinity and pH in the absorption column were the key parameters governing biomethane quality. An L/G increase from 2 to 5 entailed a higher CO_2 absorption and a slight increase of CH_4 concentration, while only IC supplementation supported a biomethane composition according to international standards (94% CH_4 and 0.2–0.7% CO_2). The high quality of the biomethane generated suggests that this technology can be implemented at full scale in municipal FW treatment plants.

CRediT authorship contribution statement

David Marín: Conceptualization, Investigation, Formal analysis, Writing – original draft. **Lara Méndez:** Investigation, Formal analysis, Writing – original draft. **Irene Suero:** Investigation. **Israel Díaz:** Conceptualization, Resources, Writing – review & editing. **Saúl Blanco:** Formal analysis. **María Fdz-Polanco:** Resources. **Raúl Muñoz:** Conceptualization, 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.

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