Maximising resource recovery from wastewater grown microalgae and primary sludge in an anaerobic membrane co-digestion pilot plant coupled to a composting process.

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Abstract

A pilot-scale microalgae (*Chlorella* spp.) and primary sludge anaerobic co-digestion (ACoD) plant was run for one year in an anaerobic membrane bioreactor (AnMBR) at 35 ºC, 70 d solids retention time and 30 d hydraulic retention time, showing high stability in terms of pH and VFA concentration. The plant achieved a high degree of microalgae and primary sludge substrate degradation, resulting in a methane yield of 370 $mLCH_4 \cdot gVS_{inf}^{-1}$. Nutrient-rich effluent streams (685 $mgN \cdot L^{-1}$ and 145 $mgP \cdot L^{-1}$ in digestate and 395 $mgNH_4\text{-}N \cdot L^{-1}$ and 37 $mgPO_4\text{-}P \cdot L^{-1}$ in permeate) were obtained, allowing posterior nutrient recovery. Ammonium was recovered from the permeate as ammonia sulphate through a hydrophobic polypropylene hollow fibre membrane contactor, achieving 99% nitrogen recovery efficiency. However, phosphorus recovery through processes such as struvite precipitation was not applied since only 26% of the phosphate was available in the effluent. Composting process of the digestate coming from the ACoD pilot plant was assessed on laboratory-scale Dewar reactors, as was the conventional sludge compost from an industrial WWTP digestion process, obtaining similar values from both. Sanitised (free of *Escherichia coli* and

Salmonella spp.) and stable compost (respirometric index at 37 ºC below 0.5 $mgO_2 \cdot g$ organic $matter^{-1} \cdot h^{-1}$) was obtained from both sludges.

Keywords

composting; anaerobic co-digestion; microalgae; resource recovery; nutrients; methane

1 INTRODUCTION

Classical Wastewater Treatment Facilities (WWTF) in which waste is derived from the purification process are now being replaced by new Water Resource Recovery Facilities (WRRF) in which waste is re-used to generate products of agronomic and commercial interest instead of being simply managed. Research in the 21st century is focused on wastewater management through anaerobic digestion (AD) processes, which is a promising approach combined with membrane separation and is generating increasing interest in the scientific community. Indeed, anaerobic membrane bioreactors (AnMBRs) are being applied to wastewater treatment for their several advantages, which allow higher resource recovery from wastewater at a lower cost than conventional biological aerobic systems (Becker et al., 2017; Dereli et al., 2012; Giménez et al., 2011; Robles et al., 2018).

Autotrophic microalgae-based technology is also being used for nutrient removal from the waterline (Acién et al., 2016; González-Camejo et al., 2020; Khalid et al., 2019). Microalgae are able to hold back a higher nutrient concentration than conventional treatments and generate better clarified effluent and sludge with a higher concentration of ammonium and phosphate (Acién et al., 2016; González-Camejo et al., 2020). Wastewater-grown microalgae biomass can be harvested and used as a substrate for AD. Indeed, microalgae biomass AD generated in a membrane photobioreactor pilot plant (MPBR) has been reported to be efficient in terms of methane production (Greses et al.,

2017) on a laboratory scale. However, this efficiency could be improved by digesting the microalgae biomass with primary sludge as a co-substrate (Serna-García et al., 2020a; Solé-Bundó et al., 2019). These authors obtained better biodegradability percentage when digesting microalgae and primary sludge on a lab-scale comparing to microalgae AD as unique substrate. However, this process needs to be evaluated at pilot-scale as a first step for future industrial application.

In this sustainable scheme for wastewater treatment, AD also has a nutrient recovery potential. Traditional wastewater treatment plants (WWTPs) used to remove nitrogen (N) from the effluent through a biological nitrification/denitrification step and phosphorus (P) through enhanced biological P removal or chemical precipitation. However, there are other techniques that make nutrient recovery from AnMBR effluents a feasible option for a circular economy-based scenario. Although struvite precipitation is a useful alternative for recovering both P and N, at least 50 ppm of phosphate (PO_4-P) are needed to make it profitable (Cornel et al., 2009). Modifications have thus been proposed in the WWTP layout to increase this PO_4-P concentration in AD (Martí et al., 2008). Although high P recovery efficiencies (80-90%) can be obtained through struvite precipitation, N recovery is not highly efficient (20-30%) and other technologies can be used such as bioelectrochemical systems, electrodialysis or hollow-fibre membrane contactors (HFMC). HFMC appears to be an interesting treatment because of its low volume and energy requirements. In these systems, free ammonia nitrogen (FAN) passes through a microporous hydrophobic membrane and a sulphuric acid solution is used as the draw solution to recover N as valuable ammonia sulphate.

As not only nutrient-rich permeate, but also nutrient-rich digestate is obtained from AnMBR processes (Nag et al., 2019, Nkoa, 2013; Seco et al., 2018), the digestate has potential agricultural applications since it could be used as a fertiliser. However, direct land application of digestate presents some drawbacks: i) large agricultural areas are needed to directly apply the large amount of digestate generated in AD plants, involving high transport costs (Fuchs and Drosg, 2013); ii) in some cases, especially when digesting substrates with high slowly biodegradable volatile solids (VS) content, such as microalgae, the digestate still contains undigested VS and needs further stabilization; (iii) the possible presence of pathogens or heavy metals (Monlau et al., 2015). These drawbacks lead to the necessity of a forward stabilisation process to produce stable organic soil improver.

Composting has been shown to be an effective process for treating different organic wastes including anaerobic digestate, municipal solid wastes and manure wastes, among others. However, the composting process of an anaerobic digestate from a microalgae and primary sludge co-digestion plant has not yet been evaluated, to the best of the authors' knowledge. In a composting process, the biological decomposition of organic waste takes place under controlled aerobic conditions, involving mesophilic and thermophilic microorganisms. Organic substrates are transformed into a stabilised material free of pathogens and ready to be used in agriculture. This process depends not only on environmental factors such as pH, aeration, moisture content or temperature, but also on sludge characteristics such as nutrient content, particle size or carbon to nitrogen (C/N) ratio (Nikaeen et al., 2015). The C/N ratio is one of the most important parameters of the composting process (Gao et al., 2010; Puyuelo et al., 2011) since is used as an initial requirement to provide the optimum conditions for development of microorganisms and, as a monitoring parameter. Due to the low C/N ratio of anaerobic digestate, especially when treating substrates with high N content such as microalgae or sludge (Solé-Bundó et al., 2019; Ullah et al., 2015), a bulking agent (BA) can be added to generate a mixture with an appropriate C/N ratio (20-25) (Huang et al., 2004).

There are a wide variety of microorganisms in a composting system, the most abundant being fungi, actinomycetes and bacteria (Silva and Naik, 2007). According to the Spanish Regulation RD 506/2013 (Annex IV), compost is required to contain less than 1000 most probable number (MPN) of *Escherichia coli* (*E. coli)* per gram of final product and *Salmonella* spp. has to be absent in 25 grams.

In this work the long-term anaerobic co-digestion (ACoD) of raw microalgae biomass and primary sludge was evaluated at pilot-scale. Potential nutrient recovery (P and N) from the ACoD permeate through struvite crystallization and HFMC was evaluated. A laboratory-scale composting process of the ACoD sludge was also applied. Composting parameters that ensured the generation of a stable organic improver were assessed. ACoD sludge composting was compared to a conventional sludge composting process to determine whether the presence of microalgae substrate in the digestate influenced the composting process in this first study to evaluate complete resource recovery from microalgae and primary sludge co-digestion.

2 METHODS AND MATERIALS

2.1 Anaerobic co-digestion pilot plant description

An ACoD pilot plant located in Cuenca del Carraixet WWTP (Valencia, Spain) was used for the ACoD experiments. This plant consisted of an anaerobic digester, with a total working volume of 900 L coupled to a 1-L membrane tank fitted with a 0.42 m^2 hollow fibre ultrafiltration membrane unit (0.03 µm pores, PURON® KMS, USA) (Figure 1a). The pilot plant was operated for one year at a solids retention time (SRT) of 70 d, while hydraulic retention time (HRT) was set at 30 d (an extensive description of the ACoD pilot plant can be found in Serna-García et al., 2020b). The ACoD pilot plant feedstock was a mixture of microalgae biomass cultivated in a MPBR plant (González-Camejo et al., 2017) and primary sludge from the WWTP thickener. Both these substrates were fed in proportions according to the results from previous studies (Serna-García et al., 2020a): 62% primary sludge and 38% microalgae based on VS content. The substrates were diluted before being fed to the reactor until the desired organic loading rate (OLR) was achieved: 0.5 gCOD·d^{-1}·L^{-1} and mixed in an equalisation tank.

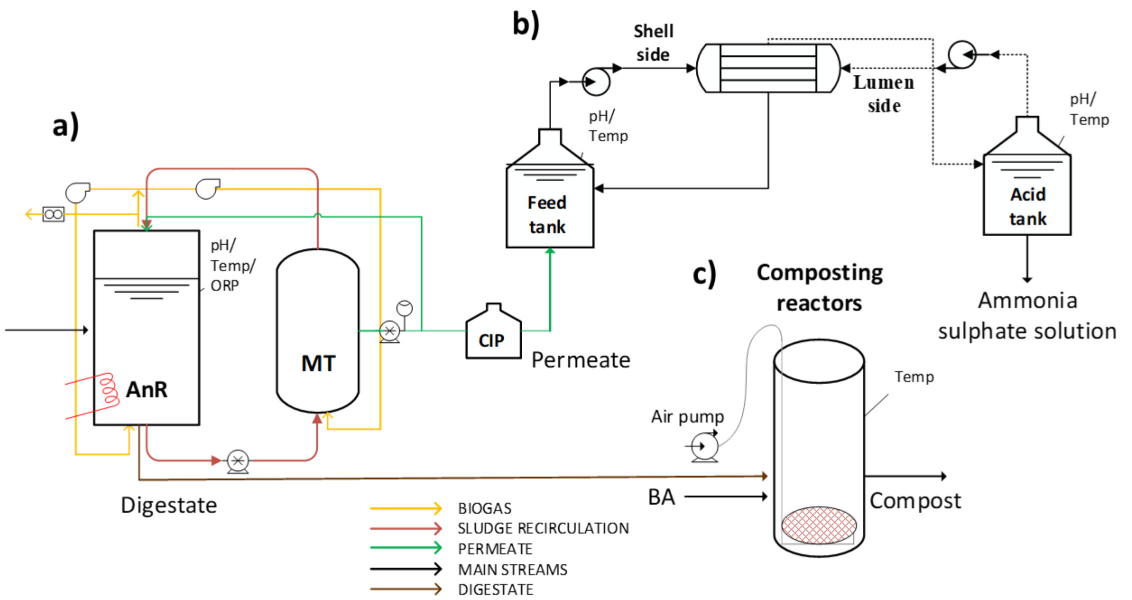

Figure 1: Layout of the experimental set-up: anaerobic co-digestion pilot plant (a), HFMC set-up (b) and an example of composting reactor (c). AnR: Anaerobic reactor, MT: membrane tank, CIP: clean-in-place tank, BA: bulking agent.

2.2 HFMC set-up

A hydrophobic polypropylene HFMC (X50 2.5x8 Liqui-Cel®, USA) with a surface of 1.4 m^2 was used for lab-scale N recovery. Two closed tanks of 1.2 L were used to store the permeate and acid solution (Figure 1b). Each tank was equipped with pH and temperature electronic sensors (SP10T, Consort®, Belgium) connected to a multiparametric analyser (Consort® C832, Belgium). The acid stream (0.05 M H_2SO_4)

circulated in the lumen side at a flow rate of 0.4 L·min^{-1} while the permeate was fed to the shell side at a flow rate of 0.6 L·min^{-1} and a pH of 10. Since the different ammonia (NH_3) concentrations on each side of the membrane are the driving force, it is necessary to work at pH over 8.6 in the feeding solution so, pH was adjusted by sodium hydroxide (1M). Both streams were recirculated and fed counter-currently. Filtration and settling were applied as pre-treatment to avoid membrane clogging.

2.3 Composting experiments

2.3.1 <u>Reactors description</u>

Seven cylindrical composting reactors (R_{A1}-R_{A4} and R_{C1}-R_{C3}) were operated at lab-scale. The composting reactors were 4 L glass Dewar flasks (KGW Isotherm, Germany) covered by an aluminium coating with cork insulation between the inside and the outside walls. A plastic mesh was incorporated at the bottom of all the reactors covered by a gravel layer to separate leachate and composted material. The Dewar flask covers were perforated to allow gas evacuation. A layout of the composting reactor is shown in Figure 1c.

2.3.2 <u>Composting substrates</u>

Two types of sludge were used to generate the mixtures in each composting reactor. The first, henceforward called ‘ACoD sludge’, was obtained from the ACoD pilot plant described in Section 2.1. The second, henceforward called ‘conventional sludge’, was an anaerobic sewage sludge from the Carraixet WWTP's conventional AD process, operated at an SRT of 20 d. Conventional sludge was used as reference substrate to compare the composting results obtained with the ACoD sludge under study. Both sewage sludges were pre-treated in a centrifuge to remove excess moisture, achieving values around 80-87% moisture. A cationic polyelectrolyte was added to conventional sludge that had been dried in the industrial centrifuge of the Carraixet WWTP. ACoD sludge was dried in a lab-scale centrifuge (Eppendorf, Centrifuge 5804) at 4350 rpm for 30 minutes and the obtained pellet was centrifuged for a further 15 minutes. The ACoD sludge was left to air-dry for 48 hours before its use.

Five different BA were characterised (Table 1). Pruning remains from the University of Valencia's garden were chosen as BA for the analytical results and their availability. These remains were shredded to achieve the correct size for assimilation by the microorganisms involved in the process.

Table 1: Bulking agent characterisation.

Bulking agent	**C/N ratio**	**Nitrogen (%)**	**Moisture (%)**
Pruning remains	49.5± 0.2	0.90± 0.00	49.1± 0.4
Lawn	13.9± 0.5	3.08± 0.09	78.9± 0.8
Olive wood	109 ± 1	0.42± 0.02	48.3± 1.1
Cypress tree	58.4± 0.3	0.81± 0.01	57.9± 0.2
Orange tree	29.4± 0.9	1.50± 0.02	30.6± 0.6

2.3.3 Experimental design

The reactors were operated for a maximum of 44 days in pairs to evaluate the effect of different operating conditions on the composting process. Three parameters were assessed: i) aeration, ii) addition of inoculum and iii) mixture of sludge and BA. Table 2 shows the main operating conditions of the seven reactors. Reactors R_{A1} to R_{A4} were fed with ACoD sludge, while R_{C1} to R_{C3} were fed with conventional sludge.

The effect of aeration was evaluated by the pairs of reactors R_{A1} and R_{A3} and R_{C1} and R_{C3}, which were run under the same operating conditions with different aeration modes: R_{A1} and R_{C1} had forced aeration while R_{A3} and R_{C3} were turned by hand (Table 2). The effect of adding inoculum to the reactor was assessed by reactors R_{A2} and R_{A4}, which had the same operating conditions, but R_{A2} had inoculum while R_{A4} had none (Table 2). The effect of mixing different proportions of sludge with BA was evaluated by the pairs R_{A2} and R_{A3} and R_{C2} and R_{C3}, which were run under the same operating conditions but with different proportions of sludge and BA (Table 2). The mixture component proportions were calculated as 'volumetric proportions' and 'theoretical proportions´. Volumetric proportions were generated with 2.5 volumes of BA per sludge volume, a commonly used ratio in sludge composting facilities. Theoretical proportions were calculated using Eq.1, in which the weight of each mixture's component is determined by setting the C/N ratio at a value of 25 and the moisture content in a value between 50% and 70%.

$$\frac{m_{BA}}{m_S} = \frac{(25 \cdot N_S(kg)) - C_S(kg)}{C_{BA}(kg) - (25 \cdot N_{BA}(kg))} \quad \text{Eq. 1}$$

being m_{BA} and m_s, proportions of BA and sludge, respectively; N_s and C_s the sludge N and carbon content, respectively; and N_{BA} and C_{BA} the BA N and carbon content, respectively.

Table 2. Main reactor operating conditions.

Reactors identification	Days in operation	Sludge	Mixture proportions	Aeration	Inoculum
R_{A1}	36	ACoD	Theoretical	Forced	✓
R_{C1}	36	Conventional	Theoretical	Forced	✓
R_{A2}	44	ACoD	Volumetric	Turned by hand	✓
R_{C2}	44	Conventional	Volumetric	Turned by hand	✓
R_{A3}	27	ACoD	Theoretical	Turned by hand	✓
R_{C3}	27	Conventional	Theoretical	Turned by hand	✓
R_{A4}	31	ACoD	Volumetric	Turned by hand	-

ACoD: Anaerobic co-digestion; A: ACoD sludge; C: Conventional sludge

Temperature was measured daily by a temperature probe inside the reactor. R_{A1} and R_{C1} were adapted to receive forced bottom-to-top aeration at a flow rate of 2 $L \cdot min^{-1}$ (by air supplied from the compressed air network). The rest of the reactors were manually turned over once a day. In all the reactors except R_{A4} (Table 2) 400 mL of inoculum from the maturation stage of a compost pile from the Vintena composting plant (Carcaixent, Valencia) were added to the mixture inside the reactor to accelerate the speed reaction of the microorganisms involved in the process.

2.4 Performance indicators

The ACoD process efficiency was evaluated in terms of biodegradability percentage, biomethane potential and methane yield according to the equations previously reported in Serna-García et al. (2020a).

To assess nutrient recovery from the ACoD permeate, P and N recovery were calculated by Eq. 2 and Eq. 3, respectively.

$$\%\,P\,recovery = \frac{P_{eff}}{P_{inf} + P_{rel}} \cdot 100 \qquad \text{Eq. 2}$$

being P_{eff} ($mgP\text{-}PO_4 \cdot L^{-1}$) the phosphate concentration in the effluent, $P_{inf,}$ ($mgP\text{-}PO_4 \cdot L^{-1}$) the phosphate concentration in the influent and P_{rel} ($mgP\text{-}PO_4 \cdot L^{-1}$) the phosphate released into the reactor during AD. This released phosphate was calculated as the influent stream phosphate degraded during AD, according to the substrate biodegradability obtained, as will be further explained in Section 3.2.

$$\%\,N\,recovery\,efficiency = \frac{TAN_{eff,0} - TAN_{eff,end}}{TAN_{eff,0}} \cdot 100 \qquad \text{Eq. 3}$$

being $TAN_{eff,0}$ ($mgN\text{-}NH_4 \cdot L^{-1}$) the initial concentration of Total Ammonia Nitrogen (TAN) in the HFMC influent (permeate from ACoD pilot plant) and $TAN_{eff,end,}$ ($mgN\text{-}NH_4 \cdot L^{-1}$) the TAN concentration at the end of the process (HFMC effluent).

To characterise the free ammonia transfer rate, the mass transfer coefficient was obtained using Eq. 4.

$$\ln\left(\frac{C_0}{C_t}\right) = \frac{k \cdot A}{V} \cdot t \qquad \text{Eq. 4}$$

being C_0 the initial total ammonia concentration in the feed solution ($g \cdot m^{-3}$), C_t the total ammonia concentration in the feed solution at time t ($g \cdot m^{-3}$), A the membrane surface (m^2), V the volume of the feed solution storage tank (m^3) and t the time (s).

C/N ratio, porosity and total nitrogen (TN) of both conventional and ACoD sludge mixtures with BA were measured in the initial composting samples. The presence of pathogens (*E. coli* and *Salmonella* spp.) was measured in both sludges (before being mixed with BA). Moisture and organic matter content, pH and electric conductivity were monitored weekly in the composting reactor samples to assess the process performance. The C/N ratio and pathogens were also analysed in each final product mixture.

According to Barrena et al. (2005), respirometric assays at the in situ temperature are suitable to monitor process biological activity since they are representative of the metabolic state of the microorganisms in the reactor. Nevertheless, assays at 37 ºC are more useful to study the stability of the process. To monitor the biological activity of the composting material several static respirometric assays at process temperature were therefore performed during the composting process in the reactors that achieved thermophilic temperatures. Static respirometric assays at a fixed temperature of 37 ºC were also performed in the same reactors to analyse the stability of the mixture. The slope of the oxygen concentration (%) versus time curves was calculated for each assay to calculate the respirometric index (RI). Respirometric assays and RI calculation were carried out according to Barrena et al. (2005).

2.5 Analytical Methods

Total solids (TS), VS, TSS (total suspended solids), VSS (volatile suspended solids), total chemical oxygen demand (TCOD), soluble COD, nutrients concentration (ammonium

(NH_4-N), TN, PO_4-P and total phosphorus (TP)), Alkalinity (Alk) and Volatile Fatty Acids (VFA) were measured in triplicate thrice a week according to APHA (2012) procedures. Methane content in the biogas produced was also determined thrice a week using a gas chromatograph equipped with a Flame Ionisation Detector (GC-FID, Agilent Technologies, USA). 1 mL of biogas was collected from the top of the reactor by a gas-tight syringe and injected into a 15 m × 0.53 mm × 1 μm TRACER column (Teknokroma, Spain) which was operated at 40 °C. Helium was the carrier gas at a flow-rate of 40 $mL \cdot min^{-1}$. Methane pure gas (99.99%) was used as standard.

Moisture and organic matter content, pH and electric conductivity were measured according to Standard Methods (APHA 2012) with the corresponding dilutions for adapting the method procedure to compost samples. For instance, for electric conductivity and pH determination, the sample was previously diluted in a ratio of 1:10. The supernatant was analysed after 30 min of agitation and 20 min of centrifugation (11000 rmp). C/N ratio was determined by measuring the elemental components of the mixture on an Elemental Analyser EA 1110 CHNS (CE Instruments Ltd, Wigan, United Kingdom). A previous pre-treatment of the sample, which consisted of drying the sample at 65 ºC in an oven and applying a milling process, was carried out to transform the heterogeneous material into a homogenous powder. Porosity was determined by the weight difference between the original sample and the sample saturated with water. TN in composting samples was determined according to APHA (2012) with previous homogenisation of the sample in a sonicator (S250D, Branson) and subsequent dilution at a ratio 1:1000.

E. coli presence was quantitatively determined by the standard method for enumeration of *E. coli* β-glucuronidase positive, following the UNE-EN ISO 9308-1:2014. *Salmonella* spp. was measured following the UNE-EN ISO 19250 standard method.

3 RESULTS AND DISCUSSION

3.1 Anaerobic co-digestion pilot plant performance

Continuous pilot-scale ACoD of microalgae biomass and primary sludge was monitored over one year. Microscopic study showed that microalgae biomass consisted primarily of *Chlorella* spp. Pseudo steady state was achieved in terms of biogas production, TS and nutrient concentration after 160 days of operation and was maintained and studied for a further period of 200 days. Table 3 shows the characterised influent and mixed liquor streams from the ACoD pilot plant during pseudo steady state. The co-digestion reactor achieved a biogas production of 78 $L \cdot d^{-1}$, with a methane percentage around 69%. Then, a higher methane yield of 218 $mLCH_4 \cdot gCOD_{inf}^{-1}$ (371 $mLCH_4 \cdot gVS_{inf}^{-1}$) was obtained than in numerous lab-scale studies of microalgae digestion as the sole substrate (González-Fernández et al., 2012; Greses et al., 2018; Ras et al., 2011, Wang et al., 2016) even when pre-treatment was applied to the microalgae biomass (Magdalena et al., 2018; Passos et al., 2014; Solé-Bundó et al., 2018, Wang et al., 2016). This methane yield corresponds to a total biodegradability percentage of 62.5% with a biomethane potential of 61.5%, which indicated that only 1% of the biodegradable organic matter was consumed by sulphate-reducing bacteria. The high AnMBR biodegradation efficiency also resulted in a high COD and VS removal of 63 and 64%, respectively. The system showed high stability since high alkalinity and no VFA accumulation was observed (Table 3), which resulted in stable non-controlled pH during the whole operation. Regarding membrane performance, pilot plant filtration was carried out at an average J_{20} of 4.5 LMH and a filtration time of 180 s, showing stability. No membrane replacement was needed during the experiment, since applying a specific gas demand of 0.15 $N \cdot m^3 \cdot m^{-2} \cdot h^{-1}$, a backwash cycle every two filtration cycles and physical cleaning was enough to control fouling formation (Serna-García et al., 2020b).

Table 3: Anaerobic co-digestion (ACoD) pilot plant influent and mixed liquor characterisation during pseudo steady state (mean ± standard deviation values).

	ACoD pilot plant
Influent	
TS ($mgTS \cdot L^{-1}$)	13086 ± 2009
VS ($mgVS \cdot L^{-1}$)	9919 ± 1592
TSS ($mgTSS \cdot L^{-1}$)	11558 ± 1323
VSS ($mgVSS \cdot L^{-1}$)	8800 ± 970
COD ($mgCOD \cdot L^{-1}$)	15895 ± 1682
TN ($mgN \cdot L^{-1}$)	578 ± 113
NH_4-N ($mgN \cdot L^{-1}$)	97 ± 20
TP ($mgP \cdot L^{-1}$)	137 ± 17
PO_4-P ($mgP \cdot L^{-1}$)	48.0 ± 14.1
SO_4-S ($mgS \cdot L^{-1}$)	194 ± 63
Mixed liquor	
TS ($mgTS \cdot L^{-1}$)	11337 ± 664
VS ($mgVS \cdot L^{-1}$)	7680 ± 457
TN ($mgN \cdot L^{-1}$)	685 ± 80
NH_4-N ($mgN \cdot L^{-1}$)	397 ± 33
TP ($mgP \cdot L^{-1}$)	145 ± 16
PO_4-P ($mgP \cdot L^{-1}$)	36.6 ± 6.1
pH	7.2 ± 0.1
VFA ($mgCH_3COOH \cdot L^{-1}$)	13.1 ± 15.4
Alk ($mgCaCO_3 \cdot L^{-1}$)	2058 ± 109

TS: total solids; VS: volatile solids TSS: total suspended solids; VSS: volatile suspended solids; COD: chemical oxygen demand; TN: total nitrogen; NH_4-N: ammonium; TP: Total phosphorus; PO_4-P: phosphate; SO_4-S: sulphate; VFA: volatile fatty acids; Alk: alkalinity.

High biogas production from microalgae and primary sludge was obtained in long-term pilot-scale operation without the need to apply costly pre-treatments to improve microalgae degradation. This biogas was rich in methane and is a renewable fuel that could be used for energy and heat generation allowing an approach to circular economy scenarios in which a WRRF would be self-sufficient in terms of energy (further research is needed).

3.2 Nutrient recovery

ACoD released N to the soluble phase, as expected; around 400 $mgNH_4$-$N \cdot L^{-1}$ was present after the ACoD process (Table 3). Ammonium remained stable at a concentration higher than that present in the influent (Table 3). Unlike ammonium, phosphate content (37 $mgPO_4$-$P \cdot L^{-1}$) in the permeate was lower than the influent content (48 $mgPO_4$-$P \cdot L^{-1}$). These results indicated that uncontrolled P precipitation processes were taking place inside the reactor, which had already been observed by several authors (Barat et al., 2009; Doyle and Parsons, 2002; Martí et al., 2017), who reported precipitation problems in the digestion stage of a WWTP when treating sludges coming from biological removal processes. For that reason, a mass P balance was applied to the anaerobic digester, considering the average influent and effluent concentrations to estimate the potential P-recovery. P balances were based on the organic P (P_{org}) content per gram of VSS ($gP_{org} \cdot gVSS^{-1}$) in microalgae and primary sludge substrates. This P_{org} content was calculated as the difference between total P concentration and phosphate concentration in each substrate. The experimental values showed a content of 0.010 $gP_{org} \cdot gVSS^{-1}$ for primary sludge and a content of 0.013 $gP_{org} \cdot gVSS^{-1}$ for microalgae. These values were in agreement with those observed in the literature: 0.013 $gP_{org} \cdot gVSS^{-1}$ in primary sludge were reported by Martí et al. (2008) and 0.011 $gP_{org} \cdot gVSS^{-1}$ in microalgae were reported by González-Camejo et al. (2020). Two mass balances were carried out (Table 4) according to the value of biodegradability used for the calculations. The first balance considered the biodegradability percentage described in Section 3.1 (62.5%) obtained digesting microalgae and primary sludge substrates together (combined biodegradability). The second balance was calculated according to the biodegradability percentage obtained when digesting each substrate alone (separated biodegradability). This was 54% for microalgae digestion and 55% for primary sludge (data not shown).

Both mass balances showed similar results, revealing meaningful phosphate precipitation (Table 4). Around 35 mgP·d^{-1} were fixed in the reactor, representing 74% of the available phosphorus. Only 26% of the phosphate was available for recovery in the effluent (Table 4). Influent and effluent calcium and magnesium concentrations (data not shown) indicated a calcium and magnesium precipitation of around 11 and 7%, respectively. This cation precipitation along with the high ammonium concentration suggests the formation of struvite or other phosphate compounds inside the reactor. Uncontrolled P precipitation hindered the recovery of phosphate through a struvite precipitation process after the AD step, reducing potential P recovery in the treatment plant. Nevertheless, a significant proportion (145 mg P·L^{-1}) was recovered in the biosolids fraction.

Table 4. Mass phosphate balances carried out in the anaerobic digester using separated and combined biodegradability. Average values and standard deviation are shown.

	Separated BD	**Combined BD**
$gP_{org}·gVSS^{-1}{}_{influent}$	0.011 ± 0.007	0.011 ± 0.007
P_{loss} (gP·kg sludge^{-1})	7.3 ± 0.7	7.4 ± 0.7
$P_{available}$ (gP·kg sludge^{-1})	2.7 ± 0.6	2.5 ± 0.6
Potential $P_{recovery}$ (%)	27.4 ± 6.5	25.5 ± 6.1

BD: biodegradability P_{loss}: phosphorus precipitated; $P_{available}$: phosphorus available for recovery.

Although there was not enough P in the ACoD plant permeate to apply struvite precipitation, the TAN content could be recovered from the permeate. N recovery processes are usually applied after P recovery, mainly struvite precipitation, in which pH is raised and some cations and P precipitate. This precipitation process leads to non-settable solids formation (fine carbonate and phosphate precipitated particles), which must be separated in order to avoid membrane clogging. In this case, as no struvite precipitation step was performed, pre-treatment was needed at the beginning of the process to raise the pH and later separate the solids formed. In these steps some N (around 15% w/w) was lost by stripping, which is a similar value to that reported by Noriega-Hevia et al. (2020). The results obtained applying HFMC to the ACoD permeate showed a recovery efficiency of 99% in an operating time of approximately 40 min. Figure 2 shows the TAN evolution during the experiment. TAN concentration first dropped by 40% after 5 min, reaching the maximum recovery rate because of the high concentration of FAN in the ACoD permeate. As the flux is closely related to the FAN concentration, which decreased, the recovery rate was slowly reduced until complete TAN recovery. Due to the FAN passing through the membrane, the pH in the feed solution storage tank decreased, so that during the experiment the pH had to be maintained at a value of around 10 by adding sodium hydroxide in order to maintain all TAN as FAN and consequently the driving force. The overall mass transfer coefficient calculated applying Eq. 4 was $1.4 \cdot 10^{-6}$ $m \cdot s^{-1}$, which is similar to the values obtained by Noriega-Hevia et al., (2020) and Kartohardjono et al., (2015). The overall NH_4-flux obtained in the experiment was 0.4 $kgNH_4\text{-}N \cdot m^{-2} \cdot h^{-1}$, being 1.43 $kgNH_4\text{-}N \cdot m^{-2} \cdot h^{-1}$ the highest flux obtained at the beginning of the experiment, when the FAN concentration was higher. The higher the N concentration in the feed solution, the lower the membrane required per kg of N recovered.

The product obtained at the end of the experiment was an ammonia sulphate solution with a maximum N richness of 4%, which is similar to that obtained by Richter et al., (2019) in a full scale plant. This ammonia sulphate solution is an inorganic salt that could be used as a substitute for the currently used chemical fertilisers. The market price of the sulphate solution obtained can vary, 2.3 €·kg N^{-1} being the value proposed by Dube et al., (2016).

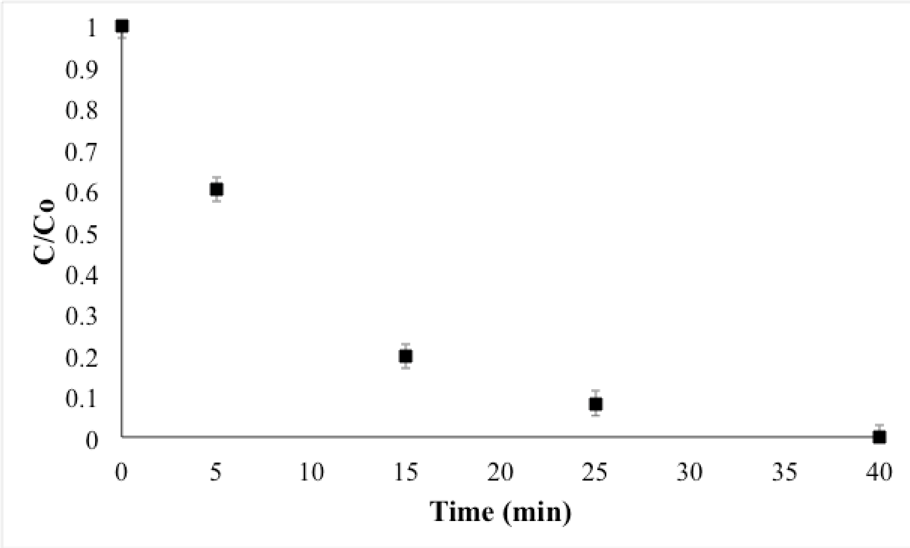

Figure 2. Total ammonium nitrogen (TAN) evolution during hollow-fibre membrane contactor tests. C is the TAN concentration and C_O is the initial TAN concentration

Although the N recovery results through HFMC are promising for future scaling up of the technology, a previous economic analysis would be required. Membrane costs are among the highest costs of the technology. For instance, the membrane cost in the present work is estimated at 0.5 € per cubic meter of N-rich stream treated. Since this stream (rejected water from sludge dewatering) usually represents less than 5% of the flow rate entering the WWTP, the membrane cost would be less than 0.025 $€\cdot m^{-3}$ of influent wastewater. Nevertheless, a detailed economic study, including the market price of the sulphate solution and the main costs of the technology (membrane and chemical reagents) is required. An optimization study is also needed to obtain the operating conditions to minimize operating costs prior to full-scale implementation of this technology.

3.3 Composting performance

The composting process of ACoD sludge and conventional sludge was evaluated. The effect of applying different aeration modes, adding inoculum to the mixtures and mixing sludge with BA in different proportions was assessed. To determine whether the composting process and stabilisation were achieved, mixtures from each reactor were characterised at the beginning (Initial characterisation) and end of the process (Final characterisation) (Table 5).

The initial characterisation showed that C/N ratio was in general lower in mixtures generated with volumetric proportions since there was a higher content of sludge in those mixtures. TN content was higher in reactors containing ACoD sludge (R_{A1}, R_{A2} and R_{A3}) than in their replicates containing conventional sludge (R_{C1}, R_{C2} and R_{C3}) due to the presence of microalgae biomass in the ACoD process, which has a high nitrogen content (Ullah et al., 2015). Initial moisture content was around 60% in all the reactors, which is an optimum value to start the composting process (Bueno et al., 2008; Diaz and Savage., 2007). Reactors containing ACoD sludge had higher moisture content associated with the dewatering method used for each sludge. It is also remarkable that in the mixtures containing volumetric proportions, moisture content was higher than in the ones generated by theoretical calculations due to their higher proportion of sludge (Table 5), except in reactor $R_{A1.}$ Initial porosity was in general higher in mixtures with theoretical proportions of sludge and BA. Initial pH and electrical conductivity of both types of mixture had typical initial values according to their ACoD and conventional sludge composition. The initial characterisation of ACoD sludge (not mixed with BA or inoculum) showed no presence of *E. coli* neither *Salmonella* spp., microorganisms commonly used as pathogen indicators. Conventional sludge (not mixed with BA or inoculum) contained no *Salmonella* spp. but was positive for *E. coli*. The inoculum added to some reactors (Table 2) also showed the presence of *E. coli*.

Table 5. Initial and final characterisation of the composting samples for the seven reactors evaluated.

	ACoD sludge				***Conventional sludge***		
	R_{A1}	**R_{A2}**	**R_{A3}**	**R_{A4}**	**R_{C1}**	**R_{C2}**	**R_{C3}**
Mixtures	Theoretical	v/v	Theoretical	v/v	Theoretical	v/v	Theoretical
Aeration / Inoculum	Forced / Yes	Turned / Yes	Turned / Yes	Turned / No	Forced / Yes	Turned / Yes	Turned /Yes
Initial characterisation							
C/N ratio	24.0 ± 0.7	14.5 ± 0.1	15.8 ± 0.5	16.4 ± 0.1	24.6 ± 0.1	12.5 ± 0.1	19.1 ± 0.1
Moisture content (%)	68.1 ± 0.8	68.7 ± 0.4	59.5 ± 0.9	62.9 ± 0.4	58.6 ± 0.5	64.3 ± 0.7	50.4 ± 0.5
O.M (%)	87.2 ± 0.6	84.1 ± 0.5	75.8 ± 0.9	87.0 ± 0.6	80.1 ± 0.8	86.9 ± 0.5	75.9 ± 0.5
Porosity (%)	36.5 ± 0 .9	13.4 ± 0.9	38.4 ± 0.6	21.6 ± 1.5	33.4 ± 2.0	24.0 ± 1.4	40 ± 1.0
TN ($gN \cdot kg_{d.m}^{-1}$)	15.0 ± 1.2	24.6 ± 1.1	15.1 ± 1.3	9.6 ± 0.7	11.6 ± 0.9	11.5 ± 0.8	9.1 ± 0.6
pH	7.8 ± 0.1	7.4 ± 0.1	8.1 ± 0.6	6.3 ± 0.1	8.5 ± 0.0	8.5 ± 0.1	8.5 ± 0.1
Conductivity ($\mu S \cdot cm^{-1}$)	1104 ± 158	1418 ± 272	1775 ± 144	2620 ± 190	1905 ± 247	1490 ± 258	1992 ± 249
*E. coli**	A	A	A	A	P	P	P
Salmonella spp.*	A	A	A	A	A	A	A
Final characterisation							
C/N ratio	14.9 ± 0.0	13.0 ± 0.2	14.9 ± 0.0	13.2 ± 0.3	13.7 ± 0.2	11.4 ± 0.1	17.6 ± 0.2
C/N ratio removal (%)	37.9	10.3	5.4	19.5	44.3	8.8	7.8
Moisture content (%)	68.3 ± 0.3	70.0 ± 1.0	61.6 ± 0.5	67.96 ± 0.6	47.8 ± 0.2	67.8 ± 0.5	66.5 ± 0.4
O.M (%)	76.6 ± 0.2	75.8 ± 0.7	74.8 ± 0.3	82.51 ± 0.5	74.8 ± 0.5	66.8 ± 0.9	72.5 ± 0.3
O.M. removal (%)	12.1	9.8	1.3	5.2	6.5	23.1	4.6
pH	8.3 ± 0.1	8.7 ± 0.1	8.3 ± 0.1	7.8 ± 0.2	8.0 ± 0.1	8.2 ± 0.2	8.11 ± 0.05
Conductivity ($\mu S \cdot cm^{-1}$)	1908 ± 196	2300 ± 253	1990 ± 181	3010 ± 173	2170 ± 260	3530 ± 224	2800 ± 250
E. coli	P	A	P	P	P	A	P
Salmonella spp.	A	A	A	A	A	A	A

O.M: Organic matter; TN: Total nitrogen; d.m.: dry matter; v/v: volumetric proportions; A: absence of microorganisms in sludge; P: presence of microorganisms in sludge; ´´ pathogens in initial samples were only measured in the sludge, not in the mixtures of sludge and bulking agent.*

Final characterisation of the samples (Table 5) showed that moisture content in general did not significantly change from the initial conditions. However, reactor R_{C1} lost a significant amount of moisture, from 59% to 48%, associated with an excess of forced aeration that contributed to the dryness of the material. On the other hand, reactor R_{C3} had increased moisture content, from 50% to 66%. This could be explained by inadequate drainage of the excess moisture. pH values were around 8, which are in agreement with typical values of mature compost (8.0 – 8.5 (Diaz and Savage, 2007)). Conductivity values showed an increase in comparison to initial values in all reactors (Table 5). According to Diaz and Savage (2007) this behaviour is typical in a composting process due to the mineralisation of the organic matter. All the samples from the reactors (except for R_{A2} and R_{C2}) contained *E. coli* after composting. Since the initial samples from the ACoD reactors were *E. coli* free (Table 5) and the initial characterisation of the inoculum showed *E. coli* to be present, it is possible that they were contaminated by the inoculum. Only reactors R_{A2} and R_{C2} achieved complete sanitation after the composting process, showing a final material without *E. coli* and *Salmonella* spp.

With regard to temperature evolution, reactors R_{A2} and R_{C2}, with volumetric mixtures, showed temperatures between 50 ºC and 53 ºC achieving the thermophilic temperature necessary for the sanitation of the composted material (Insam and De Bertoldi, 2007). In the case of reactor $R_{A2,}$ the thermophilic phase was reached during a period of 20 days and in reactor R_{C2} during a period of 24 days (Figure 3). Reactor R_{A2}, which contained ACoD sludge, had a larger acclimatisation period since it followed the same evolution as reactor R_{C2} (with conventional sludge) but with a delay of several days (Figure 3). This could be associated with a lower content of easily biodegradable organic matter due to the high SRT (70 d) of the previous ACoD process. In both reactors upper organisms such as mites were found as bioindicators of the correct progress of the composting process

(Soliva, 2001). Indeed, these two reactors also showed no presence of *E. coli* and *Salmonella* spp., as mentioned in the paragraph above. The remaining reactors did not achieve thermophilic temperatures probably due to: the excess of aeration in reactors with forced aeration (R_{A1}, R_{C1}) (Bueno et al., 2008 and Negro et al., 2000 reported that aeration should not be excessive to avoid inhibiting microbial activity), coupled with the small volumes of the reactors, which led to higher heat losses; the absence of inoculum in reactor R_{A4} (Manu et al., 2017 observed how the composting process could be improved by adding a microbial inoculum); and/or the lower sludge content in the reactors prepared with theoretical proportions (R_{A1}, R_{C1}, R_{A3}, R_{C3}).

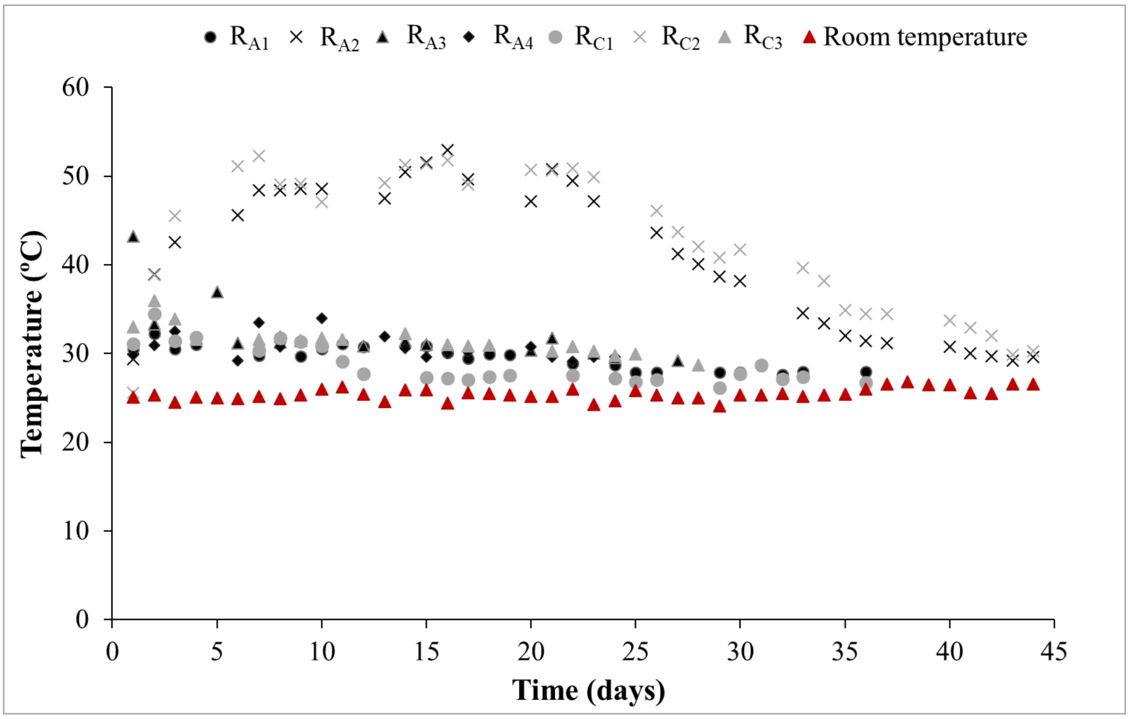

Figure 3. Evolution of mixture temperature and room temperature during composting period.

Regarding the effect of applying different aeration modes, the results indicated that forced aeration offers better composting conditions, since a higher organic matter and C/N ratio removal was observed in reactors R_{A1} and R_{C1}, compared with their replicates R_{A3} and R_{C3}, which were aerated by hand (Table 5).

The results regarding the effect of adding an inoculum source to the sludge-BA mixtures indicated that inoculum has an important effect on composting since thermophilic temperature and sanitation were achieved in reactor R_{A2} but not in reactor R_{A4}. Higher organic matter removal (9.8%) was also observed in reactor R_{A2} than in R_{A4} (5.2%).

Finally, regarding the effect of mixing sludge and BA in different proportions the results indicated that reactors R_{A2} and R_{C2}, which were prepared with volumetric proportions of BA and ACoD and conventional sludge, respectively, achieved thermophilic temperature and showed complete sanitation of the compost, suggesting that this mixture method should be used for composting. Both these reactors were aerated by hand and achieved percentages of organic matter removal and C/N ratio removal of 9.8 and 10.3 for reactor R_{A2} and 23.1 and 8.8 for R_{C2}, respectively. In contrast, reactors R_{A3} and R_{C3}, run under the same operating conditions but mixed with theoretical proportions did not achieve thermophilic temperature and showed percentages of organic matter removal and C/N ratio removal of 1.3 and 5.4 for reactor R_{A3} and 4.6 and 7.8 for R_{C3}, respectively, being these percentages lower than the ones observed for the pair of reactors R_{A2} and R_{C2} (Table 5).

When comparing all the reactors, of those fed with conventional sludge, R_{C2} showed the best performance in terms of compost sanitation, temperature achieved and organic matter removal efficiency, but not in terms of C/N ratio removal (Table 5). Reactor R_{C1} showed the best C/N ratio removal (44%) but did not achieve thermophilic temperature or compost sanitation. Of the reactors fed with ACoD sludge, even R_{A2} showed the best performance in terms of compost sanitation and temperature achieved, organic matter and C/N ratio removal were not among the highest values obtained from all the reactors. In fact, R_{A1} showed an organic matter and C/N ratio removal percentage of 12.1 and 37.9, respectively, this being the highest value for these two parameters in those reactors fed with ACoD sludge (Table 5). The two reactors mentioned (R_{A1} and R_{C1}) were prepared with mixtures calculated by theoretical proportions with forced aeration. The previous results suggest that, although reactors R_{A2} and R_{C2} were the only ones that achieved compost sanitation, and the volumetric proportions seemed to be more suitable, a higher organic matter and C/N ratio removal could be achieved by forced aeration in ACoD sludge reactors at the correct airflow to achieve the best composting conditions.

In general, it can be observed that the higher the initial C/N ratio, the greater the elimination of this parameter, which is the case of mixtures generated with theoretical proportions. However, in mixtures generated with volumetric proportions, the amount of sludge added to the mixtures is 3.2 times higher than in the theoretical ones. Therefore, it actually contains more biodegradable organic matter but it also has a much higher N content, which makes the initial C/N ratio lower. In addition, ammonia may have been lost due to the combination of ammonification of the organic nitrogen and the basic pH values reached during the composting process. The latter may result in a not significant change of the initial values of C/N ratio for mixtures generated with volumetric proportions and thus in lower C/N ratio removal. Therefore, since C/N ratio is a chemical composting parameter and not a biochemical one, it could be assumed that this alone is not a suitable indicator of the process evolution and that all the parameters (aeration, temperature, moisture content, presence of inoculum, proportions of sludge and BA) should be controlled. New BA and sludge ratios should thus be applied in future experiments to achieve correct C/N ratio removal and material sanitation. The optimum aeration rate flow should also be studied to obtain the optimum composting results.

3.3.1. Respirometry: monitoring the biological activity

Biological activity was only measured in the reactors that reached thermophilic temperatures, since this is an indicator of correct process evolution and higher biological activity in the mixture. Respirometric tests were therefore carried out in reactors R_{C2} (conventional sludge) and R_{A2} (ACoD sludge). The RIs at process temperature and 37 ºC are shown in Table 6 for each respirometric test performed.

Table 6. Respirometric index (RI) obtained in respirometric tests.

Respirometric test	Day of composting	RI ($mgO_2 \cdot gO.M.^{-1} \cdot h^{-1}$)
RI_{A2} (48ºC)	7	3.28
RI_{A2} (52ºC)	15	1.81
RI_{A2} (44ºC)	26	1.51
RI_{C2} (51ºC)	6	4.46
RI_{C2} (52ºC)	16	2.04
RI_{C2} (44ºC)	27	1.72
RI_{C2} (37ºC)	44	0.242
RI_{A2} (37ºC)	44	0.257

O.M.: organic matter

Figures 4a, 4b and 4c show the biological activity measurement for reactor R_{A2} on Days 7, 15 and 26 of operations, which were Days 2, 10 and 21 at the beginning of the thermophilic phase, respectively. Figures 4d, 4e and 4f show the measurement of the biological activity for reactor R_{C2} on Days 6, 16 and 27 of operation, corresponding to Days 4, 14 and 25 at the beginning of the thermophilic phase, respectively.

The slope of the curves marked in red colour in Figure 4 represents the oxygen uptake rate. The higher the composting time, the lower the slope of the curve. RI thus decreased as composting time increased, so that biological activity also decreased, the first few days of the thermophilic phase being the period with the highest biological activity. R_{C2}

showed the same behaviour as R_{A2}. The reactor containing ACoD sludge (R_{A2}) presented lower RI than R_{C2} at the same temperature (Table 6). This could be explained by the higher SRT (70 d) in the previous ACoD process than in the conventional AD (SRT of 20 d), which led to a lower biodegradable substrate concentration available for microorganisms, therefore lower biological activity. The thermophilic phase was also longer in R_{C2} than in R_{A2} (Figure 3), which also indicates higher biological activity in the mixture.

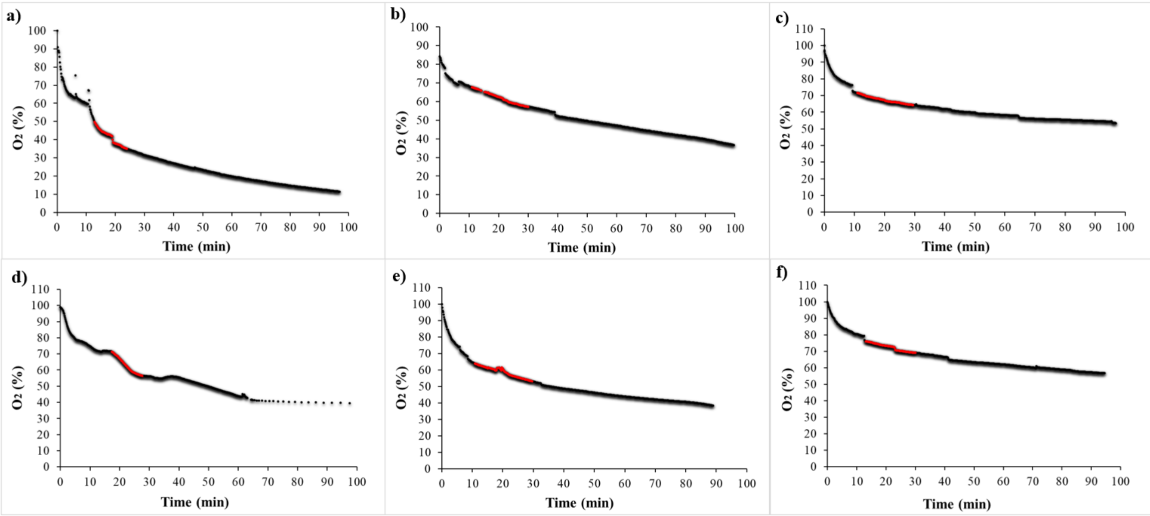

Figure 4. Oxygen percentage evolution over time at process temperature in reactors R_{A2} (a, b, c) and R_{C2} (d, e, f).

The respirometric tests at 37 ºC showed that the lower the RI, the more stable the mixture. At the end of the composting process, the biological activity decreased significantly in both reactors, showing similar RI values (Table 6). As established by the TMECC (US Department of Agriculture and Council, 2001), a composted material becomes stabilised when RI at 37 ºC is between 0.5-1.5 $mgO_2 \cdot g$ organic matter$^{-1} \cdot h^{-1}$, and is very stable when RI is below 0.5 $mgO_2 \cdot g$ organic matter$^{-1} \cdot h^{-1}$, is in this case. In terms of stability, the mixtures in reactors R_{C2} and R_{A2} were therefore stabilised (Table 6).

Composting after a previous AD step was thus achieved from both ACoD and conventional sludge. Reactors R_{A2} and R_{C2}, prepared with volumetric proportions, achieved thermophilic temperatures and complete compost sanitation. Reactor R_{C2} had higher organic matter removal (2.4-fold higher) but lower C/N ratio removal (1.1-fold lower) than R_{A2}. Both reactors showed RI values associated with a stabilised composted material and the respirometric tests indicated that the process temperature RI fell as composting time increased.

4 CONCLUSIONS

Three potentially useful by-products were generated through microalgae and primary sludge co-digestion in an AnMBR: methane-rich biogas, nitrogen-rich permeate and nutrient-rich digestate. Nitrogen was recovered from the permeate at 99% efficiency and an ammonia sulphate solution, which could be used as a commercial fertiliser, was obtained. For the first time, composting process applied to a digestate coming from a microalgae co-digestion plant was evaluated in the present work at laboratory scale. ACoD digestate composting was compared to a conventional AD digestate composting, and similar values were obtained for both. The best composting performance in terms of sanitation of the composted material and removal of organic matter and C/N ratio was obtained when mixing sludge with BA in volumetric proportions (2.5 volume of BA per 1 volume of sludge), applying forced aeration and adding an inoculum from an industrial compost plant to accelerate the biological process. Respirometric tests indicated a highly stable final compost. The combination of microalgae co-digestion with subsequent composting offers complete resource recovery (energy, nutrients and water) from sewage in a circular economy-based scenario for future WRRF implementation.

CRediT authorship contribution statement

Rebecca Serna-García: Conceptualization, Methodology, Investigation, Formal analysis, Writing - Original draft. Patricia Ruiz-Barriga: Methodology, Writing – Original draft. Guillermo Noriega-Hevia: Formal analysis, Writing – Original draft. Joaquín Serralta: Writing- Review & Editing. María Pachés: Supervision; Writing – Review & Editing. Alberto Bouzas: Supervision, Writing – Review & Editing, Funding acquisition.

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