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ORIGINAL PAPER

Enhancement of Sewage Sludge Bioconversion to Methane by the Addition of Exhausted Coffee Biowaste Liquid Fraction

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Abstract

Anaerobic co-digestion of organic rich wastes and wastewater sludge has become an attractive economic possibility for water utilities as it enhances biogas production. The suitability of the organic rich waste depends on its biodegradability as well as on its synergetic effect on the anaerobic digestion process. The feasibility of sewage sludge (SS) treatment via co-digestion was studied in a semi-continuous mode at mesophilic conditions (36 ± 1 °C), with a hydraulic retention time of 17 days and an average organic loading rate of 0.94 ± 0.05 g_{VS} $L_{reactor}$ day^{-1}, using the liquid fraction of pre-treated exhausted coffee biowaste (LECB) as a co-substrate. An anaerobic co-digestion trial (T1) was performed using as feeding mixture 80% SS and 20% LECB (v:v) and compared against a reference scenario of mono-digestion of SS (T0). The stability along assays was ensured by monitoring the digestate characteristics (pH, electrical conductivity, total alkalinity and ammonia content) and the specific energy-loading rate (SELR). Along the operation time of T1, methane yield and VS removal were significantly higher in comparison to mono-digestion of SS. Results showed that the addition of the co-substrate had a positive effect on specific methane production (3 times higher) and methane content (12% higher), indicating this is a feasible strategy towards self-sufficient wastewater treatment plants.

Keywords Anaerobic co-digestion · Exhausted coffee biowaste · Sewage sludge · Waste-to-energy

Abbreviations

AcoD	Anaerobic co-digestion
AD	Anaerobic digestion
CSTR	Continuous stirring tank reactor
ECB	Exhausted coffee biowaste
GPR	Gas production rate
HRT	Hydraulic retention time
LECB	Liquid fraction of the exhausted coffee biowaste
OLR	Organic loading rate
SCG	Spent coffee grounds
SELR	Specific energy loading rate
SMP	Specific methane production
SS	Sewage sludge
WWTP	Wastewater treatment plant

Introduction

The circular economy leads to a new vision of wastewater treatment plants (WWTP), now considered as resource recovery factories [1, 2]. Besides water reuse, there are several options of possible materials that can be recovered, namely cellulose fibres, phosphorus, nitrogen and biopolymers. Furthermore, WWTP generates large amounts of sewage sludge (SS) that can be used to recover bioenergy through anaerobic digestion (AD). However, the poor biodegradability of SS leads to low methane yields and therefore it is crucial in defining appropriate waste management scenarios to enhance biomethanisation. This can be achieved either by pre-treating the SS [3] or selecting different types of biowaste with high methanogenic potential to be co-digested with SS [4–9]. In order to promote the synergetic interactions during anaerobic co-digestion (AcoD), an optimal feeding blend ratio should be established to improve specific methane production and digestate quality [10]. The optimisation of energy recovery through AcoD enhances WWTP environmental performance and lowers its carbon footprint [11]. Furthermore, implementation of AcoD in WWTP along with other measures, for example optimisation

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of the highly energy-consuming aeration process, will contribute to energy self-sufficient WWTP [12, 13].

The food supply chain, due to its large generation of waste, is one of the targets of the Commission's Circular Economy Package [14] to stimulate transition towards a circular economy. To tackle this problem action is needed at the different stages of the supply chain including primary production, processing, distribution and consumption.

Coffee is one of the main commodities in the world, being particularly important for the economy of its producing countries, most of which are developing countries. According to the International Coffee Organization, in the coffee year 2015/2016, 151.3 million of 60 kg bags were consumed corresponding to 9 million tons of coffee. In fact, coffee is one of the most consumed beverages in the world, with 50% of the coffee produced worldwide being used for soluble coffee preparation [15]. As broadly described by Alves et al. [16], coffee processing from the field to the cup generates several by-products along the chain. The waste that remains after coffee beverage preparation (spent coffee grounds, SCG) is rich in a variety of organic compounds such as carbohydrates, lignin, fatty acids and antioxidant compounds [17, 18].

The industrial production of soluble coffee and coffee drinks includes a water-pressured extraction process of the mixture of coffee and cereals (e.g. malted barley, rye, barley in bulk and chicory), from which exhausted coffee biowaste (ECB) is generated. This biowaste contains the insoluble fraction of organic compounds present in the coffee, as happens for SCG, but also in the processed cereals (i.e. fatty acids, lignin, cellulose, hemicellulose, and other polysaccharides) [19].

Several studies on possible valorisation strategies for SCG have been published. Mata et al. [20] illustrated the implementation of the bio-refinery concept to SCG and present a review of biotechnological routes towards SCG valorisation. Murthy and Naidu [17] and Mussato et al. [18], published reviews on coffee waste valorisation for recovery of bioactive compounds and production of value-added products. Mentioned examples include production of enzymes (e.g. caffeinase, xylanase and pectinase), use as a fertiliser or as substrate for growing mushrooms, recovery of bioactive compounds (e.g. phenols), and use as an adsorbent or animal feed, among others. As SCG is rich in mannose (20–25% of its total carbohydrate content), it has been considered as a source for mannose production as presented by Nguyen et al. [21] who developed an integrated process to produce d-mannose and ethanol. This process involves pretreatment of SCG with ethanol at high temperature, hydrolysis with enzymes to produce sugars, fermentation with bioethanol-producing yeasts (that use almost all glucose and galactose to produce ethanol but maintain d-mannose in the broth), removal of coloured compounds and separation of ethanol and d-mannose through pervaporation.

More recently Karmee [22] revised the technical feasibility of producing biofuel and several value-added products from SCG. Biogas is one of the biofuels that can be produced from SCG, namely by AcoD, with waste activated sludge and/or different organic wastes (e.g. food waste, whey, etc.) [23–25]. All the above-mentioned studies have shown that AcoD of SCG is advantageous over mono-digestion, enhancing the process feasibility and stability by balancing the C/N ratio of the feedstock. This improves the buffering capacity and minimises the effect of inhibitory compounds.

Regarding ECB valorisation, few studies have been developed until now, but as its main characteristics are common to SCG [18, 19] the same valorisation routes can possibly be applied. One of the possible options is the production of biofuels, for example biodiesel, as presented by Tuntiwiwattanapun et al. [26] or recovering energy using it as a co-substrate to enhance biogas production. Regarding this last option, as far as the authors know, only Sousa [27] used ECB as co-substrate in AcoD with pig slurry.

The objective of this research was to access the potential of an industrial symbiosis between the coffee industry and the wastewater treatment sector improving the efficiency of SS biomethanisation by the addition of pretreated ECB. This approach contributes to sustainable waste management and to improving WWTP energy balance, aiming at self-sufficiency.

Materials and Methods

Feedstocks

The sewage sludge (SS) was collected from the thickening tank of a full-scale wastewater treatment plant (WWTP) designed to treat an average flow of 53,000 m^3 per day (211,000 inhabitant's equivalent), located in Lisbon, Portugal. This sewage sludge is a mixture of primary sludge and waste activated sludge (40:60, v:v).

The acclimation period lasted 68 days using diluted SS, intermittent mixing and a temperature of 36 ± 1.0 °C. During this period the influent COD was increased from 1000 to 2500 mg L^{-1}, corresponding to a variable organic loading rate (OLR) of 0.11 to 1.5 g_{COD} L^{-1} day^{-1}.

Exhausted coffee biowaste (ECB) results from the water-pressured extraction step of the soluble coffee and coffee drinks production process from Nestlé's Avanca factory. The fresh samples were placed in 5 kg plastic containers and transported to the research unit where the study was developed. Samples were stored in a refrigerator at 4 °C until pre-treatment and characterisation procedures.

ECB Pre-treatment

To optimise the feeding mixture characteristics in terms of soluble organic matter and to avoid clogging problems and floating layers inside the digester, the ECB was subjected to solid/liquid separation. This procedure was carried out by filtration using a vacuum pump (BüchiVac V-500:230 VAC; 50 Hz; 240 W) and the liquid fraction (LECB) was used as co-substrate.

Analytical Procedures

The chemical composition of the different materials used along the trials and AcoD process performance and stability was monitored based on the following parameters: pH, electrical conductivity (EC), total solids (TS), volatile solids (VS), volatile suspended solids (VSS), total chemical oxygen demand (TCOD), soluble chemical oxygen demand (SCOD), Kjeldahl nitrogen (N_K), and ammonia nitrogen ($N\text{-}NH_4^+$). These parameters were analysed according to the standard methods [28]. Total alkalinity (TA) was measured according to Forgács et al. [29] based on a potentiometric titration with 0.05 mol L^{-1} H_2SO_4 until an end-point of pH 4.0. Total organic carbon was determined according to Cuetos et al. [30]. The organic carbon was subsequently divided by the total nitrogen to obtain the C/N.

Anaerobic Digestion Trials

Anaerobic digestion trials included three different periods: start-up, mono-digestion (T0), and co-digestion (T1). The total monitoring period was 136 days, 34 days for each period. During the start-up period (68 days) the reactor was operated according to that previously mentioned in "Feedstocks". The reference scenario (T0) was developed using sewage sludge as mono-substrate keeping an organic loading rate (OLR) of 0.89 ± 0.23 g_{VS} $L_{Reactor}^{-1}$ day^{-1}.

In T1, a co-digestion regime was adopted using a feeding mixture of 80% SS and 20% LECB according to previous studies by the authors on the optimisation of VS/TS, SCOD/TCOD and C/N ratios. The OLR was kept at 0.99 ± 0.25 g_{VS} $L_{Reactor}^{-1}$ day^{-1}, which according to Tukey's test is not significantly different from the OLR kept at T0.

Each trial was kept for two hydraulic retention times (HRT, 17 days) after steady-state conditions were achieved. Trials were performed at lab-scale using a continuous stirring tank reactor (CSTR) with 12 L of working volume (Fig. 1), under mesophilic regime (36 ± 1 °C).

Operating Procedures and Process Monitoring

The AcoD performance was accessed based on several operational parameters, such as the gas production rate (GPR, mL_{biogas} $mL_{reactor}$ day^{-1}), methane content (% CH_4) and specific methane production (SMP, $mLCH_4$ $g_{VSadded}^{-1}$), as well as on the efficiencies of VS removal obtained in each trial.

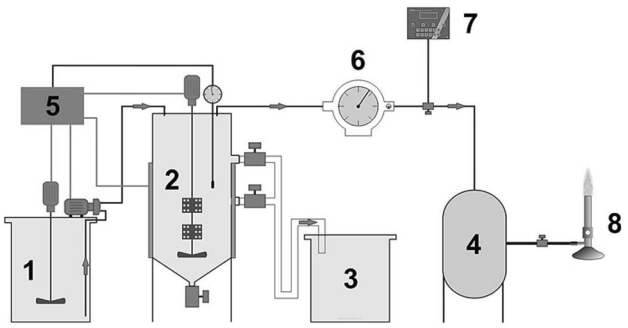

Fig. 1 Schematic of the AD lab-scale unit: 1—feeding mixture tank; 2—CSTR; 3—digestate collection tank; 4—gas holder; 5—control panel; 6—gas meter; 7—gas analyser; 8—flare system

GPR was measured daily using a gas meter (Schlumberger, Germany), and biogas composition in terms of methane (CH_4) and carbon dioxide (CO_2) was monitored weekly by an LMSxi Multifunction Gas Analyser (Gas Data, UK). Moreover, the stability of CSTR during the trials was controlled by monitoring TA, digestate pH and the specific energy-loading rate (SELR, day^{-1}). The SELR is a measure of energy loading (expressed as COD load) relative to the reactor biomass (expressed as VS content) and is a novel approach in characterising digester stability. The reactor is considered at stable conditions when SELR is below 0.4 day^{-1} [7, 31].

Statistical Analysis

All data was analysed by ANOVA at the 0.05 confidence limit followed by a Tukey's post-hoc test.

Results and Discussion

Table 1 presents the physicochemical composition of the substrate, co-substrate and feeding mixture used during the co-digestion trial.

As can be seen, LECB exhibited the highest SCOD concentration with a SCOD/TCOD ratio 19 times higher than SS, indicating the higher availability of organic matter. The VS content of LECB was significantly higher than the SS content, with significantly different VS/TS ratio. LECB C/N ratio was 3.4 times higher than SS.

Results regarding process performance and stability during the trials are presented in Table 2 and Figs. 2 and 3.

Comparing the performance of the two trials, it is noticeable that the increase in C/N, SCOD/TCOS and VS/TS ratios enhanced process yield. In fact, similar OLR at

Table 1 Physicochemical characteristics of the substrate, co-substrate and feeding mixture

	SS	LECB	Feeding mixture
TCOD (g O_2 L^{-1})	24.59 ± 6.70^a	39.29 ± 0.73^b	33.83 ± 4.12^a
SCOD (g O_2 L^{-1})	0.42 ± 0.19^a	14.70 ± 0.27^b	8.23 ± 0.19^c
SCOD/TCOD (%)	2^a	37^b	24^c
pH	5.97 ± 0.39^a	3.47 ± 0.42^b	4.84 ± 0.21^a
EC (mS cm^{-1})	10.28 ± 0.62^a	1.47 ± 0.14^b	3.51 ± 0.19^c
TS (g L^{-1})	19.59 ± 6.46^a	24.72 ± 0.99^a	20.61 ± 3.16^a
VS (g L^{-1})	15.24 ± 2.61^a	22.33 ± 1.04^b	16.87 ± 1.41^a
VS/TS (%)	78^a	93^b	82^c
TKN (g L^{-1})	1.21 ± 0.23^a	0.53 ± 0.03^b	0.92 ± 0.13^c
NH_4^+-N (g L^{-1})	0.41 ± 0.09^a	0.06 ± 0.02^b	0.36 ± 0.02^a
C/N	7^a	24^b	11^c

In each column, different letters indicate statistically different means (Tukey's post-hoc test; $P \leq 0.05$)

LECB liquor of exhausted coffee biowaste, *SS* sewage sludge, feeding mixture: 80% of sewage sludge and 20% of LECB, *TCOD* total chemical oxygen demand, *SCOD* soluble chemical oxygen demand, *EC* electrical conductivity, *TS* total solids, *VS* volatile solids, *TKN* total Kjeldahl nitrogen, *N-NH_4^+* ammonium nitrogen, *C/N* carbon/nitrogen ratio, *n* number of samples analysed = 4, ANOVA always significant at $P \leq 0.05$

Table 2 Parameters to assess process performance and stability along trials

Trial	T0 (n = 4)	T1 (n = 4)
T, ºC	35 ± 2.0^a	34 ± 1.0^a
OLR (g_{VS} $L_{reactor}^{-1}$ day^{-1})	0.89 ± 0.23^a	0.99 ± 0.25^a
Methane content (% CH_4)	59^a	63^b
SMP (mL $g_{VSadded}^{-1}$)	122 ± 52^a	276 ± 49^b
VS reduction (%)	59 ± 2^a	66 ± 4^b
TA (mg $CaCO_3$ L^{-1})	3436 ± 23^a	3712 ± 54^b
SELR (day^{-1})	0.21^a	0.33^b

In each column, different letters indicate statistically different means (Tukey's post-hoc test; $P \leq 0.05$)

T0—reference scenario; T1—trial using 80% of SS and 20% of LECB; *OLR* organic loading rate, *SMP* specific methane production, *VS* volatile solids, *TA* total alkalinity, *SELR* specific energy loading rate, *n* number of samples analysed, ANOVA always significant at $P \leq 0.05$

the same HRT promoted significantly different SMP (three times higher). The significant improvements observed, with the addition of LECB, prove the synergistic effect between biowaste and SS.

As can be seen from Fig. 2, SMP increased continuously during the trials indicating good adaptation and process improvement. Removal of VS corroborates the increase in bioconversion efficiency during the experiment, with 12% higher VS removal in T1 when compared with T0.

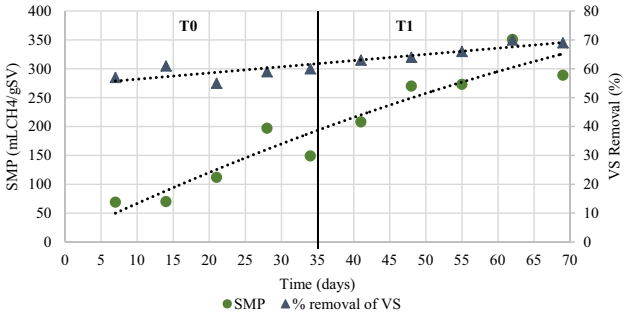

Fig. 2 Specific methane production (SMP) and volatile solids (VS) removal along trials T0 and T1

Previous research using sewage sludge and other co-substrates achieved a similar trend [5, 6, 32, 33], attesting that the bioconversion process was improved with the addition of co-substrates, once they were complementary in several parameters such as the EC, SCOD, C/N.

In Fig. 3 it is possible to see the evolution of pH, EC and ammonia nitrogen of feed and digestate along T0 and T1 trials. These expeditious parameters are relevant for process control.

Regarding pH, digestates presented values in the range 7.03–7.86, which shows a good adaptation even for the more acidic feeding in T1. This indicates the existence of sufficient alkalinity to neutralise the volatile fatty acids (VFA) produced during the process. Concerning ammonia, both digestates had values below the recommended limit of 1.7–1.8 g L^{-1} [34]. Furthermore, the digestate from the AcoD trial shows lower EC and N-NH_4^+ than the one from the mono-digestion trial, which is clearly an advantage for further use of digestate.

The above pointed stability is reassured by the alkalinity content (TA) observed during the trials (3436 ± 23 and 3712 ± 54 mg $CaCO_3$ L^{-1} for T0 and T1, respectively) being in the recommended interval for assuring a stable process (1500 and 6000 mg $CaCO_3$ L^{-1}) [32].

As previously stated, the SELR is a novel approach in characterising digester stability due to being a measure of energy loading (expressed as COD load) relative to the reactor biomass (expressed as VS content). According to Pinto et al. and Mcpherson [7, 31], the reactor is considered at stable conditions when SELR is below 0.4 day^{-1}. Therefore, the SELR values presented in Table 2 (0.21 for T0 and 0.33 for T1) support the stability of the CSTR during the trials.

In order to estimate the potential of energy recovery from the bioconversion of SS and LECB, the results were translated into energy considering the methane lower heating value (LHV) as 35.8 MJ m^{-3}. This way, the global methane produced during trial T0 allowed the recovery of 1.5 MJ, whereas in T1, 4.0 MJ were recovered. Therefore, the addition of a more bioavailable substrate as LECB helped SS

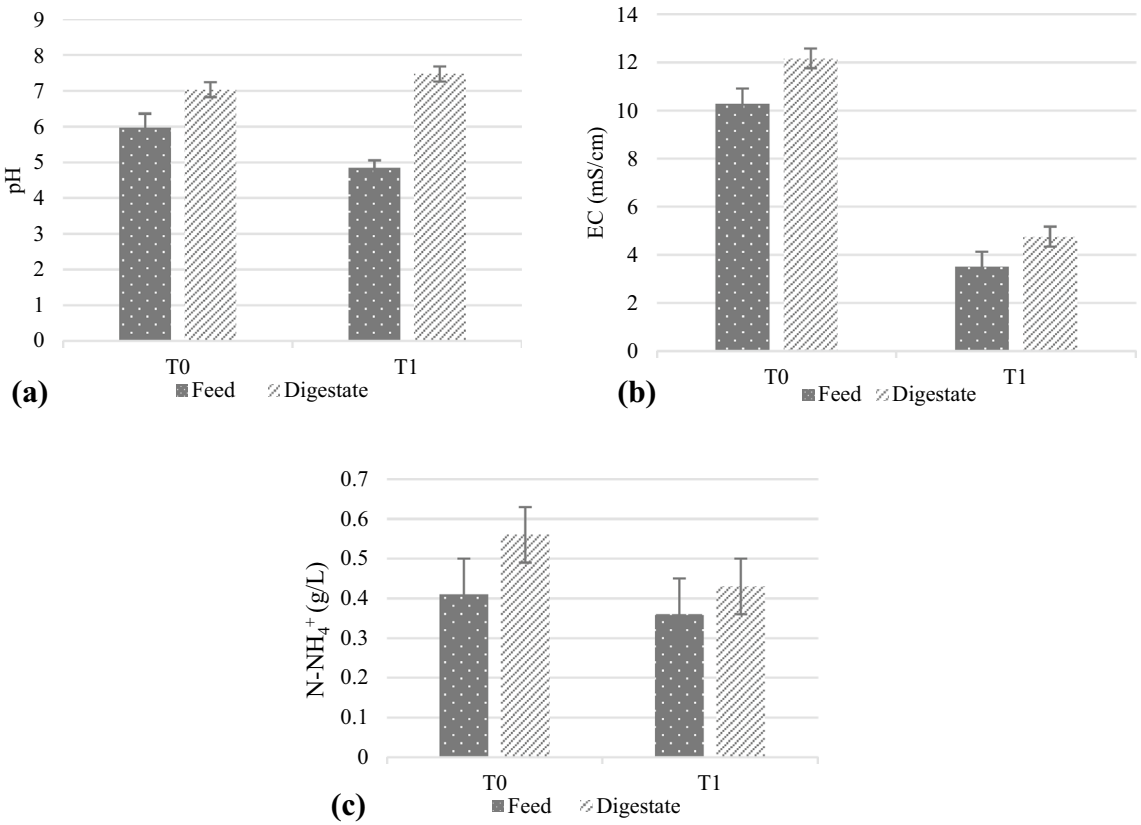

Fig. 3 Feed and digestate pH (**a**), EC (**b**), and ammonia nitrogen along T0 and T1 trials (**c**)

conversion to biomethane, recovering 3 times more energy. It is important to point out that in this work the net energy can be considered similar to the recovered energy, as the energy input for the pre-treatment of ECB (use of vacuum pump) and for heating the reactor accounts for a small share.

Conclusion

This study supports the implementation of anaerobic co-digestion of SS and LECB as a strategy towards an enhanced bioconversion process. Slow hydrolysis remains a vital constraint on the recovery of chemical energy from SS. Therefore, it is crucial to find alternatives to enhance this step. It was possible to conclude that AcoD process using a feeding mixture of 80% SS and 20% LECB (T1), keeping the same hydraulic retention time and similar organic loading rate, was optimised compared to the reference scenario (T0) using SS as mono-substrate. In fact, the addition of LECB increased specific methane production (SMP) three times; this improvement is related to the higher biodegradability of the co-substrate that has a SCOD/COD ratio almost 19 times the one from SS. It was clearly demonstrated that the feeding mixture composition had a significant influence on methane yield, suggesting that the co-substrate balances the low SS bioconversion, contributing to a better WWTP energy balance. However, it is important to highlight that further studies should be performed to test the viability of this mixture during a longer operation time (more HRT), to prove the feasibility of LECB as a co-substrate.

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