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Energy Recovery from Food Industry Sludge through Anaerobic Digestion

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Sludge generated in three Portuguese food processing companies - chestnut, dairy and fruit-cereal – were anaerobically digested, over 77-day experimental time, under batch mode and mesophilic conditions of temperature, to assess the anaerobic digestion process applicability to the energetic valorisation of these organic material surpluses. The biogas production of 106-144 mL (STP conditions) and the methane content of 61-72% indicate that all tested sludge reserve an energetic potential through anaerobic process. Comparatively, the sludge digestion from the fruit-cereal processing industry provided the largest volume of biogas, while the milk-yogurt reached the highest methane yield amount (209 L CH_4 kg^{-1} $VS_{influent}$).
Anaerobic digestion is an alternative process that, according to the obtained results, can advantageously contribute to solving the problem of excess sludge in the food industry by converting it into a digestate for agricultural purposes and an energy carrier vector (biogas/methane).

1. Introduction

The debate about future environmental conditions is on the agenda and it is promoted by a growing concern with climate change and the lack of sustainability. Countless events that reflect the current degradation of Earth's environmental conditions such as pollution, the greenhouse effect and global warming are in place. These phenomena have origin in the continuous global increase in the population, the consequent intense use of fossil fuels as the main source of energy and the harmful management of waste (Ren et al., 2017).
In the European Union (EU), the food industry represents the most economically relevant industrial sector whose importance still tends to grow (Zhang et al., 2011). In Portugal, over 1 million tons of products from the dairy industry and derivatives were produced in 2017 (INE, 2019). Dairy processing sector has already been regarded as the most significant producer of wastewater, which is often associated with the growing annual milk production (Slavov, 2017). In addition, this industry has large water requirements for the process and cleaning operations and, consequently, higher volumes of wastewater are produced. Industrial sectors related to the fruit and vegetable preparation and preservation, generating in Portugal (in 2017) more than 46 thousand tons of products (INE, 2019), are being important producers of residues and by-products, such as peels, seeds and pomace, motivated the EU to establish new goals regarding the reduction of fruit waste amounts (Campos, Ricardo, Vilas-boas, Madureira, & Pintado, 2020).
Concerning the chestnut processing industry, in 2018, five European countries were on the list of the 10 largest chestnut producers and Portugal was the seventh largest producer, with a production of more than 34 thousand tons (FAOSTAT, 2018). Portuguese chestnut processing industry has been exporting substantial quantities of chestnuts, namely frozen peeled chestnut (Rosa et al., 2017) and this activity generated a positive social and economic impact (Silva et al., 2016).
Being the food industry one of the largest sectors producing effluents, it generates large volumes of sludge from the biological treatment of the resulting wastewater (Boguniewicz-Zablocka et al., 2019). The sludge can be discharged onto agricultural land. However, when large quantities of sludge are accumulated there is the risk of GHG production and difficulties may arise with the lack of sludge outflow and with its consequent improper storage (Belhadj et al., 2014).

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Changing the global paradigm becomes urgent in order to deal with the need to develop technologies for exploiting renewable resources and sources of energy while ensuring a clean and efficient waste management (Batista et al., 2017; Castellano-Hinojosa et al., 2018). The EU and the United Nations (UN) have developed policies, targets and financing structures to encourage the use of renewable energy sources such as biogas (Scarlat et al., 2018). Effectively, anaerobic digestion (AD) is a promising and sustainable technology, applicable to the treatment and energetic/agricultural valorisation of industrial effluents, such as the food sector, under the circular economy concept. The present work aims to evaluate the potential of the applicability of AD process on sludge generated in the food industry, taking as examples the dairy, chestnut and fruit and cereal processing industries.

2. Materials and methods

2.1 Substrate and Inoculum

The potential and efficiency of energy recovery through anaerobic digestion were evaluated in samples of liquid sludge resulting from wastewater treatment from Portuguese companies, such as a chestnut processing plant, located in the North (“Cs”); a dairy processing industry plant (“Ms”) and a fruit and cereals processing industry plant (“FC”) both located in the center of the country. The sludge from a Portuguese domestic effluent anaerobic digestion plant was used for inoculum (I, 30% v/v), at a substrate/inoculum ratio of 0.75, expressed in volatile solids.

2.2 Anaerobic digestion units: experimental set-up

The tests were carried out in batch reactors (71.5 mL of volume) in triplicate, operating under mesophilic temperature conditions (37±1 °C). The digestion units were monitored by chemical and chromatographic analysis and by daily recording the volume of biogas and periodically the methane content, expressed under standard conditions of pressure and temperature (STP: 0°C and 1 atm).

2.3 Chemical and chromatographic analysis

Analytical techniques allowed to determine several parameters for the process development evaluation, such as pH, total solids (TS), volatile solids (VS), chemical oxygen demand (COD), total nitrogen (Kjeldahl, TKN), and ammonium (NH_4-N), according to the Standard Methods manual (APHA, AWWA, 2005). Humidity was determined by drying at 105±5ºC by a gravimetric method. Total sugars were determined by acid hydrolysis in autoclave and determination by molecular absorption spectrometry (phenol-sulfuric reagent) in the hydrolysate (expressed in glucose). The volume of biogas produced was determined using a pressure transducer. The composition of the biogas, in terms of CH_4 and CO_2, was obtained by gas chromatography technique (Varian 430-GC, TDC; HP-5890, FID) according to the Standard Method ASTM [2000]. The characterization of the substrates and inoculum is shown in Table 1.

Table 1: Substrate and inoculum characterization.

	Cs	Ms	FC	I
Humidity. a.r.[1]	98.9%	83.7%	81.6%	n.d.[2]
VS (g/L)	10.1	40.5	188	22.9
COD (g/L)	12.8	133.6	248	103
TKN (g/L)	0.3	3.4	1.2	1.4
N-NH_4 (mg/L N)	<4	0.8	21	360
pH	6.3	5.9	3.8	6.9
Total sugars in the hydrolysate (in glucose). d.b.[3]	3019 mg/L	2.7%	95840 mg/L	n.d.[2]

[1]a.r. – as received; [2]n.d. – not determined; [3]d.b. – dry base. Cs – chestnut sludge, Ms – milk-yogurt sludge, FC – fruit and cereal sludge, I – inoculum

3. Results and Discussion

The results concerning the amount of gas accumulated during the AD process about each substrate are shown in Figure 1 while the biogas composition, methane yield and productivity data are presented in Table 2. All tested batch reactors displayed increasing biogas volumes over the experimental period without any visible lag phase.

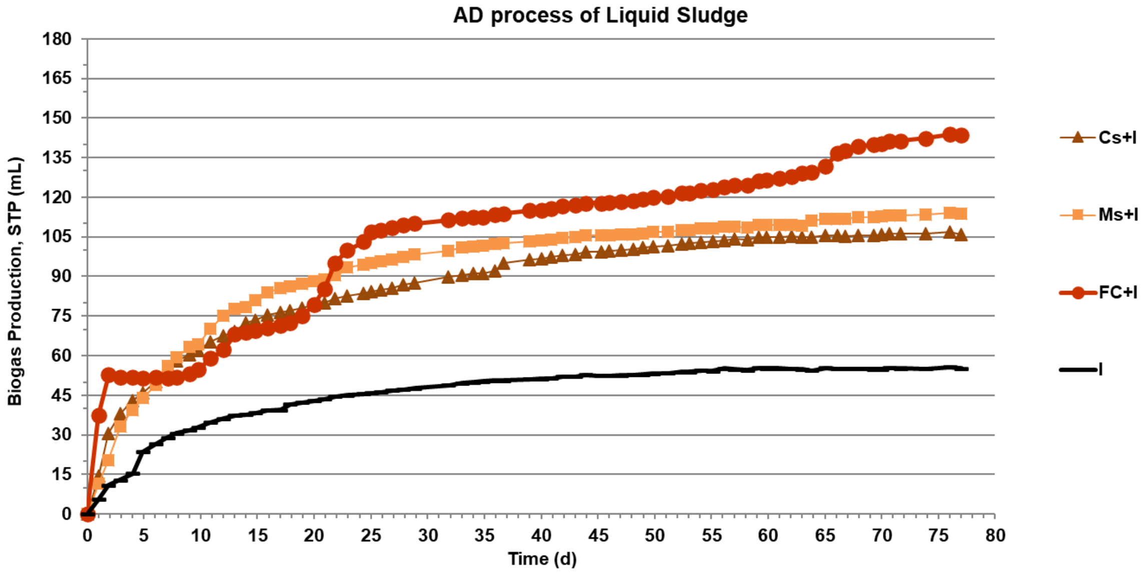

Figure 1: Volumetric biogas production registered for the chestnut sludge (Cs), milk-yoghurt sludge (Ms), fruit and cereal sludge (FC) and inoculum (I).

A more marked production progress was observed in the first testing month than in the remaining time (Figure 1). Control unit (I) exhibits a considerable biogas production due to the presence of some biodegradable material contained in the sludge that has not undergone to previous conversion. Comparatively, the tested samples provided higher biogas volumes than the I unit, showing that organic matter of substrates samples was degraded over the 77 days of experiment and converted into biogas.
FC+I reactors displayed the highest biogas production and are ranked second in terms of methane yield (143.6 mL and 190.5 L CH_4 $kg^{-1}VS^{-1}$, respectively, Table 2) comparing with the other substrates. At the beginning, FC+I reactors had a very intense biogas production, reaching high gas concentrations that required the immediate unit's depressurization (Figure 1).
This may be the consequence of a rapid hydrolysis stage due to the substrate composition. Effectively, the substrate characterization prior to this experiment (Table 1) revealed a relatively high concentration of total sugars (glucose) in the hydrolysate of FC substrate. According to Ji et al. (2017), fruit residues are considered to be one of the most suitable substrates for AD as they are easily biodegradable with high moisture. However, just after this initial peak, a sharp decrease in biogas production was observed and the curve adopted a similar behaviour to a lag phase but in two steps (Figure 1). A fast hydrolysis phase could have provided a rapid medium acidification and a consequent decrease in pH values, due to the accumulation of intermediate VFAs that led to a biogas and methane production inhibition (Ji et al., 2017). This is represented by the graphical steps portrayed in Figure 1 for the FC+I curve. After this stage, methane proportions started to increase with the biogas production once the process has recovered.

Table 2: Summary of the biogas and methane production through anaerobic digestion for each substrate.

	Cs+I	Ms+I	FC+I
Biogas production (mL)	105.8±3.6	113.6±0.1	143.6±4.2
CH_4 (%)	63.3%	70.4%	52.7%
CH_4 production (mL)	67.0	80.0	75.7
Methane yield (L CH_4 kg^{-1} $VS_{influent}$)	152.4±5.5	209.3±0.1	190.5±6.1

Concerning the Ms+I units, they presented higher biogas production than Cs+I (Figure 1) and the highest methane yield (Table 2). In a quick first inspection, this seems to be a surprising result because Ms+I solutions contained lower initial concentrations, mainly for COD, compared to the Cs+I solutions (Table 3).

However, regarding the Cs substrate composition, it derives from a chestnut processing industry and therefore constitutes a waste that may contain phenolic compounds/lignin. It is known that these types of compounds are inhibitory and recalcitrant components with the ability to delay the AD process. In relation to Ms, because it is a sludge derived from a dairy industry, it may contain relevant amounts of lipids. Lipid macromolecules have higher energy potential than proteins and carbohydrates and, accordingly, lipids provide higher volumes of biogas with higher fraction of methane. In fact, Ms+I displayed the highest methane yield of the whole experiment.

Table 3: Batch reactors initial and final content characterization and their respective removal capacity.

		pH	COD (g/L)	TS (g/L)	VS (g/L)	N-NH_4 (mg/L)	TKN (mg/L)
Cs+I	Initial	6.81	95.8±9.1	13.2±0.2	10.3±0.2	87±0	423±4
	Final	7.75	13.9±1.1	11.0±0.2	8.1±0.1	189±2	437±0
	Removal	-	85%	16	21	-118	-3
FC+I	Initial	6.70	82.3±0	12.3±0.2	9.6±0.1	71±6	381±24
	Final	7.67	10.1±0	8.5±0.2	5.9±0.2	167±2	378±4
	Removal	-	88%	31	38	-133	1
Ms+I	Initial	7.01	77.6±1.7	12.0±0	9.1±0.1	105±2	484±12
	Final	7.75	11.1±0	9.4±0.1	6.4±0.1	235±0	454±8
	Removal	-	86%	22	29	-124	6

Cs+I – chestnut sludge+inoculum, Ms+I – milk-yogurt sludge+inoculum, FC+I – fruit and cereal sludge+inculum.

The observation of Table 3 reveals that removal percentages did not vary much among the various sludge digestion processes, although the differences seem to follow trends in biogas production. Reactors with the slightly higher removal values were the ones with more biogas volume. This was the case of FC+I reactors, which provided the best removal values obtained - 88, 31 and 38% in the COD, TS and VS, respectively – evidencing the high digestibility of the substrate and its repercussion in methane production and yield (Table 2). The lowest removal capacity was observed in the Cs+I units that presented values of 85% (COD), 16% (TS) and 21% (VS), however, considering the absolute value, it was noticed that Cs+I digestion units managed the elimination of the greatest fraction of the initial COD content. Amounts of 82 g/L COD (Cs+I), against 72 and 67 g/L COD (FC+I and Ms+I, respectively) were registered. The pH that initially presented values slightly below the neutral but became basic during the process, indicating that beneficial adjustments occurred on the environmental conditions of the microorganisms, especially regarding archaea populations. This behaviour also enabled the development of the process. Increases in ammonium concentrations at the end of each testing process showed that organic matter was degraded and, consequently, validated the presence of a balanced population capable of providing this conversion.

Table 4. Methane yield and energy potential of biomass and organic waste usable as raw material in AD

Biomass	Methane Yield (L CH_4 kg^{-1} $VS_{influent}$)	Energetic Value (KWh kg^{-1} $VS_{influent}$)	Reference
Cs+I	152.4±5.5	1.52±0.06	Present work
FC+I	190.5±6.1	1.90±0.06	
Ms+I	209.3±0.1	2.09±0.01	
Organic fraction of municipal solid waste	100-400	6.18	(Roati et al., 2012)
Dairy sludge	200	n.d.	
Coffee husk	159	1.78	(Chala et al., 2018)
Potato pulp	250-400	2.49-3.99	
Vegetable waste	400	3.99	
Corn straw	174	0.66	(FNR, 2020)
Pasture	153	0.58	
Rice husk (without pre-treatment)	44	4.36	(Solarte-Toro et al., 2018)
Rice husk (with pre-treatment)	56	n.d.	

The methane yields obtained in this work (150-200 L CH_4 kg^{-1} $VS_{influent}$) are of the same order of magnitude as found in other substrates tested in other studies (Table 4), such as municipal waste, coffee husk, corn straw or pasture. The best methane yield provided by Ms+I units, 209 L CH_4 kg^{-1} $S_{influent}$, is very close to that reported by Roati et al. (2012), 200 L CH_4 kg^{-1} $S_{influent}$, digesting analogous dairy substrates. Similar observations can be made comparatively regarding the energetic value provided by the sludge digestion of food industries (Table 4).
The interest of the proposed work is reinforced by the performance of the units to digest the raw sludge in its original state, which means, without the application of any correction of the substrate or pre-treatment. From the obtained data, the sludge generated in dairy, chestnut and fruit-cereal processing industries, tested as examples of food sector, hold a potential for energetic valorization by anaerobic digestion.

4. Conclusions

Sludge from Portuguese food processing companies - chestnut, dairy and fruit-cereal – has potential to be treated/valorised by anaerobic digestion as productions of biogas of 106, 114 and 144 mL, with methane content of 63, 70, 53%, were registered in the digestion process of each sludge, respectively. The corresponding methane yields reveal a greater efficiency of the processes operating with dairy and fruit-cereal sludge (209 and 191 L CH_4.kg^{-1} $VS_{influent}$, respectively) than with the chestnut sludge which, comparatively, has been reached only 152 L CH_4.kg^{-1} $VS_{influent}$. The lower efficiency of the chestnut process was related to the recalcitrant and even inhibitory characteristics of the substrate in digestion, when compared to the other two.
These exciting data deserve further research in order to better define the operating conditions that will optimize the process of valorising the food industry surplus sludge by anaerobic digestion.

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