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STUDY ON THE EFFECT OF THERMAL PRE-TREATMENTS IN THE THERMOPHILIC ANAEROBIC DIGESTION OF A POTATO PEEL WASTE

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ABSTRACT

This work aimed to study the effect of different pre-treatments applied to a potato peel residue, in a thermophilic Anaerobic Digestion (AD) process. All samples were subjected to a mechanical pre-treatment through milling to a particle size below 2 mm. The thermal pre-treatments applied consisted of autoclaving the residue at a gauge pressure of 1.2 bar, under a temperature of 122°C, and for 20, 35 and 55 minutes: assays E122.20, E122.35 and E122.55, respectively. The control assay was performed on a ground residue, which was not submitted to any thermal pre-treatment. All pre-treated residues were subjected to an AD process in a CSTR reactor at 49±1°C. The experimental data showed that the highest methane percentages were very similar (about 92% v/v) for all samples submitted to the thermal pre-treatments. For the control assay, the highest percentage of methane was 87.9% (v/v). The highest biogas yields were recorded in the trial E122.35 (646±50 cm³.g⁻¹ COD_{removed}), against only 250±20 cm³.g⁻¹ COD_{removed} for the control assay. The highest biogas yields for VS_{removed} were attained in the assays E122.55 and E122.35, with values of 646±48 cm³.g⁻¹ VS_{removed} and 634±59 cm³.g⁻¹ VS_{removed}, respectively. Globally, the yields registered for the assay E122.35 were similar to those determined in the assay E122.55. Due to the lower energy consumption during the pre-treatment performed in the assay E122.35, this was considered to be the most suitable pre-treatment for this type of residue.

KEY WORDS: Anaerobic digestion; Biogas and Methane yields; Potato peel waste; Thermal pre-treatments.

INTRODUCTION

Among the organic wastes that can be submitted to AD it is possible to find the biodegradable residues of agriculture activities and food processing industries (Bouallagui *et al.*, 2005). According to Khalid *et al.* (2011) and Zhang *et al.* (2013), food wastes are considered a very attractive feedstock for AD, due to their high methane yields. The AD produces different products that have a high potential of valorization. For instance, the gaseous fraction – biogas – can be used to produce different forms of energy (electricity, heat, mechanical energy, among others) (Bouallagui *et al.*, 2005; Nasir *et al.*, 2012). According to Braun *et al.* (2011), biogas is a renewable source of energy, because it is obtained from a renewable source of carbon.

The AD comprises four metabolic steps: hydrolysis, acidogenesis, acetogenesis and methanogenesis. In the first step it occurs the biological decomposition of organic polymers to dimers or monomers. Mechanical, thermal, chemical and biological pre-treatments, and different combinations of them, have

been studied in several research works with the aim to increase the biodegradability of organic substrates, enhancing biogas yields and shorten the hydraulic retention times. These treatments cause the lysis of cells, allowing the release of intracellular material that becomes available to the anaerobic microorganisms. The overall efficiencies of the pre-treatments are usually evaluated through the increment of biogas yields and by the methane percentage in the biogas produced (Climent *et al.*, 2007).

A wide range of temperatures, from 60°C to 270°C (Bordeleau and Droste, 2011), had been tested in pre-treatment processes with the objective to improve the wastes digestibility, but also to improve the properties of dehydration (Kim *et al.* 2003). However, the most common thermal pre-treatments are in the range of 60°C to 180°C, since temperatures above 200° C are responsible for the formation of refractory, toxic or inhibitory compounds (Ferrer *et al.*, 2008; Bordeleau and Droste, 2011; Rodriguez-Abalde *et al.*, 2011). According to Nielsen *et al.* (2004), temperatures below 100° C are recommended in the pre-digestion steps because they increase the hydrolytic activity of certain bacteria. Valo *et al.* (2004) reported that pre-treatments at 70° C are effective to increase the biological activity of thermophilic bacteria.

Food processing industries, such as those producing chips and French fries, generate huge amounts of residues, among which are the potato peel wastes (Braun *et al.*, 2011). The biodegradability of these residues is reduced, which according to Mahmood *et al.* (1998) can be attributed to the high content of alcohol insoluble substances that are composed by pectin, cellulose, protein, starch, and ashes.

The main objective of this work was to study the effect of different thermal pre-treatments in biogas and methane yields produced in a CSTR laboratory scale unit during the thermophilic AD of a potato peel waste.

MATERIAL AND METHODS

Potato peel residue was obtained from a potato processing industry located in Central region of Portugal. This residue was characterized for different chemical parameters according to the methods of the American Public Health Association, American Water Works Association, and Water Environment Federation (APHA, AWWA and WEF, 1999), and for elemental composition according to the method CEN/TS 15104 (LECO analyzer). The residue was submitted to a milling process (mechanical pre-treatment) in order to reduce the particle size to a dimension <2 mm: 200±1 g of the potato peel residue was ground with 500 mL of deionized water (Millipore, Elix 5) in a blade mill (Philips). The thermal pre-treatments to which the ground potato residue was submitted consisted of autoclaving the residue at a gauge pressure of 1.2 bar, under a temperature of 122°C, for 20 min (E122.20), 35 min (E122.35), and 55 min (E122.55) (Table 1).

Table 1: Summary of the pre-treatments to which the potato peel waste was submitted.

Assay code	Mechanical pre-treatment (milling process)	Thermal pre-treatment (autoclaving)
EC (control)	yes	no
E122.20	yes	1.2 bar, 122°C, 20 minutes
E122.35	yes	1.2 bar, 122°C, 35 minutes
E122.55	yes	1.2 bar, 122°C, 55 minutes

The pre-treated potato peel residues were then submitted to a thermophilic AD process, at 49±1°C, in a CSTR, with a working volume of 2.1 L. The CSTR was inoculated with an active population coming from an industrial thermophilic digester (52°C) that is operated with the organic fraction of municipal solid wastes. Two replicates of each anaerobic digestion assay were performed.

The organic substrate is continuously mixed inside the CSTR with the biological active population by two paddle systems that work at 10 rpm. The reactor content is permanently warmed up by a thermostatic band, which is controlled by a thermocouple and a controlling unit. The biogas produced is directed to an acrylic cylinder (biogas reservoir), which is filled up with deionized water (Millipore, Elix 5). The biogas height is measured by a metallic calibrated scale. Samples of biogas are drawn off from the upper port of the biogas reservoir for the quantification of CH₄, CO₂, H₂, H₂S, and O₂ (Gas

Data, GFM Series 410; CH₄, and CO₂ determined by infrared sensors; H₂, H₂S, and O₂ determined by electrochemical sensors).

The pH inside the CSTR was permanently adjusted with NaOH (6N) to a value of 8.55±0.31 through a pH meter and a pH controlling unit coupled to an acid and alkaline peristaltic pumps. To avoid a temperature change inside the CSTR during the feeding step, the pre-treated residues were pre-heated in a thermostatic water bath up to a temperature of 51°C. The CSTR was then fed by a two-way peristaltic pump. This procedure kept constant the temperature inside the CSTR at 49±2° C. The peristaltic pump flowed in the pre-treated residues and flowed out an equal volume of the liquid mixture present inside the CSTR digester. In each assay, the digester was fed at once, and the anaerobic digestion cycle was allowed to develop under batch condition up to no biogas has been produced during 3 consecutive days. Each anaerobic cycle lasted for 10 to 14 days. In each feeding cycle, samples of the influent and effluent were collected for the characterization of several chemical parameters, among which were COD and VS. COD was determined according to the Method 5220 B – COD, Open Reflux Method – of the American Public Health Association, American Water Works Association, and Water Environment Federation (APHA, AWWA and WEF – Method 5220 B, 1999). SV were determined according to the Method 2540 E – Fixed and Volatile Solids Ignited at 550°C – of the same organizations (APHA, AWWA and WEF – Method 2540 E, 1999).

The volume of biogas and methane were determined according to the equations 1 and 2, respectively.

$$V_{biog} = \pi \cdot r^2 \cdot h \quad (\text{eq. 1})$$

where V_{biog} represents the biogas volume (cm³), r stands for the inner radius of the biogas reservoir (cm), and h is the height of the biogas inside the reservoir (cm).

$$V_{CH_4} = \frac{\%_{CH_4} \times V_{biog}}{100} \quad (\text{eq. 2})$$

where V_{CH_4} is the volume of CH₄ (cm³), $\%_{CH_4}$ represents the percentage of CH₄ in biogas (% v/v) and V_{biog} is as defined above.

The removal efficiencies of COD and VS were determined according to the equation 3.

$$\eta_y = \frac{\bar{x}_{influent} - \bar{x}_{effluent}}{\bar{x}_{influent}} \quad (\text{eq. 3})$$

where η_y is the removal efficiency of COD or SV (dimensionless), $\bar{x}_{influent}$ is the mean concentration of COD or SV in the influent of the CSTR (g COD.L⁻¹, or g SV.L⁻¹) and $\bar{x}_{effluent}$ is the mean concentration of COD or SV in the effluent of the CSTR (g COD.L⁻¹, or g SV.L⁻¹).

The biogas and CH₄ yields based on the COD or SV removed were determined according to the equation 4.

$$Y_{x/y} = \frac{V_x}{L_y \times V \times \eta_y} \quad (\text{eq. 4})$$

where $Y_{x/y}$ is the biogas or CH₄ (x) yield for COD or SV (y) (cm³ biogas.g⁻¹ COD_{removed}, cm³ CH₄.g⁻¹ COD_{removed}, cm³ biogas.g⁻¹ SV_{removed}, or cm³ CH₄.g⁻¹ SV_{removed}), V_x is the volume of biogas (V_{biog}) or methane (V_{CH_4}) (cm³), L_y is the volumetric load of COD or SV applied to the CSTR in each assay (g COD.L⁻¹_{reactor} or g SV.L⁻¹_{reactor}), V is the volume of the CSTR (L) and η_y is as defined above.

RESULTS AND DISCUSSION

Table 2 shows the average chemical composition of the potato peel residue, including its elemental composition. The residue was characterized by a high content of water (88.8±1.8% w/w wb). The solid fraction (11.2±1.8% w/w wb) was mainly composed by VS (10.6±1.9 % w/w wb) and a low fraction of fixed solids (0.58±0.13% w/w wb). In what concerns the organic fraction, the residue was

characterized by a high COD ($1730 \pm 293 \text{ g.kg}^{-1} \text{ db}$), but the biodegradable fraction (BOD_5) was very low ($11.6 \pm 2.7 \text{ g.kg}^{-1} \text{ db}$) when compared with the total organic content. The biodegradability of this residue, measured through the BOD_5/COD ratio, was very low (0.007). Therefore, to subject this residue to a pre-treatment might be useful to improve its biodegradability. Other authors have reported a similar chemical composition of potato peel wastes to those referred in Table 2. For example, Raynal *et al.* (1998) indicated TS and VS concentrations of 119.2 g.kg^{-1} and 105.5 g.kg^{-1} , respectively. Schieber and Saldaña (2009) reported that this type of residues can present up to 15.1% of volatile materials. Kryvoruchko *et al.* (2009) referred C-contents of 41.6% and 45.8% (w/w db) for a potato pulp residue and a mixture of potato pulp and peel residues, respectively.

Table 2: Chemical characterization of potato peel residue ($\bar{x} \pm \sigma$; n=3; wb: wet basis; db: dry basis).

Parameter	Unit	Value
Moisture content (at $103 \pm 2^\circ\text{C}$)	% w/w wb	88.8 ± 1.8
Total Solids, TS (at $103 \pm 2^\circ\text{C}$)	% w/w wb	11.2 ± 1.8
Total Fixed Solids, FS (at $550 \pm 50^\circ\text{C}$)	% w/w wb	0.58 ± 0.13
Total Volatile Solids, VS (at $550 \pm 50^\circ\text{C}$)	% w/w wb	10.6 ± 1.9
COD	$\text{g.kg}^{-1} \text{ db}$	1730 ± 293
BOD_5	$\text{g.kg}^{-1} \text{ db}$	11.6 ± 2.7
BOD_5/COD	dimensionless	0.007
pH (200±1 g : 500 mL water)	Sørensen	5.9 ± 0.61
Elemental composition		
Total Fixed Solids, FS	% w/w db	5.18 ± 1.16
C	% w/w db	49.4 ± 1.1
H	% w/w db	6.7 ± 0.4
N	% w/w db	2.5 ± 0.2
S	% w/w db	0.20 ± 0.08
P	% w/w db	0.30 ± 0.09
O + errors	% w/w db	35.72
C/N	dimensionless	19.8:1
C/N/P	dimensionless	164.7:8.3:1

Kryvoruchko *et al.* (2009) reported a C/N ratio of 23.4:1 for a potato pulp residue, and of 12.1:1 in the mixture of potato pulp and peel residues. The residue used in the present work have presented a C/N ratio (19.8:1) more close to the value reported by Kryvoruchko *et al.* (2009) for the potato pulp residue. The C/N/P ratio (164.7:8.3:1) was a little bit lower than that referred by Bouallagui *et al.* (2003) (200:9.2:1) for residues of fruits and vegetables.

Figure 1 shows the variation of the mean values and standard deviations of Temperature, pH and Redox potential in the assay E122.35. These data are shown as an example, as the variations of these parameters determined for all the other assays were similar to these depicted in Figure 1.

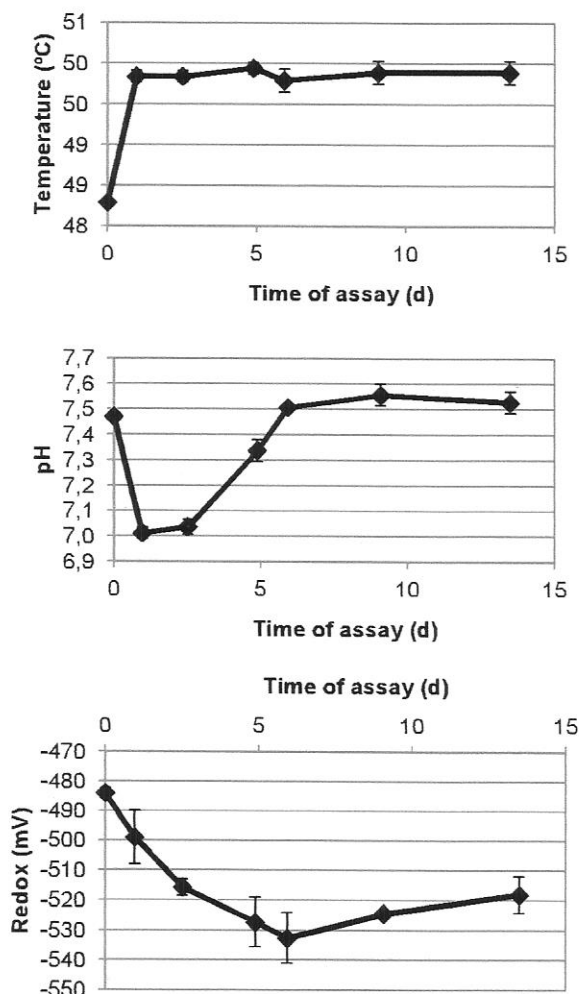


Figure 1: Variation of the mean values and standard deviations of Temperature, pH and Redox potential in the assay E122.35.

Although the pre-treated potato peel residue had been pre-heated before its introduction in the CSTR, an initial Temperature decay of about 1.5°C was registered for all the assays. Nevertheless, this did not affect substantially the biological population, as the biogas production started to occur in a couple of hours after the digester has been fed.

The initial pH decay, in the first 24 h of all the assays, can be attributed to the development of the fermentation processes that intensively produced CO₂ and volatile fatty acids (VFA) which can acidify the medium. The increase of pH, after 48 h of all the assays, was due to both the decrease of the fermentation processes and to the effect of automatic correction of pH with NaOH (6N).

After the feeding process of the CSTR, the Redox potential showed a decrease up to values of about -530 mV. The values of Redox potential were in the range of -520 to -540 mV in all assays, which maintained the reduction conditions adequate to methanogenesis.

The fermentation processes that took place in the initial 48 h of the assays are clearly represented in Figure 2 for the assay E122.35, as the mean CO₂ percentage increased up to a percentage of 41.6% (v/v) during this time of the assay. A similar trend was registered for the other anaerobic digestion assays. After the first 24 h, the percentage of CH₄ reached percentages of about 60% (v/v) and continued to increase up to the end of the assays in which percentages of about 90% (v/v) were commonly detected. The anaerobic conditions were kept all over the assays, once the O₂ percentage was always around 0.0% (v/v).

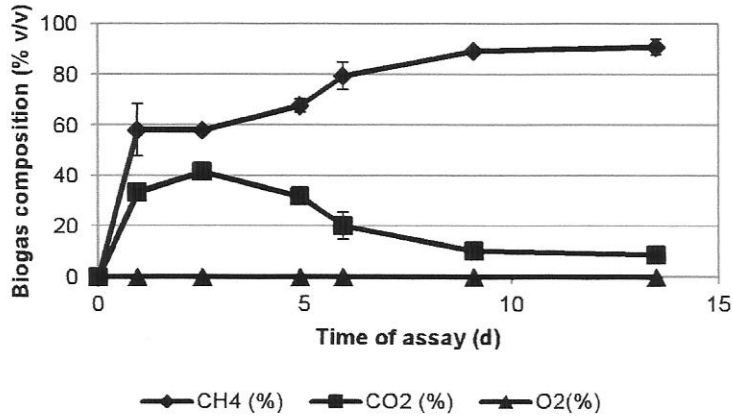


Figure 2: Variation of the mean percentages and standard deviations of CH₄, CO₂ and O₂ in the biogas produced in the assay E122.35.

Figure 3 shows the mean concentrations of H₂S, CO and H₂ in the biogas produced in the assay E122.35.

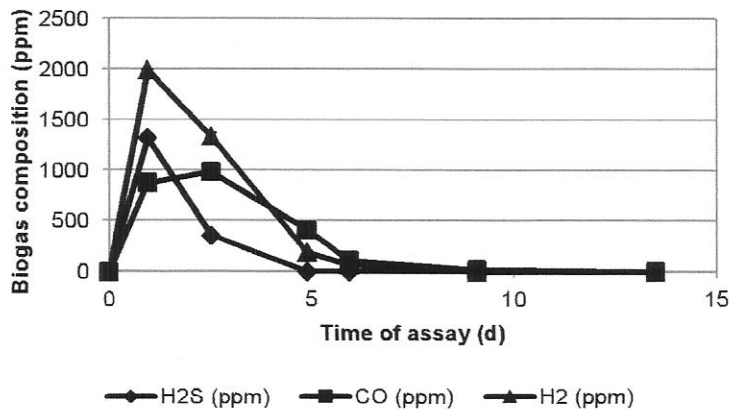


Figure 3: Variation of the mean concentrations and standard deviations of H₂S, CO and H₂ in the biogas produced in the assay E122.35.

During the most intensive step of the fermentation processes (24-48 h), it was registered the production and release to the biogas of H₂, H₂S and CO. Globally, in all the assays, the concentrations were in the range of few hundreds to few thousands of ppm. After 4 to 5 days of the assays, these gases were no longer detected. The highest concentrations of H₂S did not exceed 2000 ppm.

Figure 4 shows an example of the curves of accumulated volumes of biogas and CH₄ obtained in the assay E122.35. Similar curves were detected in the other anaerobic digestion assays. The main difference was registered on the total accumulated volumes registered at the end of the assays.

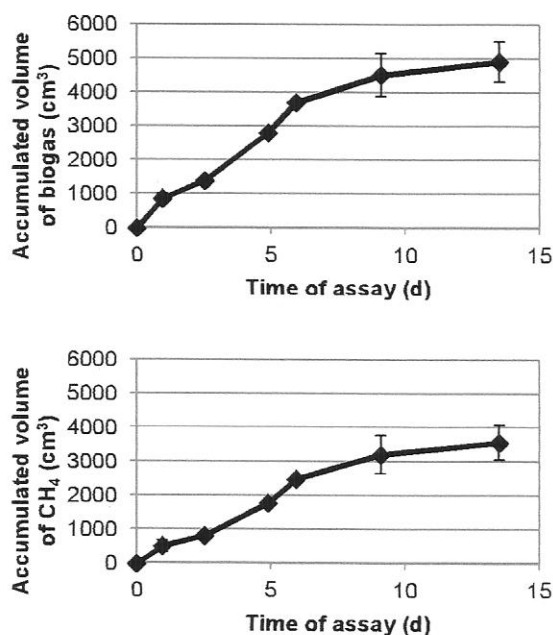


Figure 4: Accumulated volumes of biogas and CH₄ registered in the assay E122.35.

As can be seen in the curves of Figure 4, the production of biogas and CH₄ was more intensive in the first 6 days of the assays, and has decreased significantly in the next experimental days. In the assays in which a thermal pre-treatment process was applied to the potato peel residue, the accumulated volume of CH₄ represented about 68% (v/v) of the accumulated volume of biogas for this first 6 days of assay. At the end of the assays, the accumulated volume of CH₄ was about 90% (v/v) of the accumulated volume of biogas, but the production rate of biogas was very low after 6 days of anaerobic digestion.

Table 2 presents a summary on the accumulated volumes and yields of biogas and methane, and the highest percentages of CH₄ attained for all the anaerobic assays.

Table 2: Accumulated volumes and yields of biogas and CH₄ and highest concentrations of CH₄ in all the anaerobic digestion assays ($\bar{X} \pm \sigma$; n=2).

Assay	Biogas accumul. volume (cm ³)	Methane accumul. volume (cm ³)	Biogas yield (cm ³ ·g ⁻¹ COD _{removed})	Methane yield (cm ³ ·g ⁻¹ COD _{removed})	Biogas yield (cm ³ ·g ⁻¹ VS _{removed})	Methane yield (cm ³ ·g ⁻¹ VS _{removed})	Highest concent. of CH ₄ (% v/v)
EC	4413±890	3034±557	250±20	182±19	370±34	269±31	87.9±1.1
E122.20	4046±49	3318±105	638±66	524±64	531±21	435±8	93.4±0.1
E122.35	4921±592	3580±514	646±50	470±48	634±59	461±54	91.0±3.0
E122.55	5402±929	3932±802	633±29	460±36	646±48	470±50	92.9±2.1

As can be seen in Table 2, the accumulated volume of biogas reached the highest value (5402±929 cm³) in the assay E122.55, followed by the assay E122.35 (4921±592 cm³). Therefore, it can be concluded that the autoclaving process and the increase in the autoclaving time have enhanced the production of biogas. Compared to the control assay, this increase was of 22.4% in the assay E122.55 and of 11.5% in the assay E122.35.

In what concerns the accumulated volume of methane, the highest value was registered in the assay E122.55 (3932±802 cm³), representing an increase of 29.6% when compared to the control assay.

Both the autoclaving process and the increase of autoclaving time had again a positive effect in the production of methane.

The highest biogas yields for COD removed were obtained for the assays E122.35 and E122.20, with values of $646 \pm 50 \text{ cm}^3 \cdot \text{g}^{-1}$ and $638 \pm 66 \text{ cm}^3 \cdot \text{g}^{-1} \text{ COD}_{\text{removed}}$, respectively. The biogas yields in the assay E122.35 were as high as 158% of the mean value determined in the control assay.

The highest results for methane yield were obtained in the assay E122.20 with $524 \pm 64 \text{ cm}^3 \cdot \text{g}^{-1} \text{ COD}_{\text{removed}}$, followed by the assay E122.35 with $470 \pm 48 \text{ cm}^3 \cdot \text{g}^{-1} \text{ COD}_{\text{removed}}$. The pre-treatments have improved the methane yields in 254% and 158% for the assays E122.20 and E122.35, respectively, when compared to the control assay.

The highest values of biogas yield for VS removed was found in the assays E122.55 and E122.35 with values of $646 \pm 48 \text{ cm}^3 \cdot \text{g}^{-1} \text{ VS}_{\text{removed}}$ and $634 \pm 59 \text{ cm}^3 \cdot \text{g}^{-1} \text{ VS}_{\text{removed}}$, respectively. Comparing to the control assay, the biogas yield increased 74.6% and 71.3% in the assays E122.55 and E122.35, respectively.

Concerning the methane yields for VS removed, the results were very close to each other in the three assays in which the thermal pre-treatment was applied. The assays increased the methane yield of about 71%.

Regarding the highest methane concentrations in the biogas, all the percentages were very similar (about 92% v/v), and they were attained almost at the same time in each assay, i.e. at the end of the assays (10 to 14 days). This means that the autoclaving time played no significant role in the methane percentage in the biogas. Nevertheless, the autoclaving process was important to increase the methane percentage, when compared to the control assay. In the latter, the highest methane percentage was only of 87.9% (v/v).

Qiao *et al.* (2011) studied the anaerobic digestion of fruit and vegetables wastes that were subjected to a thermal pre-treatment of 170°C, for 1 hour, at atmospheric pressure. This pre-treatment increased the biogas yields by 18.5% (from 443 to 525 $\text{cm}^3 \cdot \text{g}^{-1} \text{ VS}_{\text{removed}}$). The results obtained in the present work were higher for all the assays in which a thermal pre-treatment process was used, probably because of the combined effect of temperature and pressure on increasing the lysis of potato peel cells.

Qiao *et al.* (2011) also registered an increase in methane yields by 16.1% (from 280.9 to 326.0 $\text{cm}^3 \cdot \text{g}^{-1} \text{ VS}_{\text{removed}}$). This increase is also lower than that obtained in the presented work for the assays with thermal pre-treatments.

Parawira *et al.* (2007) studied the optimum conditions to improve the methane yield in thermophilic digestion of potato wastes. The highest value was $0.31 \text{ L} \cdot \text{g}^{-1} \text{ COD}_{\text{removed}}$ which is lower than the values found in this work for E122.35 assay ($0.85 \text{ L} \cdot \text{g}^{-1} \text{ COD}_{\text{removed}}$). In the same study, the methane composition ranged from 54 to 74%. Once again, the values found in the present study were higher and reached 93.4% (v/v) for the assay E122.20, at the end of the 14 day anaerobic digestion cycle. On the contrary, Liu *et al.* (2012) reported the decay of 11.7% in methane production when fruit and vegetable wastes were submitted to 175°C, for 60 minutes, due to production melanoidins.

Globally, the best results of the accumulated volume of biogas and methane were registered for the assays E122.55 and E122.35. However, in practice, to use an autoclave at a gauge pressure of 1.2 bar, during 55 minutes, at 122°C, is an operation with an associated high cost when compared to the autoclaving process at the same pressure and temperature, but only for 35 minutes. Since the assay E122.35 also showed results as good as the assay E122.55 in terms of biogas and methane yields, the pre-treatment at a gauge pressure of 1.2 bar, during 35 minutes, at 122°C, may be advantageous to improve the anaerobic digestibility of the potato peel residue.

CONCLUSIONS

The thermal pre-treatment of potato peel wastes by autoclaving at a gauge pressure of 1.2 bar, 122°C, for different times (20, 35 and 55 minutes) increased the bioavailability of the organic fraction as

compared to the residue which was subjected only to a mechanical milling pre-treatment. The autoclaving process presented positive effects, not only in the accumulated volumes of biogas and methane, but also in the biogas and methane yields determined in the basis of the removed COD and VS.

In what concerns the effect of autoclaving time in the anaerobic digestion process, the increase of this parameter globally conducted to an increase in the production parameters of biogas and methane. Nevertheless, as the autoclaving is an energy consuming process and the differences between the results of the assays E122.35 and E122.55 were not significant, it can be concluded that an autoclaving process at a gauge pressure of 1.2 atm, 122°C, during 35 minutes, may increase the production of biogas and methane in the anaerobic digestion of potato peel wastes.

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