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Pretreatment Techniques Applied to Anaerobic Digestion of Organic Solid Waste

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Abstract

This study evaluated simple pretreatment techniques of organic solid waste of easy degradation prior to anaerobic digestion, with a view to improving performance in biogas production. The pretreatments were inoculation of organic residues with aerobic compost and inoculation of organic residues with aerobic compost associated with the permanence of residues inside compost windrows. Pretreatments didn't significantly increase the volume of biogas or the percentage of CH_4 produced, but significantly accelerated the release time of the total CH_4. Such results suggest that the proposed pretreatment techniques aren't very efficient in increasing the anaerobic digestion of easily degradable residues, being however indicated for residues of difficult degradation. On the other hand, the higher COD found at the end of the experiment in treatments that received pre-treated waste indicates the presence of substrate still available for conversion to biogas, which announces further investigations. The work also showed limitations in the evaluation of organic matter degradation based on volatile solid (VS) consumption, suggesting the use of COD, NKT, Ptot and SO_4^{-2}, to control the process and defined inhibition values for anaerobic bio-digestion due to the presence of O_2 and H_2S considering the operating conditions and the OSW used in the study. Finally, it suggests the t_{-max} concept and the COD as strong indicators for monitoring the performance of AD.

Keywords: Anaerobic Digestion; Pretreatment; Organic Solid Waste; Inoculation; Biogas Production; t_{-max}; COD

Abbreviations: AD: Anaerobic Digestion; OSW: Organic Solid Waste; LPG: Liquefied Petroleum Gas; MSW: Municipal Solid Waste; HRT: Hydraulic Retention Time; TS: Total Solids; VS: Volatile Solids; WW: Wet Weight; TC: Total Carbon; TOC: Total Organic Carbon; TKN: Total Kjeldahl Nitrogen; COD: Chemical Oxygen Demand; DOC: Dissolved Organic Carbon; DC: Dissolved Carbon; Ptot: Total Phosphorus

Introduction

Anaerobic digestion (AD) is considered to be a decentralized renewable energy alternative for the treatment and recovery of organic solid waste (OSW), especially considering the problems related to inadequate final disposal of waste in developing and underdeveloped countries, fossil fuel prices and the environmental impacts of their use [1,2]. As an example, the price of liquefied petroleum gas (LPG), popularly used for cooking food in 13 kg cylinders in Brazil, has risen much more than 100% in the last decade, reaching in February 2020 approximately US$14.00 (ANP, 2020). Unfortunately, AD and the consequent use of biogas is still incipient in Brazil regarding municipal solid waste (MSW), especially considering that more than 50% of its composition is organic (Panorama of Solid Waste in Brazil, 2018-2019). On the other hand, successful experiences have been reported in the country's South region, with agroforestry waste [3], in the Rio de Janeiro mountain region with small-scale sewage treatment [4], and for sewage treatment at the Arrudas Station in Belo Horizonte [5].

Regarding MSW from landfills, some examples of energy use are the Bandeirantes and São João landfills in the city of São Paulo, as well as other landfills in Salvador (Bahia), Minas do Leão (Rio Grande do Sul) and Caieiras (São Paulo) [6-8]. Other examples are the recent facilities installed at the Itaipu Plant to digest food waste, grass clippings and sewage [9] in Curitiba (Paraná) to treat waste from the Central Supply Market in Curitiba; the experimental anaerobic digestion plant installed in the city of Rio de Janeiro, with a capacity of 50 tons/day [10]; and a public-private partnership in Curitiba, the first one on industrial scale [11]. Although underutilized, Brazil had generation potential of 288 MW of MSW and sewage biogas in 2014, equivalent to the energy demand of 5.2 million inhabitants and 11,251,782 t CO_2/eq (ton of carbon dioxide equivalent) of avoided emissions [12]. This scenario should change in 2021 based on the National Solid Waste Policy [13]. The policy provides incentives for recycling, treatment and energy use of MSW, although it contains rules that allow the MSW to continue being buried in landfills.

Despite laudable efforts, cultural, economic, and long-term political views hinder progress in the sector, requiring efforts to develop alternative practices that stimulate its development. In this regard, simplified strategies for pretreatment of OSW before AD can be technically and economically advantageous to facilitate AD, resulting in reduced reactor size and hydraulic retention time (HRT) without requiring specialized equipment.

The limitations of access faced by hydrolytic microorganisms to OSW have been discussed for decades, resulting in research seeking to increase the particle exposure surface and/or hydrolysis rate by pretreatment of OSW before AD, allowing decreased reactor size and HRT [14,15]. These include acid dilution [16], vapor exposure [17], lime bath application [18], ammonia treatment, pressing and screening [19], exposure to enzymes [20], exposure to high temperatures via microwave and hydrogen peroxide [21], pre-aeration [22], forced aeration [23], thermochemical and biological pretreatment with inoculation of organic compounds, *Aspegillus awamori* and activated sewage sludge [24-26], microwave heating [27], steam explosion [28], wet and dry milling [29], alkaline and oxidative treatment [30], feeding mode and dilution [31] and exposure to free nitrous acid [32].

Most of these pretreatments share the advantages of increasing biogas production and digestion stabilization, although high cost and technical difficulties may limit their use, especially for small-scale facilities, aspects not observed in pretreatment involving simple inoculation of the maturing compost in OSW before AD, the objective of the present study, on-site treatment of OSW via small-scale AD. The present work showed that pre-treatments of easy application can increase the speed of CH_4 release in anaerobic bio-digestion operations of organic solid waste. It also presented the characteristics of the waste to which they are applicable and indicated parameters of inhibition of bio-digestion by H_2S and O_2, being therefore relevant for the management of urban solid waste and for the circular economy, especially in developing countries, where sophisticated pre-treatment techniques aren't viable due to the demand for equipment and/or specialized workers.

Methodology

The OSWs used were prepared synthetically in the Technical University of Dresden, Germany, focusing on the digestion of food scraps and paper towels, with an adapted composition to that described by Martin, *et al.* [33], including qualitative tropicalization based on OSW composting studies previously performed in Brazil [34]. Ten samples composed of items usually present in Brazilian OSW were prepared according to the following composition in percentage wet weight: potato 5.95%, carrot 3.4%, orange 7.65%, tomato 2.55%, onion 2 55%, leaves 8.5%, banana peels 4.25%, meat 8.5%, rice 12.75%, noodles 12.75%, beans 14.45%, bread 1.7% and paper towels 15% .

The OSWs were submitted to three pretreatment strategies accompanied by three control treatments:

Pretreatments

a. I24 (Inoculated for 24 hours): OSW inoculated with 6.6% by volume with maturing aerobic compost, kept at room temperature for 24 h.
b. IC24 (Inoculated and Composted for 24 hours): OSW inoculated with 6.6% by volume with maturing aerobic compost, kept inside an industrial composting machine at thermophilic temperature (70 °C) for 24 h.
c. IC48 (Inoculated and Composted for 48 hours): OSW inoculated with 6.6% by volume with maturing aerobic compost, kept inside an industrial composting machine at thermophilic temperature (70 °C) for 48 h.
d. Control Treatments:
e. CNT (control without pretreatment): Biodigestible OSW without any pretreatment;
f. PI (pure inoculum): Pure anaerobic inoculum, without addition of OSW, used to evaluate the remaining biogas generation of the anaerobic inoculum used and subsequent subtraction of biogas production obtained in the experiments;
g. PC (pure cellulose): Inoculum of 50 g of PC, for verification of its viability, as established in the German standard VDI4630 (2006).

The organic load inserted into the digesters was calculated following the ratio of inoculum volatile solids (VS) to substrate VS of 2: 1, as suggested in VDI 4630 (2006). The OSW samples used in all treatments underwent volume and weight reduction via quartering, according to the method of the Brazilian standard ABNT/NBR 10.007: 2004 [35], to adjust the organic load in relation to the samples submitted to composting, which had the percentage of total solids (TS) and VS reduced by dehydration and aerobic microbial consumption. Biogas production was monitored for 21 days in 12 measurements, on days 2, 3, 4, 5, 6, 7, 8, 10, 12, 14, 16 and 21 after the start of the experiment. In each measurement, three qualitative readings of the percentages of CH_4 (methane), CO_2, H_2S and O_2 and the quantities of the gases generated in each biodigester were taken.

The data were grouped for each pair of biodigesters, referring to each of the treatments, with averages and standard deviations calculated from them. Exceptions were for PC and PI, which had only one biodigester each. The volume of biogas generated was normalized considering the temperature and pressure of the measuring room in relation to the water vapor pressure table, as described in VDI 4630 (2006). These volumes, expressed as liters of biogas or liters of CH_4 per gram of VS, were, after normalization, subtracted from the remaining biogas production generated by the PI treatment. The characteristics of the OSW before and after the pretreatments regarding pH, total solids (TS), volatile solids (VS) and wet weight (WW) were investigated, as well as their characteristics after pretreatment but before AD, namely total carbon (TC), total organic carbon (TOC) and total Kjeldahl nitrogen (TKN), with means and standard deviations obtained from duplicate analyses.

The inoculum used was evaluated for pH, TS and VS before and after OSW intake, and before and after AD. Also regarding the PI, before anaerobic digestion the digestate obtained from all treatments and controls was measured to determined: dissolved organic carbon (DOC), dissolved carbon (DC), volatile organic acids (VOA), chemical oxygen demand (COD), Total Kjeldahl Nitrogen (TKN), total phosphorus (Ptot) and sulfur (SO_4^{-2}). These analyses were also performed in duplicate for the digestate of each of the CNT, I24, IC24, IC48, PI and CP treatments, as well as for a sample previously separated from the pure original anaerobic inoculum (OI) before anaerobic digestion, resulting in beans and standard deviations calculated from four results for CNT, I24, IC24 and IC48, which counted on two biodigesters each, and from two results for PI and CP, which counted on one biodigester each. The volatile acids evaluated were lactic acid, formic acid, acetic acid, propionic acid, butyric acid, iso-butyric acid, valeric acid and caproic acid. The values were summed and are presented together.

All analyses were performed in the Technical University of Dresden according to methods described in the German analytic standards, DIN EN, according to references, by parameter: total solids (TS): DIN EN 14346 [36]; volatile solids (VS): DIN EN 15169 [37]; pH: DIN EN ISO 10523 [38]; volatile organic acids (VOA): DIN EN 38404-19; dissolved organic carbon (DOC): DIN EN 1484 [39]; dissolved carbon (DC): DIN EN 1484 [39]; total carbon (TC): DIN EN 1484 [39]; total organic carbon (TOC): DIN EN 13137 [40]; chemical oxygen demand (COD); DIN 38414-9 [41]; TKN: DIN EN 25663 [42]; Ptot: DIN EN 1189 [43]; and SO4-2: DIN EN ISO 11885 [44].

For comparisons of the parameters of biogas production, percentages of CH_4, CO_2, H_2S and O_2 in biogas, VS, MO (COD, TKN, Ptot, SO_4^{-2}) and COD in anaerobic and digested inoculum were used. The data were submitted to analysis of variance (ANOVA) at a significance level of 95%. To meet the requirements for ANOVA, normal distribution and homoscedasticity of all variables were tested. Since most data did not meet these requirements, $\log_{10}(x+1)$ transformation was used before ANOVA, which was followed by the Tukey test of difference mean at 95% confidence ($p < 0.05$), to determine means that were significantly different [45].

Results and Discussion

Inoculum Viability

PC biogas production averaged 0.68 NL.gVS^{-1}, exceeding the 0.60 NL.gVS^{-1} suggested in VDI 4630 (2006) as a reference value for the viability of AD.

Biogas and CH4 Production

Total biogas production in NL.gVS^{-1}, higher in CNT and IC48 treatments compared to I24 and IC24 (Figure 1), showed no significant differences according to ANOVA. The average percentage of CH_4, higher in IC48 and I24 than in CNT (Figure 2), showed no significant differences according to ANOVA. Consequently, the production of CH_4 (Figure 3) also showed no significant differences according to ANOVA.

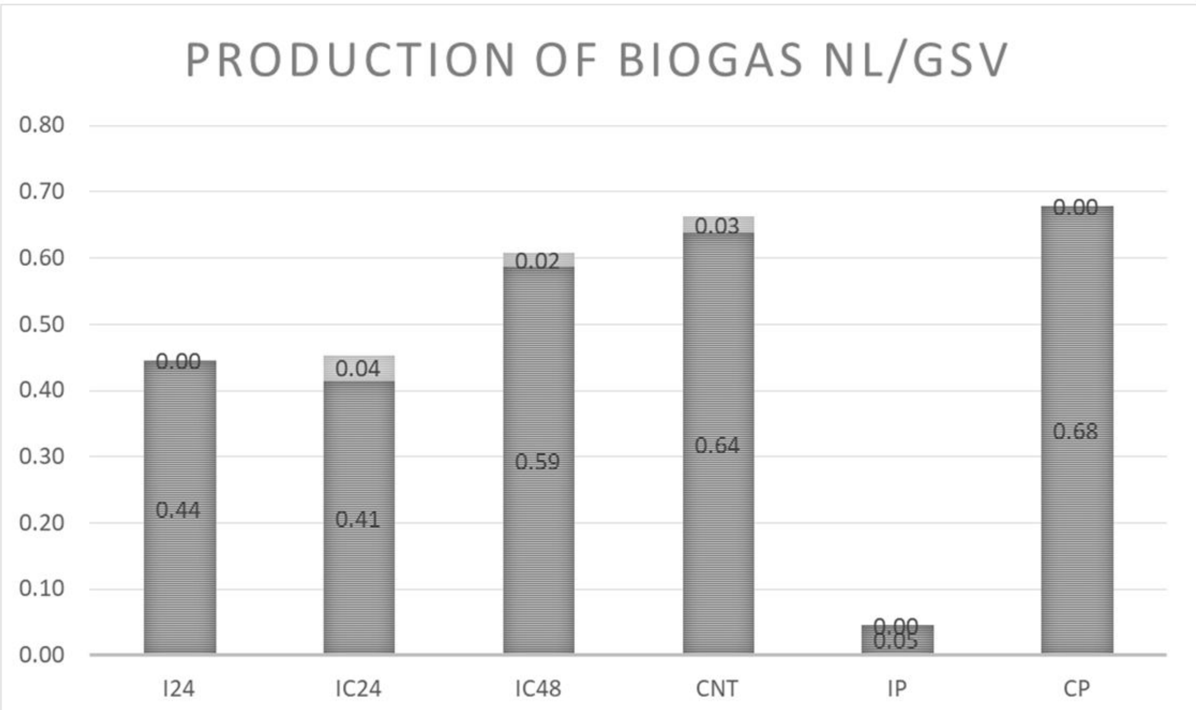

Figure 1: Mean production and standard deviation of biogas in NL.gSV^{-1}

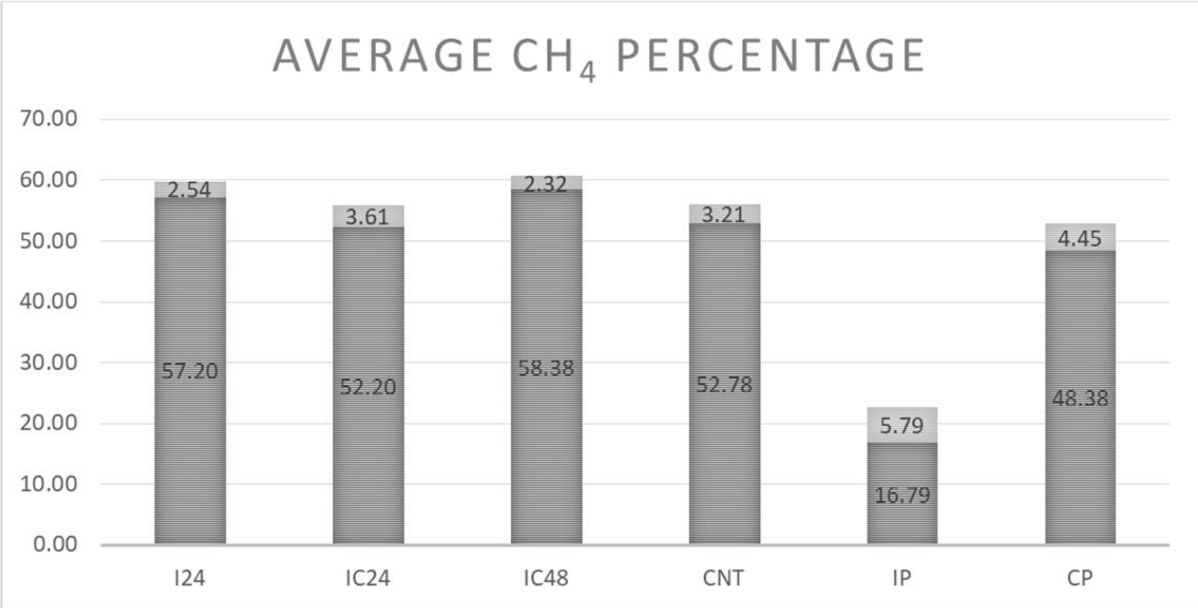

Figure 2: Average percentage and standard deviation of CH_4

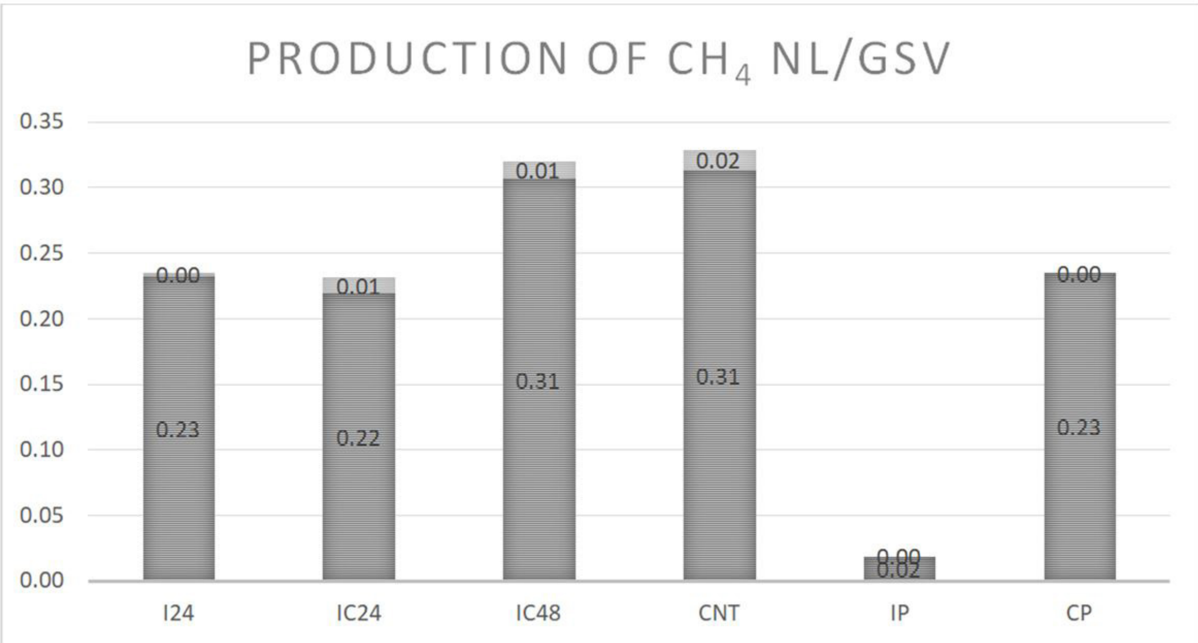

Figure 3: Average production and deviation of CH_4 in $NL.gVS^{-1}$

Treatment	CNT		I24		Ic24		IC48		IP		CP	
Parameter	**I**	**F**	**I**	**F**	**I**	**F**	**I**	**F**	**I**	**F**	**I**	**F**
VS total / g de WW(wet weight) before and afterAD	197	133	197	133	197	135	197	131	131	119	181	118
VS from OSW /g before the AD	66	NA	66	NA	66	NA	66	NA	NA	NA	50	NA
pH before and after AD	6,4	7,7	6,4	7,7	6,4	7,7	6,4	7,7	6,4	7,6	6,4	7,7
WW (wet weight) from OSW/g before and after PT	501	NA	550	546	550	407	550	314	NA	NA	NA	NA
TS % before and after PT	27	NA	27	30	27	39	27	42	NA	NA	NA	NA
VS % in DW before and after PT	97	NA	97	95	97	91	97	87	67	NA	93	NA
WW from OSW in g	245	NA	231	NA	183	NA	178	NA	NA	NA	50g pC	NA
Degradability VS/TS	0,97	NA	0,95	NA	0,91	NA	0,87	NA	0,67	NA	0,93	NA

Table 1: Initial and final physicochemical characteristics (PC, PI, AD: anaerobic digestion, I: beginning, F: end, g: grams, PT: pre-treatment, WW: wet weight, DW: dry weight , NA: not applicable, VS: volatile solids, TS: total solids)

Eliason [19], Fdéz-Guelfo, *et al.* [24] and Shahriari, *et al.* [21] observed increases in biogas production using these and other types of pretreatments in relation to their controls, but with materials with more difficult degradation and VS/TS ratio of 0.5, whereas in the present study the VS/TS ratio was between 0.8 and 0.9 (Table 1), above the 0.7 considered in VDI 4630 (2006) as the boundary between easily and difficultly degraded residues.

Side effects of pretreatments might have influenced the results, since decreases in the organic solid waste/carbon (OSW/C) ratios after the pretreatment were observed, which were 26/1 in CNT, 19/1 in I24, 18/1 in IC24 and 16/1 in IC48, in all cases significant differences at $p < 0.5$, between the control without treatment (CNT) and the others. This reflects the consumption of the easily degradable fraction of C, converted to heat and CO_2 (carbon dioxide) in the first 24 hours of composting [46], and/or consumed by the aerobic inoculum [26]. This aspect may have decreased biogas production performance in these treatments, since the C/N ratios commonly suggested in the literature for AD range from 25/1 [31] to 30/1 [14]. This fact can be observed in Table 2, indicating higher amounts of TC and TOC in the OSW used in the CNT (control without pretreatment) compared to those from the pretreatments, and the inverse in relation to TKN.

The volume of aerobic inoculum used in pretreatments, 6.6% in relation to the volume of OSW, may have been excessive regarding C/N in relation to the recommendation of Fdéz.-Guelfo, *et al.* [24], which is use of 2.5% aerobic inoculum. This aspect is relevant even though the VS values were equated after pretreatment, before AD, since the remaining VS in the OSW represents materials with more difficult degradation as well as unbalanced with respect to C/N. Thus, we suggest that the amount of aerobic inoculum used in the pretreatment be defined according to the C/N ratio and VS/TS ratio of each OSW, and that larger amounts of aerobic inoculum be used only in the pretreatment of carbon-rich OSW, or more material that is harder to degrade.

Charles, *et al.* [22] observed even more antagonistic results than those of the present study, with significantly lower production of biogas and CH_4 in one of their pre-aeration treatments compared to the control. Negative results for pre-aeration pretreatment were also observed by Krusch, *et al.* [47], using equine manure substrate, which may be related to the pretreatment already naturally promoted by the animal's digestive tract, suggesting that easily degradable materials should be viewed with caution regarding the application of pretreatment strategies. However, biogas and CH4 production in all treatments reached the same levels observed by Fdéz.-Guelfo, *et al.* [26], and Zhang, *et al.* [48], specifically using food scraps as in the present study, in wet and dry AD, respectively. Table 1 shows values of initial and final physicochemical parameters of the OSW (pH, TS, VS, wet weight and dry weight) before and after pretreatments, as well as anaerobic inoculum pH and VS values before and after AD. The TC, TOC and (TKN concentrations in $mg.g^{-1}$ in the OSW per treatment and in the aerobic inoculum (organic compound) before AD are reported in Table 2.

Treatment	CNT		I24		IC24		IC48		Compound	
Parameters for solids average in $mg.g^{-1}$/standard deviation										
TC	449,1 /	0,9	406,4 /	5,9	396,7 /	4,1	410,5	/ 3,9	251,6 / /	3,4
TOC	409,1 /	3,5	375,0 /	6,7	390,9 /	3,4	403,0	/ 2,9	231,5 /	1,0
TKN	15,6 /	0,8	21,3 /	1,1	21,3 /	0,6	25,1	/ 0,2	18,3 /	0,2

Table 2: TC, TOC and NKT in $mg.g^{-1}$ in the OSW pretreatment and in the aerobic inoculum (organic compound) before anaerobic digestion

O_2 (Molecular Oxygen)

The average percentage of biogas O_2 was higher in IC24 due to sealing problems in one of the digesters of this treatment, which may have caused a decrease in the percentage of CH_4 in this biodigester, since its O_2 percentage exceeded 1%, on the seventh day, as also observed in one of the digesters of treatment I24, indicating the onset of problems related to O_2 poisoning. O_2 contamination is known to inhibit AD, especially regarding CH_4 production [14], but the intoxication limits are not clearly defined. Laboratory practices with AD indicated a 65% reduction in biogas production 24 hours after 10 minutes of partial exposure of the anaerobic inoculum to atmospheric air, accounting for up to 2% biogas O2. Restoration of usual biogas production only occurred 6 days after this event, with O_2 concentrations below 1%, suggesting this percentage is a maximum limit of O_2 in biogas to prevent inhibitory events in CH_4 production [49,50].

CO_2

There were no significant differences between the average percentages of CO_2 obtained in the four main treatments, which presented typical AD values, except for PI (CO_2 14%), a finding consistent with the low metabolic activity imposed by the absence of feed. A lower percentage of CO_2 was noted in the PC treatment at the beginning of AD, reflecting inhibition caused by almost 7% O_2 in the same period. Similarly, higher CO_2 production and lower biogas production were noted simultaneously in I24 at the beginning of AD, suggesting possible accumulation of volatile acids due to higher hydrolytic and acidogenic activity. The above results suggest that the level of CO_2 is an indicator of the general metabolic activity of AD, being useful along with the other monitoring parameters to identify disorders in AD [51].

H_2S (Hydrogen Sulfide)

The percentages of H_2S were higher in treatments I24 and IC48, being significantly different from IC24. The limits of interference of sulfate reducing bacteria (SRB) on biogas production and methanogenesis, reflected in H_2S production, are highly variable and depend on different parameters of AD operation. However, concentrations between 200 and 1500 ppm are described in Mata-Alvarez (2003) as usual in AD. In this study, concentrations above 2000 ppm and peaks up to 3500 ppm H_2S were observed in one of the digesters (I24). At the same time, this treatment had lower biogas production compared to the other treatments and lower CH_4 percentage than the other biodigesters of the same treatment, contributing to a decrease in overall productivity of I24 [52]. Likewise, concentrations between 2000 and 2500 ppm were observed in one of the IC48 digesters, reducing its performance in relation to the other digesters of the same treatment, which remained about 300 ppm below the first one. Thus, considering the variability of H_2S toxicity patterns, we suggest that 2500 ppm H_2S in biogas is a possible limit indicating the onset of AD disorders with the characteristics of this study.

Time Distribution of CH4 Production

CH_4 production in NL.gVS^{-1}/day was more concentrated between the 3rd and 5th days for the pretreatment digesters and between the 6th and 10th days for the control treatments, with 66%, 60.5% and 58% of the total CH_4 released in 5 days of AD in I24, IC48 and IC24 respectively, in contrast to the 33% obtained for CNT (Figure 4).

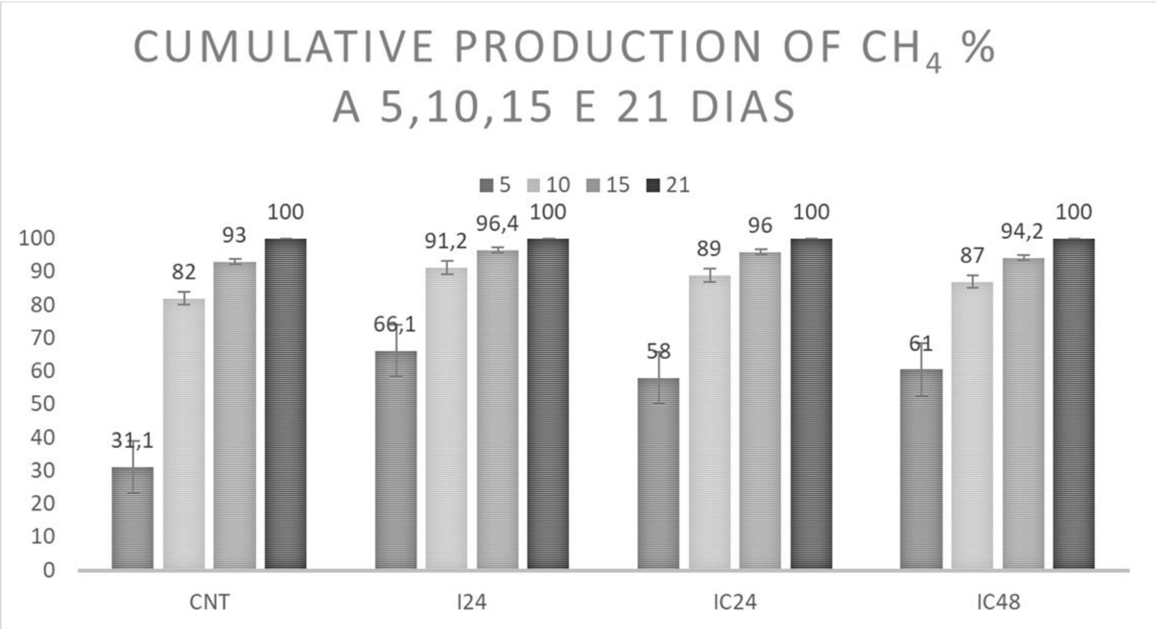

Figure 4: Percentage of total CH_4 production released at 5,10,15 and 21 days in AD

Clear advantages over the acceleration of CH4 release in AD after pretreatment of OSW were also observed by Charles, *et al.* [22], although without increased biogas production as in the present study [53].

Taking into consideration the "t-max" concept [26], where the minimization of the release time of 95% of all produced CH_4 (t_{max}) indicates better performance, the following "t_{-max}" values were observed in days in the present study: CNT = 17 days, IC48 = 15 days, IC24 = 14.5 days, I24 = 14 days. The authors cited found 15-day "t-max" for OSW pretreated with 24 h compost inoculation and 31-day "t-max" in their control experiment, but with VS/TS ratio of 0.5 in the OSW. Such acceleration in CH_4 release is due to the increased solubilization of organic matter, resulting from its partial pre-hydrolysis promoted by pretreatments, as discussed in Fdéz- Guelfo, *et al.* [26]. In this approach, there is a clear relationship between the difficulty of substrate degradation and the usefulness of using pretreatments, not only to accelerate CH_4 release, but also to increase its percentage in biogas and biogas volume itself, as also reported by Fdéz- Guelfo, *et al.* [24] and Shariari [21].

The acceleration in CH_4 production seems to be related to pretreatment, composting and inoculation of maturing compost, as also observed by Charles, *et al.* [22] in the absence of aerobic inoculum in pretreatment.

Degradation of Organic Matter

No advantages were noted regarding the increase in VS degradation by pretreatment. VS degradation, around 30%, was also small compared to the 80% degradation found by Zhang [48] in experiments very similar to those of the present study. However, the analysis of VS consumption can lead to misinterpretation by accounting for live bacterial biomass as non-consumed VS at the end of the process. Organic matter consumption data based on COD, TKN, Ptot and SO_4^{2-} can more efficiently measure the degradation of organic matter in the treatments, with degradation being above 50%, a compatible percentage for AD under mesophilic conditions according to Mata-Alvarez [14], as well as compatible with biogas and CH_4 production.

Volatile acid concentrations at the end of AD were slightly lower in the pretreatments than in the controls, with no indication of higher consumption in digesters that received pretreated OSW. However, all treatments had less than 60 $mg.L^{-1}$ VA at the end of AD, a value suggested by Fdéz-Guelfo, *et al.* [26] as indicating a stable AD processes.

The average COD values at the end of AD were 31, 20 and 19% higher in the IC24, IC48 and I24 treatments, respectively, compared to the control treatment (Figure 5), but with significant differences only in IC24 in relation to CNT. This fact suggests a larger amount of raw material available for conversion to biogas in the pretreatments compared to the control treatment, even in the case of the easily degraded OSW used in this round, which was not observed in the analyses of VS, organic matter or volatile acids, since COD also encompasses the inorganic matter available in the system [54,55].

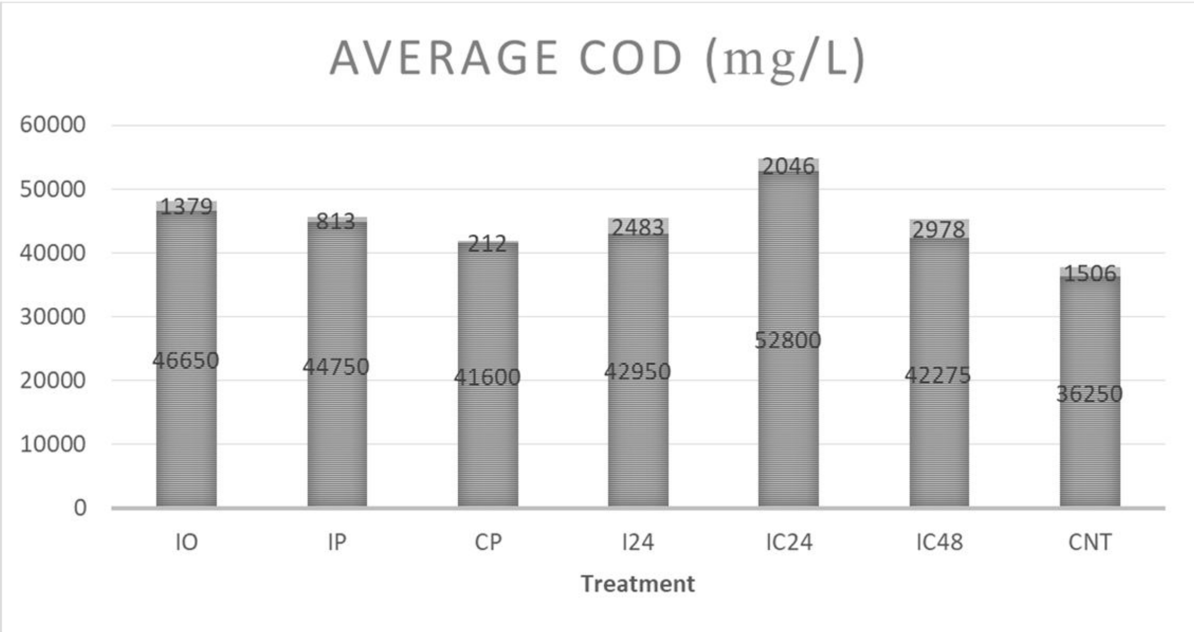

Figure 5: Mean and COD deviation in $mg.L^{-1}$ per treatment and inoculum

Thus, considering the imbalance of the C/N ratio observed in all pretreatments and the inhibitions caused in I24 and IC24, the pretreatments could have led to better performance in relation to the control treatment with simple adjustments to the method, such as a decrease in the percentage of aerobic inoculum contributed in the pretreatment. This corroborates the observation that problems of initial inhibition can have reflections throughout the AD process, not only while the inducing effects of the inhibition last [56]. The pH reached neutral values for all treatments after 21 days of AD, as can be seen in Table 1, without significant differences according to ANOVA, indicating stability of the final digestate in all treatments according to Mata-Alvarez, *et al.* [14].

Conclusions

The pretreatments tested did not increase the biogas generation, the percentage of CH_4 or the degradation of organic matter compared to the CNT (control without pre-treatment), but significantly increased the speed of CH_4 release, which may be related to the use of the pretreatment in composting (thermal effect) and for pretreatment with inoculation of aerobic compound (biological effect). All pretreatments consumed easily degradable carbon more quickly than nitrogen, unbalancing the C/N ratio and impairing the performance of AD, suggesting that such techniques be applied to residues with more difficult degradation and/ or higher C/N ratio. Or residues with easier degradation and lower C/N, percentage of aerobic inoculum used in the pretreatment should be decreased in order to increase the CH_4 release speed.

For monitoring purposes, organic matter degradation calculations based on VS consumption have limitations of interpretation. It is preferable to use COD, TKN, Ptot and SO_4^{-2}. O_2 values between 1 and 2% and H_2S above 2500 ppm in biogas can indicate inhibitory problems of AD, considering the operating conditions and the OSW used in this study. Considering the multiplicity of influential factors and the difficulties in interpreting results of AD experiments, the t-max concept and the COD can be robust indicators for monitoring the performance of AD.

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