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From Ash to Anaerobic Digestion: Optimization of Biogas Yield from Food Waste and Pineapple Peelings

Gildas David Farid Adamon[1], Matar Sylla[2], D. M. Grâce F. CHIDIKOFAN[3], Ariane Naomi Midjaya Mahouna Zounon[4]

[1,4]Department of Energy, National Higher Institute of Industrial Technology of the University National Institute of Science (INSTI), BP 133, Republic of Benin.
[2]National Higher School of Energy and Process Engineering, BP 486, Abomey, Republic of Benin.
[3]African Biomass Conversion and Energetic Efficiency, Cotonou, Republic of Benin.

Corresponding Author: David G. F. Adamon

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ABSTRACT

The acidity of essential waste makes anaerobic digestion difficult, slow and inefficient. The objective of this study is to find a substrate/catalyst dosage in order to improve the kinetics of decomposition of this waste, in particular that of food waste and pineapple peelings, from the ashes resulting from the carbonization of wood. Thus, we separately carried out a control anaerobic digestion consisting of 100% food waste, then 100% pineapple peelings. Then, we proceeded to anaerobic digestion with different dosages of food waste, kitchen scraps and ashes, then pineapple peelings and ashes in order to find the optimal dosage. The results obtained show that a dosage of 70% food waste and 30% ash gives maximum and accelerated production. For pineapple peelings, the maximum production is obtained with 35% of the ash mass concentration. A dosage of less than 20% ash has no noticeable effect on anaerobic digestion and may even constitute a brake on it, unlike a dosage greater than 40% which inhibits digestion.

Keywords: Ash; Anaerobic digestion; Biogas; pH

INTRODUCTION

Since 1990, pineapple cultivation has established itself as a promising niche and is the subject of a project to promote and develop the sector initiated by the Regional Center for Agricultural Promotion (CEPRA). In Benin, it is ranked 3rd sector after cotton and cashew. In 2014, Benin had 99 pineapple production units, 98% of which processed pineapple into pineapple juice. Each unit processes an average of 360 tons of pineapple per year. This transformation produces 35% or even 40% residues per year [(1)].

This highly digestible and recoverable waste by wet process to produce biogas comes up against a major problem: The acidity of the reaction medium, although anaerobic digestion is characterized by three (03) important parameters: the C/N ratio (ratio between Carbon and nitrogen content), temperature (T) and pH in addition to mechanical agitation [(2)]. In addition, the C/N ratio provides information on the organic matter content of the biomass (raw material), while mechanical agitation makes it possible to homogenize the substrate in the reactor as the reaction proceeds. Hydrolytic and acidogenic bacteria are little affected by pH variations, which is not the case for acetogenic and methanogenic

bacteria which do not tolerate excessive pH variations [3]. It has been shown that the degrees of inhibition of biochemical processes are distinctly related to the undissociated forms of the acids present in the media [4, 5]. For optimal methanogenic activity and proper reactor operation, pH stabilization should be between 6.8 and 7.4 [6]. Thus, a pH outside this range is toxic. In order to maintain the reactor at an optimum pH, the latter is regulated by the addition of a basic substance capable of neutralizing the acid in the reaction medium. In the literature, there are several types of basic solutions used: soda and all its derivatives (sodium bicarbonate, sodium carbonate, etc.), all chemical solutions on the market [7]. To improve the yield of anaerobic digestion, co-digestion is carried out, which consists in mixing two or more raw materials: this makes it possible to improve either the pH of the reaction medium when the pH is not close to neutrality, or to balance the C/N ratio to make it more optimal [8]. The pH is mainly related to the presence of volatile fatty acids. In addition, regardless of the nature of the raw material used, during the proper functioning of the digester, the pH is naturally buffered by the presence of the bicarbonates produced by the methanogens [9]. During stress, this buffering capacity can decrease. However, the acidification of the medium can also be caused by the solubilization of the CO_2, contained in the biogas. In the case of the methanation of a substrate rich in ammonia (degradation of proteins for example), it will react with the fraction of dissolved CO_2 to form a buffer solution. Each anaerobic digestion stage has its own optimum operating range: between 5.5 and 6.5 for the hydrolysis and acidogenesis stages; acetate production has been characterized as optimal between 8 and 11, with a maximum at pH 8, for the acetogenesis step; finally, the methanogenesis step would have its optimum at a pH between 6.7 and 7.5 [10].

Most of the solutions proposed to neutralize the effect of pH on the profitability of methane fermentation come up against obstacles such as: the (basic) solutions used come from chemical synthesis and are not free. The materials used for co-digestion are sometimes unavailable on site or not available at all in sufficient quantity for better sustainability of the sector. Added to all this is the context of the adaptability of this method to the African context: difficulty in obtaining these chemical solutions. Faced with these limits, it is necessary to develop another simple, ecological, and less expensive method, capable of improving the methane fermentation of reaction media at low pH, resulting from acidic biomasses such as food and agro-food waste or peelings pineapple. In this context, the ashes have been identified as such to neutralize the acidity of the reaction medium. Indeed, wood ash/charcoal is an essentially basic residue. The ashes resulting from the combustion of lignocellulosic material are mainly basic or alkaline. Thus, several tests are carried out in order to demonstrate its alkalizing effect on the methanation reaction medium on the one hand, and its influence on the kinetics of decomposition of the substrate contained in the digester on the other hand.

MATERIALS & METHODS

The raw materials are essentially made up of low pH biomass. In the case of the present study, these are cooking scraps and pineapple peelings (Figure 1).

They are used within 24 hours of production to avoid possible decomposition before testing. The food waste came directly from the university canteen of the National Higher Institute of Industrial Technology (INSTI) in Lokossa, Benin, while the pineapple peelings came from pineapple vendors in the city of Lokossa in Benin. Before any operation, these wastes are mixed to ensure a certain homogeneity of the sample thus formed by each of the wastes. Pineapple peels are ground into very small particles (as a paste) before use to aid in their decomposition. The ash used in this study remains the ash from the combustion of

charcoal in our homes and its pH=12. The pH values of the raw materials used are respectively 5.44 for pineapple peelings and 6.62 for food waste. They are filtered and freed of all coarse particles before use (Figure 1 below).

(a) (b)

Figure 1. Raw materials before mixing: (a) pineapple peelings and (b) food scraps.

Food waste is a mixture of condiment residues used in the preparation of meals and the rest of the cooked dishes (rice, beans, couscous, vegetables, tomatoes, corn paste, pineapple, macaroni, carrot, lettuce, onion, etc.). Methanation is carried out with 100% of the raw material, ie 90g of raw material for the reference test, then varies according to the contribution of ashes in the preparation of the constituted substrate. The pH was measured with a pH meter (model number: pH-991) throughout the methanization. In addition, the pH meter was calibrated before use. Added to this is a balance to measure the masses of the raw materials or substances used. We used three (03) identical digesters with a capacity of 150 mL each. The three (03) digesters are launched simultaneously with the same content, all immersed in a bath mounted for this purpose on a laboratory scale, in order to check the repeatability of the results and to choose the most representative data for each test (Figure 2 below).

Figure 2. Device for measuring the volume of gas produced

The three (03) digesters were introduced into a water bath set at a temperature of 37°C ± 2°C using a system consisting of a heating resistor and a thermal regulation (model of controller: Hg-802, heater model: Hg8021). The quantity of biogas produced was evaluated by measuring the volume of biogas produced using a system mounted on a laboratory scale according to the principle of ARCHIMEDES (Figure 2.). Connected to the output of the biogas produced, the system contains an inflatable balloon which is held in a graduated tube containing the water. The inflation of the balloon induced by the arrival and the accumulation of biogas in the balloon causes a displacement of the water level in the tube. Each displacement of water corresponds to a height which is then used to calculate the volume of gas produced.

The experimental procedure boils down to:

- introducing a mass m of the substrate into a suitable container;
- mix this mass with water respecting the ratio 1:1, then homogenize the mixture;

- measure a mass equal to 90 g of the mixture using a scale, then introduce this mixture into the digester (for the control or reference experiment);
- Place the ashes in a container and mix them with water respecting the ratio 1:1
- Take a mass m_1 of the previous mixture and mix it with a mass m2 of biomass (kitchen scraps/ pineapple peelings), then introduce them into the digester. In addition, the ash and raw material contents were evaluated through mc and mr representing respectively the mass percentage of ash and biomass used, knowing that $m_1+m_2=90g$ and mc+mr=100:
- $m_c = \frac{m_1}{m_1+m_2}$ et $m_r = \frac{m_2}{m_1+m_2}$ (1)
- Close the digester hermetically and connect the gas outlet to the recuperator;
- Place the digester in the bath containing the water and the heating resistor;
- Record the height of water in the recovery system every 4 hours and calculate the corresponding volume. The volume is obtained using the following formula:
- $V = \frac{\pi D^2}{4}$ (2)

D: pipe diameter; H: Displaced water height

Below is the figure of the experimental device (Figure 3). The tests are carried out in a mesophilic regime (37° C.).

Figure 3: Experimental device

RESULT

By using the cumulative production curves in the three digesters for each experiment, we were able to choose the data from the most representative digester. Thus, in the following, data from the most representative digesters have been used for each experiment. The experiments were numbered from 1 to 4: Experiment 1 corresponds to the control experiment (100% food waste or 100% pineapple peelings).

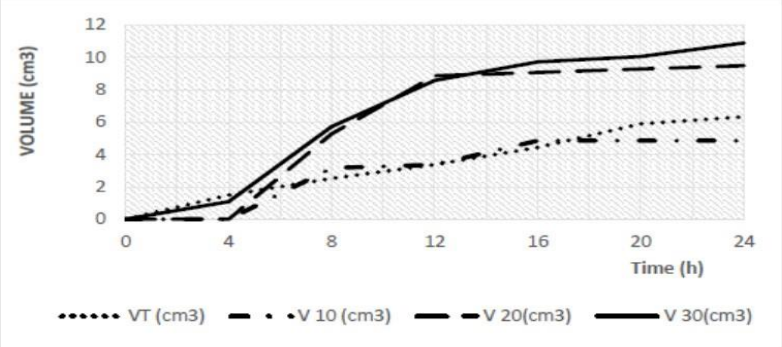

Figure 4. Cumulative production of biogas in (cm^3) of anaerobic digestion of kitchen scraps as a function of time

Figure 5 shows a very particular and very interesting aspect of the problem; it is noted that the production of biogas during the control experiment shows less variation compared to the other curves with dosage.

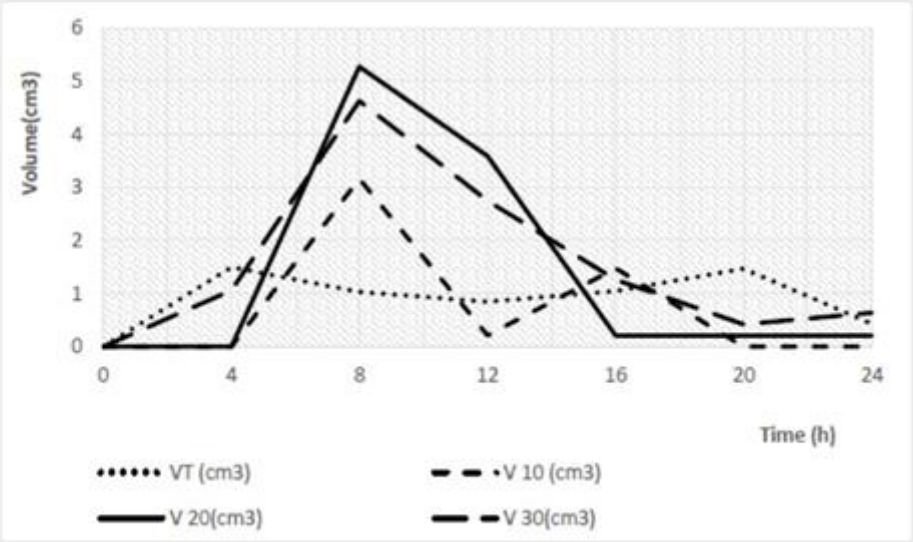

Figure 5. Representative curves of the instantaneous production of biogas from kitchen scraps.

From Figure 5 and Figure 6, it can be seen that the optimal instantaneous production of biogas (5.27 cm^3: Cf. Figure 5) is reached 8 hours after the start of methanization for the experiment whose substrate consists of 20% ash and corresponds to a pH of 11, 2 (Figure 6).

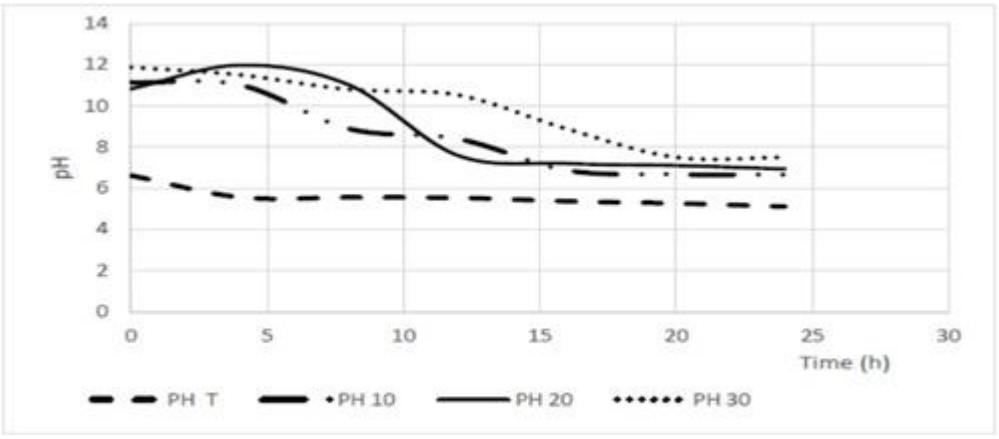

Figure 6. Evolution of pH over time

On the basis of these results obtained, we also commented on the influence of ashes on the methane fermentation of pineapple peels. To do this, the dosages were carried out ranging from 25% to 40% (relative to the total mass of the substrate: m = 90 g) Figure 7.

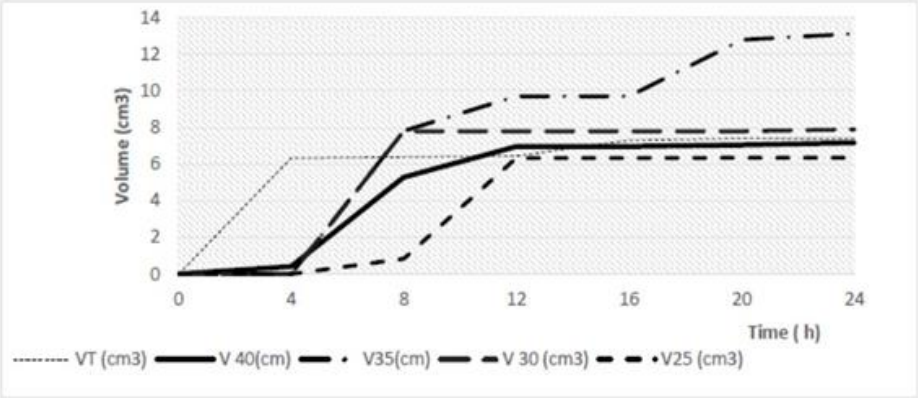

Figure 7. Cumulative production of biogas in (cm3) of methanation of pineapple peelings as a function of time

In Figure 8, we saw the rapid effect of ash on improving the reaction pH of the methane fermentation medium of pineapple peels.

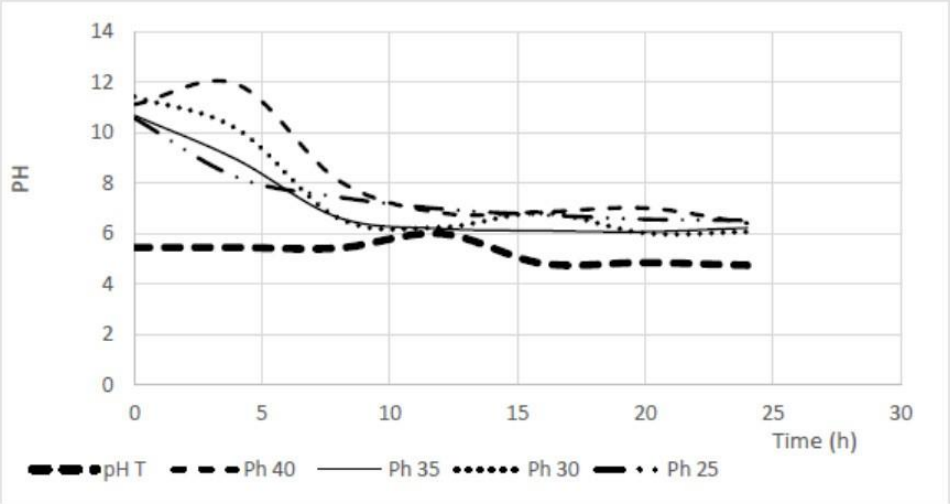

Figure 8. Evolution of the pH of the methane fermentation of pineapple peels as a function of time

The optimum is obtained at 8 a.m. and corresponds to a pH range of between 6.4 and 7.4 (for the best productions: 35% and 30%), as shown in table 1 below.

Table 1 Variation of pH as a function of time during each experiment for pineapple peels

pH measurement during the degradation of pineapple peels					
T(h)	pH-T	pH-40	pH-35	pH-30	pH-25
0	5.44	11.12	10.69	11.43	10.57
4	5.44	11.92	8.94	10.16	8.23
8	5.42	8.1	6.63	6.61	7.44
12	5.99	6.82	6.19	6.25	6.97
16	4.8	6.86	6.11	6.82	6.73
20	4.84	7.01	6.07	6.02	6.56
24	4.73	6.41	6.22	6.07	6.51

To study the influence of ash on the residence time, it was necessary to increase the volume of substrate upstream (i.e. m = 2000 g, or 2 kg of kitchen scraps on the basis of 30% ash)

while respecting the experimental protocol described above. The results obtained are shown in Figure 9.

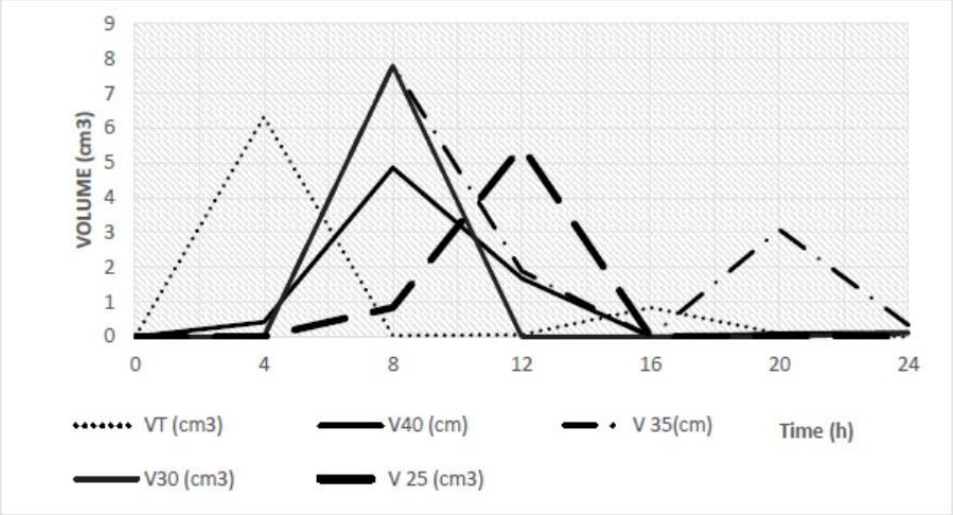

Figure 9. Representative curves of instantaneous biogas production in each experiment for pineapple peels

Through Figure 10, the hydraulic residence time remained substantially equal to 7 days, whereas in the test (food waste 30% ash).

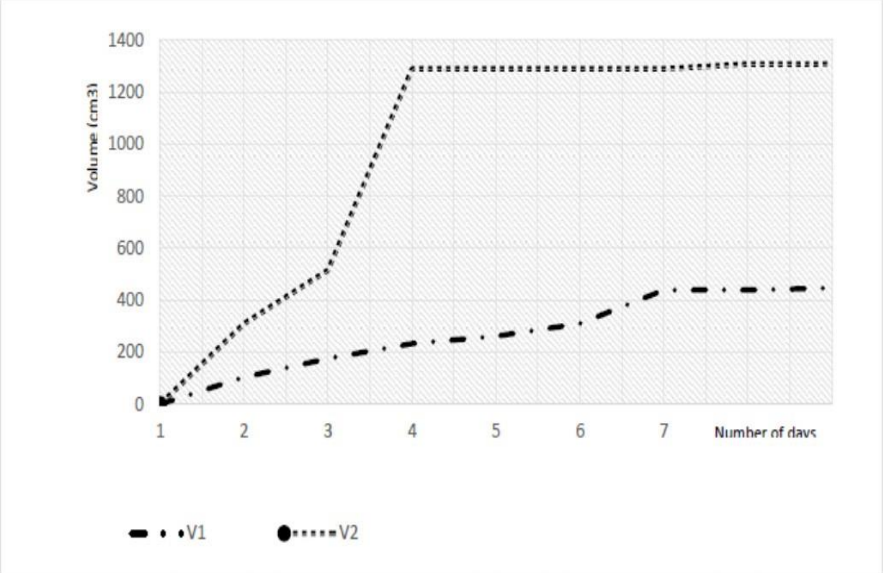

Figure 10. Curves representative of the cumulative production of biogas for kitchen scraps
V1: control test and V2: test with 30% ash

DISCUSSION

The first results come from the methane fermentation of kitchen scraps, then, based on the partial conclusions, the methane fermentation experiments of pineapple peels were validated. The results of methanization of kitchen scraps have been compiled in Figure 4. VT (cm^3), V_10 (cm^3), V_20 (cm^3) and V_30 (cm^3) denote respectively the control experiment, the experiment carried out with 10% ash, the experiment with 20% ash, the experiment made with 30% ash.

In general, we have distinguished the three (03) major stages of methanization such as the lactation phase, the exponential growth phase and the terminal phase which marks the end of the methanization process. This result is in fact consistent with most of the results obtained during the methane fermentation of biomass [(11)]. By analyzing the biogas productions in each experiment involving ash (Exp N°2, 3 and 4, respectively with 10%, 20% and 30% ash) and comparing it to the control experiment (Exp N°1: 100% leftovers from the kitchen), we find that the production of biogas in the experiments involving the ashes is higher than that of the control experiment with the exception of experiment N°2 where the production was practically stopped from 16 hours of digestion. From these different curves, it can be seen that the ashes have a particular influence on the anaerobic digestion of the wet biomass, in particular the increase in the volume of biogas produced (Figure 4).

This influence is proportional to the mass percentage of ashes contained in the mixture of the substrate consisting of kitchen scraps and ashes. In addition, the fact that experiment N^{o} 2 stopped at 4 p.m. means that the ash content is not high enough to improve the pH of the reaction medium, which negatively impacts the decomposition of the substrate, and consequently the volume of biogas produced. In order to follow the production of biogas in each experiment and to be able to clearly check the action of the ash, we grouped the instantaneous production of biogas in the three experiments involving the ash and the Control experiment in the same graph.

But the curves representative of experiments involving ash show a production peak obtained between 8h and 12h of digestion. It should also be noted that most of the volume of biogas obtained is produced during this time interval. That is 2.2/5.4 (40.74% for Exp 2), 8.86/9.97 (88.86% for Exp 3) and 7.38/10.76 (68.58% for Exp 4) (Figure 5).

Similarly, exponential growth times differ from experiment to experiment, depending on the proportion of ash. This time evolves in the same direction as the increase in the ash content in the substrate: it is from 4 a.m. to 8 p.m. for the 30% ash dosage, from 4 a.m. to 4 p.m. for the 20% dosage and finally from 4 a.m. to 8 p.m. with slight variations in volume for the 10% dosage. It can therefore be said that the ash content acts on the decomposition kinetics of the substrate. Knowing that ash is a very basic product, we are now interested in its influence on the pH during digestion. To do this, we grouped the instantaneous variations of pH as a function of time during each experiment in the same graph. According to Figure 6, the pH in the control digester (100% kitchen scraps) is much lower than 7 from the beginning to the end of methanization, with a very low production of biogas, unlike the other digesters. This result could be justified by the increase in the undissociated form of VFAs in the reaction medium, and remains in line with the results obtained by Benlensar Latifa and Abdoune Zohra in 2017 [(12)].

pH close to this (pH = 10.2) were obtained in 2011 by Nazo Edith Kpata-Konan who neutralized the acidity of the reaction medium due to the methanization of cassava effluent by adding urine human and cow dung [(7)]; This is due to the alkaline impact of these substances on the acidity of the reaction medium. Even better, this result is close to the results obtained by Laura Andre in 2016, who estimated that the optimal pH range for methane fermentation is 6 to 9 [(13)]. Furthermore, we note that despite the optimum reached during experiment No. 3 (Exp_20), the gas yield was obtained in the case of experiment No. 4 although its optimum was reached for a pH = 10.8. Indeed, the pH has direct actions on the ammonia/ammonium couple and an increase in the pH leads to an increase in the "ammonia" form in the reaction medium, which consequently leads to an inhibition of the process: this moreover justifies the

results obtained at the level of experiments N°3 and N°4. The high pH values obtained at 8 a.m. testify to the majority presence of acetate molecules whose optimum pH is between 8 and 11 [10]. However, experiments involving ashes have seen their pH vary from the acidic range (pH < 7) to the basic range (pH > 7). In 2012, to improve the production yield of methane fermentation of food waste, Volana Astérie R. [(14)] used co-digestion with agricultural waste. This has a significant impact on improving the pH of the reaction medium, which goes from 4.6 to 7. The results obtained agree well with those obtained by Moletta in 2003 [(15)]. Indeed, the latter to correct the pH of the reaction medium, opted for the use of sodium carbonate (Na_2CO_3). This work has been involved in the anaerobic digestion of acid materials such as onion, potato, carrot, beetroot, and lettuce and artichoke peelings. Using respectively cassava effluents and onion, potato, carrot, beet, lettuce and artichoke peelings, Nazo Edith et *al.* [7] then S. Kalloum et *al.* [(8)], thanks to the use of (human urine + cow dung) and (sodium carbonate), were able to double the volume of biogas produced and improve the pH of the reaction medium. In the present study, concerning the methanization of kitchen scraps, the volume of biogas resulting from the methane fermentation of kitchen scraps mixed with ashes represents more than 1.5 times the volume of biogas from the methanization of kitchen scraps alone. In short, an improvement in the production of biogas from food waste in the presence of ash is obtained for mass proportions of ash between 20% and 30%.

Figure 7 illustrates once again the influence of ashes on the methane fermentation of pineapple peels. This mixture improves the volume of biogas produced and is optimal when the mass proportion of ash is 35%.

These results are close to those obtained in 1996 by Inanc et *al.* [(16)] who estimate that the pH of a bio-digester must be between 6.4 and 7.2 in order to ensure the physico-chemical balance of the reaction medium, with an optimal value around neutrality.

A combined reading of Figures 7 & 8 leads us to see the influence of pH on the production of biogas. In fact, the optimum in the control case was obtained 4 hours after the start of methanization for a pH = 4.44, whereas the optimum in the other cases (in combination with the ashes) took place 8 hours after the start methanization for a pH = 6.63 (case of 35% ash) and therefore closer to 7.

This residence time (Figure 10) remained less than 7 days, i.e. 4 days, therefore a low stay. In 1997, KOPP et *al.* [(17)] demonstrated that the pretreatment of the raw material has the effect of reducing the residence time in the digester.

Similarly, Philippe Pouech et *al.* [(17)] showed the influence of the nature of the substrate on the hydraulic residence time during digestion. In 2013, they were able to show, using substrates of agro-industrial origin, the positive impact of co-digestion on the reduction of residence time depending on the type of codigestion [(18)].

CONCLUSION

It is clear that the use of ashes remains an effective, sustainable and ecological solution to optimize the methanation of low pH waste, in addition to being free. It made it possible to have a production yield greater than 150 % compared to normal digestion (30% mass concentration of ash for food waste and 35% for pineapple peelings).

Declaration by Authors

Conflict of Interest: The authors declare no conflict of interest.

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