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Fertilizer Value Of Digestate From Effluent Methanization And Cassava Peel Ash

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Abstract – This study examined the composition of the digestate in terms of fertilizing elements, as well as its physical, chemical and biological quality. The digestate comes from the biodigestion of cassava effluent and cassava peel ash. Two types of digestate were analyzed: digestate 1 is made up of effluent + peel ash and digestate 2 of effluent + peel ash + cow dung. The digestates showed overall basic pH values of 7.16 - 7.98 for d1 and 7.35 - 9.62 for digestates 2. These pH values are favorable for soil and plants. Total Kjeldahl nitrogen levels for digestates 1 varied from 4800 to 4860 mg/L and from 5200 to 5400 mg/L for d2. Phosphorus values ranged from 3450 to 267.33 mg/L for digestates 1 and from 3800 to 308.58 mg/L for digestates 2. Total Kjeldahl nitrogen and phosphorus values comply with the standards set by the European Commission. A sharp reduction in pollutant load was observed in digestates 1 and digestates 2, with values ranging from 2617.84 to 541.02 mg/L and 3361.26 to 982.14 mg/L respectively. A strong decrease in germs and heavy metals was observed in both digestates.

Keywords – Anaerobic digestion, Agricultural, Biofertilizer, Organic waste, Biogas.

I. INTRODUCTION

Cassava (*Manihot Esculanta* Crantz) is one of the most important food crops in Côte d'Ivoire. It plays a crucial role both in food security for rural and urban populations and in job creation and income generation for the stakeholders involved [1]. Cassava processing and the marketing of the resulting products (attiéké, placali, tapioca, gari, etc.) is a widespread activity in Côte d'Ivoire. Indeed, it is a source of household income, particularly for women and the poor [2]. In a large part of the country, cooperatives have been set up to process cassava into a semolina called "attiéké", which is a local delicacy consumed throughout the country [2]. Processing cassava pulp into attiéké generates solid and liquid waste [3]. Liquid waste, also known as effluent, is almost always discharged untreated into the environment. As a result, they emit unpleasant odours and gases harmful to health and the environment during fermentation [2]. Indeed, the production of organic waste in agri-food and other industries is a major environmental and economic issue in Côte d'Ivoire, as well as in many other countries. Efficient and sustainable management of this waste can help to reduce environmental impacts, while creating added value, "that is, new profits". The treatment of organic waste using anaerobic digestion is a promising solution. This treatment system has the advantage of producing mainly biogas (made up of methane (CH_4) and carbon dioxide (CO_2) [4], and a co-product, digestate, which is a stable, deodorized residue, mostly free of pathogens and rich in nitrogen compounds [5] and [6].

It can be used as a raw material for composting solid organic waste (poultry droppings, cattle manure), as well as for amending agricultural soils [7] and [8]. Despite its advantages, digestate remains little exploited. The composition and quality of digestates depend on the raw material combined with the efficiency of anaerobic digestion. Digestate therefore needs to be tested against quality standards before it can be used as an agricultural biofertilizer.

This study aims to characterize the digestate from anaerobic codigestion of cassava effluent with cassava peel ash as cosubstrate.

II. Materials and Methods

2.1. Materials

The biological material used in this study consisted of cassava effluent from an attiéké factory in the Tazibouo district of Daloa, ash obtained after incineration of cassava peelings and used to neutralize the pH of the effluent, and cow dung from a farm in the Kennedy 1 district of Daloa. The latter is used to seed the reactors, as it is rich in methanogenic microorganisms, and to adjust the nitrogen content to balance the C/N ratio, as the effluent is low in nitrogen.

Temperature and pH were measured using a Fl24-2 multi-parameter. Center and cow dung were weighed using a precision balance (OHAUS). For heavy metals and TKN, a flame atomic absorption spectrophotometer (SAA) and a HACH spectrophotometer (DR/2010) were used respectively. For COD, a P/SELECTA digester (Digestate Block 6) (Spanish origin) was used.

2.2. Methods

The two types of digestates were obtained with a mixture of 18L effluent (E) + 753g peel ash (PA) for digestate 1 (d1) and 18L effluent (E) + 753g peel ash (PA) + 2kg cow dung (Cd) for digestate 2 (d2).

Samples were taken twice a week using a 10-mL pipette. Samples obtained after sampling are analyzed directly in the laboratory, as the methanization study took place on the premises of the Jean Lorougnon Guédé University.

Temperature and pH were measured using a Fl24-2 multi-parameter. Center and cow dung were weighed using a precision balance (OHAUS). For heavy metals and TKN, a flame atomic absorption spectrophotometer (SAA) and a HACH spectrophotometer (DR/2010) were used respectively. For COD, a P/SELECTA digester (Digestate Block 6) (Spanish origin) was used.

With regard to the parameters to be determined, pH and temperature were measured according to [9] NF T 90-008, Total Kjedahl Nitrogen (TKN) and ammonium according to AFNOR NF T90-015 and chemical oxygen demand (COD) according to AFNOR NF T90-101. [10] analysis method was used to characterize phosphorus (P) and trace metals (copper, iron, nickel and lead). As for bacterial germs, the EMB, Hektoen, Rapid medium and MOSSEL medium agar incorporation methods were used to enumerate, respectively, *total fecal coliforms* (NF T90-414) and (NFT 90-416), *Salmonella*, *Escherichia coli* and Bacillus cereus.

Data from this study were subjected to various statistical analyses using Paleotological Statistic (PAST 4.02) [11] and Excel 2016. Differences were considered significant for values of $p \leq 0.05$.

III. Results

3.1. Physico-chemical parameters

3.1.1. pH

The pH values of the digestates recorded showed a similar evolution over time.

The pH of digestate 1 (d1) (E+PA) decreased from 7.16 to 6.45 and then increased to 7.98, with an average of 7.40.

For digestate 2 (d2) (E+PA+Cd), a decrease from 7.35 to 6.76 was recorded. The pH then rose to 9.62, with an average of 7.93.

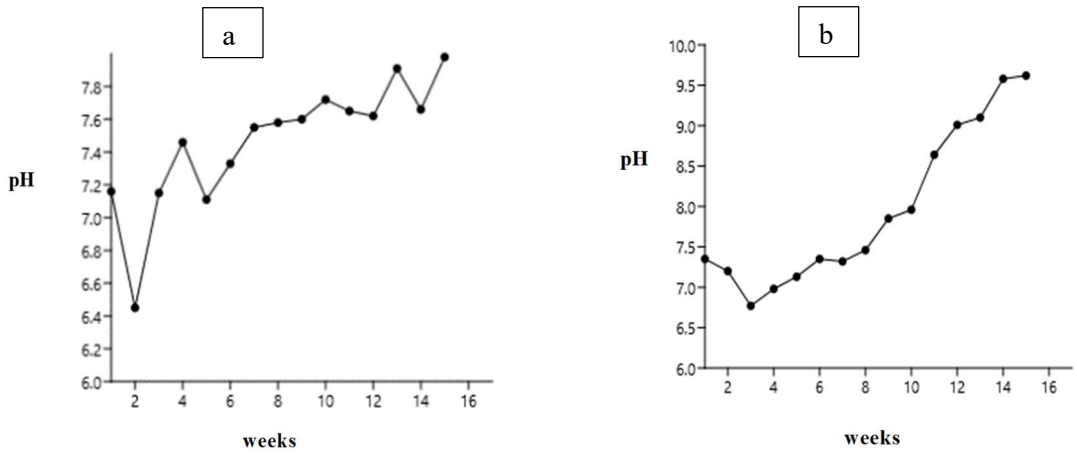

Figure 1: pH evolution as a function of time. a-(d1 (digestate 1) = E (Effluent) +As (Ash)) ; b-(d2= (digestate 2) = E (Effluent) + PA (Peel Ash) + Cd (Cow dung)).

3.1.2. Temperature

The temperature of d1 and d2 evolved in a similar way. This evolution took place in the mesophilic range from 20 to 40°C. Average temperatures were 34.48°C for d1 and 34.92°C for d2.

No significant differences ($p > 0.05$) were observed between digestate temperatures

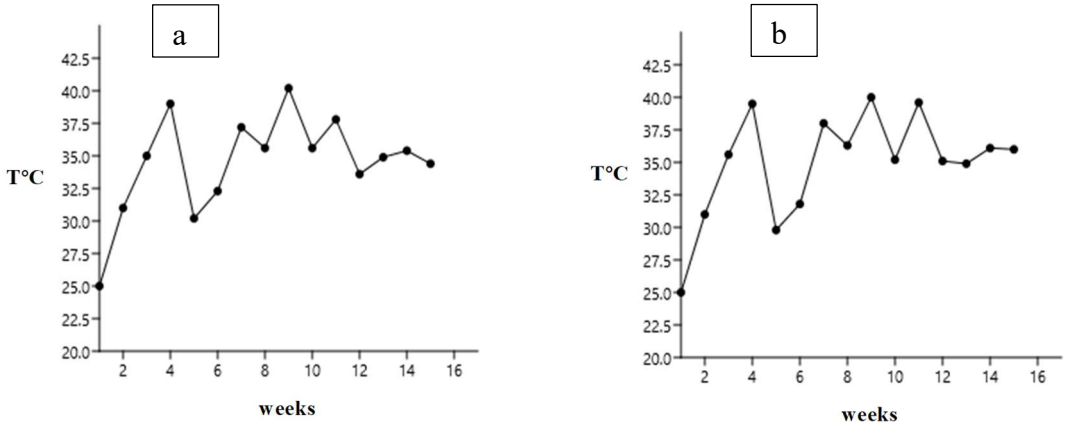

Figure 2: Temperature variation with time. a-(d1 (digestate 1) = E (Effluent) +As (Ash)) ; b-(d2= (digestate 2) = E (Effluent) + PA (Peel Ash) + Cd (Cow dung)).

3.1.3. Total Kjeldahl nitrogen (TKN)

Nitrogen levels in all digestates decreased slightly from 4800 mg/L to 2800 mg/L for digestate 1 and from 5200 mg/L to 3500 mg/L for digestate 2.

3.1.4. Ammoniacal nitrogen

The various digestates showed variations in ammoniacal nitrogen concentrations. Ammonium concentrations in d1 rose from 3450 mg/L to 8250 mg/L, before dropping to 106.1 mg/L.

That of d2 dropped from 3800 mg/L to 308.58 mg/L.

Statistical analyses show that there is no significant difference ($p > 0.05$) in ammonium between digestate types.

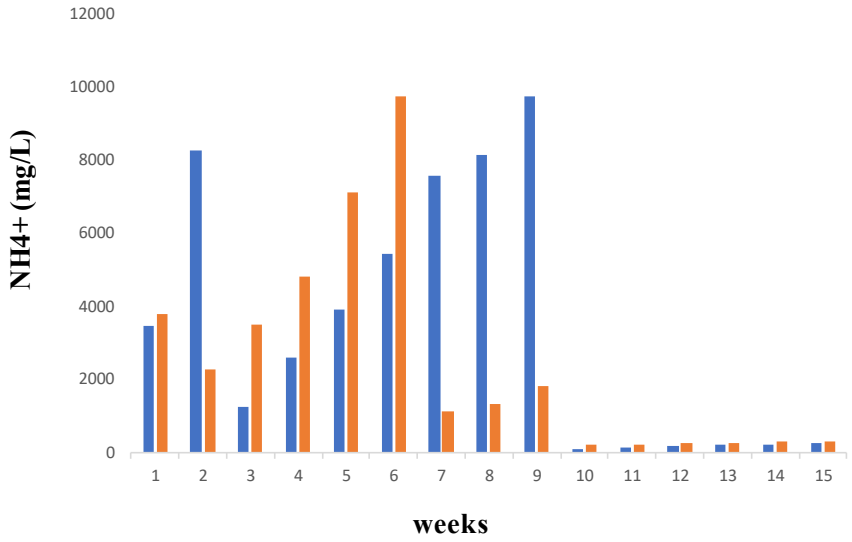

Figure 3: Evolution of ammoniacal nitrogen in digestate. Red represents the TKN of digestate 1 and blue digestate 2.

3.1.5. Phosphorus

In digestate 1, the phosphorus content was 840.5 mg/L and 360.4 mg/L. In digestate 2, it ranged from 884.5 mg/L to 621.47.

Statistical analyses showed a significant difference $p < 0.05$ between the two digestates.

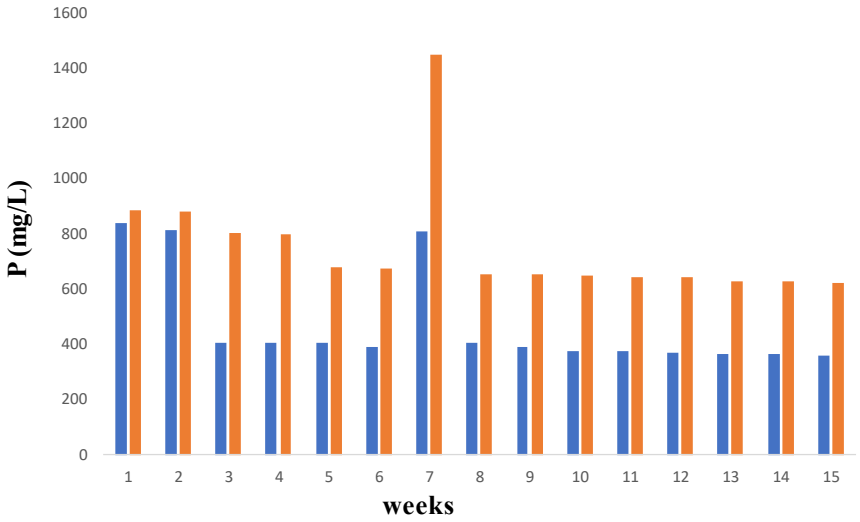

Figure 4: Phosphorus trends in digestate. Red represents the Phosphorus of digestate 1 and blue digestate 2.

3.1.6. Chemical oxygen demand (COD)

Initial effluent chemical oxygen demand (COD) values were 2617.84 mg/L and 3361.26 mg/L for digestates 1 and 2 respectively.

Values decreased for all digestates, with a reduction in pollutant load from 2617.84 mg/L to 541.02 mg/L and from 3361.26 mg/L to 982.14 mg/L respectively.

There were no significant differences in COD values in the digestates.

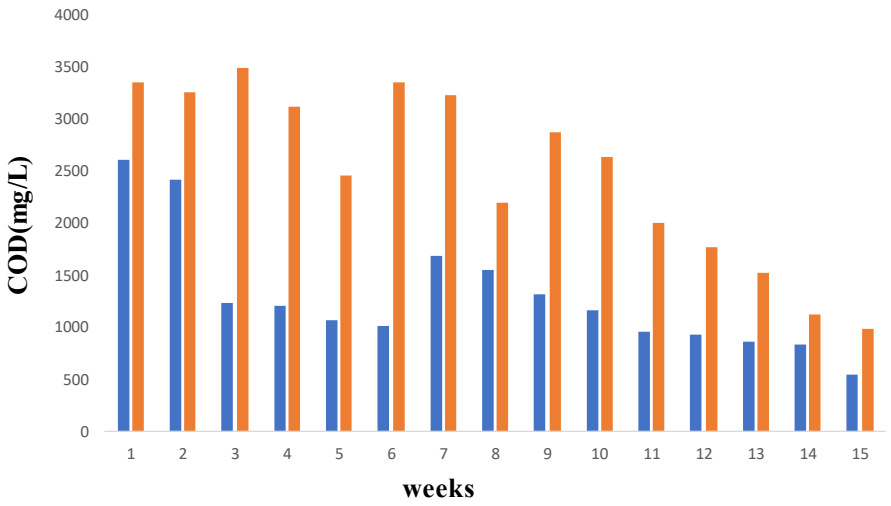

Figure 5: Evolution of COD in digestate. Red represents the COD of digestate 1 and blue digestate 2.

3.2. Microbial loads

Microbiological analysis revealed very high levels of total and thermotolerant coliforms. The values recorded were 154.10^5 CFU/100 mL and 139.10^5 CFU/100 mL and 155.10^5 CFU/100 mL and 178.10^5 CFU/100 mL respectively for digestate 1 and digestate 2.

E. coli values were 139.10^5 CFU/100mL for digestate 1 and 125.10^5 CFU/100mL for digestate 2.

Bacillus cereus levels were estimated at 56,^{105} CFU/100 mL for digestate 1 and *Bacillus cereus* for digestate 2.

No *salmonella* was observed.

After 50 days of digester operation, a sharp reduction in germs was observed.

Total and thermotolerant coliforms fell from 154,10^5 CFU/100mL to 20,10^5 CFU/100 mL and from 139,10^5 CFU/100 mL to 31,105 CFU/100 mL, i.e. abatement rates of 87.01% and 77.70%. For digestate 1. As for digestate 2, levels dropped from 155,10^5 CFU/100 mL to 24,10^5 CFU/100 mL and from 178·10^5 CFU/100 mL to 45,10^5 CFU/100mL, with abatement rates of 84.51% and 74.71% respectively.

E. coli decreased from 139,10^5 CFU/100 mL to 36,10^5 CFU/100mL for digestate 1 and 125,10^5 CFU/100 mL to 45,10^5 CFU/100 mL for digestate 2, with reduction rates of 71.20% and 74.71% respectively.

Bacillus *cereus* levels dropped from 56,10^5 CFU/100 mL to 5,10^5 $^{CFU/100mL}$ with a 91.07% abatement rate for digestate 1 and *Bacillus cereus* and 56,10^5 CFU/100 mL to 10,10^5 CFU/100 mL with an 86.84% reduction *for* digestate 2.

Table I: Microbial loads of digestates.

Germs	Total coliforms (UFC/100mL)	Thermotolerant coliforms (UFC/100mL)	*E. coli* (UFC/100mL)	*Bacillus cereus* (UFC/100mL)	Salmonelles
d1 (input)	154.10^5	139.10^5	125.10^5	56.10^5	Présence
d1 (output)	20.10^5	31.10^5	36.10^5	5.10^5	Absence
Discount rates	87,01%	77,70%	71,20%	91,07%	100%
d2 (input)	155.10^5	178.10^5	157.10^5	76.10^5	Présence
d2 (output)	24.10^5	45.10^5	56.10^5	10.10^5	Absence

Discount rates	84,51%	74,71%	64,33%	86,84%	100%
Standard (digestate)	$<10^3$ UFC/g	$<5.10^3$ UFC/g	$<5.10^3$ UFC/g		Absence dans 25g

3.3. Trace metals (TMEs)

Overall, ETMs showed a reduction in both types of digestate.

In digestate 1 (d1), Copper decreased from 0.123 mg/L to 0.027 mg/L, Chromium from 2.347 to 1.568 mg/L, Cadmium from 0.000021 to 0.00001 mg/L, Lead from 0.0431 to 0.0225 mg/L, Nickel from 0.001111 to 0.001894 mg/L. On the other hand, a slight increase was observed in Iron from 3.583 to 4.209 mg/L and Arsenic from 0.01234 to 0.03515 mg/L.

As for digestate 2, a decrease from 0.176 to 0.046 mg/L in Copper and from 0.0448 to 0.03028 mg/L in Arsenic was recorded. Chromium decreased from 5.138 to 3.458 mg/L, Zinc from 0.923 mg/L to 0.234 mg/L, Cadmium from 0.000092 to 0.00001 mg/L, Nickel from 0.002655 to 0.002049 mg/L and Lead from 0.0622 to 0.03581 mg/L were also observed.

Table II: Trace metal content of digestate

	TMEs							
Digestate	**Cu (mg/L)**	**Fe (mg/L)**	**As (mg/L)**	**Cr (mg/L)**	**Zn (mg/L)**	**Cd (mg/L)**	**Ni (mg/L)**	**Pb (mg/L)**
d1 (input)	0,123	3,583	0,01234	2, 347	0,216	0,000021	0,001111	0,0431
d1 (output)	0,027	4,209	0,03515	1,568	0,158	0,00001	0,001894	0,0225
Discount rates	78,04%	- 17,47%	- 184,84%	33,19%	26,85%	52,38%	-70,47%	47,79%
d2 (input)	0,176	6,1	0,0448	5,138	0,923	0,000092	0,002655	0,0622
d2 (output)	0,046	4,12	0,03028	3,458	0,234	0,00001	0,002049	0,03581
Discount rates	73,86%	24,39%	32,41%	32,69%	74,64%	89,13%	22,82%	42,42%
Standard (digestate)	≤ 300 mg/Kg		≤ 18 mg/Kg	≤ 120 mg/Kg	≤ 600 mg/Kg	≤ 3 mg/Kg	≤ 60 mg/Kg	≤ 180 mg/Kg

IV. Discussion

The pH values recorded showed an evolution over time. However, a short-lived drop was observed in the first few days, followed by an increase towards increasingly basic values at the end of the experiment. This drop could be explained by the formation of acids during the degradation of organic matter in the medium. These results are in line with those of [12], who argues that acidogenesis is a phase of accumulation of Volatile Fatty Acids (VFAs) that can lead to a drop in pH and dysfunction of the metabolic activity of methanogenic bacteria. According to [13], the change in pH is due to pH stabilization by acetogenic bacteria, which dissociate the organic acids produced (lactic acid, acetic acid, propionic acid, etc.). It should also be pointed out that ash plays a role in pH evolution, given its basic nature. This is in line with [14] opinion that the ash's basicity is a very important factor in increasing pH. In addition, the production of hydroxide ion (OH^-) correlative to that of ammonium raises the pH of the medium, affirming the basic characteristic of the digestate [15].

In terms of TKN, analyses showed slight variations in the digestates. We note that most of the nitrogen present is organic nitrogen, due to the virtual disappearance of ammoniacal nitrogen. This implies that the total nitrogen observed represents organic nitrogen. Indeed, the high organic nitrogen content of digestates could be explained by the conservation of TKN [16]. The

nitrogen content of methanizers is in line with that of [17], who states that the nitrogen content of digestate intended for agricultural amendment should be between 1.5 and 6.2 g/Kg. However, it should be pointed out that cow dung had no real impact on nitrogen content. Contrary results were observed by [18], who point out that cow dung is naturally rich in nitrogen.

The presence of ammoniacal nitrogen is due to the mineralization of the substrate's organic nitrogen into ammonium [19] and [20]. Indeed, according to [20], in this form, ammoniacal nitrogen gives the digestate a high fertilizing power because it is easily assimilated by plants. However, the drop-in ammonium content may be due to the microbial conversion of organic matter into biogas. Part of the ammonium is converted into gaseous compounds such as ammonia (NH_3) and dinitrogen (N_2). The conversion to ammonia takes place in the presence of a high pH, when the pH is around 7 or even higher [21]. [12] also refers to nitrification, a process by which nitrifying bacteria of the *Nitrosomonas*or *Nitrobacter* genus can oxidize ammonium to nitrate (NO^{3-}). In the digestate, complex chemical reactions can occur, involving other elements and compounds present, which can lead to a decrease in ammonium. Moreover, this decrease is a natural phenomenon resulting from the biological, chemical and environmental processes that occur during anaerobic digestion and digestate storage.

For phosphorus, the results showed a decrease after the first week for d1 and the second for d2. It remained constant in the digesters until the end of the experiment. This drop can be explained by the fact that, during anaerobic digestion, part of the phosphorus is solubilized in the liquid fraction and then forms insoluble mineral substances that precipitate on the bottom and walls of the digesters (struvite ($M_gNH_4PO_4$) and calcium phosphate (hydroxyapatite)) [22]. The observed levels of 360.4 mg/L for c1 and 621.47 mg/L for d2 are in line with those reported by [23], who show that phosphorus levels in digestate range from 0.2 to 2.6 g/Kg. Methanization increases the availability and efficiency of phosphorus in digestate [24]. It is mainly involved in **root development**, energy transfer (ATP), transmission of hereditary traits (nucleic acids), carbohydrate degradation and photosynthesis. This element is essential for flowering, fruit set, earliness, fruit swelling and seed ripening [25]. An adequate phosphate supply boosts plant maturity. Digester 2 has higher levels of phosphorus, due to cow dung.

In both reactors, COD fell progressively and considerably over the course of the experiment. This reduction in pollutant load would be linked to the potential consumption of organic matter by the purifying microflora during its natural evolution in the digesters [26] and would indicate that the digesters are functioning well.

In terms of microorganisms, the results show a very marked reduction in germs in all the digesters. All the bacteria analysed showed a decrease over the 50 days. As [27] also observed, salmonella disappeared altogether from the digestates, i.e. a total reduction (100%). As for total and thermotolerant coliforms, although they have decreased, their levels in the digestates are higher than the standards observed by [28]. *E. coli* concentrations are also above regulatory thresholds, with values below 1,000 CFU/g in 4 samples and below 5,000 CFU/g in one sample [29]. Similarly, despite their sharp decline, *Bacillus cereus* levels exceed the threshold values set by European Commission regulations. These observations may be due to the combination of temperature and residence time. In fact, anaerobic digestion took place over 50 days, during which the average

As far as TMEs are concerned, their content is one of the major concerns of biofertilizers. Methanization has the effect of concentrating them in digestates, in the same way as nutrients. The concentration factor of TMEs depends in part on the methanogenic potential of the raw materials. Indeed, the more carbon will be extracted in the form of biogas, the more mineral matter, and therefore MTEs, will be concentrated in the outgoing digestate) [30] and [4]. The results show a decrease in all digesters except for Iron, Arsenic and Nickel in digester 1. The increase in iron content may be due to the presence of rust, which has potentially been accentuated by the ash given that it has been solubilized and contains elements such as potassium oxide, which is corrosive. For Arsenic and Nickel, the increase in their levels may result from the initial composition of the inputs, chemical reactions in the digester or microbial activities. The cow dung added to the second reactor probably triggered chemical reactions that prevented an increase in the levels of these TMEs [27] consider that the use of a biofertilizer with a high TME content can lead to contamination of land and plants, remain in the soil and pollute groundwater. As a result, digestate2 is more suitable for use, as concentrations of all TMEs have decreased and are below standards.

V. Conclusion

This study was carried out with the aim of demonstrating the quality of digestates through the characterization of cassava effluent combined with ash. The digestates resulting from the experiments showed basic pH values (d1 : 7.16 - 7.98 ; d2 : 7.35 - 9.62) suitable for soil and plants, NTK (d1 : 4800 - 4860 mg/L; d2 : 5200 - 5400 mg/L) and Phosphorus (d1 : 3450 - 267.33 mg/L ; d2 : 3800 - 308, 58 mg/L) levels in line with established standards, a sharp reduction in pollutant load (d1 : 2617.84 - 541.02; d2:

3361.26 - 982.14), germs were significantly reduced or even eliminated, and trace metal elements were in line with standards, except for a few in digestate 1, which could be interpreted as a drop in quality. In view of the above, digestate2 from the second methanizer would be more suitable for use. Experiments could be carried out between digestate 2 and chemical fertilizer to find out which is more suited to nutrient requirements, and may be safe for cuttings.

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