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Characterisation of agricultural Waste for Anaerobic co-Digestion: a Case Study of Yellow and Violet Maize Bran from Katiola, Côte d'Ivoire

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Abstract: Maize is still the most widely grown cereal in the world, particularly in Katiola (Côte d'Ivoire). This study looks at the added value that can be added to its waste, a biomass that is rarely used for energy purposes. The aim of the study is to produce biogas from this waste using anaerobic co-digestion. The biomasses selected for the study are yellow and purple maize bran from Katiola. By analysis, according to the AFNOR standard, the BOD_5/COD ratio is greater than 0.6 for the infusion juice, organic matter is 67.8% in the bran, and fibres represent 80.67% for the bran. These results confirm the biodegradability of the waste. After characterisation, a discontinuous biodigester equipped with a gasometer was designed and built to produce and quantify the biogas obtained by anaerobic digestion and codigestion. The respective productivities of 41m^3/tonne OM, 167.2m^3/tonne OM and 54.5m^3/tonne OM during the anaerobic codigestion of yellow and violet bran and the mixture of yellow and violet maize bran were obtained.

Keywords: Methanisation, maize waste, biogas, co-digestion

1. Introduction

Human beings are faced with major existential challenges, including environmental pollution, the corollary of which is climate change, and energy insecurity (Mrosso *et al.*, 2023). These days, the climate is changing everywhere, with visible effects (Valérie, 2020). This ongoing change is due to past and current greenhouse gas emissions. In the short term (to 2050), our choices can reduce the risks associated with unavoidable climate change. Long-term trends (after 2050) will depend crucially on future greenhouse gas emissions (Michel, 2008). There are many options for taking action and addressing the risks, through adaptation and by reducing greenhouse gas emissions. The energy-hungry lifestyles are sustained solely by these non-renewable fossil fuels (Christian, 2015; Alaoui *et al*. 2014). In this context, according to one of the objectives of the TOKYO Protocol (1997), decision-makers and scientists are constantly seeking solutions to these major concerns for sustainable development, hence the holding of Conferences of the Parties (COP). In light of this, Circular Economy emerges as an effective tool for triggering a sustainable development process (Arruda *et al.*, 2021). In the context of the circular economy, optimising the biogas produced in the anaerobic digesters is fundamental to ensure the energy self-sufficiency (Azevedo *et al.*, 2023). In fact, Anaerobic digestion is one of the technologies that will play a key role in the decarbonization of the economy, due to its capacity to treat organic waste, recover nutrients and simultaneously produce biogas as a renewable biofuel (González *et al.*, 2022). used by the use of fossil fuels. Anaerobic digestion (AD) is practiced in various processes and has become an important part of renewable energies (Ulukardesler, 2023). However, several aspects are still pending a solution such as the profitability of the whole treatment system and the need to increase conversion efficiency to reduce installation costs (González *et al.*, 2022). For example, the accumulation of fatty acid in the digester inhibits substrate digestion. Consequently, as stand-alone it is not suitable for anaerobic digestion (Ingabire *et al.*, 2023). The addition of a co-substrate to any of these systems aids in balancing the C/N ratio and improving the global process performance allowing a better economic balance by increasing profits (González *et al.*, 2022). Anaerobic co-digestion (AcoD) is a technology fulfilling the concept of waste-to-energy (WtE) based on local resources (Fernández-Rodríguez *et al.*, 2023). In recent years, diferent studies have investigated ways of producing biogas through the codigestion of organic wastes (Mrosso *et al.*, 2023) : anaerobic co-digestion of grass, cow manure and sludge was studied under mesophilic conditions for 65 days. Experiments were performed on a feed ratio of grass/manure from 5 to 25%, respectively (Ulukardesler, 2023); anaerobic digestion (AD) of sewage sludge was optimised by adding biowastes (Mudzanani *et al.*, 2022) and trade wastes (TWs) because of their nutrient content and boost in biogas formation if non-inhibitory (Berzal De Frutos *et al.*, 2023); a mixture of papaya peels, water, cow manure, and a base produced an impressive 80.75% methane yield within 75 days under mesophilic conditions with a specific pH and temperature range (Hasan *et al.*, 2023). In addition, Omondi *et al.*, (2023) demontrated that the co-digestion of water hyacinth (WH) with ruminal slaughterhouse waste (RSW) has the potential to improve biogas production from WH through collation of processes parameters such as the C/N and C/P ratios, potassium concentration and buffering capacity.

This study looks at the use of agricultural waste, in particular yellow and purple maize bran from Katiola (Côte d'Ivoire). These waste products have interesting energy properties (FAO, 2016). The convincing results of these analyses will provide new hope for producers, despite the poor sales of agricultural products. For the specific purposes of our project, therefore, we chose katiola violet maize bran. This is obtained when the endosperm (the part containing around 90% of the starch) of the maize seed is transformed into semolina (flour). It consists mainly of the pericardium (husk) and the embryo (germ, rich in nutrients). Maize (Zea mays L.) belongs to the paoceae family of grasses and the panicoideae subfamily. It is the most widely cultivated plant in the world (Kambalé *et al.*, 2023; Guedou *et al.*, 2015). In Côte d'Ivoire in particular, it is the second most widely grown cereal after rice. Katiola in the Hambol region, in the centre-north of Côte d'Ivoire, is the main maize production area, with 1,568,000 tonnes in 2021 (Kouakou *et al*, 2010; N'da *et al.*, 2013). From harvest to consumption of maize in all its forms (semolina, braised and grilled), more than 70% of the cob is abandoned or burnt.

The contribution of this research is to add value to these maize rejects. After collection, our approach will be to determine the physical and chemical characteristics of Katiola violet maize bran, i.e. the MS, MSV, COD, BOD5, moisture, fiber and lignin content. This will be followed by an assessment of the production capacity of the selected samples, blended alone.

1. Material and methods

1.1. Nature and origin of biomass used

As biomass, we were interested only in biodegradable waste (food processing residues): yellow and purple maize bran from Katiola. This waste was collected near the small village processing units of FRONAN, which comprises five villages 7 km from Katiola in the HAMBOL region. According to geographers, this region lies between 8°10' north latitude and 5°4' west longitude. The vegetation is wooded savannah (pre-forest). The average temperatures recorded in the area vary between 26; 45°C and 33; 67°C **(Figure 1).**

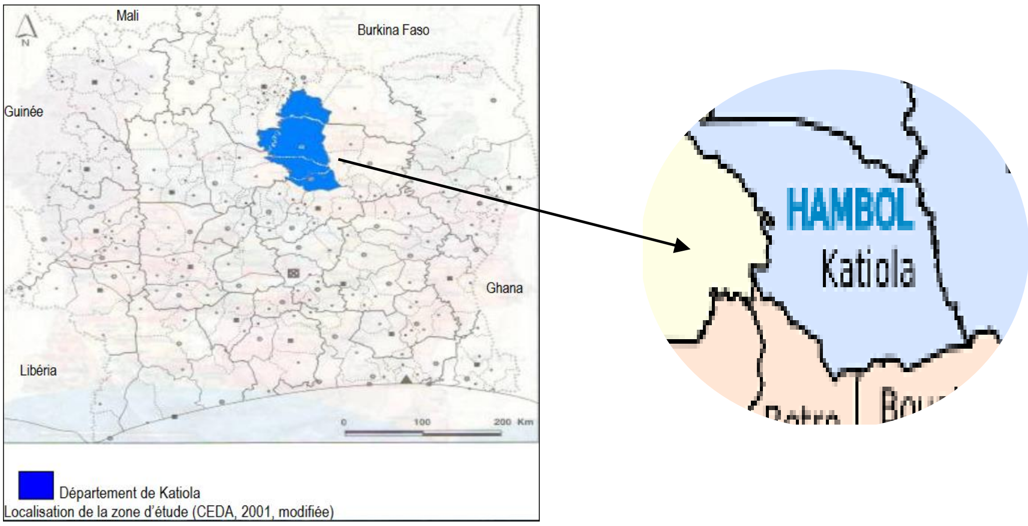

Figure 1 : Geographical location of the HAMBOL Region

Initially, the maize cobs **(Figures 2a and 2b)** are lightly dried in the sun, then the seeds are separated from the cob. This collection of kernels is washed and rinsed. At the mill, the bran (epidermis + pericardium) **(Figure 2c and Figure 2d)** is obtained from the maize kernels and the crushed maize kernels (endosperm). Our mothers used to mix the maize with fine, clean sand and a little water, and the whole mixture was crushed in a suitable mortar and then winnowed.

Figure 2a **Figure 2b** **Figure 2c** **Figure 2d**

Figure 2. Varieties of maize and maize bran

The crushed maize (endosperm) obtained is washed, rinsed and placed in hot water at around 80°C. After three days, the maize infusion juice is obtained. In the old days, in FRONAN, this juice, with its smell of alcohol, was used as a diluent for cow dung to paint the inside of round terracotta huts. This makeshift paint was used to eliminate fleas and lice. To begin with, we collected samples of freshly made maize bran from a number of village mills. After collecting them, we packaged them in plastic containers. In Yamoussoukro, the bran was laid out in the shade for two days. At the INP-HB plant, they were ground and sieved or not (to 200µm diameter).

1.2.Characterisation of samples

Once collected, the maize bran was crushed and sieved (200µm diameter). After pre-treatment of the maize rejects, physical and chemical analyses of the waste were carried out in the LCA-U-Man laboratory, in accordance with French standards.

Finally, to ensure the reliability of our plant, we have to compare the results obtained with the measurements of the biogas meter, the gas chromatograph and the theoretical equations.

The PH was measured using a HANNA multiparameter, model HI 9829.

1.2.1. Humidity (H)

The moisture content of the samples was measured using the AFNOR NFU 44-171 method of October 1982. This consists of steaming a quantity of waste equal to or greater than 100 ± 0.1 g at 105 ± 2°C for 24 hours until a constant mass is reached (AFNOR, 1996). In this study, 50g of waste was taken from households and from the landfill, then oven-dried at 105°C (Table 5). The moisture content (H) was determined by the difference between the mass of the sample before and after drying (Abollé *et al.*, 2022) (**Eqn. 1**):

$$\%H = \frac{M0-M1}{M0} 100 \qquad \textbf{Eqn. 1}$$

Where %H: percentage of moisture; M0 = initial mass of the sample before drying (50g); M1 = final mass of the sample after drying at 150°C.

1.2.2. Volatile dry matter or organic matter (OM) content

Volatile dry matter was determined using waste that had previously been oven dried at 105°C to constant mass and calcined at 550°C for 4 hours in a Nabertyerm kiln. The rate of volatile dry matter MSV or organic matter MO is obtained by **Eqn. 2**:

$$\%MO = \frac{M_1-M_2}{M_1} 100 \qquad \textbf{Eqn. 2}$$

Where %MO = percentage of organic matter; M_1 = mass of waste at 105°C;

M_2 = mass of waste at 550°C.

1.2.3. Cellulose, hemicellulose and lignin content

Analysis of extractives, hemicellulose, cellulose and acid-insoluble lignin was carried out according to the protocol described by Bakar (2017). Extractable, hemicellulose, cellulose and acid-insoluble lignin assays were carried out. The analysis was carried out several times with a maximum standard deviation of 5%. The sample was ground, sieved (< 500µm) and dried at 105°C for 12 hours. The various stages of the protocol adopted are described below.

- Extractable content soluble in toluene-ethanol

A 3g sample (M_1) was used to extract the extractives. The sample was dispersed in 150mL of a toluene-ethanol solution. The mixture was stirred for 3 hours. The residue obtained by Büchner filtration was dried at 105°C for 2 hours and then weighed (M_2). The mass of extractables corresponds to the mass loss of the sample. The fraction of extractables corresponds to the mass loss of the sample. The fraction of extractables is determined using **Eqn. 3**:

$$E_X\,(\%) = \frac{M_1 - M_2}{M_1}\;100 \qquad \textbf{Eqn. 3}$$

The residue obtained is used to determine hemicellulose and acid-insoluble lignin content.Teneur en hémicellulose

One (1) g sample (M3) of the residue was dispersed in 150mL of NaOH solution (20g/L). The mixture was placed in a flask and topped with a reflux system.

It was boiled for 3.5 hours. The solid obtained by Büchner filtration was washed 4 times with 150mL of distilled water to remove Na+ ions. It was then dried at 105°C for 24 hours and weighed (M4). The hemicellulose content H% is determined from the loss in mass of the sample following this treatment (**Eqn.4**):

$$H\% = \frac{M_3 - M_4}{M_3}\,.\,(100 - Ex) \qquad \textbf{Eqn. 4}$$

- Acid-insoluble lignin content

A volume of 30mL of 72 % sulphuric acid is gently poured over a mass of approximately 1g (M_5) of the biomass residue obtained from the toluene and ethanol extraction step. The mixture was kept at a temperature of between 8°C and 15°C for 24 hours. A volume of 300mL of distilled water was then added and the mixture was boiled for 1 hour using a reflux system. The solid obtained was filtered on a Büchner, filter-washed 3 times with 150mL of distilled water to remove SO_4^{2-} ions, dried at 105°C for 4 hours and weighed (M_6). The lignin content L% is determined from the final mass of the solid residue (**Eqn. 5**):

$$L\,(\%) = \frac{M_6}{M_5}\,.\,(100 - E_X) \qquad \textbf{(Eqn. 5)}$$

- Cellulose content

The cellulose content C (%) is determined by **Eqn. 6** :

$$C\,(\%) = 100 - E_X - H - L \qquad \textbf{(Eqn. 6)}$$

- Fiber content

The fiber content F (%) is determined by **Eqn. 7**:

$$F\,(\%) = H\% + C\% + L\% \qquad \textbf{(Eqn. 7)}$$

1.2.4. Biological oxygen demand (BOD5) content

The Biological Oxygen Demand required to oxidise organic matter biologically. This is used to assess the biodegradable fraction of the carbonaceous pollutant load in a solution. It is calculated after 5 days at 20°C in the dark. The BOD meter was used to analyse the biochemical oxygen demand (BOD_5). Preparation and pre-treatment consisted of homogenising the sample and adjusting the pH of a portion of each sample to 7±1 using an appropriately concentrated NaOH or H_2SO_4 solution. If the presence of chlorine in the sample is suspected, it is treated by adding sodium sulphite: the sample is homogenised and 1 mL of sodium sulphite solution is added per 250 mL of sample. To read the dissolved BOD_5 demand value, a portion of the sample is filtered through a Whatman 934 AH filter. **Table 1** summarises the equipment used to characterise the samples.

Table 1: The equipment used at the University of Man's central analysis laboratory (LCA-U-Man)

Designation	appareillage	Trademark
BOD_5	DBO-meter	WTW^{TM} 208210
Humidity	Oven	TCN 115
Organic Matter	Muffle furnace	NABERTHERM
Cellulose, hemicellulose, lignin, fibre	Device + centrifuge	VWR MEGA STAR 600
Biogas composition	Biogaz mètre	GEOTECH GA5000
Methane concentration	Gas chromatograph	

1.3. Monitoring biogas production

1.3.1. Experimental protocol used for biogas production

The experimental protocol is shown in **Figure 3** and **Figure 4.** Un bidon de 20L a été utilisé comme digesteur. La température interne a été maintenue autour de 34°. Une conduite flexible en plastique est reliée au digesteur à travers un perfuseur médical comportant une clé. Le canal du perfuseur est connecté à un bidon d'eau minéral gradué de 5L (gazomètre). La mesure du volume du biogaz est réalisée par la méthode de déplacement d'eau. (Figure 4).

Figure 3 : Actual installation of the biodigester and gasometer

1.3.2. Digester feedstock

The biodigester is loaded as follows:

- First, 1kg of dried and ground bran was added
- Secondly, 2L of endosperm infusion juice (the part of the grain that contains around 90% of the starch) was added
- Finally, 2 litres of tap water were added.

The biodigester is coloured black to trap the heat from the sun's rays. The organic matter is heated to between 28°C and 34°C inside the digester. The digester is constantly agitated. After 12 days, the digester is sufficiently swollen. The pipe from the biodigester to the gasometer, which is well graduated in mL, is opened (Figure 5). The volume of biogas is obtained by moving the water level.

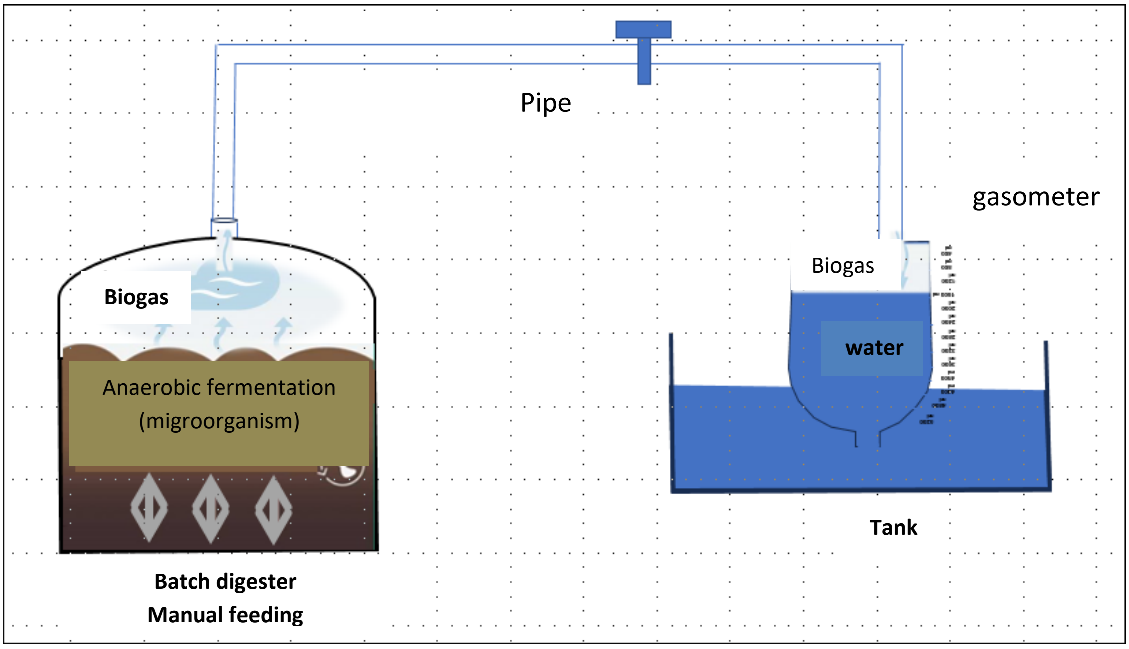

Figure 4 : Synoptic diagram

2. Results and discussion

The biodegradability of used waste was investigated using physical and chemical analyses (Tables 6, 7 and 8 below). After that, the production of biogas equivalent in CO_2 was conducted. This was done using a batch digester methanisation system.

2.1.Physical and chemical characteristics of maize bran

Table 2 presents the chemical and physical **characteristics.**

Table 2 : The results of measurements of the chemical and biological oxygen demand of infusion juices

Sample	PH	Potentiel Redox (mV)	Oxy. Dis (mg/L o2)	Cond. (µS/Cm)	TDS (mg/L)	Salinity (%)	Tur. (NTU)	T°C	COD (mg/L O2)	BOD_5 (mg/L O_2)
Violet bran	3.97	0	0.35	4291	2148	2.27	742	28.71	24.46	16.87
Yellow bran	3.87	47.7	0.38	3004	1502	1.55	737	28.92	22.82	13.56

The ratios BOD5/COD = 0.59 for yellow and BOD5/COD = 0.69 for violet. When the BOD5/COD ratio > 0.3, the waste is rich in organic matter. Mesophilic biodegradation can therefore be envisaged at a temperature of around 28°C. This solution has an acid pH. The addition of cow dung can raise the pH to between 6 and 7 in order to increase the populations of methanogenic agents (Kouame *et al.*, 2023). Turbidity is around 700 NTU. This turbidity is made up of suspended matter and colloidal particles to which many micro-organisms favourable to anaerobic co-digestion attach themselves. The presence of finely divided suspended matter is quite significant. The redox potential averages 47 Mv. The relatively low redox potential favours the maintenance and growth of methanogenic bacteria. Redox reactions provide microorganisms with chemical energy (AERMC, 2012). Conductivity of around 300µS/cm reflects the normal degree of mineralisation of the infusion juice, leading to a moderate salinity level of around 1.5. This quality makes it possible to control the concentration of mineral molecules that could inhibit methanisation (Moletta, 2009).

2.2.Moisture content, ash content and organic matter content (solid waste)

Table 3 presents Humidy, Ash content, organic matter content, carbon content and pH. Looking at the results presented, the first observation is that the waste is rich in organic matter (OM) (%MSV greater than 60%) and relatively rich in carbon (%COT greater than 45% on average). This waste is predisposed to being biodegradable. These values are close to those found in the literature (USAMA 2010; Joao A Amarante, 2010). Our waste therefore has a high content of non-soluble sugar (Afilal et al., 2014). This makes the pH of the mixture somewhat low and consequently an increase in methanogenic bacteria will be observed. This sugar content enables us to estimate their biogas production capacity at around 400mL/g MSV (Lacour et al., 2011). However, this gas production is still low. To remedy this, it is necessary to control and maintain the pH of the medium at around 7. We recommend codigestion in the presence of cow dung, or sewage sludge from WCs, or slaughterhouse waste or manure, which are basic. (Mata-Alvarez J. et al., 2014; Gildas-David, 2010).

Table 3 : Results of measurements of organic matter levels in purple maize bran

Sample	Humidity (%)	Ash Content (%)	Organic Matter (%)	%COT	pH
Violet bran	14.6	17.6	67.8	39.30	5.5
Yellow Bran	17	17.2	65.8	38.14	5.2

2.3. Fibre content: hemicellulose, cellulose and lignin

The values for hemicellulose, cellulose and lignin content are given in **Table 4.**

Table 4. Results of measurements of the fibre content of maize bran

Samples	Extraction rate (%)	Hemicellulose (%)	Lignin (%)	Cellulose (%)	Fiber (%)
Violet bran	19.33	17.01	18.55	45.11	80/67
Yellow bran	21	14.91	16.8	47.29	79

It appears that the relatively high levels of cellulose and fibre in our solid waste. However, lignin and hemicellulose are relatively low. These results confirm the biodegradability potential of our dried and ground solid waste. The presence of cellulose and hemicellulose contributes to and improves the anaerobic degradation of composite materials (Sujeto, 2011). The high fibre content gives an acceptable probability of degradable fibre.

2.4.Production of biogas by methanisation

2.4.1. Daily biogas production: the case of yellow maize bran

Daily biogas production with yellow maize bran is shown in **Figure 5.** Twelve (12) days after loading the biodigester, we obtained the results shown in Figure 5 above. The high biogas rate observed on the 13th day can be explained by another aerobic phenomenon called composting, which is simultaneous with methanisation, since our biodigester was loaded to 70%. The 30% of air allowed transient composting according to **Eqn. 8**:

$$MO + O_2 \longrightarrow MO_{stable} + CO_2 + H_2O \qquad \textbf{Eqn. 8}$$

This happens during 1 to 3 days.

However, methanisation continued according to the equation **Eqn. 9**:

$$MO \longrightarrow MO_{stable} + CH_4 + CO_2 + H_2O \quad \textbf{Eqn. 9}$$

Production then stabilised at 500mL on days 14, 15 and 16. A slight drop from 380 mL on day 17 to less than 200 mL on day 24. The stabilisation of organic matter was noticeable from day 20. In short, during this codigestion, 41 mL was released, i.e. 41 m^3/ton of organic matter (OM). According to the literature, biogas from agricultural waste contains around 60% methane (Jonathan Hess, 2007). This quantity corresponds to 24.6 mL (24.6 m^3/ton OM) of CH_4.

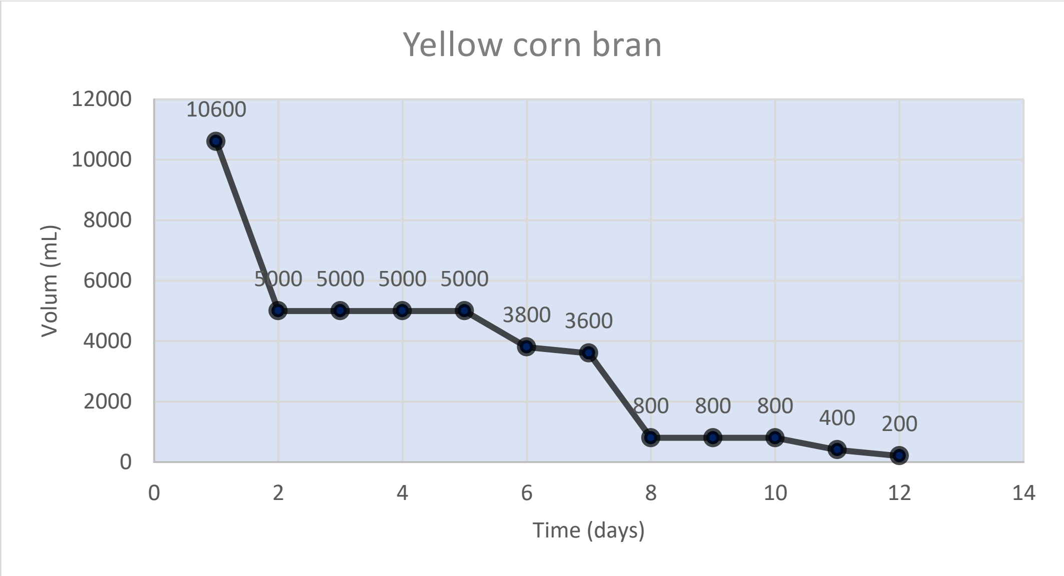

Figure 5 : Biogas production curve as a function of time: the case of yellow bran

2.4.2. Daily biogas production: the case of purple maize bran

Daily biogas production with purple maize bran is shown in **Figure 6.**

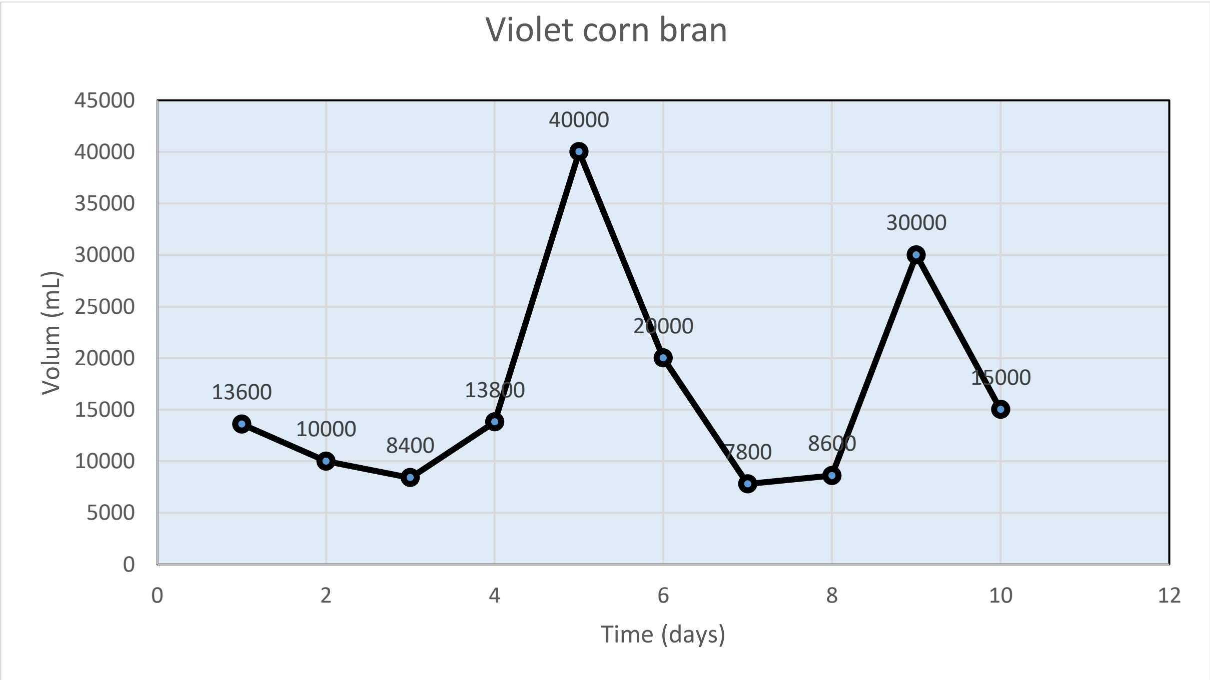

Figure 6. Daily biogas production curve for purple maize bran

Twelve (12) days after loading the digester, the biodigester is well swollen. The pipe connecting the digester and the gasometer is opened.

Day 1: we record a production of 13.6 mL of biogas. This high biogas rate can be explained by the simultaneous composting (aerobic codigestion) and methanisation (anaerobic codigestion) of the biomass.

From the 2nd day onwards, only anaerobic codigestion takes place. Hence the low level of biogas production (from 13600 to 8.400 mL).

From the 5th day onwards, production peaks at 40 mL, before dropping back to 8.60 mL 3 days later. The micro-organism population and digestion activity had reached their peak.

On day 9, another average peak of 30 mL was observed, before halving on day 10 to 15 mL.

Much of the organic matter stabilised.

In short, the biogas productivity of purple maize bran is around 167.2 m^3/ton of organic matter MO of biogas, giving a methane value of 100.2 m^3/tonne of organic matter.

2.4.3. Production of the mixture of yellow and purple maize bran

In addition to the individual productions, a mixture of the two types of maize bran was produced. The results of monitoring the production of this mixture are shown in **Figure 7.**

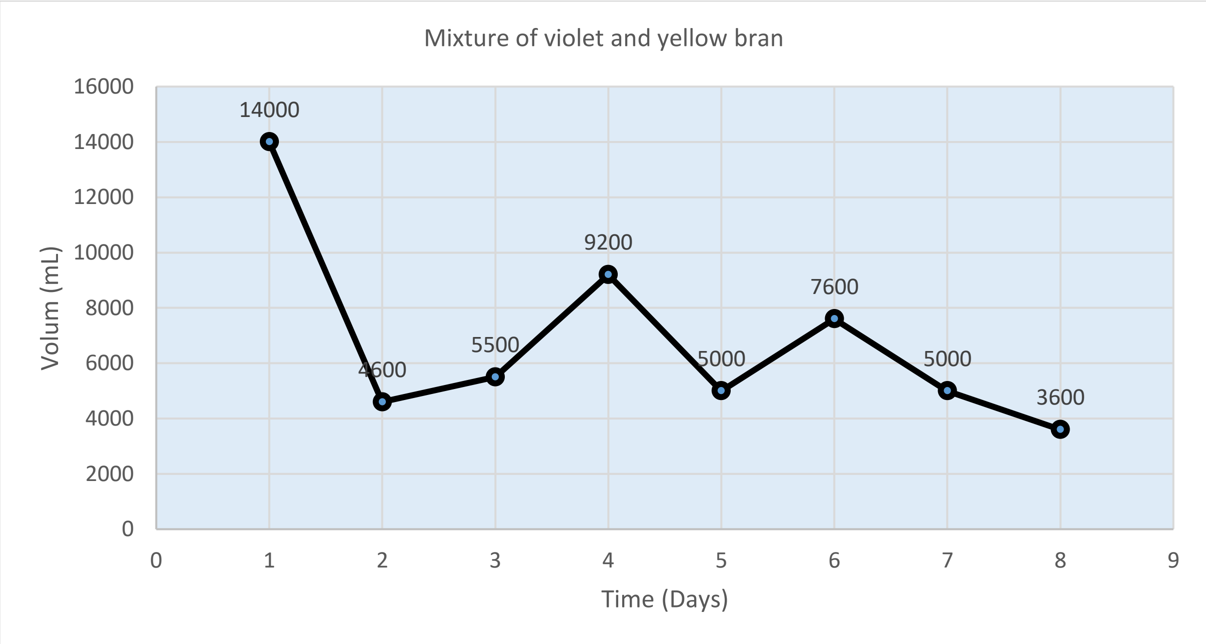

Figure 7 : Production curve for the mixture of yellow and purple maize bran.

The curve in Figure 8 shows that the mixture produces an intermediate amount of biogas. This biogas production is generally lower than that of the violet bran and higher than that of the yellow bran. This result seems reasonable. In sum, this productivity of 54.5m^3/tonne OM (41<54.5<167.2 m^3/ton OM) gives a methane productivity of 32.7m^3/ton of organic matter.

2.5.Comparison of daily biogas production

The results of biogas production in the different cases are shown in **Figure 8**, for comparison. **Figure 8** above shows a comparison of the productivity of the conventional mixing plan. It clearly shows that purple maize bran is more biodegradable than yellow maize bran. This observation can be explained by the high OM (67.8%) and fiber (80.7%) content of purple maize bran. Mixing these two types of waste produced intermediate productivity (41<54.5<167.2 m^3/ton OM). Adding more lignin to the mixture increases the level of non-degradable lignin, which we believe is a limiting factor in the hydrolysis phase of methanisation. It is not feasible to make the mixture knowing that it will produce a quantity of biogas close to that of yellow maize bran.

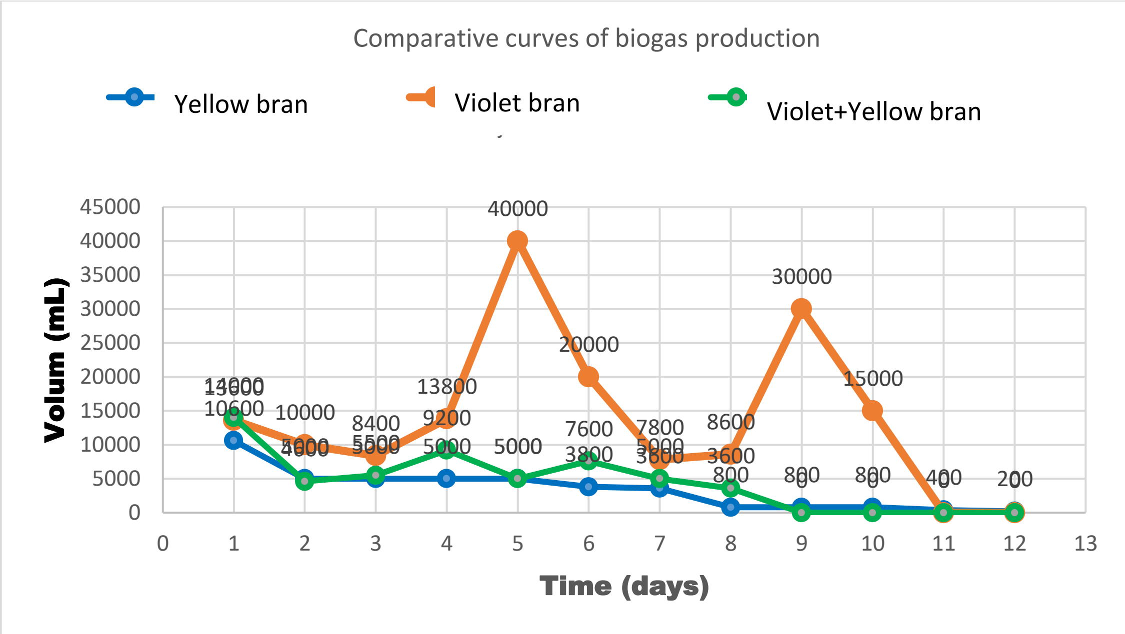

Figure 8: Comparison of the biogas productivity of yellow and purple maize bran

Conclusion

At the end of this study, it is demonstrated that the maize bran of the two yellow and violet varieties is biodegradable. Their anaerobic codigestion in the presence of inoculum (maize kernel endosperm infusion juice, cow dung and some vegetable peelings) is fairly productive in terms of biogas. Purple maize bran, on the other hand, is more easily and roughly more biodegradable. Its productivity of around 167.2 m^3/ton OM of biogas, including 100.32 m^3/ton OM of methane gas, will generate more than 826 kWh of clean, green energy. The CO_2 equivalent of the biogas should also add to the level of CO_2 in our immediate environment, in the context of global warming.

From an economic point of view, the energy potential of this purple Katiola maize variety will add value to this cereal. As a result, production of this cereal will increase and the purchasing power of this population, which is very vulnerable economically because of the poor sales of cashew nuts, will be improved.

Once the biogas has been analysed, we plan to use the methane from the biogas to create renewable domestic sources of electricity and heat in the maize-growing areas. This is because these production areas have always been isolated and plagued by unprecedented energy insecurity. This self-sufficiency in energy will lead to the protection of flora, and therefore to the protection of the environment.

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Disclosure statement: *Conflict of Interest:* The authors declare that there are no conflicts of interest. *Compliance with Ethical Standards:* This article does not contain any studies involving human or animal subjects.

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