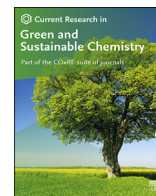


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## Biomethane potential and physicochemical characterization of cassava vinasse from ethanol distillery



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### ABSTRACT

In this study, the biochemical methane potential (BMP), biodegradability index (BI), competitiveness index (CI), physicochemical properties and elemental compositions of cassava vinasse (CV) obtained from an ethanol distillery were investigated using experimental as well as instrumental analyses. Results showed that at room temperature of 28 °C, the CV was very watery with a specific gravity of 1.03. The relatively high Chemical Oxygen Demand, COD (62000–736500 mg/l) and Biochemical Oxygen Demand, BOD (21620–25000 mg/l); acidic pH (4.2–4.8) and cyanuric acid content (386–450 mg/l), emphasized its potential as a pollutant when discharged directly on land and water. A volatile solids content, VS (71580–72250 mg/l) and brix (5.9% sucrose) respectively indicate the presence of biodegradable organic matter and residual sugars in addition to many macro and micro-nutrients. At standard temperature and pressure (STP), BMP was experimentally determined to be 247.10 Nml/gVS which shows that the CV is a bioenergy resource. The results of BI and CI determination indicated that 82% of organic matter in CV is biodegradable and with a CI value above 10, there will be no competition between the methanogens and the sulphate-reducing bacteria (SRB) if the CV is subjected to anaerobic digestion (AD). This work improved existing knowledge and provided information for distillery operators and environmental regulators on alternative utilization and treatment routes for CV.

### 1. Introduction

The high volume of cassava vinasse (CV) being generated per volume of ethanol produced continues to be of serious concerns to distillery operators. Vinasse is generated at 10–15 l per volume of ethanol produced [1]. It is associated with a foul, putrefaction smell [2] and fugitive release of methane into the atmosphere [3]. Its adverse effects on the environment have been reported to include: heavy metals remobilization and soil salinity [4]; eutrophication in water bodies due to high phosphorus and nitrogen concentrations [5]; and its brownish colour can prevent sunlight from reaching aquatic plant and fauna to inhibit photosynthesis [6].

Physicochemical methods such as coagulation/flocculation, wet oxidation, and thermolysis, among others [7] and the biological method of AD [5,8], have been widely studied as treatment options for vinasse. In a review of vinasse treatment options, Prajapati and Chaudhari [9]

concluded that the physicochemical methods are effective in both colour and COD removal. However, excessive use of chemicals, high energy input and sludge generation with subsequent disposal problems are major disadvantages. The two most common applications of vinasse are in fertirrigation [10] and biogas production [11]. Studies however, suggest that both still have potentials to cause soil and underground water pollution [12]. The concept of waste biorefinery to simultaneously produce biochemicals and energy [13] and the use of eutectic solvents to recover natural bioactive compounds that can be used in the pharmaceutical, agrochemical, nutraceutical, and cosmetic industry, from distillery wastewater [14] are both regarded as vital element of sustainable development with huge potentials for economic advantage [15]. Nonetheless, these applications require a deeper understanding of the properties of the target feedstock. Excellent knowledge and understanding of the various characteristics of cassava vinasse (CV) will help find tailored alternative treatment routes and utilizations [13,16]. Cassava and

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sugarcane are the most used feedstock for bioethanol production in Nigeria, Brazil and most Asian countries [17]. Whereas many studies have been carried out to investigate the various properties (Parsae et al., 2019), treatment options [5,9], and possible utilizations [18] of sugarcane-based vinasse, cassava vinasse on the other hand, has been under-studied and under-explored [19] such that data on its characterization is very scanty in the literature. Most studies determined the physicochemical properties of CV as a prelude to its use in AD experiments [2,20], leaving a huge information gap on other categories of properties. In this study therefore, experimental and instrumental analyses were conducted to determine CV properties that include the biochemical methane potential (BMP), biodegradability index (BI), competitiveness index (CI), physicochemical properties and elemental compositions. As far as we know, this is the first time such a comprehensive investigation is conducted on CV, in a single study. Results from this work will advance knowledge and provide information for distillery operators and environmental regulators on alternative utilization and treatment routes for CV.

## 2. Materials and methods

### 2.1. Materials

The CV used in this work (Plate 1) was collected from the distillery of Addin Mills Foods Limited, Osogbo, Nigeria (7.4905° N, 4.5521° E) which processes fresh roots of cassava into bioethanol and fructose syrup. The ruminal fluid of a freshly slaughtered cow was collected from the 'Odo-Eran' Abattoir in Ile-Ife, Nigeria. Sampling was carried out following standard guidelines reported in Chapleur et al. [21] with sample pH taken onsite.

### 2.2. Methods

#### 2.2.1. Physicochemical properties and elemental composition

Table 1 lists out all the physical and chemical parameters of interest that were investigated in this study and for analysis, Standard methods for the examination of water and wastewater [24] was employed, except where otherwise stated.

#### 2.2.2. BMP and BI

Four automated digester systems (PDANC0007/144, manufactured by the Edibon International S. A., Spain) were simultaneously operated in batch mode at 37 °C, for the BMP experiments. While temperature and pH inside the reactors were read directly on a linked computer system,

the digesters used inserted balls to achieve slurry mixing and homogeneity. Methods described in Refs. [28,29] were adopted to determine the BMP for a CV sample that had pH (4.2), TS (77200 mg/l) and VS (73650 mg/l). Ruminal fluids of cattle (RFC) that had been pre-degassed for 5 days was used as the seed at an inoculum-to-substrate ratio (ISR) of 3 based on volatile solids contents. Its physicochemical characteristics were pH (7.8), TS (71500 mg/l), VS (58500 mg/l) and TA (7200 mg-CaCO<sub>3</sub>/l). It has been reported to be a good source of methanogens during anaerobic digestion [30]. A set of two digesters were loaded with CV and RFC inoculum mix and another set loaded with RFC inoculum only (blanks). Loading was based on a total 50 gVS of CV (B) and the respective equivalent volumes of CV (Q<sub>sub</sub>) and the RFC (Q<sub>inoc</sub>) loaded were calculated using Equations (3) and (4) where VS<sub>CV</sub> and VS<sub>RFC</sub> respectively represents the specific volatile solid contents in the CV and RFC (gVS/l) and ISR is the gVS ratio of inoculum to substrate.

$$Q_{Sub} = \left( \frac{B}{VS_{CV}} \right) \quad (3)$$

$$Q_{inco} = \left( \frac{B}{VS_{REC}} \right) * ISR \quad (4)$$

The digester content was then brought up to the 4 l mark with distilled water in each reactor. Anaerobic condition was created by adding 2 drops of 1 M disodium sulphide (Na<sub>2</sub>S.9H<sub>2</sub>O) solution [31], while the stability of each digester was maintained by adding 10 ml of 0.05 M potassium phosphate buffer [32]. Biogas produced was allowed to bubble through a 3 M NaOH solution so that mostly methane, the volume of which was equivalent to the volume of alkali displaced, was collected and recorded daily [33]. The experiments were concluded when daily gas production became <1% of cumulative volume for three consecutive days [34]. Equation (5) was used to calculate the BMP as the average volume of methane produced by the substrate-inoculum systems less that from the inoculum only systems and reported as Nml CH<sub>4</sub> (gVS)<sup>-1</sup> at STP condition [35]. The VS of all digester effluents were measured on completion of the BMP experiments and the average biodegradability index was calculated according to Equation (6) where, V<sub>Sub+inco.avg</sub> is the average volume of methane gas produced in the digesters with CV and RFC; V<sub>inco.only.avg</sub> is the corresponding value for digesters with RFC only.

$$BMP_{Sub} = (V_{Sub+inco.avg} - V_{inco.only.avg}) / gVS \text{ mass of sub.} \quad (5)$$

**Table 1**

Methods and comments on the physicochemical properties determination for cassava vinasse.

s/n	Characterization	Variable	Method	Comments
1.	Physicochemical (Experimental analysis)	(i). COD, BOD <sub>5</sub> , (ii). Moisture content (MC), total solids (TS), volatile solids (VS), ash content (AC), and total carbon (C <sub>T</sub> ) (iii). Volatile fatty acid (VFA), total alkalinity (TA)	5210B/5220B (APHA,2012) 2540G (APHA,2012) Dilallo and Albertson [23]	Total carbon was calculated using the formula [22]. C <sub>T</sub> = {100 - Ash Content }/1.8 (1) First stage- total alkalinity (Titration from initial pH to 4.0): 2NH <sub>4</sub> HCO <sub>3</sub> + H <sub>2</sub> SO <sub>4</sub> → (NH <sub>4</sub> ) <sub>2</sub> SO <sub>4</sub> + H <sub>2</sub> O + H <sub>2</sub> CO <sub>3</sub> Second stage- volatile acid (Back titration from pH 4.0 to 7.0): (NH <sub>4</sub> ) <sub>2</sub> SO <sub>4</sub> + 2CH <sub>3</sub> COOH + 2NaOH → 2CH <sub>3</sub> COO.NH <sub>4</sub> + Na <sub>2</sub> SO <sub>4</sub> + H <sub>2</sub> O
2.	Elemental compositions (instrumental analysis)	Nitrogen (N), Nickel (Ni), Calcium (Ca), Copper (Cu), Phosphorus (P), Potassium (K), Magnesium (Mg), Manganese (Mn), Iron (Fe), Zinc (Zn), Aluminium (Al), Chromium (Cr), Molybdenum (Mo), nitrates (NO <sub>3</sub> <sup>-</sup> ), phosphates (PO <sub>4</sub> <sup>2-</sup> ), sulphates (SO <sub>4</sub> <sup>2-</sup> ) and cyanuric acid (HCN)	APHA [24] methods	The analytical instrument used for this purpose was the Pallintest <sup>®</sup> Advanced Digital Readout Photometer (model PHOT.1.1.AUTO.71) with an accuracy of ±1%, as described by Refs. [25,26].
3.	Competitiveness Index (CI)		Cruz-Salomón et al. [27]	This was calculated as a ratio of concentrations. Thus, CI = [COD]/[SO <sub>4</sub> <sup>2-</sup> ] (2)

$$BI = \left[ \frac{VS_{\text{before digestion}} - VS_{\text{after digestion}}}{VS_{\text{before digestion}}} \right] * 100 \quad (6)$$

### 3. Results and discussion

#### 3.1. Physicochemical properties of CV

Two batches of CV samples were collected, and each was analysed in duplicates within a space of 4 weeks in-between. The analyses presented in Table 2 showed some slight variations even in the COD and BOD values, which perhaps was due to the type of cassava varieties and processing routes used at different times [36]. The CV was acidic with a pH of 4.2–4.8 and a poisonous cyanuric acid content (386–450 mg/l) that is well above the safe consumption limit of 10 mg HCN/Kg set by the World Health Organization (WHO) [37]. The relatively high COD (62000–74000 mg/l) and BOD (21000–25000 mg/l) values were within the range reported in the literature for CV [55] but lower than the corresponding values of 58500–109700 mg/l and 23200–87700 mg/l reported for sugarcane vinasse [38]. Nearly 6% sucrose content and a large VS (71580–72250 mg/l) respectively indicate the presence of carbon source for microbial processes [12] and a significant quantity of organic matter that can be converted to biogas [5].

#### 3.2. Nutrients and elements in cassava vinasse

The various nutrients and elements detected in the analysed CV samples are listed in Table 3. High C/N ratio (43:1–45:1) could mean lack of enough nitrogen for the methanogens and so, effective treatment via AD would require the use of a protein/nitrogen-rich cosubstrate [39]. Nutrients such as C, P, N, K as well as the salts of  $\text{NO}_3^-$ ,  $\text{PO}_4^{2-}$ , and  $\text{SO}_4^{2-}$  were found in quantities that could support plants, animals, and microbial growth [40]. The presence of a large number of trace/heavy metals suggests that CV is even a more complex substrate because of possible synergistic and antagonistic effects of their complex interactions on microbial-driven processes [41,42]. Although, there are variations in reported optimum quantities of each of these elements for microbial processes, including biogas production, the concentrations of  $\text{Fe}^{2+}$ ,  $\text{Ca}^{2+}$ ,  $\text{Mg}^{2+}$ , and  $\text{Mn}^{2+}$  in CV are regarded as generally low and within optimum range for anaerobic digestion processes [43,44] whereas,  $\text{Ni}^{2+}$ ,  $\text{Mo}^{2+}$ ,  $\text{Cr}^{3+}$ ,  $\text{Cu}^{2+}$  and  $\text{Zn}^{2+}$  concentrations are higher than the respective recommended values of 0.005–0.5, 0.005–0.05 and 0.005–50 [45–47]. It was however, noted that despite the presence of most of these trace elements in quantities above the recommended optimum, no significant inhibition was observed in digesters during the anaerobic digestion experiments for BMP determination. This could be attributed to complex synergistic/antagonistic interactions and the ability of some methanogens to survive toxins for more extended period [48]. For a CI value greater than 10, no competition should be expected between the archaea

**Table 2**  
Physical and chemical properties of cassava vinasse.

Parameter	Unit	CV	
		<sup>a</sup> Batch 1	<sup>a</sup> Batch 2
pH	–	4.80	4.20
Cyanuric acid (HCN)	mg/l	386.00	450.00
Specific gravity	–	1.03	1.03
Refractive Index	–	1.32	1.34
Brix	%Sucrose	5.72	5.88
TS	mg/l	77810.00	77200.00
Volatile solids	mg/l	71580.00	72250.00
COD	mg/l	62000.00	73650.00
BOD	mg/l	25000.00	21620.00
VFA (acetic acid eq.)	mmol/l	178.00	165.00
Total Alkalinity	mmol-CaCO <sub>3</sub> /l	52.00	50.00
Moisture content	%	92.60	92.50

<sup>a</sup> Results were the average of two analyses.

**Table 3**  
Elemental compositions of cassava vinasse.

Parameter	Unit	CV	
		<sup>a</sup> Batch 1	<sup>a</sup> Batch 2
Total carbon, C	mg/l	29.92	30.69
Total nitrogen, N	mg/l	660.00	705.00
C/N ratio	–	45.30	43.50
Total phosphorus, P	mg/l	229.00	268.00
Total phosphate ( $\text{PO}_4^{2-}$ )	mg/l	121.00	126.00
Nitrates ( $\text{NO}_3^-$ )	mg/l	29.60	32.00
Sulphate ( $\text{SO}_4^{2-}$ )	mg/l	860.00	920.00
Calcium, Ca	mg/l	100.00	133.00
Magnesium, Mg	mg/l	120.00	148.00
Potassium, K	mg/l	48.00	53.00
Nickel, Ni	mg/l	47.50	53.00
Aluminium, Al	mg/l	7.60	8.60
Copper, Cu	mg/l	28.50	39.00
Iron, Fe	mg/l	51.00	66.00
Zinc, Zn	mg/l	295.00	325.00
Manganese, Mn	mg/l	0.32	0.48
Molybdate	mg/l	196.50	197.40
Chromium, Cr	mg/l	4.60	5.10
Competitiveness Index CI	–	72.10	80.05

<sup>a</sup> Results were the average of two analyses.

methanogens and the sulphate-reducing bacteria [27]. Generally, values recorded in this study are at variance with those reported for sugarcane vinasse [49,50].

#### 3.3. BMP and BI for cassava vinasse

Results from the digestion experiments are graphically represented in Fig. 1(a and b). It showed that all digesters experienced a lag phase of 1–2 days and that the maximum methane production was reached between the 17th and 19th day of digestion. After 30 days, an average cumulative methane volume of 16,178 ml was recorded for digesters loaded with CV and an inoculum of RFC whereas, it was 4854 ml for digesters loaded with inoculum only. Table 4 presented the numerical data from the experiments in addition to showing how both the BMP and BI were calculated.

The BMP value of 247.10 ml/gVS for CV was very close to the value of



**Fig. 1.** Cumulative and daily methane production profiles. (a) RFC inoculum only (b) CV and RFC inoculum.

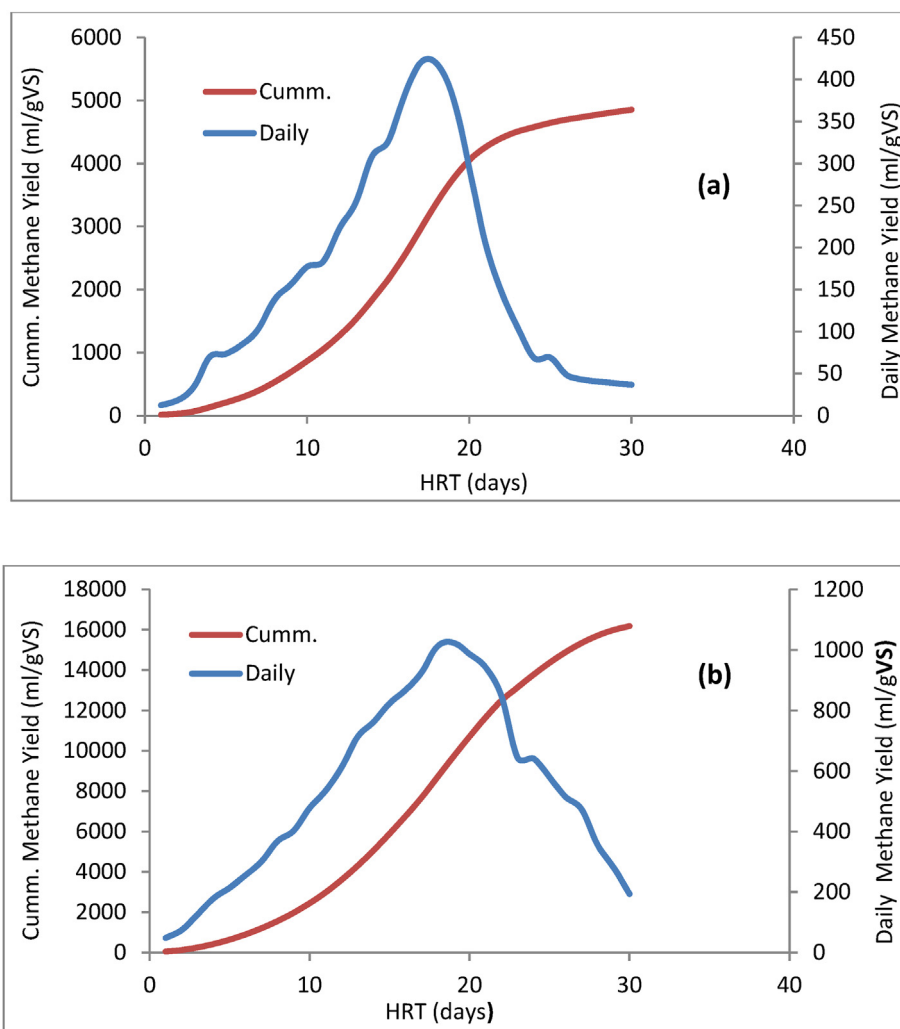


Plate 1. Sample of cassava vinasse from bioethanol production.

Table 4

Summary of digester loadings with the BMP and BI test results for cassava vinasse.

Systems		Digester loadings			Results		
Digesters		CV (ml)	RFC (ml)	Distilled water (ml)	Average cumulative CH <sub>4</sub> volume (ml)	VS of before digestion (mg/l)	VS of after digestion (mg/l)
Set 1	Digesters A1 & A2	692.00	2564.00	744.00	16177.50	73650.00 (for CV)	19257.00 (CV + RFC)
Set 2	Digesters B1 & B2	0.00	2564.00	1436.00	4853.50	58500.00 (for RFC)	6000.00 (RFC)

\*Operating conditions: T<sub>1</sub> = 310.15 K; P<sub>1</sub> = 1.15 bar; V<sub>A</sub> = 16177.50 ml; V<sub>B</sub> = 4853.50 ml  
 \*STP conditions: T<sub>2</sub> = 298.15K; P<sub>2</sub> = 1.01325 bar; V = V<sub>A</sub> - V<sub>B</sub>

$$V_{STP} = \frac{P_1 \cdot T_2 \cdot V_1}{P_2 \cdot T_1} = 12,355 \text{ ml}$$

$$\text{BMP} = \frac{V_{\text{subs+inoc}} - V_{\text{inoc}}}{\text{gVS basis}} = 247.10 \text{ Nml/gVS}$$

$$\text{BI} = \left[ \frac{73650 - (19257 - 6000)}{73650} \right] \cdot 100$$

$$\text{BI} = 82\%$$

\* T, P, V respectively represents the temperature, pressure and cumulative methane volume.

248 ml/gVS observed by Zhang et al. [51]. It also compares favorably with the values of 267.4, 260–390, 208, and 175–240 ml/gVS respectively recorded for sugar beet vinasse [52], sorghum energy crop and poultry manure [53] and bovine slurries [54], respectively. About 82% of the organic matter (VS) in the CV was biodegraded from an initial concentration of 73,650 to 13,257 mg/l at the end of the digestion processes.

#### 4. Conclusion

This study was carried out to comprehensively characterize CV

obtained from a local ethanol distillery with a view to suggest alternative treatment and utilization routes. Results showed that CV had appreciable amounts of trace elements, mineral salts, and residual sugars that can serve as nutrients for microbial growth and processes. A BMP of 247.1 Nml/gVS compared favorably with those of known substrates already established as feedstock for biogas production and thus indicate the potential of CV for bioenergy recovery. Its amenability to treatment via AD was further strengthened by a biodegradability index of 82% and a competitiveness index » 10. Therefore, CV could be used for many applications including as carbon/nutrient source for microbial-driven

bioproduction and bioenergy recovery. Results from this work served to update existing knowledge on CV characteristics and would be very useful to distillery operators and environmental regulators with regards to treatment and utilization options.

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## CRediT authorship contribution statement

**Taiwo Hassan Ibrahim:** Conceptualization, Methodology, Investigation, Writing – original draft, Visualization, Formal analysis. **Julius Olusegun Oyedele:** Resources, Supervision. **Eriola Betiku:** Methodology, Software, Validation, Writing – review & editing, Supervision. **Bamidele Ogbe Solomon:** Methodology, Project administration, Supervision. **Samuel Olatunde Dahunsi:** Supervision, Resources, Validation. **Rose Suniso Gidado:** Resources.

## Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

## Data availability

Data will be made available on request.

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## Abbreviations

AD	anaerobic digestion
BI	biodegradability index
BOD	biochemical oxygen demand
BMP	biochemical methane potential
CI	competitiveness index
COD	chemical oxygen demand
RFC	ruminal fluids of cattle
CV	cassava vinasse
E10	a blend of gasoline and fuel-grade ethanol in the ratio 9:1
STP	standard temperature and pressure
TS	total solids
VFA	volatile fatty acids
VS	volatile solids

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