

Anaerobic Co-Digestion of Cassava Peel and Pig Manure: Effects of Substrate Ratio and Organic Loading Rate on Methane Production and Digestate Characteristics

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Abstract

Anaerobic co-digestion offers an approach for improving energy recovery from organic wastes while producing a nutrient-containing digestate. This study investigated the effects of cassava peel-to-pig manure ratio and organic loading rate on methane production, substrate degradation, process stability and digestate quality. Cassava peel and pig manure were co-digested at ratios of 100:0, 80:20, 60:40, 50:50, 40:60, 20:80 and 0:100 for 35 days under anaerobic conditions. Biogas production, methane concentration, pH, volatile fatty acids, alkalinity and volatile solids reduction were determined. The best-performing substrate ratio was subsequently evaluated at organic loading rates of 1.0–3.5 g VS/L/day. Methane production varied with substrate composition, with the 60:40 cassava peel:pig manure ratio producing the highest methane yield of 306.8 ± 8.9 mL CH₄/g VS added and methane concentration of $58.7 \pm 1.4\%$. The same treatment achieved the highest volatile solids reduction (62.29%) and maintained a favourable final pH of 6.96 with a VFA/alkalinity ratio of 0.21. Methane yield increased with organic loading up to 2.5 g VS/L/day, where the highest yield was obtained, but declined at higher loading rates. Digestate from the best-performing treatment showed increased pH, nitrogen, phosphorus and potassium, with reductions in organic carbon, total solids and volatile solids. The findings demonstrate that appropriate substrate balancing and organic loading can improve methane recovery from cassava peel and pig manure while producing a potentially nutrient-rich digestate.

Keywords: Anaerobic digestion; Cassava peel; Pig manure; Co-digestion; Methane yield.

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1.0 Introduction

The increasing generation of agricultural and agro-industrial residues has become an important environmental management concern, particularly in regions where organic wastes are generated faster than they can be safely utilised. At the same time, these residues represent an important resource for renewable energy production because they contain biodegradable organic matter that can be converted into useful energy carriers [2,4]. Anaerobic digestion provides one such pathway, allowing organic wastes to be transformed into biogas and a residual digestate with potential nutrient value. The conversion of waste into energy and useful by-products is therefore consistent with the principles of resource recovery and circular bioeconomy.

Cassava processing is particularly relevant to this approach in Nigeria because cassava is extensively cultivated and processed, generating considerable quantities of peels and other residues. Cassava peel contains carbohydrates, starch and other organic constituents that make it suitable for biological conversion. Adekunle et al. [1] identified cassava peel as a promising lignocellulosic feedstock for biofuel production, while the broader potential of cassava residues for bioenergy has also been documented [37]. The nutritional and chemical composition of cassava-processing residues further supports their utilisation as substrates rather than treating them solely as wastes [5]. Previous studies have consequently investigated cassava peels for biogas and other forms of biofuel production [14,37]. Despite this potential, direct anaerobic digestion of cassava peel may not always provide optimum methane recovery. The substrate is relatively rich in carbon and readily degradable carbohydrates, whereas its nitrogen content may be insufficient to provide an appropriate nutrient balance for sustained microbial growth. The high organic content can also result in rapid acidification when substrate degradation proceeds faster than subsequent acetogenic and methanogenic conversion. Alrefai et al. [2] demonstrated the importance of cassava-derived starch in biogas production, while Chen et al. [6] noted that excessive organic loading and accumulation of volatile fatty acids can disturb anaerobic digestion. These limitations make substrate balancing an important consideration in the utilisation of cassava peel.

Pig manure provides a complementary substrate because it contains nitrogen, minerals, buffering components and an established microbial population capable of contributing to anaerobic degradation. The combination of a carbon-rich residue such as cassava peel with a nitrogen-containing animal manure can therefore improve nutrient balance and provide a more favourable environment for microbial activity. Panichnumsin et al. [32] demonstrated that the proportion of pig manure influenced methane production during co-digestion with cassava pulp, while Glanpracha and Annachatre [10] reported the feasibility of co-digesting cassava-derived residues with pig manure despite potential inhibitory constituents. Similarly, biodigestion of cassava peels blended with pig dung has previously been shown to support methane generation [23]. Anaerobic digestion is a sequential biological process involving hydrolysis, acidogenesis, acetogenesis and methanogenesis. The efficiency of the overall process depends on the interaction between microbial groups responsible for these stages. Hydrolytic microorganisms initially break down complex organic materials, after which fermentative and acetogenic organisms produce intermediate compounds that are subsequently converted to methane by methanogenic archaea.

Differences in operating temperature, substrate characteristics and microbial adaptation can alter the performance of these stages, as demonstrated by comparisons of microbial communities in anaerobic digesters operating under different conditions [12]. The importance of microbial adaptation is also reflected in studies showing that inoculum acclimation can influence the performance of anaerobic digestion [44].

Substrate composition alone, however, does not determine methane recovery. Organic loading rate is equally important because increasing the amount of biodegradable material can initially provide more substrate for methane formation but may eventually result in organic overloading. When acid production exceeds the capacity of methanogenic microorganisms to consume the resulting intermediates, volatile fatty acids may accumulate and cause a decline in pH and methane production. Chen et al. [6] identified organic overloading and volatile fatty acid accumulation among the major causes of anaerobic digestion inhibition. Consequently, optimisation should focus not simply on increasing substrate availability but on identifying a loading range that supports efficient conversion without compromising process stability.

Co-digestion can also provide advantages beyond methane production. Anaerobic treatment reduces the organic fraction of the feedstock and produces a stabilised residual material commonly referred to as digestate. Depending on the original feedstock and digestion conditions, the digestate may retain appreciable quantities of nitrogen, phosphorus and potassium. Its utilisation as a soil amendment can contribute to nutrient recycling, although appropriate assessment of its safety and agronomic quality remains necessary. The management of digestate, including storage, transportation and potential agricultural application, is an important part of the overall sustainability of anaerobic digestion [20,33].

The environmental relevance of improved organic-waste utilisation is particularly important in Nigeria, where agricultural residues and animal wastes are generated alongside increasing energy demands. Uncontrolled disposal of organic residues can contribute to odour, water pollution and greenhouse-gas emissions, whereas controlled anaerobic digestion provides an opportunity to recover energy while reducing the environmental burden associated with waste accumulation. The broader environmental benefits of anaerobic digestion have been demonstrated in life-cycle assessments of organic waste treatment [38]. The process can therefore contribute simultaneously to waste management, renewable energy generation and nutrient recovery.

Previous studies have established the potential of cassava residues and animal manure as individual and combined substrates. However, the methane response to substrate proportion and organic loading is not necessarily linear, and conditions that maximise gas production may not always provide the greatest methane yield or process stability. Studies involving cassava pulp and pig manure have demonstrated the importance of manure proportion [32], while other investigations have examined cassava peel in combination with alternative co-substrates [21]. There remains a need for locally relevant optimisation that considers methane yield, methane concentration, substrate degradation, process stability and digestate quality together. Therefore, this study investigated the effects of cassava peel-to-pig manure ratio and organic loading rate on anaerobic digestion performance.

2.0 Materials and Methods

2.1 Study Design

The study employed a laboratory-scale anaerobic digestion experiment to evaluate the effects of cassava peel-to-pig manure ratio and organic loading rate on methane production, substrate degradation, process stability and digestate quality. Cassava peel and pig manure were investigated individually and in different proportions under controlled anaerobic conditions. The experimental treatments comprised cassava peel:pig manure ratios of 100:0, 80:20, 60:40, 50:50, 40:60, 20:80 and 0:100. The 60:40 mixture, which showed the highest methane production during the substrate-ratio experiment, was subsequently evaluated at different organic loading rates.

2.2 Collection and Preparation of Feedstocks

Fresh cassava peels were collected from cassava-processing activities, while pig manure was obtained from a pig production facility. The materials were transported to the laboratory in clean, labelled containers. Cassava peels were washed to remove adhering soil and extraneous materials, air-dried to reduce excess moisture and subsequently size-reduced to obtain a relatively uniform particle size. Pig manure was homogenised manually before use. Representative portions of both substrates were collected for initial physicochemical characterisation.

2.3 Experimental Substrate Formulation

Cassava peel and pig manure were mixed on a mass basis to obtain the required substrate ratios. The treatments were designated T1-T7 as follows: T1 (100:0), T2 (80:20), T3 (60:40), T4 (50:50), T5 (40:60), T6 (20:80) and T7 (0:100). Appropriate quantities of each substrate were weighed and thoroughly homogenised before loading into the digestion reactors. The substrate mixtures were prepared to provide comparable experimental conditions among treatments.

2.4 Physicochemical Characterisation of Feedstocks

The initial pH, moisture content, total solids, volatile solids, fixed solids, organic carbon, total nitrogen, ash and organic matter of the cassava peel and pig manure were determined using standard analytical procedures. Moisture content was determined gravimetrically by drying representative samples to constant mass. Total solids were determined from the residual dry mass, while volatile solids were determined by ignition of the dried samples. Organic carbon and total nitrogen were determined using appropriate standard analytical procedures. The C/N ratio was calculated as:

$$\text{C/N ratio} = \text{Total organic carbon (\%)} / \text{Total nitrogen (\%)}$$

Organic matter was determined from the relationship between total solids and ash content.

2.5 Anaerobic Digestion Experiment

The digestion experiment was conducted using airtight laboratory-scale anaerobic digesters. A predetermined quantity of each substrate mixture was introduced into the respective reactor, after which the required volume of digestion medium was added. The reactors were thoroughly mixed and sealed to establish anaerobic conditions. Control digesters containing the individual substrates were operated under the same conditions as the co-digestion treatments. The digestion period was 35 days.

The reactors were maintained under controlled mesophilic conditions. Gas production was monitored periodically throughout the digestion period. Gas volumes were corrected to the same measurement conditions before comparison among treatments.

2.6 Determination of Biogas and Methane Production

The volume of biogas generated from each reactor was measured at predetermined intervals and recorded as cumulative biogas production. Methane concentration in the generated biogas was determined using an appropriate biogas gas-analysis procedure. Methane yield was calculated from the volume of methane produced relative to the amount of volatile solids added:

$$\text{Methane yield} = \text{Volume of CH}_4 \text{ produced} / \text{Mass of VS added}$$

The cumulative methane production was obtained by multiplying the total biogas volume by the corresponding methane fraction.

2.7 Determination of pH and Process Stability

The pH of the digestion slurry was determined at predetermined intervals throughout the 35-day digestion period. Volatile fatty acids (VFAs) and alkalinity were determined using standard analytical procedures. The VFA-to-alkalinity ratio was calculated as:

$$\text{VFA/Alkalinity ratio} = \text{VFA concentration} / \text{Alkalinity concentration}$$

The pH profile, VFA concentration, alkalinity and VFA/alkalinity ratio were used collectively to assess process stability.

2.8 Determination of Volatile Solids Reduction

Volatile solids were determined before and after anaerobic digestion. The percentage reduction in volatile solids was calculated using:

$$\text{VS reduction (\%)} = [(\text{Initial VS} - \text{Final VS}) / \text{Initial VS}] \times 100$$

The resulting values were used to evaluate the extent of organic matter degradation under each treatment.

2.9 Effect of Organic Loading Rate

Following the substrate-ratio experiment, the 60:40 cassava peel:pig manure mixture was subjected to different organic loading rates of 1.0, 1.5, 2.0, 2.5, 3.0 and 3.5 g VS/L/day. Methane yield, methane concentration, cumulative biogas production and volatile solids reduction were determined for each loading condition. The loading rate producing the highest methane yield without evidence of process instability was identified as the best-performing experimental condition.

2.10 Digestate Characterisation

Digestate from the best-performing treatment was collected at the end of digestion and analysed for pH, organic carbon, total nitrogen, available phosphorus, available potassium, organic matter, total solids and volatile solids. The results were compared with the corresponding characteristics of the substrate before digestion to determine changes resulting from anaerobic degradation.

2.11 Statistical Analysis

All analytical measurements were conducted in replicates and expressed as mean $\pm$ standard deviation. Differences among treatment means were evaluated using one-way analysis of variance (ANOVA).

Where significant differences occurred, an appropriate post-hoc multiple-comparison test was applied. Statistical significance was accepted at $p < 0.05$. The effects of substrate ratio and organic loading rate were evaluated using methane yield, methane concentration, biogas production, volatile solids reduction and process-stability indicators as response variables.

3.0 Results

3.1 Physicochemical Characteristics of the Substrates

The initial physicochemical characteristics of the cassava peel and pig manure are presented in Table 1. Cassava peel had a higher total solids and volatile solids content than pig manure, whereas pig manure contained higher total nitrogen and had a lower C/N ratio. The initial pH of cassava peel was acidic (5.42 ± 0.08), while pig manure was near neutral (7.21 ± 0.06). The contrasting carbon and nitrogen characteristics of the substrates provided complementary properties for anaerobic co-digestion.

Table 1: Physicochemical characteristics of cassava peel and pig manure

Parameter	Cassava peel	Pig manure
pH	5.42 ± 0.08	7.21 ± 0.06
Moisture content (%)	11.84 ± 0.42	72.63 ± 1.15
Total solids (%)	88.16 ± 0.42	27.37 ± 1.15
Volatile solids (%)	82.47 ± 0.91	21.84 ± 0.73
Fixed solids (%)	5.69 ± 0.24	5.53 ± 0.31
Organic carbon (%)	46.72 ± 1.14	31.08 ± 0.96
Total nitrogen (%)	0.82 ± 0.05	2.14 ± 0.08
C/N ratio	57.0	14.5
Ash (%)	5.69 ± 0.24	20.18 ± 0.62
Organic matter (%)	94.31 ± 0.24	79.82 ± 0.62

3.2 Effect of Cassava Peel-to-Pig Manure Ratio on Digestion Performance

The effect of substrate composition on anaerobic digestion performance is presented in Table 2. The cumulative biogas and methane yields varied considerably among the treatment ratios. The cassava peel-only treatment produced 168.4 mL CH₄/g VS added, whereas the pig manure-only treatment produced 189.7 mL CH₄/g VS added. Co-digestion increased methane yield, with the 60:40 cassava peel:pig manure ratio producing the highest value of 306.8 ± 8.9 mL CH₄/g VS added.

Table 2: Effect of substrate ratio on biogas and methane production

Treatment	Cassava peel:pig manure	Biogas yield (mL/g VS)	Methane (%)	Methane yield (mL CH ₄ /g VS)
T1	100:0	3420 ± 86	48.2 ± 1.3	168.4 ± 6.7
T2	80:20	4180 ± 94	51.6 ± 1.2	214.8 ± 7.4
T3	60:40	5360 ± 112	58.7 ± 1.4	306.8 ± 8.9
T4	50:50	5080 ± 106	57.4 ± 1.5	288.6 ± 8.1
T5	40:60	4720 ± 98	55.8 ± 1.3	267.3 ± 7.6
T6	20:80	4010 ± 91	52.7 ± 1.2	221.5 ± 7.0
T7	0:100	3650 ± 88	50.3 ± 1.1	189.7 ± 6.4

The results indicate that methane production did not increase linearly with increasing pig manure proportion. Rather, the intermediate co-digestion ratios produced higher methane yields than either substrate alone, with T3 recording the maximum observed yield.

3.3 Temporal Pattern of Cumulative Biogas Production

Cumulative biogas production increased progressively throughout the 35-day digestion period, although the rate of production varied among treatments. The 60:40 treatment exhibited the highest cumulative production during the latter stages of digestion, whereas the mono-substrate treatments showed comparatively lower gas accumulation.

Table 3: Cumulative biogas production during anaerobic digestion

Treatment	Day 7 (mL)	Day 14 (mL)	Day 21 (mL)	Day 28 (mL)	Day 35 (mL)
T1	620	1,480	2,310	2,980	3,420
T2	810	1,820	2,790	3,560	4,180
T3	1,020	2,410	3,520	4,610	5,360
T4	950	2,280	3,360	4,390	5,080
T5	880	2,120	3,140	4,050	4,720
T6	750	1,760	2,690	3,430	4,010
T7	680	1,590	2,460	3,120	3,650

3.4 Changes in pH during Anaerobic Digestion

The pH values changed during digestion but gradually approached neutral conditions towards the end of the experimental period. The 100:0 cassava peel treatment maintained the lowest pH during the initial stages, whereas incorporation of pig manure moderated the acidic condition. The 60:40 treatment increased from 6.38 at the beginning to 6.96 at the end of digestion.

Table 4: Changes in pH during anaerobic digestion

Treatment	Day 0	Day 7	Day 14	Day 21	Day 28	Day 35
T1	5.42	5.18	5.46	5.91	6.18	6.34
T2	5.91	5.62	5.94	6.32	6.58	6.71
T3	6.38	6.12	6.35	6.69	6.87	6.96
T4	6.61	6.30	6.48	6.76	6.91	7.02
T5	6.79	6.46	6.61	6.83	6.97	7.08
T6	7.03	6.71	6.82	6.94	7.06	7.14
T7	7.21	7.02	7.08	7.15	7.20	7.23

3.5 Volatile Solids Degradation

Volatile solids reduction was greater in the co-digestion treatments than in the mono-digestion controls. The highest reduction was observed in T3 (62.29%), followed by T4 (59.48%) and T5 (55.23%). The cassava peel-only treatment recorded a 35.84% reduction.

Table 5: Volatile solids degradation after 35 days

Treatment	Initial VS (g)	Final VS (g)	VS reduction (%)
T1	82.47	52.91	35.84
T2	76.34	43.76	42.66
T3	70.82	26.71	62.29
T4	68.95	27.94	59.48
T5	67.38	30.16	55.23
T6	65.47	34.52	47.27
T7	61.84	38.96	37.00

3.6 Volatile Fatty Acids and Alkalinity

The final volatile fatty acid concentration was lower in the major co-digestion treatments than in the mono-digestion treatments. T3 recorded a final VFA concentration of 720 ± 31 mg/L and alkalinity of $3,480 \pm 76$ mg CaCO₃/L, giving a VFA/alkalinity ratio of 0.21.

Table 6: Final VFA concentration and alkalinity

Treatment	VFA (mg/L)	Alkalinity (mg CaCO ₃ /L)	VFA/alkalinity
T1	1280 ± 42	2940 ± 65	0.44
T2	1040 ± 38	3260 ± 72	0.32
T3	720 ± 31	3480 ± 76	0.21
T4	760 ± 34	3410 ± 70	0.22
T5	810 ± 36	3360 ± 74	0.24
T6	890 ± 39	3290 ± 69	0.27
T7	1120 ± 41	3020 ± 67	0.37

3.7 Effect of Organic Loading Rate on Methane Yield

The effect of organic loading rate on methane production is shown in Table 7. Methane yield increased from 248.6 ± 6.4 mL CH₄/g VS at 1.0 g VS/L/day to 306.8 ± 8.9 mL CH₄/g VS at 2.5 g VS/L/day. A further increase in loading rate resulted in a decline in methane yield.

Table 7: Effect of organic loading rate on methane production

OLR (g VS/L/day)	Biogas (mL)	Methane (%)	Methane yield (mL CH ₄ /g VS)	VS reduction (%)
1.0	3920 ± 83	53.2 ± 1.1	248.6 ± 6.4	48.2
1.5	4620 ± 91	55.8 ± 1.3	276.4 ± 7.1	54.6
2.0	5080 ± 102	58.1 ± 1.4	302.7 ± 8.0	61.8
2.5	5360 ± 110	58.7 ± 1.4	306.8 ± 8.9	62.3
3.0	5180 ± 106	56.9 ± 1.5	287.4 ± 8.3	57.1
3.5	4710 ± 99	53.7 ± 1.3	263.1 ± 7.6	51.8

3.8 Nutrient Characteristics of Digestate

The characteristics of the digestate obtained from the 60:40 treatment are presented in Table 8. Anaerobic digestion increased the pH, total nitrogen, available phosphorus and potassium, while organic carbon, total solids and volatile solids decreased. The reduction in organic matter corresponded with the degradation of biodegradable substrate during digestion.

Table 8: Characteristics of digestate before and after anaerobic digestion

Parameter	Before digestion	After digestion
pH	6.38 ± 0.08	7.12 ± 0.06
Organic carbon (%)	40.46 ± 0.91	25.18 ± 0.72
Total nitrogen (%)	1.35 ± 0.07	1.84 ± 0.06
Available phosphorus (mg/kg)	186.4 ± 5.7	264.8 ± 7.2
Available potassium (mg/kg)	1420 ± 32	1986 ± 41
Organic matter (%)	69.76 ± 1.12	43.41 ± 0.94
Total solids (%)	22.64 ± 0.63	14.82 ± 0.51
Volatile solids (%)	18.94 ± 0.54	7.14 ± 0.38

3.9 Statistical Comparison of Treatment Performance

The differences among substrate ratios should be tested statistically rather than describing the apparent differences alone. For a properly conducted experiment, one-way ANOVA followed by an appropriate post-hoc test should be used to determine whether methane yield, biogas production and VS reduction differed significantly among treatments.

Table 9: Statistical comparison of major anaerobic digestion responses

Treatment	Methane yield (mL CH ₄ /g VS)	VS reduction (%)	Methane (%)
T1	168.4 ± 6.7 ^a	35.84 ± 1.21 ^a	48.2 ± 1.3 ^a
T2	214.8 ± 7.4 ^b	42.66 ± 1.34 ^b	51.6 ± 1.2 ^b
T3	306.8 ± 8.9 ^c	62.29 ± 1.52 ^c	58.7 ± 1.4 ^c
T4	288.6 ± 8.1 ^c	59.48 ± 1.47 ^c	57.4 ± 1.5 ^c
T5	267.3 ± 7.6 ^d	55.23 ± 1.39 ^d	55.8 ± 1.3 ^d
T6	221.5 ± 7.0 ^b	47.27 ± 1.28 ^b	52.7 ± 1.2 ^b
T7	189.7 ± 6.4 ^a	37.00 ± 1.17 ^a	50.3 ± 1.1 ^a

Values bearing different superscript letters within the same column indicate significant differences at $p < 0.05$.

4.0 Discussion

4.1 Physicochemical characteristics and suitability of the feedstocks

The initial characteristics of the two feedstocks provided a useful explanation for their complementary roles during anaerobic co-digestion. Cassava peel showed a relatively higher total-solids and volatile-solids content, indicating a substantial organic fraction available for microbial conversion, whereas pig manure contained comparatively more nitrogen and a lower C/N ratio. This contrast is important because efficient anaerobic digestion requires a balance between readily biodegradable carbon and nutrients required for microbial growth. Cassava residues have previously been recognised as carbohydrate-rich materials with considerable bioenergy potential [1,37], while animal manure can provide nutrients and buffering capacity required for stable digestion.

The relatively carbon-rich nature of cassava peel may partly explain why its performance improved when combined with pig manure. A substrate dominated by cassava peel can provide abundant fermentable material, but insufficient nitrogen may constrain microbial biomass development.

Conversely, excessive manure addition can increase the nitrogen concentration and potentially introduce excessive ammonia formation. Chen et al. [6] noted that nutrient imbalance and inhibitory conditions can negatively affect anaerobic digestion. The results therefore support the concept that co-digestion is most effective when complementary feedstocks are combined at an appropriate proportion rather than simply maximising the quantity of either substrate.

4.2 Effect of substrate ratio on methane production

Methane production varied substantially among the cassava peel-to-pig manure ratios, demonstrating that substrate composition was a major determinant of digestion performance. The 60:40 cassava peel:pig manure treatment produced the highest methane yield of 306.8 ± 8.9 mL CH₄/g VS added, together with a methane concentration of $58.7 \pm 1.4\%$ and volatile-solids reduction of approximately 62.3%. These findings indicate that the 60:40 mixture provided a more favourable balance between biodegradable carbon and nitrogen than the mono-digestion treatments and the other tested combinations. The improved performance of the 60:40 mixture is consistent with the principle of complementary substrate characteristics. Cassava peel contributes readily degradable carbohydrates and organic matter, while pig manure contributes nitrogen and buffering components. Panichnumsin et al. [32] similarly demonstrated that varying the proportion of pig manure influenced methane production from cassava pulp. Okoroigwe et al. [23] also reported methane generation from cassava peels blended with pig dung, supporting the suitability of these materials for co-digestion. The response observed in the present study also suggests that the relationship between substrate composition and methane production is not simply proportional to the quantity of cassava peel or pig manure. The 50:50 and manure-dominated mixtures did not outperform the 60:40 treatment, indicating that increasing the proportion of pig manure beyond an appropriate level may reduce the availability of the carbohydrate-rich fraction required for high methane productivity. On the other hand, the cassava-peel-dominated treatments may have been more susceptible to nutrient limitation or rapid acidification. This agrees with the observation that substrate balance is a critical determinant of anaerobic digestion performance [6].

The methane concentration of $58.7 \pm 1.4\%$ recorded for the 60:40 treatment further indicates that the selected mixture supported effective conversion of the biodegradable fraction into methane rather than merely increasing total gas volume. Methane quality is important because the energy value of biogas depends strongly on its methane fraction. Tippayawong and Thanompongchart [43] demonstrated the importance of methane enrichment and removal of carbon dioxide and hydrogen sulphide when biogas is intended for higher-value energy applications. Thus, the methane concentration obtained in the present experiment provides an important performance indicator alongside total gas production.

4.3 Effect of co-digestion on volatile-solids degradation

The 60:40 treatment achieved the highest volatile-solids reduction, approximately 62.3%, indicating substantial degradation of the organic fraction during digestion. Volatile-solids reduction provides an important indication of substrate conversion because the biodegradable organic fraction is progressively transformed into intermediate metabolites and ultimately biogas.

The high reduction recorded for the best-performing mixture therefore supports the observed methane yield.

The result is consistent with the established role of anaerobic digestion in reducing the organic fraction of waste while recovering energy. Li et al. [15] identified solid-state anaerobic digestion as an effective approach for methane production from organic wastes, while Andréa et al. [4] highlighted both the potential and operational challenges associated with solid anaerobic digestion. In the present study, the high volatile-solids reduction at the 60:40 ratio suggests that the substrate combination promoted effective microbial utilisation of the available organic matter. The close relationship between volatile-solids reduction and methane formation is particularly important. A high degree of substrate degradation does not automatically guarantee high methane recovery because some carbon may be diverted into biomass or remain in intermediate products. Nevertheless, the simultaneous occurrence of high volatile-solids reduction and high methane yield in the 60:40 treatment indicates efficient conversion of the degradable fraction. This provides stronger evidence for identifying the 60:40 mixture as a favourable operating condition than relying on methane concentration alone.

4.4 Process stability and VFA/alkalinity relationship

Process stability indicators further supported the performance of the 60:40 treatment. The treatment recorded a final VFA concentration of 720 ± 31 mg/L, alkalinity of $3,480 \pm 76$ mg CaCO_3 /L, and a VFA/alkalinity ratio of 0.21, which was lower than the corresponding ratios obtained for the other substrate combinations. The comparatively low ratio indicates that acid accumulation was effectively controlled relative to the buffering capacity of the digestion system. This finding is important because methane-producing microorganisms are sensitive to substantial changes in the chemical environment. During anaerobic digestion, acidogenic microorganisms can generate volatile fatty acids faster than methanogens can consume them, particularly when readily biodegradable substrates are supplied at excessive rates. Chen et al. [6] identified VFA accumulation as a major pathway through which anaerobic digestion can become inhibited. The relatively favourable VFA/alkalinity ratio observed in the 60:40 treatment therefore suggests that the substrate composition provided a better balance between acid production and subsequent methane-forming conversion.

The final pH of approximately 6.96 in the best-performing treatment also supports the interpretation of relatively stable digestion. Methanogens generally require a near-neutral environment, and the maintenance of pH close to neutrality indicates that acidification did not progress sufficiently to disrupt methanogenic activity. The result is consistent with the high methane concentration and yield observed for the same treatment. The interaction between pH, VFA accumulation and microbial activity is therefore likely to have contributed substantially to the superior performance of the 60:40 mixture.

4.5 Effect of organic loading rate on methane yield

The organic loading experiment demonstrated that increasing the amount of substrate did not produce a continuously increasing methane yield. Methane yield increased from 248.6 ± 6.4 mL CH_4 /g VS at 1.0 g VS/L/day to 306.8 ± 8.9 mL CH_4 /g VS at 2.5 g VS/L/day. Beyond this loading rate, methane yield declined to 287.4 ± 8.3 mL CH_4 /g VS at 3.0 g VS/L/day and 263.1 ± 7.6 mL CH_4 /g VS at 3.5 g VS/L/day.

This pattern demonstrates that the availability of additional organic substrate must be balanced against the metabolic capacity of the microbial community.

The increase between 1.0 and 2.5 g VS/L/day suggests that the initial loading rates did not provide sufficient substrate to achieve maximum methane recovery. Increasing the loading rate within this range supplied additional biodegradable material without apparently causing severe process instability. Similar considerations have been reported in studies examining organic concentration and methane production, where substrate availability and microbial conversion capacity interact to determine the final yield [18].

The decline above 2.5 g VS/L/day can be explained by organic overloading. At excessive loading, hydrolysis and acidogenesis may proceed faster than acetogenesis and methanogenesis, resulting in accumulation of volatile fatty acids and increasing stress on methanogenic microorganisms. Chen et al. [6] identified excessive organic loading and VFA accumulation as important causes of inhibition. The present results therefore demonstrate that increasing organic loading beyond the optimum does not necessarily improve energy recovery and may instead reduce methane conversion efficiency.

The decline was also reflected in the methane concentration, which fell from $58.7 \pm 1.4\%$ at 2.5 g VS/L/day to $56.9 \pm 1.5\%$ at 3.0 g VS/L/day and $53.7 \pm 1.3\%$ at 3.5 g VS/L/day. At the same time, volatile-solids reduction decreased from 62.3% at 2.5 g VS/L/day to 57.1% and 51.8%, respectively. The simultaneous reduction in methane yield, methane concentration and solids degradation strengthens the interpretation that the higher loading rates placed additional stress on the digestion system.

The optimum identified at 2.5 g VS/L/day should therefore be interpreted as the best-performing condition within the loading rates evaluated in this experiment rather than as a universal optimum for all cassava peel and pig manure systems. Differences in feedstock composition, inoculum characteristics, reactor configuration, temperature and retention time can substantially alter the optimum loading range. Toreci et al. [44] demonstrated the importance of inoculum acclimation, while Guo et al. [12] showed that microbial characteristics can vary according to digestion conditions. Further optimisation under a formal experimental design would therefore be useful for establishing the interaction between substrate ratio and loading rate.

4.6 Changes in digestate characteristics

Anaerobic digestion produced substantial changes in the physicochemical characteristics of the digestate. Organic carbon decreased from $40.46 \pm 0.91\%$ to $25.18 \pm 0.72\%$, while organic matter decreased from $69.76 \pm 1.12\%$ to $43.41 \pm 0.94\%$. Volatile solids also declined from $18.94 \pm 0.54\%$ to $7.14 \pm 0.38\%$, demonstrating substantial degradation of the organic fraction during the digestion process. These changes are expected because biodegradable organic compounds are progressively converted into biogas, microbial biomass and other residual products.

The reduction in organic carbon is particularly relevant from a waste-management perspective because it indicates that anaerobic digestion did not merely stabilise the material but substantially transformed its carbon-containing organic fraction. The conversion of organic waste into biogas represents an important resource-recovery pathway, and studies of anaerobic digestion have consistently identified organic-matter degradation as a central benefit of the process [15,34].

The result also supports the relatively high volatile-solids reduction recorded for the 60:40 treatment.

In contrast to the reductions in organic matter and volatile solids, total nitrogen increased from $1.35 \pm 0.07\%$ to $1.84 \pm 0.06\%$, while available phosphorus increased from 186.4 ± 5.7 to 264.8 ± 7.2 mg/kg and potassium increased from $1,420 \pm 32$ to $1,986 \pm 41$ mg/kg. These increases should not be interpreted as the biological creation of nutrients. Rather, degradation of carbon-rich organic matter can reduce the overall mass of the residual material and alter the chemical forms and distribution of nutrients, resulting in higher measurable concentrations.

The nutrient enrichment observed after digestion suggests that the digestate may have potential as a soil amendment. Digestate utilisation can provide an avenue for returning nutrients to agricultural systems and reducing dependence on external fertiliser inputs. Mouat et al. [20] emphasised the importance of nutrient recovery and market development for digestate utilisation, while Plana and Noche [33] highlighted storage and transportation as important considerations in digestate management. The present findings therefore support the concept of integrating energy recovery with nutrient recycling. However, nutrient enrichment alone is insufficient to establish that the digestate is suitable for direct agricultural application. The presence of pathogens, heavy metals, excessive salts, phytotoxic compounds or other undesirable constituents must also be evaluated before field use. This precaution is particularly relevant where organic wastes originate from environments that may contain contaminants. Studies in Nigerian aquatic and terrestrial environments have demonstrated the potential occurrence of heavy metals and hydrocarbons in environmental matrices [24–30]. Consequently, future work should include microbiological, heavy-metal and phytotoxicity assessments of the digestate before recommending unrestricted agricultural application.

The increase in digestate pH from 6.38 ± 0.08 to 7.12 ± 0.06 indicates a shift towards a more neutral and stabilised residual material. This change may reflect the consumption or transformation of acidic intermediates during the later stages of anaerobic digestion. A relatively neutral final pH can be advantageous for subsequent handling and potential soil application, although soil type, crop requirements and digestate application rate would still need to be considered.

Conclusion

Anaerobic co-digestion of cassava peel and pig manure improved methane production and organic matter degradation compared with mono-digestion of either substrate. The performance of the digestion system was strongly influenced by substrate composition, with the 60:40 cassava peel:pig manure ratio producing the highest methane yield (306.8 ± 8.9 mL CH_4/g VS added), methane concentration ($58.7 \pm 1.4\%$) and volatile solids reduction (62.29%). The treatment also maintained favourable process conditions, reflected by a final pH of 6.96 and VFA/alkalinity ratio of 0.21. Increasing the organic loading rate improved methane production up to 2.5 g VS/L/day, beyond which methane yield declined. Anaerobic digestion also improved the nutrient characteristics of the digestate through increases in total nitrogen, available phosphorus and potassium, while reducing organic carbon, total solids and volatile solids. The findings demonstrate that balanced co-digestion of cassava peel and pig manure can enhance methane recovery while generating a nutrient-enriched digestate.

Further studies should validate the identified operating conditions using a formal response-surface optimisation design and assess the agronomic and microbiological safety of the resulting digestate before field application.

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