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Biogas production from waste pulps of cassava (Manihot esculenta Crantz) via anaerobic digestion

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Abstract The huge volume of wastes generated from industries kindles immediate attention, especially those wastes that bring adverse effects to humans and the environment. For one, cassava waste pulps (CWPs) from starch-producing industries are needing attention for its alternate disposal by making value-adding products out of it. In this work, the CWP with pig manure as inoculum was anaerobically digested for the possible production of biogas. The effect of the concentration of pig manure (CPM) and biomass to water ratio (BMR) was scientifically analyzed in relation to biogas yield. The central composite design of the response surface methodology was used as the design of the experiment. Biogas yield was modeled and characterized according to essential properties. The result of the batch experiment obtained a biogas yield of 4.9–7.3 L per kg of CWP. At optimized conditions of 250 gVS of CPM and 1:1.22 BWR (kg/L), the optimum biogas volume was 7.43 ± 0.58 L per kg of CWP. Analysis of the produced biogas via gas chromatography showed a significant concentration of biohydrogen (18.69 \pm 1.71%), a highly desirable upshot considering that this gas is highly flammable with less emissions when combusted. Other percent components of the produced biogas include carbon dioxide (38.02 \pm 0.71), nitrogen (20.77 \pm 1.59), and a trace of methane (0.73 ± 0.28) . This work, therefore, proved that CWP can be used for the production of biogas and would eventually provide practical solutions to starch processing industries as it gives promising lucrative routes of CWP with added commercial worth in the production of high-value energy resources like the desirable H_2 gas. It

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poises high potentials with less socio-economic apprehensions while offers numerous environmental advantages.

Keywords Anaerobic digestion · Biogas · Biohydrogen · Cassava waste pulps \cdot CO₂ emissions \cdot Hydrogen \cdot Steam methane reforming

1 Introduction

The increasing demand for starch from cassava (Manihot esculenta Crantz) has been generating tons of wastes during production (Ekop et al. [2019;](#page-8-0) Jha et al. [2013\)](#page-8-0). It is estimated that 20–25% by mass of the cassava during starch production is discarded as cassava waste pulps (CWPs) (Ahou et al. [2019](#page-8-0)). For instance, in the Philippines, there is significant CWP generation considering the production of cassava of 773.15 thousand metric tons in 2019 (Mapa [2019\)](#page-8-0). Among the industries that have difficulty in CWP disposal is the PhilAgro Industrial Corporation in Baungon, Bukidnon, Philippines with a daily generation of 100 tons CWP. In the global scene, CWP disposal is a common dilemma considering world cassava production of 291 million tons in 2017 (Otekunrin and Sawicka [2019](#page-8-0)). This percentage of CWP is inevitable, considering that starch has various uses in food industries, including food manufacturing and food establishments. As such, proper disposal of CWP is likewise inevitable to safeguard human health and the environment. The improper disposal of CWP would make nearby inhabitants at risk, especially in local areas due to possible pollution it would bring to water and air, and even results in soil degradation (Fawole and Ibikunle [2019\)](#page-8-0). It may release \boxtimes Reymar H. Jaro
foul odors and lead to pH dropping of water bodies up to \cong Reymariano S@nmail.com

2.6—making aquatic organisms difficult to survive (Etta et al. [2019](#page-8-0)), and even give unwanted aesthetic views (Ochu and Okwori [2019\)](#page-8-0). Additionally, cassava contains hazardous cyanide, a mutagenic and carcinogenic substance that makes CWP disposal more challenging.

Various approaches have been explored in utilizing CWP into value-added products such as surfactants, fertilizers, feed livestock, briquettes, and others (Oyewole [2019\)](#page-8-0). Another value-added product is in the bio-energy area (Ekop et al. [2019](#page-8-0)), such as the production of bioethanol (Elemike et al. [2015;](#page-8-0) Srimuang and Polprasert [2019](#page-8-0); Talemporos et al. [2018](#page-8-0)). An additional promising alternate route is the production of biogas from CWP to take advantage its high composition of starch (50–60%) in dry matter, and high moisture content (60–70%), a desirable state of CWP that allows favorable feeding of various microorganisms during anaerobic digestion (Akaracharanya et al. [2010;](#page-8-0) Cremonez et al. [2020;](#page-8-0) Jha et al. [2013](#page-8-0); Panichnumsin et al. [2010\)](#page-8-0). In the process, microorganisms break down organic matter into smaller molecules under anaerobic conditions starting from hydrolysis, acidogenesis, acetogenesis, up to methanogenesis (Srivastava [2020](#page-8-0)). In previous studies, anaerobic digestion produced biogas composing 50–75% methane, 35–40% carbon monoxide/carbon dioxide, 0–10% nitrogen, 0–1% biohydrogen, 0–2% oxygen, and 0–3% hydrogen sulfide (Balat and Balat [2009\)](#page-8-0). Aside from the desired methane gas that dominated the biogas product, it is also worth working to explore the possibility of producing high-value components such as biohydrogen gas from CWP. As far as literature provides, no exhaustive study has been done that uses CWP as a raw material in the production of biogas. It has not been explored and optimized, a significant knowledge gap because operating conditions of the production might have synergistic or antagonistic effects in the yield, characteristics, and compositions of the biogas from wastes.

This work explores the use of CWP in the production of high-quality biogas through anaerobic digestion. It would pace forward in the crusade for renewable energy production worldwide that would eliminate debates on food versus fuel competition (Dale [2017;](#page-8-0) Valenti et al. [2018](#page-8-0)). It likewise embraces the need to elevate the biogas knowhow, a technology introduced for more than three decades but not fully embraced and thus remains a topic of continuing concern, particularly its application in developing countries. Specifically, this work optimized the biogas volume via anaerobic digestion considering the effects of the concentration of pig manure (CPM) and biomass-water ratio (BWR) as chosen important variables. The widely used central composite design of the response surface methodology was employed with a criterion of maximizing biogas yield. The produced biogas was characterized to determine if high-value and desirable gases were generated during anaerobic digestion. Overall, this work addresses the call for a new alternate disposal route of CWP by making a valuable product—the biogas, a promising innovation that would give a share in answering the need for renewable resources.

2 Materials and methods

2.1 Collection and preparation of cassava waste pulps and pig manure

The cassava waste pulp (CWP) samples were collected from PhilAgro Industrial Corporation at Baungon, Bukidnon, Philippines. The samples were put in the icebox during transport to avoid the degradation of the biomass. In a very short time before experimentation, the samples were kept in the refrigerator at 20 $^{\circ}$ C. The pH of the CWP was measured using a pH meter (Yieryi, TPH01608, China), before the anaerobic digestion.

The fresh pig manure was collected from USTP farms at Ane-i, Claveria, Misamis Oriental, Philippines. It was added directly to the CWP biomass and the mixture was then loaded for anaerobic digestion.

2.2 Parametric study of biogas extraction

The initial range of values of the examined variables was based on recent literature: the concentration of pig manure or CPM (50, 100, 150, 200, and 250 gVS) as inoculum (Panichnumsin et al. [2010\)](#page-8-0) and biomass-water ratio or BWR (1:1.1, 1:1.3, 1:1.5, 1:1.7 and 1:1.9 kg/L) where CWP biomass was held constant at 1 kg. A parametric study was done by taking the center of one variable as constant while varying the other one. The peaks in the graph of the examined variables were used as the centers (level 0) in the subsequent optimization study (Gumaling et al. [2018\)](#page-8-0).

2.3 Experimental design and set-up of biogas extraction

The results of the parametric study were used in the design of the experiment that generated experimental runs via Design Expert 7.0 software. A total of 25 set-ups were made during the parametric study. Each set-up consisted of four pieces of plastic bottles connected in series. A biodigester cap with 2 cm diameter was drilled, and the 46 cm plastic tubing was inserted carefully into each bottle. The plastic tubing connected the plastic bottles. The 1st bottle (fermentation vessel) contained the substrate, while the 2nd bottle (water vessel), filled with water and saturated with carbon, served as the product containment vessel. The

displaced water in the second bottle was transferred in the 3rd bottle, while the 4th bottle served as a reservation space whenever much gases were generated, and displaced water could not be contained in the 3rd bottle. The 3rd and 4th bottles were both calibrated to monitor and measure the amount of the displaced water. All connections were sealed with rubber tubes and sealant to prevent leakages and made the whole bio-digester system airtight. Daily monitoring of the set-ups was observed throughout the duration of the experiment. The bio-digester was agitated daily to enable digestion to take place in the entire medium. The experimental set-up is best shown in Fig. 1.

Foreign matters in the substrate were carefully removed before these were loaded into the digester. Before each run, the substrate was stirred for 5 min using a mechanical stirrer. Subsequently, the compositions of the substrate mixture that were set according to the experimental runs generated by the CCD were put into the bio-digester for 30 days of retention time under ambient room conditions.

2.4 Determination of product yield, and biogas compositions

The biogas yield was determined via water displacement, a method that determined the volume of the gas that filled the bottles. When biogas was produced, the built-up pressure pushed the water in the 2nd bottle to the 3rd bottle, thereby displacing the water and was replaced by biogas products. The volume of the displaced water was measured through the calibrated 6-L plastic bottle, and the data was used to estimate the equivalent volume of the biogas (Otaraku and Anaele [2020\)](#page-8-0). The percent composition of gases that composed the biogas was analyzed through gas chromatography (HP-GC 78200) at Pilipinas Kao, Incorporated in Jasaan, Misamis Oriental, Philippines. Among the important compositions of biogas that were measured include methane, hydrogen, nitrogen, and carbon dioxide.

2.5 Statistical analysis, modeling, and optimization

Statistical analysis and modeling were done through the central composite design of the response surface methodology using Design Expert 7.0 software. The built-in analysis of variance determined a model that best fits the gathered data during batch experimentation. The percent biogas yield of the experimental runs underwent graphical modeling (3D model) to determine the interactive effects of CPM and BWR. By using the same software, numerical optimization was done and determined the optimum conditions that could result in the optimum volume of biogas as affected by CPM and BWR.

3 Results and discussion

3.1 pH of cassava waste pulps

The pH of the CWP of 6.4 ± 0.13 or nearly neutral is close to the favorable pH of 6.7–7.5 for biogas production (Adekunle and Okolie [2015](#page-8-0)). The near-neutral pH is suitable to produce biogas since methanogens use organic acids as the source of food, which leads to better fermentation activity (Jørgensen [2009](#page-8-0)). In this work, a more acidic environment might promote the production of biohydrogen $(H₂)$ because it would deactivate hydrogen-consuming bacteria (i.e., methanogens) in the process (Sriroth et al. [2015](#page-8-0)). The production of H_2 gas has numerous advantages over the known methane product in biogas technology.

Fig. 1 Experimental set-up for the biogas production process

Among the benefits include higher energy content on a mass basis, minor greenhouse emission during production, and a very environment-friendly by-product during combustion with only water vapor (Balat et al. [2008](#page-8-0)).

3.2 Parametric study result of biogas production

As shown in Fig. 2, the increase of CPM from 50 to 200 gVS results in an increasing trend of biogas yield from 4.2 to 7.63 L per kg of CWP, respectively. However, when the CPM is increased further, the biogas yield drops significantly.

The increasing trend is expected, considering that the increase of the concentration of pig manure as inoculum provides necessary microorganisms to initiate the digestion process (Rizwan et al. [2015](#page-8-0)). This means that the conversion of CWP to volatile fatty acids was minimized, considering that the digestion process took place immediately in the substrate with pig manure as inoculum. Without starup microorganisms from pig manure, there might be a production of high concentration of volatile fatty acids that could cause inhibition to methanogenesis and would lead to anaerobic digestion failure (Rizwan et al. [2015\)](#page-8-0). For optimization purposes of this work, the 200 gVS was set at the center value (level 0) of the design of the experiment.

Another important variable in anaerobic digestion is the ratio of the biomass and water (BWR) in the making of the slurry. This is important considering that the amount of water present in biodegradable waste affects and influences the biogas yield as it boosts the process of biodegradability of the substrate, which alters either in the increase or decrease of biogas yield (Babatola [2011;](#page-8-0) Jha et al. [2013\)](#page-8-0).

The result of the parametric study with variable BWR at constant CPM (200 gVS), as shown in Fig. [3,](#page-4-0) reveals maximum biogas yield at 1:1.1 kg/L BWR (1 kg CWP, 1.1 L water). This result is congruent to a previous study that used a 1:1 ratio in the production of biogas using cow dung and water (Sambo et al. [1995](#page-8-0)). Further investigation of this previous study showed that 1:0.5 and 1:2 biomass (cow dung) to water ratio resulted in less biogas yield compared to a 1:1 ratio. Hence, the result of this study agrees on the literature cited. For optimization purposes, the 1:1.1 ratio was used as the center (level 0) of the design of the experiment.

In the succeeding batch experiments to optimize biogas yield, the above-known center points were considered in the design of the experiment using the central composite design of response surface methodology. Particularly, the ranges of the two variables were as follow: CPM (100, 150, 200, 250, 300 gVS), and BWR (1:0.7, 1:0.9, 1:1, 1:3, 1:5 kg/L).

3.3 Batch experiment results of biogas production from cassava waste pulps

At variable CPM and BWR that were set according to the design of the experiment, the biogas yield varies from 4.90 to 7.30 L for every kilogram of CWP used (Table [1\)](#page-4-0). The highest yield of 7.30 L per kg of CWP was obtained at CPM of 250 gVS and 1:1.3 BWR.

The strong potential in the production of biogas from CWP is apparent, considering its huge volume discarded in the starch production area. For example, the PhilAgro Industrial Corporation, a starch processing plant in the Philippines, with a daily generation of 100 tons CWP, can potentially generate 490–730 thousand liters of biogas daily, a highly probable business in a large scale. When concerned industries embrace the potential of CWP for

8.0 7.63 7.10 7.0 Biogas yield (L) Biogas yield (L) 6.15 6.0 5.50 5.0 4.20 4.0 0 50 100 150 200 250 300 Concentration of pig manure (gVS**)**

Fig. 3 Biogas yield from a kilogram cassava waste pulps at varied CWP biomass–water ratio

Table 1 Production of biogas from cassava waste pulps

 a^2 Biomass = 1 kg cassava waste pulps

^bRatio of cassava waste pulps to water volume (1 kg biomass: L of water)

biogas production, such an act would contribute to the call for augmentation of renewable energy resources in the local, national, and global settings.

3.4 Modeling in predicting the volume of biogas

Based on ANOVA, a reduced surface quadratic model is most suited to predict the total volume of biogas from CWP with p-value of \lt 0.0001 (Table [2](#page-5-0)). There is strong evidence to build upon that biogas yield can be correctly estimated using the model equation derived from the experimental data gathered. There is only $\lt 0.01\%$ chance that an error could be committed in the calculation due to unpredictable data swings and variation from one value to another. Additionally, the lack of fit p-value of 0.2381 discloses no statistical significance relative to the pure error which supports the claim that the reduced quadratic model generated could accurately estimate the biogas volume when the values of the chosen variables are known. This reduced surface quadratic model equation is given in Eq. (1) where A represents CPM (gVS) and B represents BWR (kg/L).

Biogas yield
$$
(\%) = -10.3866 + 0.0559A
$$

+ 16.8491B - 0.0001A² - 6.4655B² (1)

Using Eq. (1), the biogas yield can be computed by plugging in the numerical values of A and B . Further, the ANOVA table reveals significant p -values of the terms $(A,$ $B, A², B²$ of Eq. (1) signifying its significant effects in the volume of biogas produced. Such effect of the terms to biogas yield can be determined through its numerical coefficient and algebraic sign that are both shown in the equation. The terms A and B have positive numerical coefficients signifying that when these two terms are taken singly, the increase of both CPM and BWR results in the increase of the volume of biogas. Of the two variables, the far greater numerical coefficient of BWR (16.8491) implies that this is a more influential variable than CPM with only 0.0559 numerical coefficient. On the significant square terms $(A^2$ and $B^2)$, the equation reveals both negative numerical coefficients implying that the increase of the values of A and B would otherwise result in the decrease of the volume of biogas with B having greater numerical coefficient than A. Hence, it can be deduced that a peak volume of biogas can be derived at a certain spatial point of A and B because increasing the values higher would result in a decrease of the yield. This can be explained further in quadratic model of biogas yield from cassava waste pulps

^bNot significant

the parabolic 3D plot whereby optimum biogas yield peaks at observable points (Fig. 4).

Based on graph modeling, a parabolic 3D is generated with a yield that peaks at high BWR and high CPM. Since it is parabolic, increasing further the values of the two variables after peak yield would gradually result in the decrease of biogas yield. This is a further explanation of the importance of the model equation with different signs of numerical coefficients. The increase of CPM, up to a certain point, results in the increase of biogas volume

because of the buffering capacity and nitrogen source for the microorganisms to prosper, thereby stabilizing the anaerobic digestion process (Panichnumsin et al. [2010](#page-8-0)). Similarly, the increase of BWR, up to a certain point, results in a significant rise in yield up to an observable point, which implies that the mixing conditions were rightly chosen considering that a parametric investigation was carefully conducted before the actual batch experiment. Generally, the 3D plot emphasizes the interactive

Fig. 4 3D plot showing the effects of the concentration of pig manure and biomass to water ratio on biogas yield

Table 3 Actual versus predicted biogas yield from cassava waste pulps

| Run ^a | Operating variable | | Biogas yield (L) Actual 6.10 5.00 | |
|------------------|--------------------|------------------|--|-----------|
| | CPM (gVS) | BWR^{b} (L/kg) | | Predicted |
| 1 | 250 | 1:0.9 | | 6.34 |
| 2 | 200 | 1:0.7 | | 4.88 |
| 3 | 150 | 1:0.9 | 5.20 | 5.44 |
| 4 | 200 | 1:1.1 | 7.30 | 7.54 |
| 5 | 100 | 1:1.1 | 4.90 | 4.78 |
| 6 | 200 | 1:1.1 | 7.10 | 6.96 |
| 7 | 200 | 1:1.5 | 7.10 | 6.98 |
| 8 | 150 | 1:1.3 | 6.10 | 6.34 |
| 9 | 200 | 1:1.1 | 7.20 | 6.96 |
| 10 | 200 | 1:1.1 | 7.10 | 6.96 |
| 11 | 300 | 1:1.1 | 7.00 | 6.88 |
| 12 | 200 | 1:1.1 | 6.70 | 6.96 |
| 13 | 250 | 1:1.3 | 7.30 | 7.54 |

 a^2 Biomass = 1 kg of cassava waste pulps

^bRatio of cassava waste pulps to water volume (1 kg biomass: L of water)

effects of the CPM and BWR in obtaining optimum biogas yield.

The reliability of the equation in estimating the biogas yield is further validated by the comparison of actual and predicted values (Table 3). It can be seen that the actual and predicted volumes of biogas are nearly equal. It proves the claim that the generated equation of the reduced surface quadratic model is robust and dependable in estimating the biogas yield.

The reliability of the model equation is further exemplified through the diagnostic graph of the actual versus

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predicted biogas yield (Fig. 5). The actual and predicted values are close to the trend line of the biogas yield. It supports the claim that the reduced surface quadratic model generated in this study is correct and reliable.

3.5 Numerical optimization and validation

The RSM suggested optimum conditions in numerical modeling analysis to attain theoretical biogas yield. Based on the analysis, the theoretical optimum yield of 7.43 L can be obtained at given optimum conditions: BWR 1:1.22 kg/ L, and CPM 250 gVS. Actual experimentation conducted at given optimum conditions reveals a nearly equal biogas yield of 7.43 \pm 0.058 L (Table 4). This further reinforces the reliability and accuracy of the reduced surface quadratic model with an insignificant percent error between the actual and theoretical yields.

3.6 Biogas composition

Results of the gas chromatography analysis of the produced biogas, as posted in Table [5](#page-7-0), reveal the following percent

Table 4 Comparison of theoretical versus validated biogas yield

| Source | Operating variable | Biogas yield (L) | |
|--------------------------------|--------------------------|------------------|------------------|
| | BWR (kg/L) CPM (gVS) | | |
| CCD _(theoretical) | 1:1.22 | 250 | 7.43 |
| Validation _(actual) | 1:1.22 | 250 | 7.43 ± 0.058 |

Biomass $= 1$ kg cassava waste pulps

Fig. 5 Diagnostic graph on the actual versus predicted biogas yield from cassava waste pulps

compositions: carbon dioxide or $CO₂$ (38.02 \pm 0.71), nitrogen or N₂ (20.77 \pm 1.59), biohydrogen or H₂ (18.69 ± 1.71) , and a trace of methane or CH₄ (0.73 ± 0.28) . It is noted that the volume of H₂ gas is higher compared to $CH₄$ gas, a different result in typical biogas production with $CH₄$ dominating the composition at 65% and only a trace amount of H_2 concentration (Zhu et al. [2009\)](#page-8-0).

Most likely, the produced CH₄ during aerobic digestion, with the presence of H_2O and under pressure in the airtight container, was converted to $CO₂$ and $H₂$ gases in the process called steam methane reforming or SMR (Kong et al. [2020\)](#page-8-0). The presence of water that was purposely used to determine biogas volume by displacement in the 2nd container, by chance, served as water scrubber as it reacted the produced $CH₄$ gas in the 2nd container. In here, the $CH₄$ reacted with H₂O and produced H₂ and CO. After this, the CO was further converted into H_2 and CO₂. This is the reason that a high concentration of $CO₂$ and $H₂$ were detected from the collected biogas. The same reaction was proposed in related studies whereby a high concentration of H2 gas was produced during anaerobic digestion (Capa et al. [2020;](#page-8-0) Jechura [2015;](#page-8-0) Parente et al. [2020\)](#page-8-0).

The production of H_2 is looked-for considering the lower explosive limit (LEL) of 4% and the upper explosive limit (UEL) of 75% when compared to $CH₄$ gas with 5% LEL and 15% UEL (Ceper [2012](#page-8-0)). Also, the calorific value of H_2 (121–142 MJ/kg) is higher than that of CH₄ (50–56 MJ/kg) (Rajpara et al. [2018\)](#page-8-0). This result triggers a further investigation of exploiting CWP for optimum H_2 production, thereby contributing to the emerging biohydrogen technology. Anaerobic digestion may be modified by exploring the effects of relevant variables such as pH, temperature, and light (e.g., dark fermentation). In this way, the use of CWP for H_2 production may become the solution to the possible energy crisis in the future and the environmental problems faced by industries.

4 Conclusion

This study demonstrated the benefits of exploiting the potentials of cassava waste pulps (CWPs) for biogas and biohydrogen $(H₂)$ production. The concentration of pig

manure (CPM) and biomass to water ratio (BWR) have significant effects on biogas yield, as revealed in the coefficient of the terms in the model equation generated. Optimum biogas yield of 7.43 \pm 0.058 L per kg of fresh CWP was achieved at optimized conditions: CPM at 200 gVS and 1:1.3 kg/L BWR. Interestingly, the $18.69 \pm 1.71\%$ of the produced biogas is H₂ that is formed via steam methane reforming, an attractive outcome because H_2 is not only a high-value product but also environment-friendly biofuel with water as a by-product during combustion. Additionally, exploiting the CWP for biogas production would not only contribute to lessening the underutilized wastes but, more importantly, in its role in the current crusade of finding cutting-edge biofuel technology in the renewable energy sector. The outcome showed the immense potential of CWP for the production of valuable H_2 gas, a desirable biofuel for various applications. Although the study provides good insights for the production of biogas with high H_2 concentration, a further experiment that would optimize the yield of $H₂$ via suitable technique, like dark anaerobic digestion, maybe explored to better exploit CWP for the production of highvalue and environment-friendly $H₂$ gas. Overall, the study provides novel technology of producing H_2 gas at low-cost processing. The upshot of this work may serve as the basis of government instrumentalities to jump-start in the articulation of plans and innovative strategies that would lead to economic growth, environmental safety, energy sufficiency, and national security.

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Compliance with ethical standards

Conflict of interest The authors declare that they have no conflict of interest.

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