



EFFECTS OF TEXTILE WASTEWATER PRE-TREATMENT ON LIGNOCELLULOSIC BIOMASS FOR SOLID-STATE ANAEROBIC DIGESTION

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ABSTRACT

Researchers are looking into sustainable and inexpensive energy sources due to the constant rise in the price of fossil fuels, greenhouse gas emissions, and dependence on non-renewable energy sources. In this study, wastewater from the textile industry is co-digested with lignocellulosic biomass (wheat straw) to produce biogas. During 21 days, five anaerobic digesters were run at room temperature (28 to 30 °C). Wheat straw inoculated with cow manure was put into wheat straw operating in five digesters at five different ratios of wastewater to distilled water. It has been discovered that the slurry digested with mere wastewater (not diluted with distilled water) has the lowest production, while the slurry digested with wheat straw and cow dung has the highest production.

Keywords: Textile wastewater, lignocellulosic biomass, Solid-state anaerobic digestion

INTRODUCTION

In addition to raising energy demand, rapid urbanization and industrialization have also resulted in enormous waste release in a variety of forms. By 2030, there will be a 45–60% increase in the world's energy consumption, however today, 85% of that energy comes from traditional sources (Zheng *et al.*, 2014). The promise of biomass-based energy generation must be acknowledged by researchers who have found biomass to be a dependable and affordable energy source. A variety of biomass has been utilized for the production of biofuels depending on the constituents or the type of bioenergy product depends upon the process applied for this conversion, for example, fermentation results in bio-ethanol or biodiesel while anaerobic digestion produces biogas. Among different bioenergy products, anaerobic digestion for the production of biogas is preferred over other chemical or biological methods due to its good output/input ratio. Organic matter is broken down by a variety of bacteria during anaerobic digestion (AD), a biological process that results in biogas (about 50-75% CH₄ and 25-50% CO₂) and organic waste. Municipal solid waste and lignocellulosic biomass are two examples of feedstocks with a high solid content that are frequently treated using solid-state anaerobic digestion (SS-AD). SS-AD provides a number of advantages over liquid-state anaerobic digestion (LS-AD), including a high organic loading, less digestate production, and a low heating energy need. Nevertheless, lengthy solid retention times, insufficient mixing, and an inhibitor build-up are the key drawbacks limiting SS-AD effectiveness. For a successful and efficient SS-AD, it is important to control operation parameters such as nutrient levels, C/N ratio, feedstock-to-inoculum ratio, pH, temperature, and mixing. Biogas production in SS-AD performance can be enhanced by feedstock pre-treatment, co-

digestion, and supplement of additives such as biochar. This study provides a comprehensive summary of the current development in SS-AD as an effective way of treating solid waste materials. Lignocellulosic materials (that is agricultural wastes and crop residues) normally have high fiber content and primarily consist of lignin, cellulose, and hemicellulose. The cellulose, which is the main component of lignocellulosic material is a linear polysaccharide cellobiose with strong linkages through the β-1, 4 glycosidic bonds. These chains of cellulose are also connected via hydrogen bonds due to the presence of the hydroxylic group as well as van der Waals forces creating microfibrils with high tensile strength (Paul and Dutta, 2018; Zheng *et al.*, 2014). The cellulose microfibrils are further connected by hemicellulose. The hemicellulose-bound network of cellulose microfibrils is further bound together by lignin which is a large and complex aromatic hydrophobic amorphous heteropolymer constructed of phenylpropane units (Figure 1), and it acts as protection for the plant material from microbial attack (Paul and Dutta, 2018). This system leads to a three-dimensional network that is exceptionally rigid, water-insoluble, and optically inert, making it difficult for degradation by microbes and enzymes (Zheng *et al.*, 2014). The key, therefore, to unlocking the carbon in lignocellulosic biomass is by removing the grip of lignin on the readily biodegradable carbohydrates of cellulose and hemicellulose through physical, chemical or biological means (Sawatdeenarunat *et al.*, 2015). Pure alkaline pre-treatment is expensive and uneconomical in developing countries like Nigeria. Industrial alkaline materials are secondary resources that are cheaply available for the pre-treatment of lignocellulosic biomass.

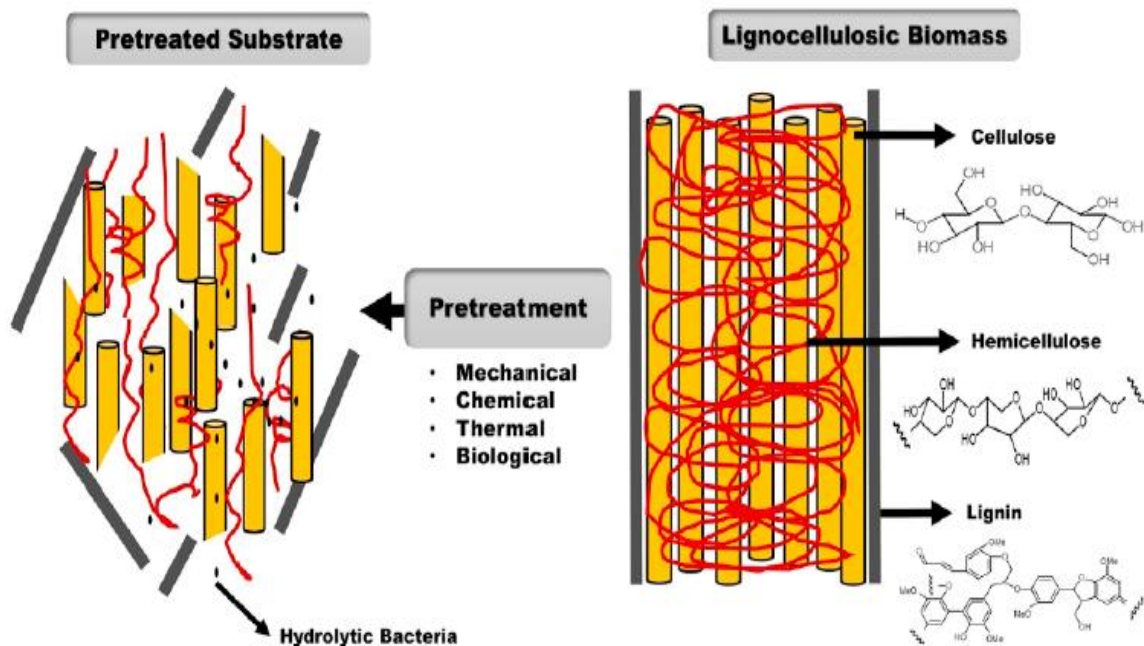


Figure 1: Role of pre-treatment on lignocellulosic biomass (Hosseini Koupaie *et al.*, 2019)

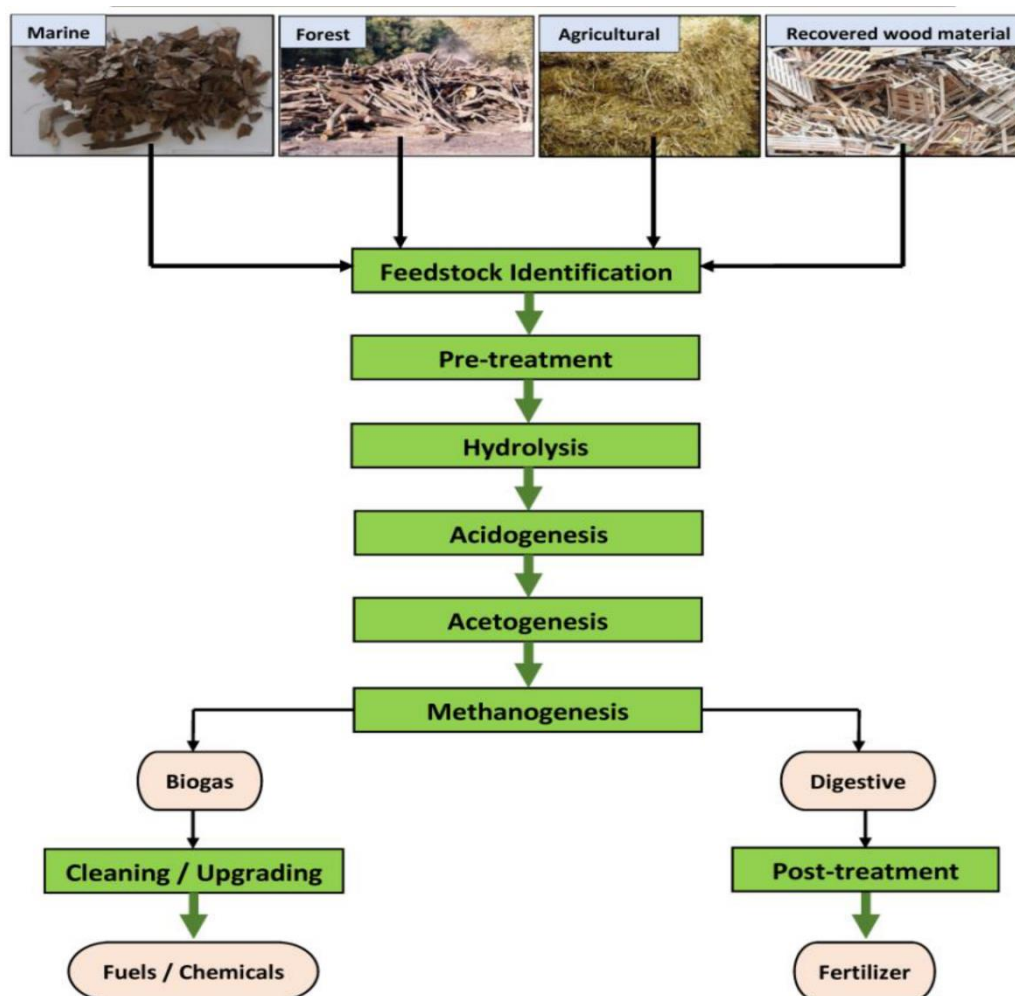


Figure 2: Anaerobic digestion of lignocellulosic biomass

A lot of work has been done for improvements in biogas production and is also in continuation. As in the process of anaerobic digestion, a huge amount of water is required for

slurry formation, and thus water wastage is a drawback of anaerobic digestion. This can be improved by replacing distilled water with wastewater from various sources. As the

living standards of people have been enhanced, this has resulted in the growth of industrialization. Hence, industrialization is increasing day by day, which releases more industrial wastewater. The type of wastewater depends upon the type of industry or the generally used process. Industrial wastewater can be categorized as inorganic and organic wastewater. Inorganic wastewater mainly disposed of in coal, steel, and metallic industries. Organic wastewater is mainly produced by the pharmaceutical, beverages, and textile industries (Holkar *et al.*, 2016). Textile wastewater was used as a solvent for making the slurry from lignocellulosic biomass and inoculums. Lignocellulosic biomass gives better result when integrated with textile wastewater for the production of biogas. The process of bio-methanation has been boosted up by the use of textile wastewater. This study aimed to determine the potential of textile wastewater as a pre-treatment resource for lignocellulosic biomass for biogas generation through anaerobic digestion. Specifically, the determination of the effect of textile wastewater on digestibility and biogas generation through solid-state AD. The study could provide insight into the potential of

establishing solutions that could reduce the impact of two important waste streams on the environment.

MATERIALS AND METHODS

Collection and Characterization of Wheat Straw, Cow dung and Wastewater

Wheat straw (*Tritium aestivum*) was collected from Agricultural and Environmental Engineering (AGE) departmental farm plots. The straw was crushed into a smaller size of than 20 mm before used. Cow dung was collected from a central abattoir in Kano city and used as an inoculum for the source of methanogens. The characterization of wheat straw and inoculum was performed in the AGE departmental laboratory.

Textile wastewater collected from African Textile Company (Plate 1) and kept at 4 °C without the addition of any chemicals to avoid further oxidation and degradation of wastewater constituents. The physio-chemical characteristics of textile wastewater samples were determined in the laboratory as per Standard Methods for the Examination of Water and Wastewater, 21st Edition.



Plate 1: Textile waste and lignocellulosic biomass samples collection

The analytical methods used in the study include

- i. Fibre content was determined in accordance with Van SOEST (1967) characterisation for fibre analysis as used by Zheng *et al.* (2014) using the British Standards stated below:
- ii. Sample preparation for fibre analyses in accordance with ISO 6498.
- iii. Amylase-treated neutral detergent fibre content (NDF) was conducted according to BS EN ISO 16472, which isolates the total fibre content of the substrate that includes lignin, cellulose and hemicellulose.
- iv. Acid detergent fibre (ADF) content and acid detergent lignin (ADL) content was determined according to BS EN ISO 13906:2008 standard. The first stage involves the removal of all insoluble carbohydrates, and protein complexes not removed by neutral detergent, leaving only the lignin, cellulose and hemicellulose fibres known as ADF. The second stage involves the removal of both cellulose and hemicellulose fibres using a stronger sulfuric acid leaving only lignin fibre as residue called ADL.
- v. Total solids (TS) and volatile solids (VS) was conducted based on loss of ignition method.
- vi. Total organic carbon (TOC) was determined based on loss of ignition method as described according to Dean, W. E. Jr., 1974.

- vii. Total nitrogen (TN) was determined based on the Methods for the Examinations of Water and Associated Materials (1985).
- viii. Total Ammonia Nitrogen (TAN) was determined based on the ISO/TS 14256-1 Soil quality – Determination of nitrate, nitrite and ammonium in field moist soils by extraction with potassium chloride solution.

Pre-treatment and biogas measurements

- i. The pre-treatment procedure was adapted from the work of Vazifekhoran *et al* (2018) at ambient temperature conditions in Kano, Nigeria is 35°C, and the pre-treatment was conducted in a temperature-controlled incubator at 35°C.
- ii. The BMP tests were based on the German Standard (VDI 4630, 2016) which has useful data analysis and reporting procedures. The BMP tests were conducted in an incubator under the mesophilic condition at 35°C,

involving 1 L glass culture vessels with 0.5 L headspace. The digestion was solid-state AD due to the advantages of easier material handling and lower retention times. The digestion was batch digestion where the reactors were sealed after loading and only emptied after digestion is completed. Gas ports were used, where one socket was used for the evacuation of biogas produced during digestion to the water displacement apparatus. The second socket was used for nitrogen gas purging to ensure anaerobic conditions in the reactor (Plate 4).

- iii. For biogas measurements, biogas volume was determined using an inverted measuring cylinder to collect gas through water displacement water. The GA 5000 landfill gas meter (manufactured by Geo-Tech) was used to determine the composition of the biogas (Plate 5).



Plate 2: Oven and Furnace of the determination of TS, TV and TSS



Plate 3: Oven and Furnace of the determination of TS and TV



Plate 4: Running BMP reactors

RESULTS AND DISCUSSIONS

Result of Characterization of Wheat straw, Cow dung and Wastewater

The characteristics of wheat straw and cow dung are reported in Table 2. Wheat straw and inoculate (cow dung) were analyzed for evaluation of total solids (TS), total-Kjeldahl nitrogen (TKN), pH, and volatile solids (VS). TS was determined by drying in an oven at 100 °C for 48 h. VS

content was determined by the mass of sample remaining after heating the dried milled sample at 550 °C for 4 h. The TS and VS of the substrate were determined before and after AD by 2540B method. The total carbon by Walkey and Black method, total nitrogen 4500-C, and chemical oxygen demand open reflux method (COD) by 5220B of the substrate were analyzed. The pH value was detected by a digital pH meter (Cph-102). Tables 1 and 2 present the characterization of

textile wastewater, wheat straw and cow dung. The textile sector produces large amount of wastewater with lots of contamination and high pH and COD value. The pH of textile wastewater samples is 8.2, is alkaline in nature. COD and TOC of sample are appropriate after dilution for growth and

activity of microbial population. Oil and grease are low in value, but there is sufficiently high amount of other water pollutants, that is sulphides, chlorides, and phosphates. Total suspended solids and total dissolved solids are 170 and 3400 mg/L, respectively.

Table 1: Characterization of textile wastewater sample

S/N	Parameter	Wastewater sample
1	pH	8.2±0.15
2	COD (mg/L)	1392±3.7
3	BOD (7 days) (mg/L)	762±2.6
4	TDS (mg/L)	3400±7
5	TSS (mg/L)	170±1.5
6	Sulphides (mg/L)	7±0.15
7	Chlorides (mg/L)	76±0.25
8	Nitrates (mg/L)	4.6±0.13
9	Phosphates (mg/L)	5.9±0.12
10	Electrical conductivity	4.4±0.13
11	Oil and grease (mg/L)	15

Table 2: Characterization of Wheat straw and Cow dung as a substrates

S/N	Parameter	Wheat straw	Cow dung
1	pH	7.5±0.15	6.2±0.12
2	TS (%)	75.5±1.25	32.4±0.11
3	VS (%)	87.5±1.35	22.2±0.01
4	TKN (%)	0.5±0.05	0.23±0.01
5	TOC (%)	8.90±0.25	4.75±0.21
6	COD (mgL-1)	200.0±4.5	80±1.2
7	C/N ratio	47.5±0.80	20.7±0.57
8	Celluloses (%)	40.5±0.56	19.6±0.16
9	Hemicelluloses (%)	27.9±0.35	21.8±0.24
10	Lignin (%)	6.8±0.14	1.9±0.14

The most crucial factors in building and running an anaerobic digester are the feedstocks. During anaerobic digestion, the early properties of the beginning material play a significant impact in process commencement, uniformity, and bioenergy output. Hence, the target of Hence, the goal of increased biogas generation can be accomplished. Whereas cow dung had an initial pH of 6.2, wheat straw had a pH of 7.5. The characterisation of wheat straw and cow dung are shown in Table 2. Wheat straw has a far better volatile solid content than cow dung, at 87.5% versus 22.2%. The cow dung had already undergone the process of digestion, so it had a low value of volatile solids. C/N ratios of inoculate used in the study were in between the optimal range of C/N ratio for anaerobic digestion that is 20 to 50, while that of substrate was quite high. The co-digestion of lignocellulosic biomass with cow dung can adjust the C/N of slurry to an appropriate level.

Biogas Yield from Different Digesters

During the anaerobic digestion all the five digesters, a significant change in all selected physico-chemical parameters was observed. Among all the five digesting set ups of wheat straw, the highest percent reduction of pH, TS, TOC, VS, COD, TKN, and C/N ratio for 75% dilution of wastewater has been encountered at room temperature with maximum biogas production (590 mL) (Figure 1) (Table 3) and (Plate 5). As the ratio of wastewater in the slurry increases, the activity of methanogens decreases, thus falling off the production of biogas. It has been found that the slurry containing wheat straw and cow dung digested with 75% diluted wastewater has maximum production, while the slurry digested with only wastewater (not diluted with distilled water) has the minimum production.

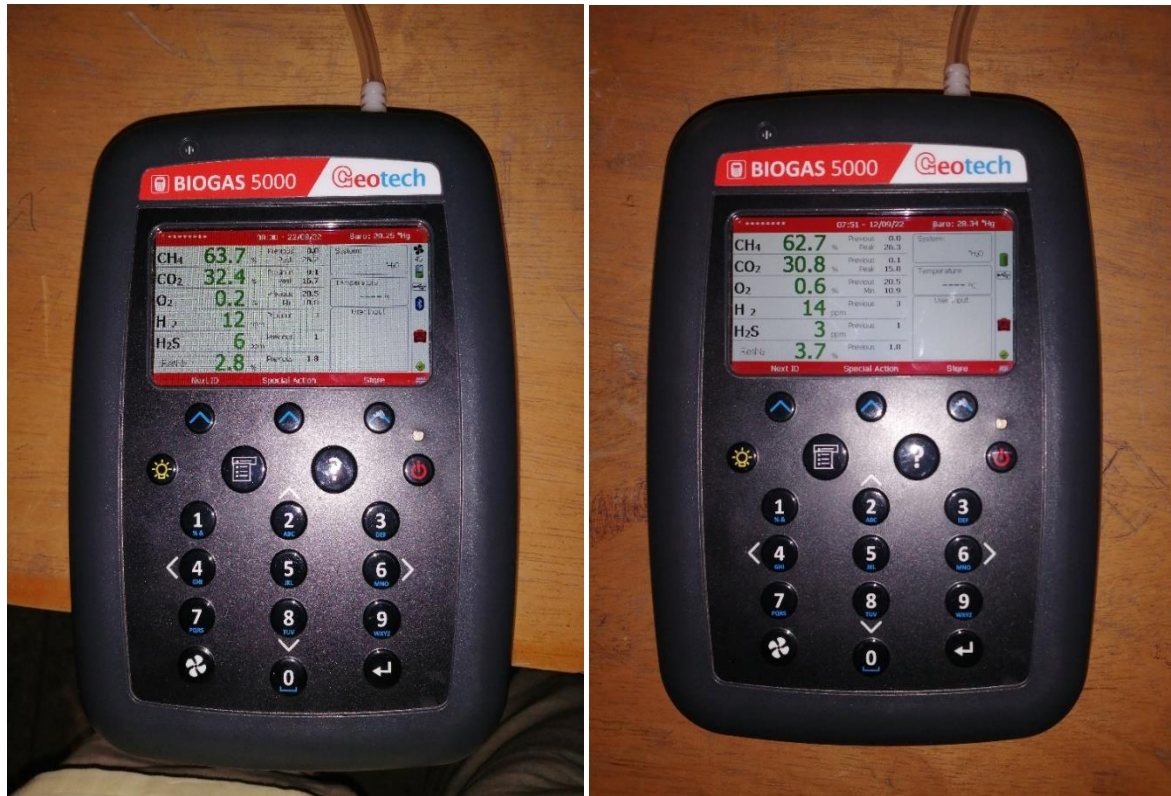
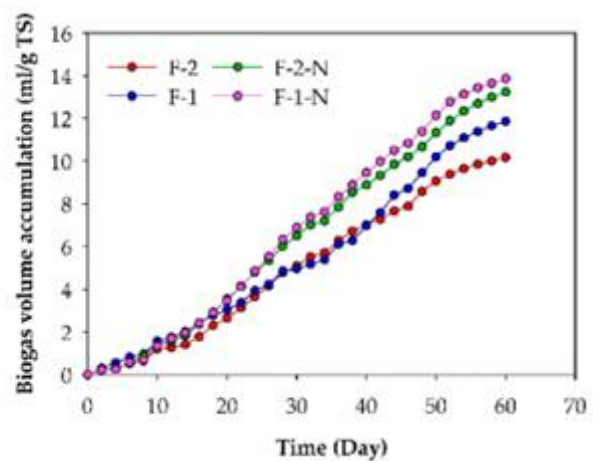
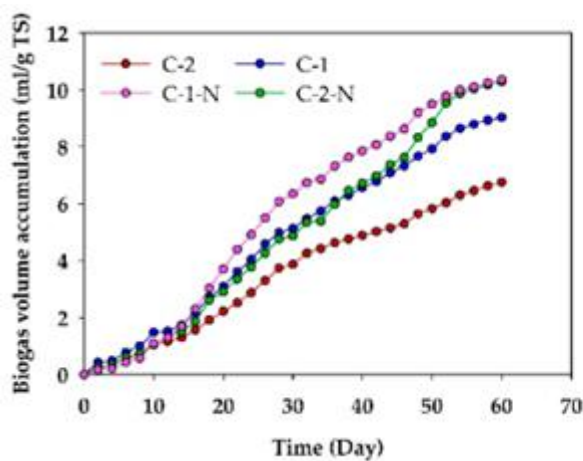


Plate 5: Result of Gas composition displayed by the biogas meter

Table 3: Experimental set ups for the digestion of wheat straw with cow dung and yields obtained

Set-up	Wheat straw: cow dung	Dilution of textile wastewater (%)	Distilled water (mL)	Textile wastewater (mL)	HRT (day)	Biogas production (mL/kg VS)
BMP1	1:1	Control	600	0	21	420
BMP2	1:1	100	0	600	21	415
BMP3	1:1	75	450	150	21	590
BMP4	1:1	50	300	300	21	520
BMP5	1:1	25	150	450	21	460



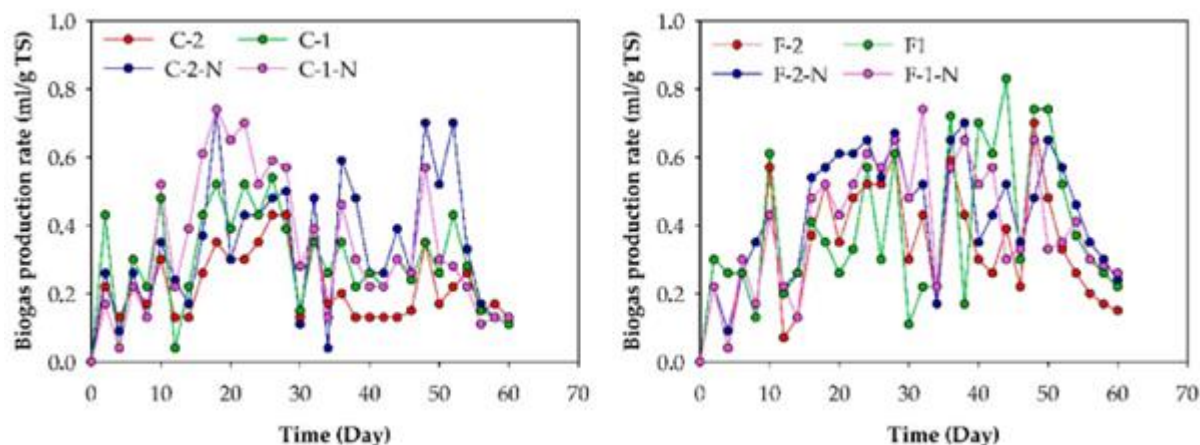


Figure 3: Biogas production for 21 days HRT

Effect of Wastewater Composition on Biogas Generation

The ions in the slurry play an important role in the stability and granulation of the reactor. Methanogens therefore behave in accordance with the environment that they are given. Methanogens require certain nutrients to support the ability of the microorganisms to produce methane. The results of the experimentation reveal that a greater wastewater content reduces biogas production by inhibiting the activity of the methanogens. Also, because the concentration of wastewater is kept at a suitable level, it gives bacteria the right nutrients, enhancing the production of biogas. The difference in CODs between before and after slurring each sample can help you understand this. The oxygen equivalent of the organic matter content is calculated as the Chemical Oxygen Demand (COD) of any biomass or slurry. The amount of oxygen removed by hanging organic compounds is measured as a change in COD, which includes a sizable amount of CO₂, H₂, and a minor number of other gases like H₂S. This demonstrates that sample MBP5's digester, which has a wastewater content of 25%, experiences the greatest reduction in CODs. Accordingly, that the precise ions in little quantities can increase methanogens' potential and activity for greater performance is easily reached. This is agreement with Paul and Dutta (2018) findings which demonstrated how increasing biogas production was impacted by a change in CODs.

Pollution Reduction in Textile Wastewater

Textile wastewater has pollutants that can cause a bad effect on aquatic life. Thus, reduction in these pollutants must be made before reviving to water bodies. Anaerobic digestion helps in the reduction of these pollutants. Analysis of pre and post characterizations of textile wastewater in this study has shown that the chemical moieties that may cause water pollution can enhance the biological activities of microbes.

CONCLUSION

The wheat straw biomass co-digested with cow dung using industrial wastewater was explored in this study. It has been determined that the production of biogas can be increased with the aid of textile wastewater. By giving bacteria the right nutrients, the creation of biogas with the right amount of wastewater increases the depolymerization of polysaccharides. The production of biogas has been increased by adding the right amount of textile wastewater. Changes in CODs and TOCs before and after each slurry sample indicated the effect of improved biodegradability. A study on textile wastewater pollution reduction has demonstrated that

using textile wastewater to produce biomethane aids in the reduction of water pollution.

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