Original Research Article

Evaluation of Biogas Productivity from the Mixtures of Vinasse, Wastewater and Filter Mud

Fathelrahman Ahmed Hamid Oboody1*, Hassan Beshir Elamin2 and Hanan Moawia Ibrahim3

1Department of Research & Development – Industrial Research & Sugar Technology Section - Kenana Sugar Scheme, Sudan Academy of Science, Sudan
2Department of Microbial Biotechnology, Commission for Biotechnology & Genetic Engineering Sciences, Sudan
3Ministry of High Education & Scientific Research, Central Laboratory
Associate Prof, Fermentation Technology & Industrial Biotechnology, Sudan

*Corresponding author

A B S T R A C T

This study was conducted in R&D department, Kenana Sugar Company, Sudan on June 2014 and January 2015, to treat the sugar and alcohol industry waste as a source of biomass converted into alternative renewable energy as a biogas from the mixing of Vinasse, wastewater and filter mud using anaerobic digestion process and rumen fluids as inoculum starter for 28 working days at room temperature. Experiments were carried out to find optimum condition and parameters of biogas production process, Substrates (Vinasse: filter mud: wastewater) were mixed into three different ratios A (1:3:1), B (2:2:1), C (3:1:1) experiments were conducted in summer and winter. In summer (A, TS=12.07a, COD=89.10a, Biogas yield=1718.3ml/l, B, TS=8.96ab, COD=55.30a, Biogas yield=1751.7ml/l, C, TS=5.9, COD=82.3, Biogas yield=2470ml/l), C had the highest biogas production and COD removal rate in summer. In winter (A, TS=13.16b, COD=119.66a, Biogas yield=2514.7ml/l, B, TS=15.96a, COD=18.28b, Biogas yield=2335.0ml/l, C, TS=10.3, COD=72, Biogas yield=2265ml/l). A had the highest biogas production and COD removal rate in winter. So the average of biogas yields in summer 1777ml/l, 2371ml/l in winter, both 2070 ml/l yield/year. That means 3752*106 m3/year of waste produce 7766*105 m3/year of biogas that equal 4660*105 kWh, also reduce odor, organic load, produce better fertilizer, minimize waste treating cost, power saving and less polluting. Results showed not significant different in biogas production and COD removal rate in summer and winter. Study concluded that utilization of mixed biomass derived from sugar industry byproducts and wastewaters for biogas production is applicable and feasible.

Keywords
Biogas Productivity, Vinasse, Wastewater and Filter Mud COD.

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Introduction

At the turn of the 20th Century, The evolution of the alcohol sector in Kenana Sugar Company is a remarkable fact.

It is known that for each liter of ethanol produced is generated 10-14 liters of vinasse, which is a highly polluting waste and problematic for treatment.
The objective of this study is to propose a rational use of distillery stillage generated in medium/large, which produces on average 200,000 to 180,000 liters of alcohol per day, and that consequently generates around 2,400,000 to 2,160,000 liters of vinasse/day (Ahmed, 2014).

The biological treatment of wastewater is a widely acceptable process that takes place aerobically or anaerobically. There is a huge source of biomass from wastewater disposal in Kenana Sugar Company; the factory with different sections such as mills, labs…etc. Produce more than 137,000 m³, town ship, hospital and other departments produces more than 10,000 m³ of wastewaters; so the total average of wastewater is more than 147,000 m³ per day; some of the sewage collected in sewerage with capacity 65,000 m³ per day (Adil, 2014).

The filter mud (also called filter cake and press mud) is a solid waste, generated on clarification of cane juice before its concentration and sugar crystallization. It is a soft, spongy, lightweight, amorphous, dark brown to black colored material. It generally contains 60-85% moisture (w/w); the chemical composition depends on cane variety, soil condition, nutrients applied in the field, process of clarification adopted and other environmental factors. During the clarification of cane juice using sulphitation process, most of the non-sugar components are precipitated by addition of milk of lime and sulphur dioxide. The precipitate is allowed to settle in a clarifier and the settled sludge is filtered by using rotary vacuum filter. The amount of sulphitation process mud cake is about 3-4% of the weight of the cane crushed. It is generally used as a fertilizer (Elhadi, 2014).

Problems

There is a reasonable quantity of industrial waste (vinasse, wastewater and filter mud) led to cause harmfully pollution affecting the human, animal, plant, water and soil.

There is a concern about difficulty of these problems solutions and highly cost of treatment.

Hypothesis

Variation and denser of biomass types will increase biogas yield.

There is an ability to convert (vinasse, wastewater and filter mud) into organic fertilizer (compost).

The main objectives of this study includes that, to produce biogas from the selected substrates materials (vinasse, wastewater and filter cake) by anaerobic digestion. To reduce high pollution caused by this byproduct contaminates water and soil. To determine the digestion of the optimum mixing of Vinasse, wastewater and filter cake to produce. And also o reduce COD, GHG emissions and filter cake treatment to achieve high efficiency of anaerobic digestion. To evaluate the sludge as a fertilizer (C, N, P, K).

Materials and Methods

Materials

The following material and equipment were used in experiment:

- Vinasse.
- Wastewater.
- Filter mud.
- Rumen fluids.
- Glass Bottles 2 liters capacity.
- Glass Bottles 0.5 liters capacity.
- Plastic bottles 1 liter capacity.
- Plastic container 20 liter capacity.
- Flexible hoses.
Soluble salt in water.
- COD meter.
- TS (Brix meter).
- PH meter.

**Substrates**

The substrates used were a mixture of (vinasse, wastewater and filter mud) vinasse obtained from Kenana Ethanol Plant, wastewater which left from sugar mill washing, labs, all factory water disposal and all township wastewater received in lagoon bond sewerage and filter mud or press mud is a solid residue, obtained from sugarcane juice before crystallization of sugar. The Sugar Factory and Ethanol Plant were located in Kenana Sugar Company, White Nile State, Sudan, which produced sugar from cane and Ethanol from molasses. Properties of substrates that were used as biogas feedstock are shown below.

**Inoculum**

The fresh rumen fluid was used as inoculum; the rumen was obtained from the butchery at Kenana, White Nile State, Sudan.

**Methods**

The Mixtures samples were analyzed for total solids (TS), Chemical Oxygen Demand (COD), total organic carbon (TOC), total nitrogen (TN) and minerals. The total volume of the biogas generated was measured. All the analysis was performed by standard procedures (APHA, 1998) unless otherwise noted.

**Chemical Oxygen Demand (COD)**

COD determined: 2ml deionized water (blank), 2ml sample, mix, heated in reactor 120 min at 150 °C, cool to 60 °C mixed and measured in sample chamber using TINO METER.

**Total Nitrogen (TN) Ratio**

Total Nitrogen (TN) was determined by Kjeldahl method using acid digestion, steam distillation and acid titration procedures.

**Total Solid (TS)**

The refract meter used by calf facilities is designed to measure the Brix value of a solution, used to indicate the sugar content of a solution, but this scale has been adapted for measuring total solids in Vinasse range 9.42, Wastewater 3.09 and Filter Mud13.22.

**pH**

PH meter was used, Vinasse range 4.56, Wastewater 6.67 and Filter Mud 4.50. Sodium hydroxide (NaOH 10) was used to adjust pH to 6.

**Total Organic Carbon (TOC)**

Total organic carbon was determined according to the method described by Standard methods for the examination of water and wastewater (2000).

**Biogas Measurement**

The determination of gases was carried out by displacement of water from Fermenter and flame test.

**Experimental Design**

The study was based on Completely Randomized Design (CRD) replicated three times. The treatments (Table 3.3) include loading three different mix ratios of 1:3:1, 2:2:1 and 3:1:1 of vinasse and filter mud respectively diluted with the same amount of wastewater (Carmen et al., 2006).
**Laboratory Digester Set up**

Anaerobic digestions of experimental laboratory using a 2-liter Digester were operated in batch system. One liter mixed substrate was put in the digester. Rumen fluid mixed inoculum was added into the digester 10% w/w substrate. An inverted measuring cylinder was used to measure the biogas volume through displacement theory.

**Preparation of Substrate**

Experiments were done into two separate environmental conditions: 1- summer experiment (old substrate materials), 2- winter experiment (freshly new substrates). The study was conducted on Research and Development (R&D) as follows:

200g of vinasse and 600g of filter mud were mixed with 200g of waste water and loaded into digester A1 replicated into A2 and A3. 400g of vinasse and 400g of filter mud were mixed with 200g of wastewater and loaded into digester B1 replicated into B2 and B3. 600g of vinasse and 200g of filter mud mixed with 200g of wastewater and loaded into digester C1 replicated into C2 and C3.

**Experimental set up**

Anaerobic digesters were made from a glass bottles which have a volume of 2 L. The bottles were plugged with rubber plug and were equipped with valve for biogas measurement. Anaerobic digesters were operated in batch system and at room temperature. Biogas formed was measured by liquid displacement method as also has been used by the other authors (Budiyono et al., 2013).

**Experimental Procedures**

Biogas formed was measured every once in two days to know biogas production with water displacement method. PH substrates in the digester were measured by pH meter every once in two days to know pH profile daily (Budiyono et al., 2013).

**Result and Discussion**

**Part1**

**Summer Experiment**

Summer experiment was conducted in Research and Development Department (R&D) in June 2014 and results shown below.

**Part2**

**Winter Experiment**

Was conducted on R&D January 2015 with freshly new substrates and result shown below.

**The Experiments Analysis**

**Content of Digestate (Effluent) as a Direct Fertilizer or Compost**

**Effect of Total Solid (TS) Ratio**

Total solid in vinasse and filter mud is very high, stated that feedstock of biogas with 7-9% solid concentration was best-suited. Investigated the manure: water ratios to find the best solid concentration in the production of biogas from cow manure (Budiyono et al.). Whereas investigation on the ratio of vinasse: filter mud: wastewater to find the optimum total solid content to biogas production rate has not yet been studied. This research was focused on investigating the effect of vinasse: filter mud: wastewater ratio against biogas production rate.

In anaerobic digestion, organic materials...
were decomposed by microorganisms through four stages, namely: hydrolysis, acidogenesis, acetogenesis and methanogenesis. The product of the process was biogas. This research was done to investigate the effect of wastewater addition on total solid (TS %). Variation of vinasse: filter mud: wastewater ratio caused change in solid concentration. The figure 1 shows the decreasing of total solid after anaerobic digestion.

**Summer experiment**

In this research the average of treatments replicates (A) which are (1:3:1) (V: W: F), parameter TS (8.7) % was decreased to (2.5) and recorded lowest productivity of biogas (1110) ml/l. Average of treatments replicates (B) which are (2:2:1) (V: W: F), parameter TS (9) % was decreased to (3.6) and recorded medium productivity of biogas (1751) ml/l. Average of treatments replicates (C) (3:1:1) parameter TS (5.9) reduced to (3) and recorded highest yield of biogas (2470). Figure 1 shows how TS effect on biogas productivity in summer.

Winter experiment highly increasing of TS was observed due to new freshly substrates and suitable temperature on winter that followed by highly biogas productivity. (A; TS 13.2, biogas 2514ml/l), (B; TS16, biogas 2335ml/l) and (C; TS10.3 biogas 2265ml/l). Fig 2 shows how TS effect on biogas productivity in winter.

Budiyono *et al.* reported that the ratio cattle manure: water: rumen fluid produced the biggest total biogas which has TS between 7.4 – 9.2%. Budiyono’s result confirmed the report of Zennaki *et al.* that solids concentration 7 – 9% in the substrate produced biogas optimally.

The result of this study was similar with those of the other authors. This research, authors did not use solid waste such as filter mud but research used liquid waste, vinasse and filter mud. Authors conclude that total solid level of either solid waste or liquid waste need to be considered.

**Effect of COD Ratio**

This treatment uses digester to transform COD of biomass into biogas at the anaerobic condition, the high COD can be destroyed by methanogenic bacteria in the digester, and COD is amount of oxygen needed for waste material in the water that can be oxidized through a chemical reaction. Anaerobic digestions of experimental laboratory using 1-liter volumes were operated in batch system.

Summer Experiment known that there is highly ratio of COD in treatments (A) which are (1:3:1), (V, F, W)average (89.1mg/l) with highly COD removal rate due to concentration of vinasse and filter mud so that produce a little amount of biogas range from (1110ml/l). Treatments (B), (2:2:1) that give lowest rate of COD range from (55.6mg/l) due to balancing of vinasse and wastewater quantity also achieved lowest rate of COD removal and generate more biogas than pervious treatment (1751ml/l). Treatment (C), (3:1:1) was recorded medium rate of COD ranged from (82.3mg/l) and removed to highest rate also achieved highest biogas yield (2470ml/l).

Winter Experiment treatment A (1:3:1) average COD before digestion (119.7) with highly removal (29.24) and highly biogas productivity (2514ml/l), B (2:2:1) average COD (48.4), low removal (18.3) and recorded optimum yield of biogas (2335ml/l), C (3:1:1) average COD (72) with optimum removal (25.3) and low productivity compared with A (2265ml/l).
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Influent COD and effluent COD of substrates were measured by using COD meter. COD of influent substrates and COD of effluent substrates were used to calculated COD removal (%). COD removal (%) = [(Influent COD – Effluent COD)/Influent COD] x 100%

COD ratio of substrate is necessary parameter to produce biogas optimally. So, if ratio between carbohydrate (C) and protein (N) are appropriate, biogas will be produced optimally. The value of C (carbon) can be obtained from COD, where C = COD value*(12/32). Mixtures used in this study contained large amount of carbohydrate and small amount of protein which converted

Table 1. Treatments with Different Mixing Ratio

| Digesters | Mix Ratios | Replications | V g/l | F g/l | W g/l |
|-----------|------------|--------------|-------|-------|-------|
| A         | 1:3:1      | A1,A2,A3     | 200   | 600   | 200   |
| B         | 2:2:1      | B1,B2,B3     | 400   | 400   | 200   |
| C         | 3:1:1      | C1,C2,C3     | 600   | 200   | 200   |

(V: Vinasse, F: Filter mud, W: Wastewater), Digester A with mixing ratio 1:3:1 and replicated into A1, A2, A3. Digester B with mixing ratio 2:2:1 and replicated into B1, B2, B3. Digester C with mixing ratio 3:1:1 and replicated into C1, C2, C3

Table 2. Summer Experiment Parameters and Biogas Yield

| Parameters | T.S. a (%) | T.S. b (%) | COD a mg/l | COD b mg/l | PH a | PH b | Biogas y/v ml/l |
|------------|------------|------------|------------|------------|------|------|----------------|
| Digesters  | A1(1:3:1)  |            |            |            |      |      |                |
| A1         | 12.5       | 3          | 80.9       | 31         | 4.5  | 7.5  | 1825           |
| A2         | 13.7       | 1.2        | 93         | 89         | 3.8  | 6.8  | 1560           |
| A3         | 10         | 3.3        | 93.4       | 20         | 4.6  | 7.6  | 1770           |
| Average    | 12.07a     | 2.50       | 89.10a     | 46.67      | 4.30a| 7.30a| 1718.3         |
| B1(2:2:1)  |            |            |            |            |      |      |                |
| B1         | 8.4        | 5.2        | 60.7       | 10         | 3.7  | 7    | 1330           |
| B2         | 10.5       | 3.5        | 46.2       | 20.4       | 3.2  | 6.8  | 1945           |
| B3         | 8          | 2.2        | 59         | 45.3       | 4    | 8.2  | 1980           |
| Average    | 8.96a      | 3.63       | 55.30a     | 25.23      | 3.63a| 7.33a| 1751.7         |
| C1(3:1:1)  |            |            |            |            |      |      |                |
| C1         | 5.6        | 1.5        | 92         | 62.4       | 5.5  | 9    | 1965           |
| C2         | 4.5        | 2.4        | 77.6       | 52         | 5.8  | 9.2  | 2385           |
| C3         | 7.5        | 4.4        | 77.3       | 64.2       | 4.9  | 8.5  | 3060           |
| Average    | 5.86b      | 2.76       | 82.30b     | 59.53      | 5.40b| 8.90b| 2470.0         |

COD a = COD before anaerobic digestion, COD b = COD after anaerobic digestion, TS a = Total Solid before digestion, TS b = Total Solid after digestion, Temp = the average of temperature while digestion, Biogas y/v = Biogas yield/ volume, COD = Chemical Oxygen Demand
Table 4.5 Winter Experiment Parameters and Biogas Yield

| Parameters | Digesters | T.S \(^a\) | T.S \(^b\) | COD \(^a\) mg/l | COD \(^b\) mg/l | PH \(^a\) | PH \(^b\) | Biogas \(\text{y/v} \text{ml/l}\) |
|------------|-----------|------------|------------|----------------|----------------|---------|---------|-----------------|
| A1(1:3:1)  |           | 13         | 5          | 119            | 20.30          | 4.8     | 6.8     | 2170            |
| A2         |           | 13.7       | 1.2        | 128            | 35.12          | 5.2     | 6.4     | 3199            |
| A3         |           | 12.8       | 2.8        | 112            | 32.30          | 5.5     | 6       | 2175            |
| Average    |           | 13.16\(b\)| 3.00       | 119.66\(a\)   | 29.24\(a\)     | 5.16     | 6.40\(b\)| 2514.7         |
| B1(2:2:1)  |           | 16         | 6.8        | 57.8           | 17.20          | 5.8     | 7.8     | 2170            |
| B2         |           | 16.4       | 4.5        | 42.3           | 15.43          | 5.4     | 9.2     | 2390            |
| B3         |           | 15.5       | 1.6        | 45             | 22.23          | 4.9     | 7.2     | 2445            |
| Average    |           | 15.96\(a\)| 4.30       | 48.36\(c\)    | 18.28\(b\)     | 5.36     | 8.06\(a\)| 2335.0         |
| C1(3:1:1)  |           | 9.2        | 1.2        | 65.2           | 17.40          | 6       | 9       | 2560            |
| C2         |           | 11         | 3.7        | 77.3           | 30.12          | 6.2     | 9.4     | 3097            |
| C3         |           | 10.7       | 4          | 74.2           | 28.28          | 5.9     | 8.9     | 1140            |
| Average    |           | 10.30\(c\)| 2.96       | 72.23\(b\)    | 25.26\(ab\)    | 6.03     | 9.10\(a\)| 2265.7         |

\(\text{COD} \(^a\) = \text{COD before anaerobic digestion}, \text{COD} \(^b\) = \text{COD after anaerobic digestion}, \text{T.S} \(^a\) = \text{Total Solid before digestion}, \text{T.S} \(^b\) = \text{Total Solid after digestion}, \text{Tempt} = \text{the average of temperature while digestion}, \text{Biogas}\ \(\text{y/v} = \text{Biogas yield/volume}, \text{COD} = \text{Chemical Oxygen Demand}\)
**Table.4** The Average of Replicated Treatments and Parameters for Summer and Winter Experiments

| Parameters          | T.S<sub>a</sub> | T.S<sub>b</sub> | COD<sub>a</sub> mg/l | COD<sub>b</sub> mg/l | pH<sub>a</sub> | pH<sub>b</sub> | Biogas y/v ml/l |
|---------------------|----------------|----------------|----------------------|-------------------|----------------|----------------|----------------|
| **Summer Experiment** |               |                |                      |                   |                |                |                |
| Treatments          | A             | B             | C                   |                   |                |                |                |
| T.S<sub>a</sub>     | 12.07<sup>a</sup> | 8.96<sup>b</sup> | 5.86<sup>c</sup>     | 20.53<sup>c</sup> | 1.06           |                |                |
| T.S<sub>b</sub>     | 2.50          | 3.63          | 2.76                | 52.99             | 0.90           |                |                |
| COD<sub>a</sub>     | 89.10<sup>a</sup> | 55.30<sup>a</sup> | 82.30<sup>b</sup>   | 11.80             | 5.13           |                |                |
| COD<sub>b</sub>     | 46.67         | 25.23         | 59.53               | 61.80             | 15.62          |                |                |
| pH<sub>a</sub>      | 4.30<sup>a</sup> | 3.63<sup>b</sup> | 5.40<sup>a</sup>   | 11.13             | 0.28           |                |                |
| pH<sub>b</sub>      | 7.30<sup>b</sup> | 7.33<sup>b</sup> | 7.68               | 7.68              | 0.34           |                |                |
| Biogas              | 1718.3        | 1751.7        | 2470.0              | 16.70             | 191.38         |                |                |
| **Winter Experiment** |               |                |                      |                   |                |                |                |
| Treatments          | A             | B             | C                   |                   |                |                |                |
| T.S<sub>a</sub>     | 13.16<sup>b</sup> | 15.96<sup>a</sup> | 10.30<sup>c</sup>   | 4.20              | 0.31           |                |                |
| T.S<sub>b</sub>     | 3.00          | 4.30          | 2.96                | 68.03             | 1.34           |                |                |
| COD<sub>a</sub>     | 119.66<sup>a</sup> | 48.36<sup>c</sup> | 72.23<sup>b</sup>  | 10.80             | 4.98           |                |                |
| COD<sub>b</sub>     | 29.24a        | 18.28b        | 25.26ab             | 18.73             | 2.62           |                |                |
| pH<sub>a</sub>      | 5.16          | 5.36          | 6.03                | 7.34              | 0.23           |                |                |
| pH<sub>b</sub>      | 6.40a         | 8.06a         | 9.10a               | 6.87              | 0.31           |                |                |
| Biogas              | 2514.7        | 2335.0        | 2265.7              | 24.4              | 334.1          |                |                |

**Table.4.7** Content of Digestate (Effluent) as a Direct Fertilizer or Compost

| Experiments         | Content | DM% | N% | P(P₂O₅)% | K(K₂O)% |
|---------------------|---------|-----|----|-----------|---------|
| Summer experiment    | slurry  | 30  | 10 | 8.7       | 3.7     |
|                      | solid   | 2.6 | 4.9| 0.5       | 2.5     |
| Winter experiment    | slurry  | 24  | 5.7| 1.6       | 2.6     |
|                      | solid   | 6.4 | 1.5| 1.5       | 0.9     |

Chen *et al.* reported that COD/N ratio of 70 has been suggested for the stable performance of anaerobic digestion. This report could approach with the result of this study, which the range of COD/N ratios of 71.4 – 85.7 (500/7 – 600/7) showed the satisfactory results. In control variable, availability of nitrogen source was too few. Hence, bacteria in the digester could not build cell structures well and the rate biogas production was low.

This research was confirmed with Chen *et al.* finding compared COD removal and biogas productivity.
Effect of Digestate

Application of digestate as fertiliser must be done on the basis of a fertiliser plan. The fertiliser plan is elaborated for each agricultural field, according to the type of crop, the planned crop yield, the anticipated utilisation percentage of nutrients in digestate, the type of soil (texture, structure, quality, pH), the existing reserve of macro and micro nutrients in the soil, the pre-crop and the irrigation conditions and the geographic area. (Mariana, 2014).

Effect of Environmental Impact

This study showed the reducing of COD, pH, OLR and TS reflecting environmental impact on reduced odor, GHG, climate change, saving water, soil, plant and human safe.

Effect of Economic Impact

My result can determine the economic benefits in cost minimizing from the quantity of waste can be treated by anaerobic digestion, saving energy cost by converting biogas into electricity also utilizing of digestate as a direct fertilizer or compost. Experience from Denmark indicates that the most economic and environmental friendly strategy of application of digestate as fertiliser is by fulfilling the phosphorus requirement of the crops with phosphorus from digestate. Application of digestate to fulfill the phosphorus requirement implies also a partial fulfillment of nitrogen requirement of the crops. The remaining nitrogen requirement can thus be completed by application of mineral fertiliser (Dumitru, 2012).

In conclusion, experiments were conducted in summer and winter
- Substrates (Vinasse: filter mud: wastewater) were mixed into three different ratios A (1:3:1), B (2:2:1), C (3:1:1) the optimum mixture of substrates was obtained (3:1:1).
- Industrial waste treatment using anaerobic digestion converted organic compounds into biogas.
- Highly pollution from COD, GHG emissions, odors reduced and filters cake treatment achieved
- Treatments with high rate of filter mud were high rate in TS.
- Digestate has optimum characteristic of compost (C, N, P, and K).

Recommendations

Considering the conclusions described above, the following recommendations may be considered:

1. Utilization the useless bulk of biomass derived from sugar industry byproducts and wastewaters for biogas production.
2. Set targets for gas demand to be met with biomethane considering the environmental issues.
3. Monitoring of anaerobic process parameters such as TS, COD and selecting the suitable conditions.
4. Utilize the digestate (Effluent) as direct fertilizer or compost.
5. Pilot scale should be making to investigate operational parameters and further studies should be done.

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