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Energy-efficient enrichment of bioslurry and methane content of biogas from wheat flour in a bio-electrochemical anaerobic digester

H. M. Jariwala[1], Y. C. Rotliwala[2*]

[1]*Research scholar, Chemical Engineering Department, Government Engineering College, Valsad, Gujarat Technological University, Ahmedabad 382424, India*

[2]*Chemical Engineering Department, Pacific School of Engineering, Surat, Gujarat Technological University, Ahmedabad 382424, India*

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The methane-rich biogas resulting from an anaerobic digester (AD) was utilized as a potential source of energy, whereas the bioslurry, due to its rich nutrients content, was explored as a fertilizer. The effect of a bio-electrochemical system (BES) integrated with a conventional anaerobic digester (AD) for biogas enrichment is widely reported. This paper presents a comparative study of the characteristics of bioslurry resulting from two different digestors, i.e., only-AD and AD-BES. In contrast to only-AD, AD-BES-derived bioslurry showed a significant increase in nitrogen, phosphorus and potassium content to the tune of 14.29%, 37.04% and 19.26%, respectively. Moreover, it showed an increment in pH and reduction of total solid, volatile solid, C:N ratio. This paper also includes a comparison of two different methods for analyzing methane and carbon dioxide concentration in the biogas, i.e., statistical analysis-based method using ORSAT apparatus and gas chromatographic (GC) method. The comparison showed little difference in methane and carbon dioxide concentrations between the two methods (1.66 % and 2.79%, respectively). The comparison of the energy requirement (kWh) of AD-BES and of other methane-enriching conventional methods demonstrated a noteworthy reduction in energy requirement of AD-BES (43-90%). The results showed that AD-BES provides significantly enriched nutrients of bioslurry, and increased the concentration of methane in the biogas at lower energy requirement.

Keywords: Anaerobic digester, BES (bio-electrochemical system), bio-fertilizer, digestate (bioslurry), enrichment

INTRODUCTION

In order to improve crop productivity and maintain soil fertility, frequent uses of chemical fertilizers and pesticides become common practices in agriculture fields leading to harmful effects on human health and also cause various environmental issues. In India, application of nitrogenous chemical fertilizers becomes the costlier affair due to increasing price of raw materials (petroleum) for chemical fertilizers production. Under the circumstances, it is creating a large gap of actual supply for supplementing nitrogenous fertilizer as a nutrient to plant growth [1]. Thus, conventional practices demand replacement in farming methods through the use of sustainable approaches. Presently, organic farming as a sustainable agriculture practice has gained global attention. In the year 2022, a FiBL (Institute of Organic Agricultural Research) survey reported that India ranked first in numbers of organic farmers (about 25 lakh), which increased by 93 percent as compared to year 2021 after Uganda (4 lakh) and Thailand (about 2 lakh). Despite numerous advantages of organic farming practice, poor crop productivity and high cost of production limits its acceptability as a routine farming practice [2].

In terms of resource recovery from biowaste, anaerobic digestor is considered to be a clean, efficient and sustainable technique for generation of energy from organic wastes (dung, cattle waste, food waste, agriculture waste and municipal solid waste) [3]. The products of the anaerobic digestor mainly consist of methane-enriched gas known as biogas utilized as a domestic cooking fuel/commercial fuel (compressed natural gas) and residual slurry known as bioslurry [4]. The bioslurry, originating from high-solid content waste materials, is a complex mixture composed of various dissolved organic matters, salts, microbes, and other suspended solids (SS) [5, 6]. It contains nutrients such as nitrogen (N), phosphorus (P), potassium (K), and other important elements for plant growth, which are utilized for soil quality improvement and crop yield [4, 7, 8]. Also, the bioslurry is reported to be creating a closed-loop system where nutrients are recycled from biowaste creating zero waste valorization of residues to accomplished circular economy and reduced environmental impacts [9, 10]. A significant improvement in crop yield of soybean, sunflower, wheat, rice and sweet corn is reported using bioslurry on agricultural fields [9, 11-13].

* To whom all correspondence should be sent:
E-mail: *yrotliwala@yahoo.com*

The utilization of bioslurry as an effective soil conditioner is widely reported. The high water content (~93%) in the bioslurry imparts bulkiness and raises handling challenges [14-16]. Also, some of the authors demonstrated that the anaerobic digestion favors phosphorous precipitation leading to increased pH of the resulting bioslurry [17, 18]. Moreover, few studies reported that alkaline pH encourages loss of nitrogen in the form of volatile ammonia. Due to the typical nature of alkaline pH of bioslurry [19,20], transformation rate of C/N decreases. Thus, application of bioslurry in its present form limits its uses as a bio fertilizer at agricultural fields and demands further enrichment and quality improvement in bioslurry. To the best of our knowledge, very few studies (mainly on mechanically dried slurry) are reported in terms of bioslurry enrichment. Thus, the idea came to look for alternative methods for enrichment of bioslurry through AD-BES using wheat flour (carbohydrate - 82%, protein - 14% and fat - 4%) as a substrate. The application of AD-BES for enrichment of bio slurry is not yet reported.

Our previous study [21] revealed that integration of BES with an AD digester improves volume and quality (methane- enrichment) of the resulting biogas. It showed that BES-integrated AD increases the volume of biogas by 66.7% and the concentration of CH_4 in raw biogas by 81.5 v/v% (from 64 v/v% CH_4) at a supplied voltage of 0.3 V. Further, in our previous study a statistical analysis-based method using ORSAT apparatus was used for the determination of CO_2 and CH_4 in biogas. This paper presents a comparative study of bioslurry characteristics (mainly N, P and K) resulting from only-AD and AD-BES. This paper also includes a comparison of two different methods, i.e., statistical analysis-based ORSAT apparatus (reported in earlier publication) and gas chromatography (GC) for analyzing the concentration of methane in the biogas. Besides, a comparative study of the energy requirements for the operation of AD-BES and other conventional method of methane enrichment is addressed.

MATERIALS AND METHODS

Feedstock

Cow dung was used as an inoculum with wheat flour substrate-to-water ratio of 1:1 (by weight). A 20 kg homogeneous mixture of inoculum was created by combining equal weights of cow dung and water. This mixture was tested for total solids (TS) and volatile solids (VS), resulting in measurements of 10% and 7.5%, respectively [21]. After starting biogas generation within 4 to 5 days, substrate was added at a frequency rate of 3 g per day for the duration of 25 days.

Digester setup

As shown in Figure 1, the digester has two cylindrical floating domes fabricated with high-density polyethylene (HDPE); two digesters (only-AD and AD-BES) of 20 L capacity were used to carry out experimental runs at 30ºC ± 5ºC for the duration of 25 days. Conventional Only-AD digester was without electrodes, whereas the AD-BES digester included two carbon (99.9% pure graphite, length - 127 mm, diameter -25.4 mm, surface area - 11147.56 mm^2) electrodes (anode and cathode) installed at the bottom at a distance of 210 mm. A DC power supply to regulate the voltage (Tektronix, PWS2185, 0-18 V, 5A, India) was installed across these two electrodes. Detailed drawing of the AD-BES digester along with all accessories and specifications (high-density polyethylene (HDPE, cylindrically shaped floating dome type (capacity 20 L)) can be found in our earlier publication [21].

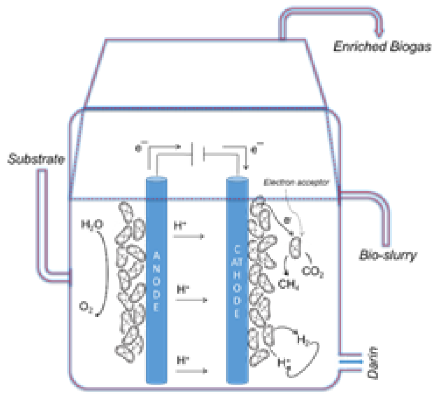

Figure 1. Schematic diagram of a bio-electrochemical integrated anaerobic digester

Procedure

The overflow of the bioslurry (supernatant) was obtained at an interval of 5 days from the drain line installed at the outer side of the digester. In order to remove solids from the bioslurry, it was filtered out through Whatman-597 (30 µm) filter paper and further utilized for characterization. The resulting biogas from anaerobic digesters was collected from a sampling line fitted at the top of the floating dome and analyzed in a gas chromatograph (GC).

Determination of energy supply to digester

The energy requirement of AD-BES digester was calculated in kwh for the total run time of 25 days at 30 ºC ± 5 ºC (laboratory operating condition). Figure 2 shows the representative circuit diagram of the

AD-BES digester. A DC power supply regulator was used to regulate the supplied voltage across the electrodes. The experimental runs were conducted in an AD-BES digester at a supplied range of voltage 0.1 V to 1.5 V. The current was measured using A-meter attached with digester. The measured current was in the range of 0.0095 A to 0.095 A. The energy supplied was calculated using equation 1:

$$E_{kwh} = \frac{V_V \times I_A \times t_h}{1000} \quad \ldots (1)$$

where, E_{kwh}= energy supplied in kwh, V= voltage supplied, volt, I = current generated, ampere, t = duration of energy supply, h.

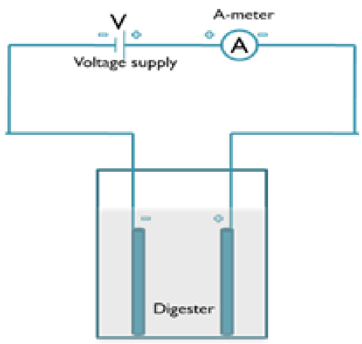

Figure 2. Circuit diagram of AD-BES digester

Determination of biogas slurry and biogas

The Soil Science Department, Navsari Agriculture University, Gujarat, India kindly supported for the characterization of N, P and K of bioslurry. The modified Kjeldahl's method (IS 3025 (Part-34) 2019) was used for nitrogen determination in the bioslurry. Phosphorus (P) content was determined after wet digestion [22]. Flame photometry (IS 3025 (Part 45) 2019) was adopted for potassium measurement in the bioslurry [22, 23]. The pH was measured with pH meter (LABMAN LMPH-10, India). Total solids (TS) and volatile solids (VS) were determined by the standard method (APHA 2540, 2012). CHN elemental analyzer (Leco CHN 2000) was used to measure total organic carbon and nitrogen (C/N) [24].

In our previous study, ORSAT apparatus was used for statistical analysis-based determination of CO_2 and CH_4 in biogas [21]. In order to compare the results of the statistical method, this paper addressed the determination of CO_2 and CH_4 in biogas using gas chromatographic analysis carried out at the Quality Control Laboratory, BEIL Infrastructure Ltd., Ankleshwer, India. Biogas produced from only-AD and AD-BES was collected in gas bladders and analyzed through Shimadzu (GC 2010 plus) GC equipped with an FID detector and a capillary column (DC -10, 25 m × 0.32 mm × 0.5 µm (detector film thickness)). The temperatures of the column chamber, inlet chamber and detector were maintained at 40 ^{0}C, 60 ^{0}C and 180 ^{0}C, respectively. The carrier gas, high purity nitrogen, was used at a flow-rate of 3 ml/min. The flow-rate of the sample was 30 ml/min, maintained injection volume was 1 µl at total run time of 10 min. The split ratio of gas sample in the inlet chamber was 1:1.15. The area under the curve 20520 was considered for standard methane of 250 mg/L.

RESULTS AND DISCUSSION

Characterization of the biogas

Figures 3 (a) and (b) show the chromatograms of biogas samples derived from AD and AD-BES, respectively. The peak areas of the tested biogas profiles were used to determine biogas composition. The chromatogram of only-AD showed methane and carbon dioxide peak area of 55383193 ppm and 5490952 ppm at retention time of 3.883 min and 4.588 min, respectively. As shown in chromatogram, in contrast to only-AD, AD-BES digester derived biogas indicated higher peak area of methane gas, i.e. 68194099 ppm and lesser peak area for carbon dioxide gas, i.e. 3802695 ppm. In order to determine the concentration of methane and carbon dioxide in biogas for respective area under the curve of chromatogram, the calibration was established using standard mixture of methane premixed carbon dioxide of concentration of 250 ppm and 350 ppm, respectively. The resulting area under the curve was 20520 ppm for methane and 28730 ppm for carbon dioxide. Accordingly, the area under the curve for the peaks of AD showed concentration of methane and carbon dioxide to the tune of 67.27 % and 29.80 %, respectively whereas AD-BES indicated concentration of methane and carbon dioxide to the tune of 83.08 % and 15.75 %, respectively. The study of other gases in biogas not considered in a chromatogram. The conventional AD derived biogas from cow dung reported to contain 60.29 % methane and the cooked starch releases 85.1% [25].

- *Deviation of ORSAT apparatus and gas chromatographic method for analyzing methane concentration in biogas.* In order to compare the two different methods, i.e., statistical analysis-based method using ORSAT apparatus (reported in earlier publication [21]) and gas chromatographic (GC) method for analyzing methane concentration in the biogas, three sets of experimental data were considered.

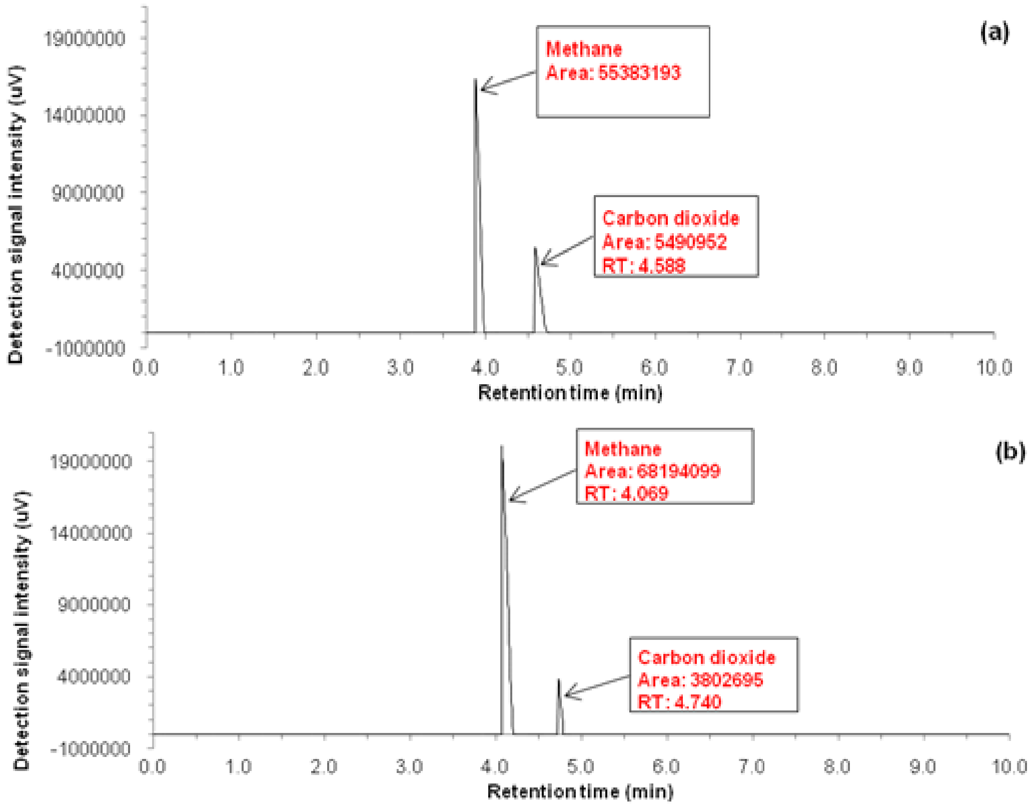

Figure 3. Chromatograms of biogas produced from (a) Only- AD and (b) AD-BES

Out of three data, one set of experiment using ORSAT apparatus method, AD-BES showed concentration of methane and carbon dioxide to the tune of 81.5 v/v% and 15.5 v/v %, respectively, whereas, the chromatographic study showed concentrations of methane and carbon dioxide to the tune of 83.08 % and 15.75 %, respectively. Thus, negligible deviation in the concentration of the two major gases using two different methods was observed, i.e. deviation of 1.66 % for methane and 2.79 % in carbon dioxide. Comparison between these two methods refereed for three sets of experiments.

Characterization of the bioslurry

Table 1. Characteristics of bioslurry obtained from AD-BES and only-AD digesters

Characteristics	Only-AD	AD-BES	% Change
pH	6.7	7.5	10.67
Water, %	89.0	92.1	3.37
Total solids, %	9.5	8.7	-9.20
Volatile solids, %	8.75	7.45	-17.45
C:N ratio	10.08	9.13	-10.41

Table 1 shows the characteristics of bioslurry obtained from two different digesters, i.e., only-AD and AD-BES. It is observed that pH (alkaline nature) of AD-BES-derived bioslurry increased by 10.67%. Improvement in solubility of phosphate and potassium concentration in the bioslurry of AD-BES is believed to be responsible for such increment. It was observed that in contrast to only-AD, bioslurry obtained from AD-BES demonstrated reduced percentage of total solids (9.2%) and volatile solids (17.45%). Such a reduction occurred due to the improvement in decomposition of fatty acids and total ammonium nitrates of the digester inoculum in BES. Some authors reported that solids consisting of manure in the digester were consumed more effectively for biogas generation and mineralization of organic carbon during the operation of AD-BES, leading to increased volume of biogas and methane concentration [26]. A similar reason was demonstrated for the reduction in TS and VS in a bioslurry obtained from AD-BES operated for treatment of sewage [27]. The AD-BES boosts the conversion of digester inoculum. Also, improvement in retention of nitrogen in bioslurry is believed to be responsible for reduction in C/N ratio by 10.41%. In contrast to AD, the bioslurry obtained from AD-BES has lower carbon-to-nitrogen ratio. The acceleration of the nitrogen mineralization process is to be responsible for such a change [28].

Nutrient composition of the bioslurry

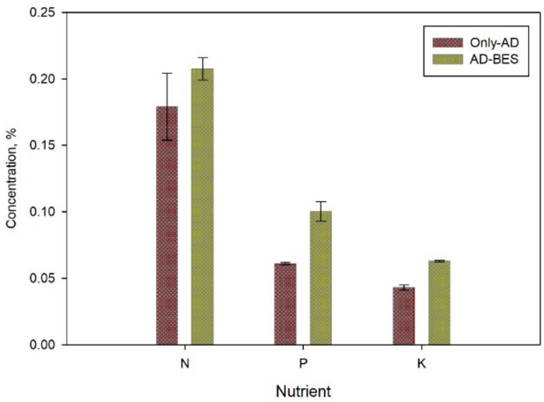

Figure 4, NPK content of bioslurry obtained from AD-BES and only-AD digesters

As shown in Figure 4, in contrast to AD, the bioslurry obtained from AD-BES demonstrated significant improvement in concentration of nutrients such as N, P and K. The nitrogen content of bioslurry increased by 14%, attributed to mineralization of complex organic nitrogen compounds to ammonium ion (NH_4^+) [29, 30]. As shown in equations 2 and 3 the ammonium ion is assumed to be oxidized through oxidizing bacteria at the anode surface of AD-BES producing nitrate (NO_3^-) followed by releasing nitrogen in the bioslurry [31, 32].

$$NH_4^+ + 2O_2 \rightarrow NO_3^- + H_2O + 2H^+ \quad \text{... (2)}$$
$$2NO_3 + 10e + 10H^+ \rightarrow N_2 + 2OH + 4H_2O \quad \text{... (3)}$$

During the anaerobic digestion process, phosphorus and potassium in the digestate are retained in the bioslurry [33]. The inorganic phosphate remains in the bioslurry, whereas the solid phase of the digestate contains both inorganic and organic phosphate [34]. The phosphorus concentration of the bioslurry from AD-BES increased by 37.04% compared to conventional only-AD. The remarkable improvement in the concentration of phosphate was due to the electrochemical precipitation of inorganic phosphate in the form of hydrate of magnesium ammonium phosphate ($MgNH_4PO_4 \cdot 6H_2O$) [35]. Upon further conversion of such a hydrate, it produces phosphate in the aqueous phase, which resulted in increased concentration of phosphate in the bioslurry of AD-BES. In the case of AD-BES, the concentration of total potassium in the bioslurry was by 19.26 % higher compared to conventional only-AD. The pH (alkaline nature) of the bioslurry is also believed to be responsible for the improvement in solubility of phosphate and potassium in the bioslurry of AD-BES. Thus, overall nutrientx in terms of nitrogen, phosphorus and potassium (NPK) were improved by an average of 28% using AD-BES.

- *Comparative energy requirement of AD-BES and other conventional methods for methane enrichment.* The study showed that for the production of 1 m³ of biogas enriched with 83% methane concentration, the energy requirement was 0.085 kWh (calculated from equation 1) at the supplied voltage of 0.3 V (current density of 0.34 mA/cm^2). As shown in Table 2, the energy required for AD-BES was lower by 43-90% when compared to conventional biogas production methods. This suggests a considerable improvement in energy efficiency using the AD-BES technique. The study is confined to laboratory-scale experiments, meaning real-world applications might show different results. The further research to explore these energy requirements and the potential of AD-BES on a larger scale will be the future scope of work.

Table 2. Comparative energy requirements (kWh) for AD-BES and other conventional methods for methane enrichment

Method	Energy requirement for 1 m^3 biogas generation (kWh)
AD-BES	(0.016 - 0.085) This study
Chemical absorption	(0.05 - 0.25) [36]
Membrane technology (gas/gas or gas/liquid)	(0.2 - 0.38) [37]
Pressure-swing absorption	(0.16 - 0.43) [36]
Absorption (water scrubbing)	(0.2 - 0.5) [36]
Cryogenic separation	(0.42 - 1.0) [37]

CONCLUSION

The results of the statistical analysis-based method using ORSAT apparatus and gas chromatographic analysis for determination of methane and carbon dioxide concentration in biogas are in agreement with each other showing a marginal deviation between the two methods for methane and carbon dioxide in the range of 1.66 % and 2.79%, respectively. The comparative study of bioslurry characteristics resulting from the two digesters, i.e., only-AD and AD-BES, showed that in contrast to only-AD, AD-BES-derived bioslurry demonstrated a significant increase in nitrogen, phosphorus and potassium concentrations (14.29%, 37.04% and 19.26%, respectively). Apart from this, it showed increment in pH and reduction in total solid, volatile solid, C:N ratio. Moreover, the energy requirement (kWh) of AD-BES demonstrated a noteworthy reduction (by 43-90%) as compared to conventional enrichment methods. The results showed that AD-BES significantly enriched nutrients of bioslurry, improved concentration of methane in biogas at lower energy requirement.

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