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## Effect and efficiency of two different purification systems for production of enriched biogas

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#### Abstract

Anaerobic digestion being a complex process produces biogas which varies in its composition depending on the type of the substrate and operational conditions of the digester. Apart from methane (CH<sub>4</sub>) the other gases are considered as impurities and their separation improves the calorific value of biogas. Thus, treatment of biogas mainly involves two steps *viz.*, biogas cleaning during which trace unwanted gases are removed; and biogas upgrading during which CH<sub>4</sub> is separated from carbon-dioxide (CO<sub>2</sub>). Thus, a study was carried out to upgrade the biogas produced from single stage anaerobic digester using two different purification systems *viz.*, Treatment-1 (T<sub>1</sub>) and Treatment-2 (T<sub>2</sub>). In T<sub>1</sub>, water vapour removal was by condensation mechanism, hydrogen sulphide (H<sub>2</sub>S) using iron scrap and CO<sub>2</sub> using 30% ethanolamine while in T<sub>2</sub>, water vapour was removed using activated alumina balls, H<sub>2</sub>S by 100 cm bed of activated charcoal and CO<sub>2</sub> using 0.25 M NaOH. The water vapour removal percentage by condensation mechanism and activated alumina balls was 93.99% and 99.28%, respectively. The H<sub>2</sub>S removal efficiency was 72.9% for iron scrap and 95.55% per cent for activated charcoal. The CO<sub>2</sub> removal percentage was 82.02% and 85.99% using ethanolamine and 0.25M NaOH solution, respectively. Overall, the results revealed that T<sub>2</sub> was 2.07% more efficient than T<sub>1</sub>.

Keywords: Biogas, carbon-dioxide, methane, purification, upgrading

### 1. Introduction

Anaerobic digestion of the organic matter produces two valuable products *viz.*, methane rich combustible gas called biogas and digestate. The process being a complex one helps in extracting carbon from the waste materials to be used as fuel and leaves behind the nitrogen in the digestate, to be used as an organic fertiliser. Biogas is a mixture of 50-70% methane (CH<sub>4</sub>) and 30-50% carbon-dioxide (CO<sub>2</sub>) (Angelidaki *et al.*, 2018) <sup>[3]</sup>. Apart from these gases, other components are present in traces which include water vapour (5-10%), hydrogen sulphide (0.005-2%), oxygen (0-1%), nitrogen (0-2%), ammonia (< 1%), siloxanes (0-0.02%) and carbon-monoxide (<0.6%) (Ryckebosch *et al.*, 2011) <sup>[15]</sup>. The composition of biogas varies depending on the type of substrate used and operational conditions of the digester. All the other gases apart from CH<sub>4</sub> are considered as impurities and their separation from CH<sub>4</sub> is important to improve the calorific value of biogas. Thus, treatment of biogas mainly involves two steps *viz.*, biogas cleaning during which trace unwanted gases are removed; and biogas upgrading during which CH<sub>4</sub> is separated from CO<sub>2</sub> (Adnan *et al.*, 2019)<sup>[1]</sup>.

Water vapour is present in traces in biogas which is mainly added at the time when biogas is leaving the digester. The water vapour contained biogas when used as such, causes corrosion of the engines thus, it is necessary to remove it. The removal of water vapours can be done by compression, cooling, absorption or adsorption (Petersson and Wellinger, 2009) <sup>[12]</sup>. The different adsorbing materials like molecular sieves or activated carbon can be used and the different absorbing materials are activated alumina balls and glycol solutions.

Different substrates like agricultural residues, animal manure, human excreta, kitchen waste, fruit and vegetable waste can be used for biogas production. The characteristics of substrates vary depending on the type of substrate. The protein rich wastes are high in both organic and inorganic sulphur. Upon anaerobic fermentation, the organic sulphur and sulphates present in the feedstock follow different pathways to produce hydrogen sulphide (Rasi *et al.*, 2011 and Peu *et al.*, 2012)<sup>[13, 14]</sup>. The H<sub>2</sub>S upon combustion, reacts with oxygen to form sulphur dioxide (SO<sub>2</sub>), followed by sulphurous acid (H<sub>2</sub>SO<sub>3</sub>) and sulphuric acid (H<sub>2</sub>SO<sub>4</sub>) thereby causing corrosion (Kuo and Dow, 2017)<sup>[9]</sup>. The H<sub>2</sub>S thus produced can be reduced pre-digestion, digestion or post-digestion (Ahmad *et al.*, 2019)<sup>[2]</sup>.

Pre-digestion removal can be by anaerobic co-digestion process. During digestion, the production of H<sub>2</sub>S can be reduced either chemically or biologically. In chemical method, the addition of FeCl<sub>2</sub>, FeCl<sub>3</sub>, or FeSO<sub>4</sub> to the digesters reduces H<sub>2</sub>S production where, the Fe ions react with H<sub>2</sub>S to form insoluble iron sulphide which can be removed along with the digestate (Shetty et al., 2020) [16]. A dose of air into the digester brings about aerobic oxidation reaction with the help of certain sulphate oxidising bacteria like sulpholobus and thiobacillus which reduces the H<sub>2</sub>S concentration in the biogas leaving the digester, biologically (Ryckebosch et al. 2011) <sup>[15]</sup>. After digestion treatments include- absorption, adsorption and biological scrubbing. The absorption of H<sub>2</sub>S can be done physically using water or chemically using FeCl<sub>2</sub>, NaOH etc. Adsorption by activated carbon, charcoal or iron sponge and bio-trickling filters can be used for biological method (Cebula, 2009 and Ahmad et al., 2019)<sup>[4, 2]</sup>.

CO<sub>2</sub> being an inert gas reduces the heating value of biogas. The calorific value of biogas can be increased by upgrading the biogas during which the  $CO_2$  will be removed in order to obtain biomethane as the final product which will be having around 90-95% methane and 1-5% CO2. There are several technologies for the removal of CO2 and they can be categorised into sorption and sepration. The sorption removal technologies include absorption and adsorption. Absorption of CO<sub>2</sub> can be physically using water or other liquids like polyethylene glycol or selexol and chemically using different amines like monoethanolamine, di-methyl ethanolamine and alkaline solutions of sodium hydroxide (NaOH), calcium hydroxide  $(Ca(OH)_2)$  or potassium hydroxide (KOH). Adsorption of  $CO_2$  can be done by Pressure Swing Adsorption process where the different adsorbents like activated carbon, zeolites etc, can be used. The separation technologies include the membrane technology and cryogenic separation (Adnan et al., 2019)<sup>[1]</sup>. Among the different methods, chemical absorption of CO<sub>2</sub> is most commonly followed as the method is simpler and with greater removal efficiency due to high reaction between the chemicals used and CO<sub>2</sub>.

In this study, different absorbing and adsorbing agents are used to remove different impurities from the biogas.

## 2. Materials and Methods

## 2.1 Experimental setup

The study was carried out at the Biogas Research Laboratory, School of Bio-energy and Farm Waste Management, College of Veterinary and Animal Sciences, Pookode, Wayanad which was situated at  $11^{\circ}32'18.5$  (North) longitude and  $76^{\circ}$  01'14.15 (East) latitude, and at an altitude of 867 m above the Mean Sea Level (MSL).

The study involved two fabricated single stage anaerobic digesters of 3000 l capacity which were daily fed with kitchen

waste and cow dung diluted with water and mixed in 1:1 ratio. The biogas thus produced was analysed for its composition using a biogas analyser (Model No. L-314 Precision scientific). The analysed biogas was made to pass through two different purification systems *viz.*, Treatment-1 ( $T_1$ ) and Treatment-2 ( $T_2$ ).

## 2.2 Treatment-1 (T<sub>1</sub>)

In this treatment, the biogas produced was passed through biogas purification system where, moisture was removed by condensation mechanism, carbon-dioxide by bubbling the gas through ethanolamine (Krumdieck *et al.*, 2008) <sup>[8]</sup> and hydrogen sulphide was removed by passing the gas through iron scrap (Feroskhan and Ismail, 2017) <sup>[5]</sup>. The schematic diagram and model diagram of the purification system is shown in Fig. 1.

The biogas leaving the digester was passed through the biogas flow meter which regulated the flow rate of 1 m<sup>3</sup> per hour. At first, the biogas was passed through the top of H-shaped PVC pipe of ---cm height and ---cm diameter to remove the moisture by condensation mechanism. The moisture dried biogas was then passed through the top of cylinder of ---cm height and ---cm diameter which was filled with iron scrap in order to remove H<sub>2</sub>S. The removal is by adsorption mechanism where, the H<sub>2</sub>S will be oxidised to sulphur contents. The sulphur contents will be trapped on the iron scrap by Vander wall forces.

$$2 \operatorname{Fe}_2 O_3 + 6 \operatorname{H}_2 S \to 2 \operatorname{Fe}_2 S_3 + 6 \operatorname{H}_2 O \tag{1}$$

$$2\mathrm{Fe}_2\mathrm{S}_3 + 3 \mathrm{O}_2 \longrightarrow 2 \mathrm{Fe}_2\mathrm{O}_3 + 6 \mathrm{S}$$

The biogas leaving the second cylinder from bottom will be made to pass through top of the third cylinder of ---cm height and ---cm diameter which contained 30% ethanolamine. The  $CO_2$  was separated from  $CH_4$  by chemical absorption process.

$$2RNH_{2} + CO_{2} \rightleftharpoons RNH_{3^{+}} + RNHCOO^{-}(Carbamate route)$$
(1)
$$RNH_{2} + CO_{2} + H_{2}O \rightleftharpoons RNH_{3^{+}} + HCO_{3^{-}} (Bicarbonate route)$$
(2)

Absorption of  $CO_2$  by the amine solutions is both by physically into the liquid as well as chemically. When the chemical reaction between the  $CO_2$  and amine increases, there will be mass transfer of  $CO_2$  from gaseous phase to liquid phase. Thus the chemical reaction helps in maintaining the concentration gradient of  $CO_2$  by consuming it in the liquid phase (Abdeen *et al.*, 2016).

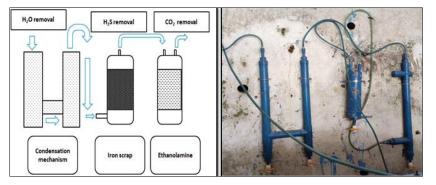


Fig 1a: Schematic diagram 1b. Model diagram of  $T_1$  purification system  $\sim$  1776  $\sim$ 

#### 2.3 Treatment-2 (T<sub>2</sub>)

The biogas produced was passed through the biogas purification system where, moisture was removed by passing the gas through activated alumina balls, carbon-dioxide by bubbling the gas through 0.25 molar sodium hydroxide solution and hydrogen sulphide was separated from biogas by passing it through a 100 cm bed of nano activated charcoal (Pallan *et al.*, 2018) <sup>[11]</sup>. The schematic diagram and model diagram of the purification system is shown in Fig. 2a & 2b.

The biogas leaving the digester and passing through the flow meter was made to enter the first PVC material cylinder from the bottom which had dimensions of ---cm height and ---cm diameter which contained activated alumina balls. The alumina balls being hygroscopic removed the moisture from the biogas. The dried biogas leaving the water removal cylinder from top was made to pass through second cylinder of ---cm height and ---cm diameter from bottom which contained a 100 cm bed of activated charcoal. The contents of sulphur from H<sub>2</sub>S were adsorbed on the activated charcoal and the gas left the cylinder from top. Then, the biogas was made to enter a U-shaped PVC pipe from the top which contained 0.25 M sodium hydroxide solution. The biogas while bubbling through the caustic solution reacted with it to form sodium carbonate.

 $2 \operatorname{NaOH}_{(aq)} + \operatorname{CO}_{2(g)} \operatorname{Na2CO}_{3(aq)} + H_2O_{(l)}$ 

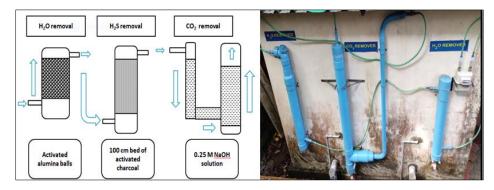


Fig 2a: Schematic diagram 2b. Model diagram of T2 purification system

The upgraded biogas from both the treatments were again analysed for its composition using the biogas analyser.

#### 3. Results and discussion

The single stage digesters by co-digesting kitchen waste and cow dung in 1:1 ratio produced the biogas whose composition was 53.62% CH<sub>4</sub>, 41.77% CO<sub>2</sub>, 4.33% water vapour and 0.27% H<sub>2</sub>S. The experimental values were in correlation with that of Pallan *et al.* (2018) <sup>[11]</sup> who got the composition of 53% CH<sub>4</sub>, 46% CO<sub>2</sub> and 1124 ppm of H<sub>2</sub>S by co-digesting cow dung and vegetable waste.

As the concentration of  $CH_4$  was low and concentration of  $CO_2$ ,  $H_2S$  and water vapour was high it would cause corrosion of the engines so it was decided to purify the raw biogas and increase the concentration of  $CH_4$ .

#### 3.1 Removal of water vapour

In T<sub>1</sub>, water vapour removal was by condensation mechanism while in T<sub>2</sub> it was by using alumina balls. The water vapour removal percentage by activated alumina balls was 99.28% which was higher than the condensation mechanism whose removal percentage was 93.99%. Results obtained are depicted in Fig. 3 which clearly indicates that activated alumina balls were more efficient in water vapour removal than by the condensation mechanism. The higher water vapour removal percentage from activated alumina balls was mainly due to hygroscopic nature of the balls which helped in removal of more amount of water vapour than by the condensation mechanism.

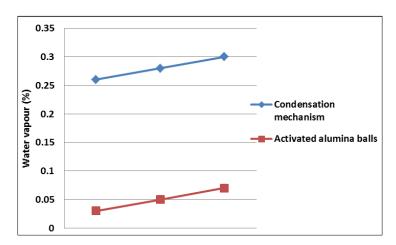


Fig 3: Water vapour removal (Condensation mechanism v/s Activated alumina balls)

#### 3.2 Removal of H<sub>2</sub>S

The  $H_2S$  was removed by using iron scrap and 100 cm bed of activated charcoal in  $T_1$  and  $T_2$ , respectively. The concentration of  $H_2S$  was reduced from 0.27% to 0.07% in  $T_1$ 

and to 0.01% in T<sub>2</sub>. Thus the removal percentage was 72.9% for iron scrap and 95.55% per cent for activated charcoal bed suggesting that charcoal was much efficient in  $H_2S$  removal than the iron scrap. The increased efficiency of activated

charcoal was due to its physical property of high adsorption capacity. The results thus obtained are depicted in Fig. 4. A similar trend in the purification system was reported by Pallan *et al.* (2018)<sup>[11]</sup>.

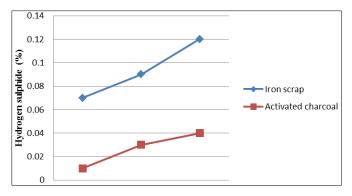


Fig 4: Hydrogen sulphide removal (Iron scrap v/s 100 cm bed of activated charcoal)

Within the scrubbing unit,  $H_2S$  reacts with oxygen during the catalytic oxidation process and the elemental sulphur thus formed during the activity will be adsorbed by the activated charcoal (Klein and Henning, 1984). The studies also reveal that larger the pore volume greater will be the adsorption capacity with increased level of  $H_2S$  removal.

In case of metal scrap, sulphur from the H<sub>2</sub>S is bound as the

metal sulphide releasing water vapour. Inside the scrubbing unit, when the concentration of the deposited sulphur in the form of metal sulphide increases, the rate of purification decreases thereby, lowering the H<sub>2</sub>S removal efficiency (Singhal *et al.*, 2017) <sup>[17]</sup>. Kapdi *et al.* (2005) <sup>[6]</sup> reported that, the lower removal efficiency of iron scrap was due to precipitation of sulphur on the iron scrap within the scrubbing unit.

The removal rate can be enhanced either by increasing the contact time of the biogas with the adsorbent or by increasing the absorption section. With the increase in the absorption section, the bed size can be increased which further increases the pore volume thereby achieving greater removal efficiency (Maile *et al.*, 2017)<sup>[10]</sup>.

#### 3.3 Removal of CO<sub>2</sub>

In T<sub>1</sub>, CO<sub>2</sub> was removed using 30% ethanolamine while in T<sub>2</sub> it was removed using 0.25 M NaOH solution. The initial concentration of CO<sub>2</sub> in raw biogas was 41.77% and upon up gradation the concentration of CO<sub>2</sub> was 7.51% in T<sub>1</sub> and 5.85% in T<sub>2</sub>. The removal percentage was 82.02% using ethanolamine and 85.99% using 0.25 M NaOH solution. The results obtained reveal that 0.25 M NaOH solution was more efficient in the removal of CO<sub>2</sub> than ethanolamine and the results are shown in the fig. 5.

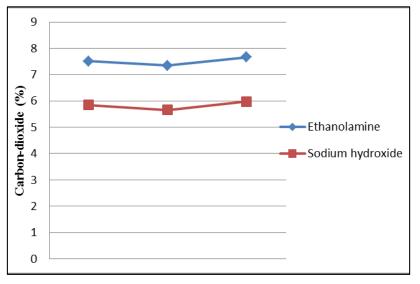


Fig 5: Carbon-dioxide removal (Ethanolamine v/s Sodium hydroxide)

By increasing the concentration of NaOH, the rate of absorption of  $CO_2$  can be increased (Maile *et al.*, 2017)<sup>[10]</sup>. With the increase in the concentration of NaOH, more OH<sup>-</sup>ions will be available in the solution thereby, increasing the  $CO_2$  absorption. The concentration of the  $CO_3^{2^-}$  ions also increases with the increase in the  $CO_2$  absorption. By increasing the molarity of the NaOH solution we may have obtained higher percentage of CH<sub>4</sub>. When ethanolamine and NaOH are compared, the use of NaOH is more beneficial because of its easy availability, cheaper and more efficient removal of  $CO_2$  than ethanolamine.

#### 3.4 Composition of upgraded biogas

The final composition of the biogas upon up gradation varied in  $T_1$  and  $T_2$ . The raw and upgraded composition of biogas obtained from  $T_1$  and  $T_2$  are mentioned in the Table 1/ Fig 6.

Table 1: Composition of raw and upgraded biogas

Components	Raw biogas (%)	$T_1(\%)$	$T_2(\%)$
Methane (CH <sub>4</sub> )	53.62	92.16	94.11
Carbon-dioxide (CO <sub>2</sub> )	41.77	7.51	5.85
Water vapour (H <sub>2</sub> O)	4.33	0.26	0.03
Hydrogen sulphide (H <sub>2</sub> S)	0.27	0.07	0.01

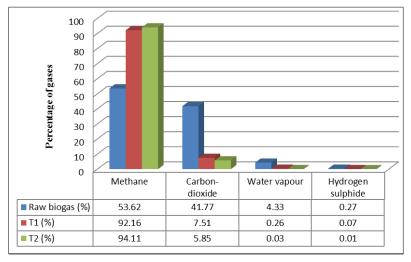


Fig 6: Composition of raw and upgraded biogas

The  $CH_4$  percentage obtained from  $T_2$  was higher than that obtained from  $T_1$ . Thus, it can be seen that  $T_2$  was more efficient in up gradation of biogas than  $T_1$ .

## 4. Conclusion

The composition of the raw biogas varied depending on the type of raw materials used. By co-digesting cow dung and kitchen waste, the raw biogas obtained had higher impurities and the use of such biogas with low calorific value for different purposes may cause corrosion of the engines. To avoid such problems, the obtained raw biogas was upgraded by using two different purification systems. The T<sub>2</sub> upgraded biogas obtained a final composition of 94.11% CH<sub>4</sub> which was 2.07% higher than T<sub>1</sub>. Thus, it can be concluded that use of activated alumina balls for water vapour removal, activated charcoal for H<sub>2</sub>S removal and 0.25 M NaOH solution for CO<sub>2</sub> removal is more efficient in the up gradation of biogas.

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