

# Experimental Analysis on Anaerobic Digestion of Municipal Solid Waste

Aromal Thampan<sup>1</sup>, Munish Kumar Chandel<sup>2</sup>

aromal.iitr@gmail.com

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## Abstract

The application of anaerobic digestion of municipal solid waste, though well established in many European and other developed countries, the full scale anaerobic digestion of MSW is still in its infancy in India. A total of six different samples of MSW has been taken and anaerobically digested in Oxitop serum bottles and their biogas and methane generation potential is studied. It is inferred that around 45% of the biogas generated is methane taking into account that the gas collection was carried out with glass syringe method, which is an approximate method. The theoretical methane and biogas generation are compared to the experimentally derived results.

Also, the characterization of the MSW had been studied for the influent and effluent samples experimentally. It had been found that the concentration of ammonical nitrogen increases and the concentration of nitrate and total phosphorus in the samples decreases at the end of anaerobic digestion.

## Keywords

Anaerobic digestion; Municipal solid waste; Oxitop serum bottle; Methane; Biogas; Ammonical nitrogen; Nitrate; Total phosphorus.

## Introduction

Anaerobic digestion is a complex biological process in which microorganisms break down biodegradable organic matter in the absence of oxygen and thus producing biogas [16]. It is the processes where the wastes are segregated and are added to a closed chamber inside which under anaerobic conditions, the organic fraction of the wastes undergo biodegradation producing biogas and sludge.

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<sup>1</sup>Faculty Member, The ICFAI University, Dehradun, Uttarakhand, India.

<sup>2</sup>Assistant Professor, IIT Mumbai, MH, India.

The biogas produced is an energy source and replaces:

- a) Petrol for refueling vehicles
- b) Coal for heat and energy production
- c) Natural gas for municipal gas supply

The effluent of this process, i.e. biomass/sludge after stabilization can be used as a soil conditioner. More over the anaerobic digestion process offers the possibility to recycle nutrients, reduce green house emissions, reduce odours and controlled waste disposal [2].

## Method and Methodology

### Municipal solid waste collection, grinding and mixing

The raw materials consisting of bio-degradable fraction of MSW of different composition were collected and shredded to finer particles. The wastes were collected, segregated, shredded and weighed as per the different compositions. The shredded MSW samples were characterized for moisture content, total solids, volatile solids, total carbon, total nitrogen, C/N ratio and COD using the standard methods.

This substrate was mixed with the fresh anaerobic seeding sludge collected from the Sewage treatment plant in Saharanpur, Uttrakhand. The resultant mixture, called feedstock was used in the reactors for anaerobic digestion process. A portion of this feedstock was analyzed for its chemical composition.

### Characterization of Sample

**Moisture content:** The moisture content for the waste samples and sludge was determined by weight loss of compost sample (105°C for 24 hour) using the gravimetric method [1].

$$\text{Moisture content (\%)} = \frac{\text{mass of water}}{\text{mass of sample}} * 100$$

**Total solids and Total volatile solids:** The total solids and total volatile solids were determined for all the influent and the effluent samples as per the standard method

$$\text{Total solids (\%)} = 100 - \text{moisture content}$$

$$\text{Total volatile solids} = \text{Total solids} - \text{Fixed solids}$$

**pH:** As pH is an important parameter for an efficient anaerobic digestion process, regular monitoring of pH has been done at an interval of every two days for the anaerobic reactors. The pH was measured using a pH meter with a glass electrode, previously calibrated and corrected for temperature. Sodium hydrogen carbonate (NaHCO<sub>3</sub>) was added as to the reactors, as a buffer, for maintaining the pH in the desired range.

**COD:** The Chemical oxygen demand was determined for all the influent and effluent samples as per the standard test procedure. A spectrophotometer was used for COD determination [1].

**Ammonical Nitrogen (NH<sub>3</sub>-N) and Nitrate (NO<sub>3</sub>-N):** 50 ml samples were prepared and the ammonical nitrogen and nitrate nitrogen were determined in a spectrophotometer [1].

**Total phosphorus (TP):** 100 ml samples were prepared for the determination of total phosphorus. Total phosphorus (TP) was analyzed using stannous chloride method [1].

### Experimental Setup

**Oxitop apparatus and samples used:** Oxitop bottles (WTW, Germany) of 312 ml with working volume of 250 ml were used as anaerobic batch reactor. The Oxitop bottles were washed thoroughly with chromic acid solution and distilled water. The bottles were dried and placed in incubator in order to maintain the desired temperature conditions. Six Oxitop bottles were used as reactors with different waste samples of composition as shown in the table below.

Table 1: Compositions of MSW samples used for Oxitop experiment [20]

Sr. No.	Population range (in millions)	Paper	Rubber, leather and Synthetics	Glass	Metal	Compostable Matter	Inert material
1.	0.1–0.5	2.91	0.78	0.56	0.33	44.57	43.59
2.	0.5–1.0	2.95	0.73	0.56	0.32	40.04	48.38
3.	1.0–2.0	4.71	0.71	0.46	0.49	38.95	44.73
4.	2.0–5.0	3.18	0.48	0.48	0.59	56.57	49.07
5.	5.0 and above	6.43	0.28	0.94	0.8	30.84	53.9
6.	-	-	-	-	-	100	-



Figure 1: Oxitop bottle set-up

The total composition of each sample was made to 100g. The 6<sup>th</sup> Oxitop bottle was completely filled with biodegradable fraction of the Municipal solid waste (100g). Anaerobic sludge was used as feed (50g) and 100 ml water was added to make slurry and the samples were kept at 30<sup>o</sup>C in the incubator.

## Anaerobic transfer and incubation of reactors

The procedure used by [23] was followed to anaerobically transfer the contents to bottles. Oxitop bottles were flushed with 30% CO<sub>2</sub> and 70% N<sub>2</sub> gas prior to the sealing of the Oxitop head. Oxitop bottles were sealed with pressure sensors. Total slurry volumes containing (sample 100g, sludge 50g and water 100ml) 250 ml were used in case of Oxitop bottles in order to maintain appropriate liquid to void ratio for precision and accuracy of results. Bottles were incubated in a temperature controller chamber at (30 + or - 2)<sup>o</sup> C.

## Biogas collection and analysis

Gas produced in Oxitop bottles was measured at regular intervals. Gas sampling and removal during incubation was performed with glass syringes (5-20 ml, depending on gas volume) equipped with 20-gauge needles. When the pressure within the Oxitop bottles exceeds, the gas was released with the help of glass syringe and added cumulatively. The main constituents of the biogas produced were methane and carbon dioxide. Methane content in the biogas was measured and recorded after passing the total biogas through Sodium hydroxide (NaOH) solution. The biogas when inserted through syringe into the Sodium hydroxide solution, the carbon dioxide gas, dissolves in the solution and the methane gas was collected in the syringe. The amount of methane reported throughout is after blank correction.

The theoretical methane gas production was calculated from the COD of the MSW samples and compared with the experimental results.

## Results and Discussion

### Proximate analysis

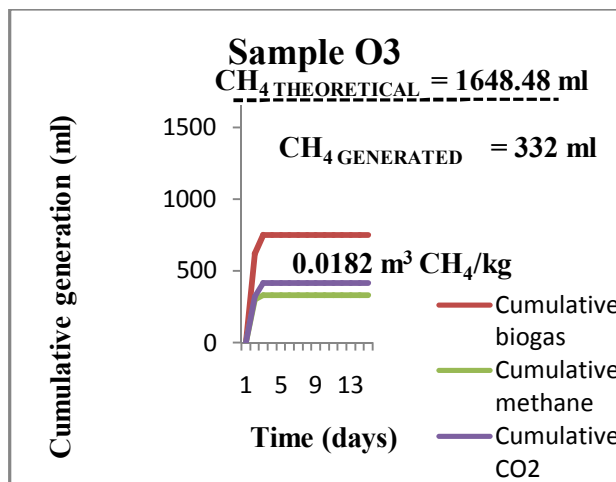
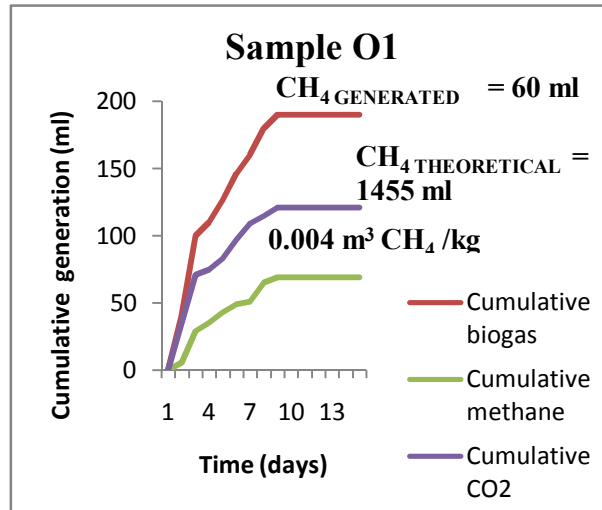
The proximate analysis for the six Oxitop samples was determined experimentally and is tabulated in table below.

Table 2: Proximate analysis result for Oxitop samples

Sample No.	Sample	Moisture content (%)	T.S.S (%)	V.S.S (%)	Ash content (%)	
1.	O1	Initial	32.9	67.1	53.08	5.31
		Final	44.15	44.4	27.70	2.77
2.	O2	Initial	29.24	70.96	56.41	5.64
		Final	37.38	64.20	55.2	5.52
3.	O3	Initial	32.69	68.31	54.17	5.42
		Final	24.7	65.30	46.2	4.62
4.	O4	Initial	38.53	69.47	54.14	5.41
		Final	34.32	37.8	20.70	2.07
5.	O5	Initial	21.46	78.54	64.31	6.43
		Final	24.5	67.00	22.20	2.22
6.	O6	Initial	71.35	28.65	6.39	6.4
		Final	25.52	64.8	27.6	2.8

The samples O1, O2, O3, O4, O5 and O6 represent Oxitop samples of different compositions as discussed above. O2 was the Oxitop bottle that did not generate any biogas and this is evident from its V.S.S values for its initial and final samples.

The samples were kept in the incubator under a controlled temperature of  $30 \pm 2^{\circ}$  C and the biogas production, the methane and the carbon dioxide generated from it was recorded daily and tabulated. The graphs showing the cumulative biogas, methane and carbon dioxide generation for all the six Oxitop bottles were plotted and shown below.



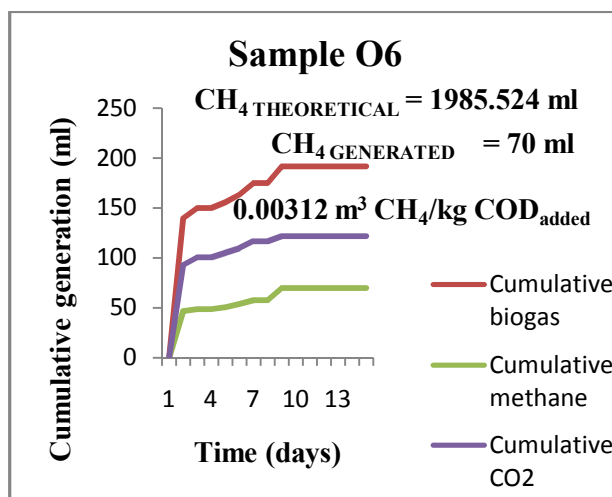
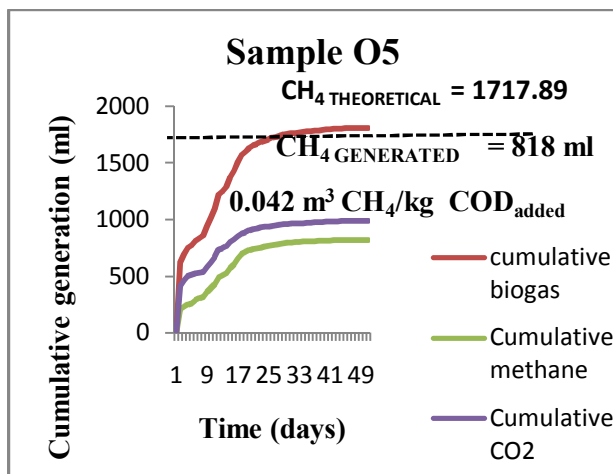
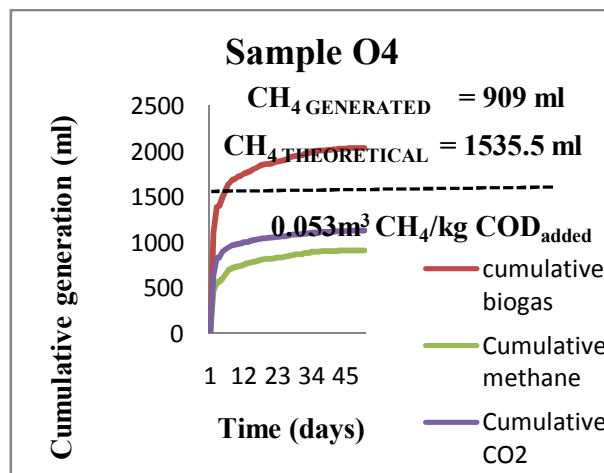


Figure 2: Cumulative Biogas and methane generation for Oxitop samples

## Characterization of the sample

The characterization of the Oxitop bottle samples was done before and after the experiment and is tabulated in below.

Table 3: Characterization of Oxitop bottle samples

Sample No.	Sample	pH	COD (mg/L)	Nitrate nitrogen (mg/L)	Ammonical nitrogen (mg/L)	Total Phosphorus (mg/L)	
1.	O1	Initial	7.21	16432	282	174	6.0
		Final	5.55	11324.28	105.5	215	3.0
2.	O2	Initial	7.07	22739.50	260	214	15.8
		Final	5.65	19193	152	230	15.5
3.	O3	Initial	7.00	18619	292.5	122.5	10.5
		Final	5.40	9866	205.5	220	5.5
4.	O4	Initial	6.99	17343	366.3	198.5	2.8
		Final	5.68	3028.43	290	355	1.5
5.	O5	Initial	7.10	19403	297	192	7.8
		Final	5.12	3864.04	205.5	270	5.0
6.	O6	Initial	6.6	22422.64	316.5	228.5	15.9
		Final	5.25	10943	292	455	12.0

Samples O1, O2, O3, O4, O5 and O6 represent Oxitop samples.

## Discussion

Six samples of municipal solid waste has been prepared according to the population of Indian cities as discussed above and was kept in incubator under controlled temperature range of  $30 \pm 2^{\circ}$  C. Anaerobic bacteria has been brought into the reactors in the form of seeding sludge brought from the UASB reactors of the Saharanpur sewage treatment plant, Uttarakhand in the ratio of 20% of the total volume. All the Oxitop bottles produced initial biogas for a period of 5-6 days after which no gas was produced. The whole samples seized within a week. The pH of all the samples has been checked and it was well below the optimum range (6-8) (Table 4).

Table 4: Oxitop samples pH after 6 days

Sample	O1	O2	O3	O4	O5	O6
pH	3.8	3.2	4.1	3.6	4.4	3.9

The whole experiment was repeated with a biodegradable fraction to sludge ratio of 50%. The samples shown good results for the initial 10 days. The biogas production seized as happened in the initial set-up. The pH of the samples has been checked and was in the range of 3 to 4. The seizing of the biogas generation for the other samples were probably that the anaerobic medium was not able to further increase the pH after the acidogenic phase.

A third set of samples has been installed with a waste to sludge ratio of 1:1. Sodium hydrogen carbonate ( $\text{NaHCO}_3$ ) was added to the Oxitop bottles as a buffer for maintaining optimum pH inside the reactors. The Oxitop bottles produced good results for sample 4 and sample 5. Sample 2 produced no biogas and were probably due to the



problem with the Oxitop bottle. Sample 1, 3 and 6 shown good results for two weeks after which it seized again. The samples 4 and 5 generated good amount of biogas and the reading was taken for 45 days. The graphs have been plotted above for the biogas generation of all the samples.

The experimental study of the Oxitop bottles revealed that pH of the media is a main parameter concerning the life of the anaerobic process. Also a minimum waste to sludge ratio of 1:1 or more is required for the efficient anaerobic digestion of MSW.

The characterization of all the samples has been done for all the samples before and after the experiment. The chemical characteristics such as pH, moisture content, TVS, VSS, Ash content, Chemical oxygen demand (COD), nitrate, ammonical nitrogen and total phosphorus has been found out experimentally by standard procedures and is tabulated and shown in table 4.3. From the table it is clear that that the COD of municipal solid waste samples are high varying 16000 - 23000 mg/L. The COD removal has been maximum for Oxitop sample 4 (82.54%), followed by Oxitop sample 5 (80.0%) (Table 5). This indicates that in these two samples, the biodegradation of organics has been maximum and hence the higher breakdown of COD.

*Table 5: Result for COD removal of samples*

Sample No.	O1	O2	O3	O4	O5	O6
COD removal (%)	31.08	15.59	47.01	82.54	80.0	51.2

The Oxitop sample O2 produced no gas and that may be due to the leakage of the Oxitop bottle. The samples other than O4 and O5 generated good initial gas and after 14 days, it production suddenly stopped. The Oxitop samples O4 and O5 shown good results and continued for around 45 days.

The characterization results of the samples clearly show the increase in the concentration of ammonical nitrogen in the effluent samples and decrease in case of nitrate and phosphorus concentrations (Table 3).

The Oxitop bottle samples 4 and 5 continued for around 50 days and shown good results. The biogas generated for these samples were calculated as 0.12 m<sup>3</sup>/kgCOD<sub>added</sub> and 0.093m<sup>3</sup>/kgCOD<sub>added</sub> respectively. The methane of these samples was found to be 0.053m<sup>3</sup>/kgCOD<sub>added</sub> and 0.042m<sup>3</sup>/kgCOD<sub>added</sub>. Sample 4 was able to generate 44.56% CH<sub>4</sub> of the total biogas produced and sample 5 generated 45.2% CH<sub>4</sub> of the total biogas generated.

## Conclusion

The initial trial experiments using Oxitop bottle samples with biodegradable fraction of MSW to sewage sludge ratios of 5:1, 2:1 and 1:1 inferred that a minimum ratio of 1:1 or more should be used as seed for efficient anaerobic digestion as the trials using the ratios of 5:1 and 2:1 failed to continue more than ten days.



The initial set of experiments stopped working within ten days. The pH of all these samples was found to be less than the optimum range and hence pH is a very important parameter that governs the anaerobic digestion.

The theoretical values for methane generation for Oxitop bottles were calculated as O1 = 1455 ml, O3 = 1648.48 ml, O4 = 1535.5 ml, O5 = 1717.89 ml and O6 = 1985.524 ml. At the end of the experiments, it could be inferred that 40-45% of the total biogas produced was methane gas.

Ammonia nitrogen accumulates in the reactor during the course of anaerobic digestion as observed from the results. This accumulation is caused from the protein fraction of the OFMSW. The OFMSW, which contains an average 4% of protein content (major source of nitrogen) is removed via ammonification process and is accumulated as ammonia-N [32].

Nitrate nitrogen (NO<sub>3</sub>-N) and phosphorus concentrations decreased in the characterization of the effluent samples of the reactors. This decrease in concentration is because, the nitrogen and phosphorus in the samples act as nutrients for the growth of anaerobic bacteria and are used up during the course of the process [32].

The study infers that the municipal solid wastes are rich in organic content and its potential for the generation of huge amount of biogas having methane in surplus quantity. This energy can be a viable option and can be replaced for other sources of energy such as petrol, coal and natural gas. Anaerobic digestion is a stable and is the prime option for waste treatment in many of the developed countries. Though there are some concerns regarding the failure of the process due to volatile fatty acids and ammonia nitrogen accumulation and lowering of the pH, this is one technology to look forward as these concerns would be overcome in the near future especially in developing countries including India.

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