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# Biogas production from co-digestion of orange peel waste and jatropha de-oiled cake in an anaerobic batch reactor

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Anaerobic co-digestion of jatropha deoiled cake and orange peel waste for biogas production was carried out in the batch scale (500 ml serum bottle) under anaerobic condition at ambient temperature (at various mixing ratios of two substrate). The experimental data showed a maximum gas output of 1140 ml of gas production at (1:2) ratio of jatropha deoiled cake with orange peel waste obtained for a period of 17 days. The modified Gompertz equation was used to adequately describe the cumulative biogas production for this reactor. The  $CH_4$  content was 75%,  $CO_2$  content was 16% and CO content was 9%. The biogas production was measured by liquid displacement system on daily basis. The digested slurry can be used as a fertilizer for agricultural purpose.

Key words: Co-digestion, orange peels waste, jatropha, biomethanation, Gompertz equation.

# INTRODUCTION

Energy is one of the most important factors to global prosperity. The dependence on fossil fuels as primary energy source has lead to global climate change, environmental degradation, and human health problems. By the year 2040, it is predicted that the world will have a population of 9 to 10 billion people that must be provided with energy and materials (Okkerse et al., 1999). Moreover, the recent rise in oil and natural gas prices may drive the current economy towards alternative energy source such as biogas. Anaerobic digestion is a process in which microorganisms break down biodegradable material in the absence of potassium. Anaerobic digestion can be used to treat various organic wastes and recover bio-energy in the form of biogas, which contains mainly CH<sub>4</sub> and CO<sub>2.</sub> Methane could be a source of renewable energy producing electricity in combined heat and power plants (Clemens et al., 2006). The process of anaerobic digestion has the potential of converting biodegradable organics into biogas which comprises methane (55 to 75%) and carbon dioxide (25 to 45%) with calorific value of 20 MJ/m<sup>3</sup> (Steffen et al., 2000; Myles et al., 1985). The organic loading rate (OLR) and

hydraulic retention time (HRT) are two major parameters used for sizing the digesters and their optimum values are specific to the substrate, as well as the operating temperature of digesters (Rowena et al., 2007). Orange juice is one of the most widely-consumed beverages today. Therefore, the cultivation of oranges has become a major industry and an important economic sector in the United States (Florida and California), Brazil, Mexico, Pakistan, China, India, Iran, and most Mediterranean countries. Anaerobic digestion, in which both pollution control and energy recovery can be achieved, is another possible way to treat and revalorize abundant orange peel waste (Martin et al., 2010).

India produces a host of non-edible oils, which are essentially under-utilized and can be used for oil extraction and Bio-diesel production. These non-edible oils can be obtained from Rice-bran, Sal, Neem, Mahua, KaranjaCastor, linseed, Jatropha, honge, rubber-seed etc. Most of these trees and crops grow well on wasteland and can tolerate long periods of drought and dry conditions. Among them, jatropha has several more desirable properties such as hardiness, wide environmental tolerance, can grow on any type of soil, adapt well to wasteland easy propagation and high oil content and require minimal care. It also has less gestation period, rapid growth and is not browsed by animals. This

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Table 1. Analysis of sewage sludge.

Parameter	Content
pН	6.8
Total solids (mg/L)	47800
Total Suspended solids (mg/L)	37500
Volatile Solids (mg/L)	15000
Carbon/ Nitrogen ratio	13:1

plant provides ample opportunities for wealth generation among marginal sections of the society, women empowerment and rural employment (Agarwal et al., 2007). One hectare of jatropha curcas plantation on an average will produce 3.75 metric tonnes of seed yielding 1.2 metric tones of oil. At the end of two years, jatropha curcas plant will give seed to its full potential. Hence four lakh hectares will produce 0.48 million metric tonnes of oil and 1.02 million metric tonnes of oil cakes (Braun, 2002). One of the major problems arising in the coming years is the disposal of cake after expelling oil from seed. The cake can neither be used for animal feeding nor can it directly be used in agricultural farming due to its toxic nature. Hence, the generation of biogas from these cakes would be a best solution for its efficient utilization. Biogas from cake provides energy for heating, cooking, lighting and engine operation and digested cake slurry can be directly put for agricultural farming.

Co-digestion is the simultaneous digestion of a homogenous mixture of two or more substrates. The most common situation is when a major amount of a main basic substrate (example manure or sewage sludge) is mixed and digested together with minor amounts of a single, or a variety of additional substrate (Mata-Alvarez et al., 2000). The use of co-substrates usually improves the biogas yields from anaerobic digester due to positive synergisms established in the digestion medium and the supply of missing nutrients by the co-substrates (Callaghan et al., 1999). Co-digestion was used by researchers such as (Gelegenis et al., 2007; Chellapandi., 2004; Desai et al., 1994) to improve biogas yield by controlling the carbon to nitrogen ratio. Co-digestion can provide a better nutrient balance and therefore better digester performance and higher biogas yields. In this research, the utilization of substrate in the form of jatropha deoiled cake was co-digested with orange peel waste in order to obtain optimum biogas production rate kinetics at ambient temperature. The objective of this study was to treat the organic waste and produce methane gas from the orange peel waste and jatropha deoiled cake.

# MATERIALS AND METHODS

#### **Collection of raw materials**

The raw materials employed in this study are sewage sludge,

jatropha deoiled cake and orange peel waste. The deoiled cake was collected from the oil mill and the orange peel waste was collected from the Anna University canteen.

#### Preparation of substrate for analysis

Both jatropha deoiled cake and orange peel waste were used for analysis. The physical and chemical properties such as moisture content, total solids, volatile solids were analyzed.

# Sludge collection and their activity

Bio-sludge was collected from a sewage treatment plant (STP) at Chennai. The sludge had methanogenic activity as would be discussed. The characterization of sludge was made, which is tabulated in Table 1.

### Total solids, volatile solids and non-volatile solid content

Sludge contains water and solids. The sludge concentration was expressed in terms of kg TS (Total solids)/m<sup>3</sup>. The total solids (TS) consist of organic material (volatile solids = VS) and ash. Sludge with high ash percentage consists of more inert material. While sludge with low ash percentage will contain less inert material, but will possibly have poor settling characteristics.

## **Total solids content**

The total solids content of feed materials were determined as per the standard method. The initial weight of the samples of 50 g biomass with pre-weighed porcelain boxes were taken by using an electronic balance with least count of 0.001 g. The samples were first heated at 60°C for 24 h and then at 103°C for 3 h using a hot air oven. The final weight or dried samples weight with pre-weighed porcelain boxes were recorded. The percentage total solids content of the sample was then calculated using the formula:

$$TS = \left(\frac{Wd}{Ww}\right) \times 100 \tag{1}$$

Where, TS is the total solids in percentage (%); Wd is the weight of oven dried sample and Ww is the weight of wet sample in gram (g).

#### Volatile solids and non-volatile solid content

The volatile solids and non-volatile solids content of feed materials were determined as per the standard method. The oven dried samples used for the determination of total solids content were further dried at  $550 \pm 50^{\circ}$ C temperature for 1 h in a muffle furnace and allowed to ignite completely. The dishes were then transferred to desiccators for final cooling. The weight of the cooled porcelain dishes with ash were taken by the electronic balance. The volatile solids content and non-volatile solids content of the sample were calculated using the formulas:

$$VS = \left(Wd - \frac{Wa}{Wd}\right) \times 100 \tag{2}$$

$$NVS = \left(\frac{Wa}{Wd}\right) \times 100 \tag{3}$$



Figure 1. Schematic diagram for methanogenic activity test and reactor setup.

Where, VS is the volatile solids in dry sample, %; NVS is the nonvolatile solids in dry sample, %; Wd is the weight (g) of oven dried sample; Wa is the weight (g) of dry ash left after igniting the sample in a muffle furnace.

#### Methanogenic activity test for sludge

In this test, activity was not determined directly as the substrate utilization rate; rather, the methane production rate was noted. The higher the methane production rate, the higher the activity. A sludge sample of 1.8 g VS was placed in a serum flask of 500 ml (120 ml of sludge + 280 ml of water). Water (preferably saturated with nitrogen) was added to a level of 3 cm from the top of the flask, and then 5 ml of the stock solution of acetic acid are added. The rubber stopper was placed and the flask was then connected to the liquid displacement system. The serum flask for the blank (containing only water, in the same volume of the liquid in the serum flask containing the sludge sample) was also connected to a liquid displacement system of the blank could be comparable to the volume of the liquid displacement system that was connected with the serum flask that contains the sample as shown in Figure 1.

The first reading of gas production was performed after one day (overnight incubation). This reading is the 'zero reading'. The volume of displaced NaOH is not only the result of gas production, but also of the realization of an equilibrium between liquid displacement system and ambient pressure. Therefore, the amount of liquid produced in the zero reading is not included in the calculation of the methanogenic activity. After the zero reading, reading should be executed three times a day, and before every reading, the sludge flask has to be mixed thoroughly. The liquid displaced by the blank should be measured for every reading.

Methane production sludge = displaced liquid by sample – displaced liquid by blank.

After every reading, the accumulated methane production was calculated .The second feeding was added when the total produced methane exceeds 200 ml as shown in Figure 2.

After the addition of the second feeding, the procedure was continued as before. The experiment was completed after the gas production (350 ml) as shown in Figure 3, where the velocity decreases and the major part of the second feeding was consumed. Approximately, 500 to 600 ml of methane was produced. After completing the readings, the exact sludge amount in the serum flask was determined by measuring the TS and VS content of sludge. A graph was prepared with X – axis representing the cumulative time and Y – axis the cumulative gas production.

## **Experimental design**

The experimental design for the anaerobic digestion of jatropha deoiled cake and orange peel waste was carried out at ambient temperature that ranged from 27°C to 32°C in four batch reactors Labeled 1 to 4:

Reactor 1 : 2 g jatropha deoiled cake + 4 g orange peel waste (1:2) (120 ml of sludge + 280 ml of saturated water).

Reactor 2 : 2 g jatropha deoiled cake + 2 g orange peel waste (1:1) (120 ml of sludge +280 ml of saturated water).

Reactor 3 : 2 g jatropha deoiledcake (120 ml of sludge + 280 ml of saturated water).

Reactor 4 : 2 g of orange peel waste (120 ml of sludge + 280 ml of saturated water).

The reactors were set up as described by (Yusuf et al., 2011) and biogas measurement was carried out by using the water displacement method. The NaOH solution in the liquid displacement system of the blank should be comparable to the volume of the liquid displacement system that is connected with the serum flask that contains the sample. Ambient temperature mea-



Figure 2. Feed-1 .Methane production during methanogenic activity (250 ml).



Figure 3. Feed - 2. Methane production during methanogenic activity (350 mL).

surement was determined with a mercury bulb thermometer.

The scope of this research was to study the cumulative biogas generation using the modified Gompertz equation in studying the kinetics of biogas production.

$$B_t = B * \exp\left[-\exp\left[\frac{R_b}{B} * \exp(\lambda - t) + 1\right]\right]$$
(4)

Where, B<sub>t</sub> is the cumulative biogas produced (mL) at any time (t); B is the biogas production potential (ml); R<sub>b</sub> is the maximum biogas production rate (ml/day);  $\lambda$  is the lag phase (days), which is the minimum time taken to produce biogas or time taken for bacteria to acclimatize to the environment in days. The constants B, Rb and  $\lambda$  were determined using the non-linear regression approach with the aid of the MATLAB 7.9 model software function. In many biological fields, the basic knowledge of phenomena is insufficient to build a mechanistic model. In this study, the effect of mixing ratios of jatropha deoiled cake and orange peel waste to methane production in the anaerobic digester was analyzed using a

Gompertz model (Momirlan. 1999) as shown in Equation 4 above. This equation was utilized by researchers to study the cumulative methane production in biogas production. Zwietering et al. (1990) and Lay et al. (1996) applied this equation to study bacteria growth. Recently, Budiyono et al. (2010) utilized this modified equation to describe biogas yield from cattle manure.

#### Analysis of jatropha de-oiled cake and orange peel waste

The substrate analyses were made according to the APHA of standard methods (APHA, 1989). The parameters like, total solids, volatile solids and also nutrient content of substrate like nitrogen, carbon, hydrogen, sulpur, oxygen were analyzed and the values are shown in Table 2.

#### Gas measurement

The biogas produced from the batch reactor (serum bottle (500) ml)

 Table 2. Nutrient content of raw material.



Figure 4. Gas chromatography analysis for jatropha deoiled cake with orange peel waste.

was measured by liquid displacement system on daily basis over a period of 17 days.

#### Carbon dioxide and methane content

In the liquid displacement system 1.5% NaOH was filled in the serum bottle during the production of gas which allows only methane, while other gases are absorbed by sodium hydroxide solution. For the analysis of biogas, the gas was collected in the rubber balloon and it was estimated by gas chromatography analysis.

## Gas chromatography analysis

Biogas was produced from the anaerobic digestion of mixed orange peel waste with jatropha deoiled cake. It was quantified in liquid displacement system. Volume of biogas was measured by volume of water displaced in graduated measuring jar. JEOL GCmate instrument was used for the analysis of biogas. JEOL GCmate instrument parameters were injection temperature 220°C, temperature range 40 to 100°C, flow rate of temperature 2°C/min and helium gas was used as a carrier gas. The column of JEOL GCmate HP5 (Hewlett Packard) was used. The biogas analysis was done at IIT, Madras. The gas composition was CH<sub>4</sub> -75%, CO<sub>2</sub>-16% and CO – 9% as shown in Figure 4.

# **RESULTS AND DISCUSSION**

In this experiment, agro industrial wastes are used for biogas production at room condition (such as ambient temperature without any physical treatment). The volatile solids content for jatropha deoiled cake and orange peel waste used in this research were determined to be 96.3, 94.2, 85.4 and 94.5%, respectively while the room temperature ranged between 27 to 32°C. Biogas production was monitored and measured until biogas production reduced significantly. The modified Gomperzt equation was used to fit the cumulative daily biogas production which was observed to adequately describe



**Figure 5.** Comparison of experimental data and modified Gompertz model for cumulative biogas production.

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Reactor	Weight of orange peel waste (g)	Weight of jatropha deoiled cake (g)	Total solid (%)	Volatile solid (%)	Biogas produced (ml)	B (ml)	R⊳∖ (ml/h)	λ (h)	R <sup>2</sup>
1	2	4	93	96.3	1140	1159	6.678	-19.35	0.9908
2	2	2	91	94.2	490	490.4	3.175	6.221	0.9970
3	-	2	92	85.4	244	238.5	1.433	-19.35	0.9701
4	2	-	87.3	94.5	447	464.6	3.349	7.96	0.9971

B is the biogas production potential (ml);  $R_b$  is the maximum biogas production rate (ml/day);  $\lambda$  is the lag phase (days), which is the minimum time taken to produce biogas or time taken for bacteria to acclimatize to the environment in days,  $R^2$  is the goodness of fit.

the biogas production from these substrates as shown in Figure 5. The study of biogas production from jatropha deoiled cake and orange peel waste and their mixtures were conducted in the reactor labeled 1 to 4 .The estimated kinetic constants using non-linear regression and other characteristics of the reactors 1 to 4 are shown in Table 3.

At the end of 17-days period, it was observed that reactor1 produced the highest cumulative biogas production potential (B) of 1159 ml at a maximum biogas production rate ( $R_b$ ) of 6.678 ml/h, with a lag phase ( $\lambda$ ) of -7.626 h. Reactor 3 had biogas production potential estimated to be 238.5 ml at a maximum biogas production rate of 1.433 ml/h, with a lag phase of -19.35 h. While in reactor 2, which comprised equal amount of jatropha deolied cake and orange peel waste, the biogas production potential was 490.4 ml at a maximum biogas production rate of 3.175 ml/h with a lag phase of 6.221 h.

Finally, reactor 4 also had biogas production potential estimated to be 464.6 ml at a maximum biogas production rate of 3.349 ml/h with a lag phase of 7.96 h. The modified Gompertz equation was observed to adequately describe biogas production with a goodness of fit ( $R^2$ ) of 0.9908, 0.9970, 0.9701 and 0.9971 for reactors 1, 2, 3 and 4, respectively.

# Conclusion

Biogas production from jatropha deoiled cake and orange peel waste was established here to be feasible at room temperature. The application of the modified Gomperzt equation in studying the biogas production was able to predict the pattern of biogas production with time. It was observed that the maximum biogas production could be obtained from the reactor 1 (2 g jatropha deoiled cake + 4 g orange peel waste (1:2)). In reactor 2, the biogas production was less amount than the reactor 1. Likewise reactor 3 and 4 produced less amount of biogas than the reactor 2. Hence, we conclude that the biogas production varied due to various substrate concentration of the reactors. The digested slurry can be applied for the agriculture purposes.

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