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# **Anaerobic Batch Digestion of Cattle Manure under Various Oscillatory Flow Mixing**

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#### **ABSTRACT**

The feasibility of an anaerobic digestion of cattle manure for biogas production is studied in this paper. A batch thermophilic oscillatory flow anaerobic bioreactor (OFBR) operated in thermophilic (55°C) condition was used. Within the experimental conditions set in this study, the effect of mixing intensity on volatile solids removal was found out to be significant. Results demonstrated that increasing the level of mixing decreased the digester performance. Low intensity mixing at oscillatory Reynolds number (Re<sub>o</sub>) of 100 achieved an increase of 37% in biogas yields compared to high mixing intensity, Re<sub>o</sub> of 500. It was observed that the mixing intensity effect interacts with the methane composition in the biogas. The benefit of decreasing mixing intensity emerges to significantly increase the methane composition in the biogas. These experiments established that high intensity mixing was not essential for good performance of oscillatory flow anaerobic bioreactor. In addition, the effect of mixing intensity might be reduced through the use of a slightly lower total solid concentration, hence, lowering the operational cost of the process. Although the study was lab scale a pilot-scale system where mixing retention times are longer would be useful.

Keywords: Anaerobic digestion, biogas, cattle manure, mixing intensity, oscillatory flow mixing

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#### **INTRODUCTION**

In recent times eco-friendly reliable waste disposal methods have gained popularity in the animal husbandry industry to protect the environment. An alternative animal manure treatment approach is anaerobic biological treatment bioreactors. Anaerobic digestion is a biological operation that breakdown organic matter such as animal waste in the absence

of free oxygen into biogas, and produces a digestate that can be used for soil conditioning (Nasir et al., 2012a). There are two major bioreactor designs common to farm-scale operations, which are the plug-flow, and mixed-flow bioreactors (Wu & Chen, 2008). The mixed-flow reactors are more advantageous than the plug-flow reactors, because of the homogeneous dispersion of the substrate induced by agitation. These, therefore, provides effective use of the total bioreactor volume, prevent stratification and temperature gradients, disperse metabolic end products and toxic materials in the influent, and sustain close contact between the bacteria, and their substrate (Wu & Chen, 2008). Adequate mixing provides a uniform environment for anaerobic bacteria, which is one of the major factors in achieving optimal digestion (Amani et al., 2010). Mixing is recognised as among the most common unit operations in process manufacturing industries and several categories of mixers and impellers have been formed for different operations (Nunhez et al., 2005). However, the major disadvantage of this type of bioreactors is the additional energy and mechanical complexity related with the pump or impeller.

The conventional types of mixing in digesters include slurry-recirculation, gas-recirculation, and mechanical agitation. However, mechanical agitation is leading the market for stirring fermenting substrates especially from agricultural origin (Wu & Chen, 2008). A study conducted by Karim, Hoffmann, Klasson & Al-Dahhan (2005) on the influence of mixing in the anaerobic degradation of animal waste showed that mechanical, hydraulic and pneumatic accounted for 29%, 22% and 15% higher biogas yields compared to the unmixed digesters. In addition, Stroot, McMahon, Mackie & Raskin (2001) reported that by treating animal manure, similar effects on biogas production rates and yields at steady-state conditions of four different mixing intensities (50, 350, 500 and 1500 per min) could be obtained in continuously stirred bioreactors. They observed a higher methane production by 1.3% and 12.5% with intermittent and minimal mixing strategies compared to continuous mixing of manure in the bioreactor. According to Kaparaju, Buendiaa, Ellegaardb, & Angelidaki (2007) mixing in manure fed anaerobic digesters is very important, and the mixing intensity was found to have a small effect on biogas yield. On the other hand, mixing showed to have an effect on the anaerobic digestion of manures.

Although, much research had been conducted in this field using different mixing, but it must be stated that oscillatory flow mixing (OFM) has never been reported in the anaerobic digestion of animal manure. OFM use a combination of flow oscillation and baffled tube geometry to ensure efficient mixing and effective heat transfer. Therefore, the objective of this study was to investigate the effect of different oscillatory flow mixing intensity on the anaerobic digestion of cattle manure for biogas production.

### MATERIALS AND METHODS

#### **Substrate**

The cattle manure used in this study was collected from a dairy farm in Taman Pertanian Universiti (TPU) in the Universiti Putra Malaysia campus in Serdang, Selangor, Malaysia. The cattle manure was mixed (1:1) with water then filtered through a screen (0.5cm x 0.5cm) and stored at 4°C until use. The characteristics of the initial substrate are shown in Table 1.

Table 1
Chemical Composition of Cattle manure

Parameter	Unit	Cattle manure	
pН	-	7.65	
TS	%	11.7	
VS	%TS	83.7	
NH <sub>3</sub> -N	mg/L	2733	
COD	mg/L	640.3	
DM	%dry base	16.7	
CF	g/kg DM	34	
CP	g/kg DM	11.8	
NDF	g/kg DM	83.36	
ADF	g/kg DM	71.77	
Lignin	g/kg DM	30.61	
Cellulose	g/kg DM	41.16	
Hemicellulose	g/kg DM	11.59	
Acetic acid	mg/L	399.4	
Propionic	mg/L	277.5	
Butyric	mg/L	120.4	
Isovaleric	mg/L	83	

#### **Experimental Setup**

Experiments were performed in a stainless steel jacketed oscillatory flow bioreactor (OFBR) mounted on an oscillator base unit, with a working volume of 4.5 litres (L). The internal diameter and length of the tube were 98 mm and 732 mm, respectively, and it is fitted with a baffle insert assembly using outside diameter of 80 mm baffles with 28 mm diameter orifices spaced at 1.5 tube diameter. A variable-frequency, variable-amplitude oscillator was used, in which a stainless steel pistons provided fluid oscillation at the base of the reactor tube (Figure 1). Operating temperature was maintained at 55±1 °C, controlled and monitored circulating heated water through the OFBR jacket. While, pH was set in the range between 6.8 and 7.1, by a pH controller that continuously monitors the pH and adds acid or base, as required.

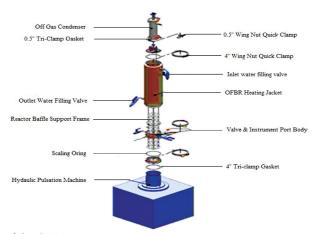


Figure 1. Schematic of the OFBR system

In the experimental period; three different mixing intensities were tested (Harvey, Mackley, & Stonestreet, 2001) as batch at different times. Three batches were processed at different oscillatory Reynolds number ( $Re_o$ ) of 100, 300 and 500, which is equivalent to frequencies of 1Hz, 2Hz and 5Hz, respectively. Further, a control batch experiment without mixing was performed to provide a baseline for comparison. Within each batch, the effect of mixing on the bioreactor performance was evaluated. Composition and volume of the biogas produced from the bioreactor was measured daily until the biogas production seizes. Biogas was collected in tedlar bags (Supelco, Bellefonte, PA) and the volume produced was measured by water displacement.

#### **Analytical Methods**

Total solids (TS), volatile solids (VS), chemical oxygen demand (COD), and ammonia nitrogen (NH<sub>3</sub>-N) were analysed according to the standard method (APHA, 1998). The fibre compositions such as neutral detergent fibre (NDF), acid detergent fibre (ADF), crude fibre (CF), crude protein (CP) and lignin were analysed using the reflux apparatus. Methane content was analysed using a gas chromatography (HP 6890N) equipped with a thermal conductivity detector (TCD) and a HP Molesieve column 30 m x 0.5 mm ID. The injector, detector, and oven temperatures were 60°C, 200°C and 70°C, respectively. Argon served as the carrier gas. The biogas yield was expressed as the volume of biogas produced based on the initial total VS in the feedstock. Samples for volatile fatty acid (VFA) determination was collected every other day and centrifuged at 11,000 rpm for 20 min. The supernatant was used in the analysis. A gas chromatography (Agilent 7890N), using a Supelco SP 2560 capillary column of  $100m \times 0.25$  mm ID  $\times 0.2$ - $\mu$ m film thickness (Supelco, Bellefonte, PA, USA) was used for the analyses. It was equipped with a split/splitless injector, flame ionization detector (FID) and an auto sampler (Agilent Auto Analyzer 7683 B series, Agilent Technologies, Santa Clara, CA, USA). The temperature of the injector was 250 °C, and the detector temperature was 270 °C. The carrier gas was nitrogen at a flow rate of 1.2 mL/min. The peaks of samples were identified, and concentrations calculated based on the retention time and peak area of known standards (Sigma Chemical). The fatty acid concentrations are expressed as gram per 100 gram of the sum of identified peaks measured in each sample.

#### RESULTS AND DISCUSSION

## Biogas and methane production

Biogas was generated from day 1 and varied significantly during the different batch studies. The daily biogas production increased steadily during the oscillatory Reynolds number (Re<sub>o</sub>) of 100 experiment until day 10, after which the production dropped gradually until the digestion process was stopped at day 21 (Figure 2). Similarly, the biogas production during the Re<sub>o</sub> of 300 and 500 experiments appeared to increase until day 6, and then decreased continuously until day 20 when the biogas production was observed to seize. However, the highest amount of average daily biogas was observed during the experiment using Re<sub>o</sub> of 100, as compared

to  $Re_o$  of 300 (1.46 L/L/day) and  $Re_o$  of 500 (1.28 L/L/day). The methane content of biogas exceeded 50 % in all the experiments except for the control. The average biogas production of  $Re_o$  of 100 and  $Re_o$  of 300 operations amounted to 0.28 and 0.17 L/g VS added with an average methane content of 52 % and 44 %, respectively, which were higher than the values of  $Re_o$  of 500 and control operations (Figure 3).

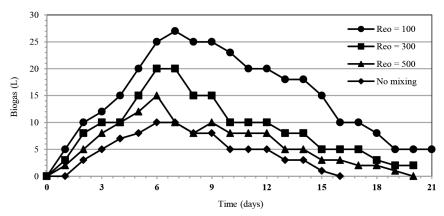


Figure 2. Biogas production in different mixing conditions

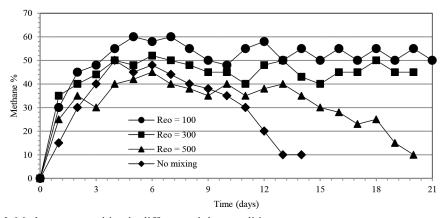


Figure 3. Methane composition in different mixing conditions

Additionally, over the 20 days of operation, the highest methane yield was obtained for  $Re_o$  of 100, followed by  $Re_o$  of 300 and 500 experiments with values of 0.144, 0.074 and 0.038 L  $CH_4/g$  VS added respectively, which was higher than the control of 0.034L  $CH_4/g$  VS added.

The significance of mixing in accomplishing effective substrate conversion has been reported in the literatures (Karim, Hoffmann, Klasson & Al-Dahhan, 2005; Kaparaju, Buendiaa, Ellegaardb & Angelidaki, 2007). Though, the readily obtainable facts available in the literature on the influence of mixing intensity on the performance of anaerobic bioreactors is contradictory. Adequate mixing was shown to enhance the uniform distribution of substrates, enzymes and microorganism all through the digester; on the other hand, inadequate mixing

leads to the non-uniform distribution of substrates, enzymes and microorganisms which was to result in stratification and formation of floating layer of solids (Amani, Nosrati & Sreekrishnan, 2010; Jha, Li, Nies & Zhang, 2013). In this study, the results obtained suggest that high intensity mixing may prevent excellent performance of the OFBR. Whereas, low intensity mixing exhibited higher methane yield and better performance, which is attributed to sufficient substrate distribution and producing direct contact with the microbes in the bioreactor. Results show that the biogas and methane yield decreased with increasing mixing intensity. This coincides with the results achieved by Kaparaju, Buendiaa, Ellegaardb & Angelidaki (2007) who found that the vigorous mixing would result in delaying and lowering the methane production.

#### Effects of performance parameters on OFBR stability

**pH.** As can be seen from figure 4, the pH was stable and remained in the neutral range (between 6.8 and 7.5) until the end of the experiment for Re<sub>o</sub> of 100 and 300, but the pH for Re<sub>o</sub> of 500 and the control decreased continuously at the beginning of the experiment from 6.8 to the range of 4.7–5.3 after 4 days (Figure 4). The observed pH was reported as unsuitable for anaerobic digestion as the suitable pH for methanogenic bacteria ranges from 6.5 to 7.5 (Nasir et al., 2012b). In addition, the ammonia nitrogen values in different experiments were all below 1000 mg/L indicating that it was in the safe range for anaerobic digestion as reported by Calli, Mertoglu, Inanc & Yenigun (2005).

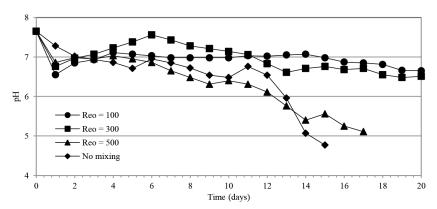


Figure 4. pH profile during different mixing conditions

**Volatile solids (VS).** Figure 5 shows the VS concentration profile with different Re<sub>o</sub> and no mixing. The VS concentration reached stability after day 18 during the experiments for Re<sub>o</sub> of 300 and 500, which indicates the microbial transformational activity. However, for Re<sub>o</sub> of 100 there is still tendency for further degradation of VS. Further, it was observed that the VS degradation during the experiments except the control was not over after 20 days of operation. As evidence in Figure 2, the best values of biogas production were for all experiments except the control. This is because the VS degradation took place during days 0 to 5, after which the

degradation dropped drastically. Finally, the VS removal was the highest for  $Re_o$  of 100, with the value of 57 % after 20 days of operation, which was almost 3 times higher than that of the control, followed by  $Re_o$  of 300 and 500 with 42 % and 30 %, respectively.

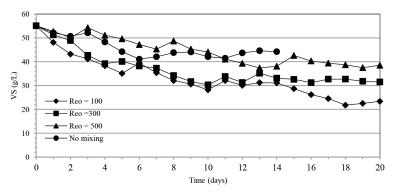


Figure 5. VS concentration during different mixing condition

**Volatile fatty acid (VFA).** The concentration of the total VFA (TVFAs) in a bioreactor is usually obtained by their rate of production and removal. During this experiment, the rate of acetic acid removal was higher to its production rate because almost 70 % of the methane generated is originated from acetate, while the remainder of the methane is originated essentially from the reduction of carbon dioxide with hydrogen (Chynoweth et al., 2001). During the experiments, it was observed that the concentration of propionic acid increased slowly for Re<sub>o</sub> of 100, 300 and 500 with almost no accumulation of butyric and valeric acids. However, the butyric and valeric acids were observed to accumulate in the control experiment, this is evident as pH was observed to drop fast as such stop the digestion after day 14. Table 2 summarises the average concentration of VFAs data during the study.

Table 2
Reactor Performance Data on the volatile fatty acids concentration

	Concentration (mg/L)					
	Acetic acid	Propionic acid	<b>Butyric acid</b>	Valeric acid	TVFA	
$Re_o = 100$	620.25	440.87	181	64.62	1306.7	
$Re_o = 300$	645.2	467.5	194.1	73.1	1380	
$Re_o = 500$	682.9	548.5	213.5	84.3	1529.2	
No mixing	673.1	586.4	319	116.6	1695.1	

#### **CONCLUSION**

The experiments with different mixing strategy at different oscillatory Reynolds number (Re<sub>o</sub>) of 100 and 300 during the anaerobic digestion of cattle manure for biogas production had been performed successfully, except for Re<sub>o</sub> of 500 and no mixing. Results demonstrated that Re<sub>o</sub>

of 100 and 300 resulted in excellent performance after 20 days of the experiment. High biogas production rates of 1.46 L/L/day and 1.28 L/L/day, with methane yield of 0.144 L/g VS added and 0.074 L/g VS added were observed for these operational conditions, respectively. This shows that minimal mixing could enhance the digestion process by improving the concentration of VFAs in a safe range. On the other hand, Re<sub>o</sub> of 100 and control experiments were found to be inhibitory during the experiment, possibly due to the disruption of syntrophic relationships between the microorganisms. This is evident as VFAs mainly of valeric and butyric acids were observed to accumulate, which degraded slowly and finally seized the digestion. In all the experiments, an optimum performance for the methane yield was achieved by adapting the mixing condition at Re<sub>o</sub> of 100.

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