

# Effect of false flax oilcake in thermophilic biogas production

HANS MARTEN PAULSEN<sup>1</sup>, WEI XIAOMING<sup>2</sup>, SUN YONGMING<sup>3</sup>, GEROLD RAHMANN<sup>1</sup> UND HERWART BÖHM<sup>1</sup>

<sup>1</sup> Johann Heinrich von Thünen-Institute (vTI), Institute of Organic Farming, Germany

<sup>2</sup> China Agriculture University, College of Water Conservancy and Civil Engineering, Beijing, China

<sup>3</sup> China Agriculture University, College of Resources and Environmental Science, Beijing, China

## 1 Conclusions

False flax oilcake has been found to be suitable for anaerobic fermentation in mixtures with cattle slurry and straw. In organic farms, digestion of cattle dung and wheat straw with 8 % dry matter content mixed with 5 % of total material weight false flax oilcake is a feasible option for utilizing false flax oilcake to produce farm-own renewable energy and offering farm-own high nitrogen (ammonia) content fertilizer (2.48 g kg<sup>-1</sup> wet wt).

In field digesters, the biogas yield of 8 % dry matter pure material under thermophilic condition was 0.24 l g<sup>-1</sup> VS fed. The biogas yield could be increased by mixing 5 % false flax oilcake to get 0.37 l g<sup>-1</sup> VS fed and a VS conversion efficiency with 0.83 l g<sup>-1</sup> VS destroyed. Under laboratory controlled conditions, the biogas yield of slurry with 0.5 % oilcake was a little higher than biogas yield of the digestion of pure material, which was 0.26 and 0.24 l g<sup>-1</sup> VS fed, respectively. Compared with the field experiment, only small amounts of biogas were produced in the lab-scale when 5 % oilcake was mixed in. The mixing can improve the biogas yield and substrate reduction in the digesters which have sufficient material.

Further research is needed to find out the best controlled conditions (high-efficient bacteria, mixing frequency and time) and best equipment (for example: two phases digesters).

## 2 Abstract

Anaerobic fermentations are especially suitable for use of organic wastes from agriculture. Agricultural wastes are needed by organic farms to be recycled in production and for use as farm own energy and fertilizer. Biogas digestion can be considered a good way to meet these needs.

Oilcakes are interesting substrates for biogas production due to their carbon, nitrogen, and high dry matter content. Up until now the use of false flax (*Camelina sativa* (L.) Crantz) oilcakes in animal production has been limited by the EU fodder regulations. But false flax is seen as a crop suitable to produce oil for human consumption and as a renewable energy source from mixed cropping systems.

Field and laboratory experiments were conducted to determine the effect of the use of false flax oilcakes in thermophilic biogas production. In the field experiments biogas yield increased from 0.24 to 0.37 L g<sup>-1</sup> volatile solid (VS) when 5 % false flax oilcake were added to the original substrate of cattle dung and wheat straw (8 % dry matter, DM). The VS conversion efficiency in the digester enriched with 5 % oilcake (0.83 L g<sup>-1</sup> VS destroyed) was higher than in the digester with the original material (0.63 L g<sup>-1</sup> VS destroyed). The addition of 5 % oilcake enriched the ammonia-nitrogen content in slurry (2.48 g kg<sup>-1</sup> wet wt).

Biogas production rates were measured in the laboratory at three different levels using a water displacement method. Biogas yields from A) cattle dung and straw (8 % DM) B) addition of 0.5 % oilcake and C) addition of 5 % oilcake were 0.24, 0.26 and 0.07 L g<sup>-1</sup> substrate, respectively. The insufficient biogas yield in variant C) with addition of 5 % oilcake in the laboratory test can be explained by insufficient mixing of the substrate during the process, a decreased pH value and its hindrance to the methanogenesis. Field tests with continuous stirring allow to conclude that 5 % oilcake addition can be recommended for raising biogas production and to obtain higher ammonia-nitrogen content in the slurry which might be advantageous for the use as fertilizer for organic farming.

### 3 Introduction

With the shortage of fossil energy, governments have launched policies to develop renewable energies. In the EU, targets were made to increase the share of renewable energy to 12 % by 2010. Especially, in Germany, the German Renewable Energy Law was passed in 2000 in order to establish a framework for doubling the share of renewable energy by 2010.

False flax (*Camelina sativa* (L.) Crantz) is a low-input energy crop whose seeds contain 37 % oil by weight on average, meaning it can be used as a renewable fuel or for human consumption (Harndorf et al. 2008, Bernardo et al. 2003, Zubr 1992). In organic farming, false flax cultivation is established in mixed cropping systems (Paulsen, 2007). As feedstuff for monogastric animals in meat production, the false flax oilcake can affect the sensory quality of meat and may cause metabolic disorders evident in enlarged organs (Böhme and Flachowsky 2005, Böhme et al. 1997, Jaskiewicz and Matyka 2003, Paulsen et al. 2005, Weißmann et al. 2006). So the use of the false flax oilcake of in animal production has up to now been limited by the EU fodder regulations and will have dosage limitations even in future (Richtlinie 2008/76/EG). So alternative uses also have to be considered, like the direct use of oilcakes as a nitrogen source in plant production (Laber 2003).

No research has been conducted on false flax oilcake in biogas fermentation. Besides energy production, biogas fermentation with farm-own manure, straw and oilcakes is especially valuable for organic farms to create a mobile nitrogen fertilizer pool. This can be applied with normal slurry technique (Paulsen and Rahmann, 2004). Therefore, the purpose of this study was to investigate the effect of mixing false flax oilcake in biogas substrates and to determine the effects on thermophilic biogas production.

### 4 Materials and Methods

The research consisted of a laboratory and a field experiment. Cattle manure, wheat straw and false flax oilcake used in the experiments was collected from the experimental dairy farm of the vTI Institute of Organic Farming (Schaub et al. 2007). The wheat straw was chopped into 2-3 cm pieces to enhance digestion (Zhang and Zhang, 1999). Inoculum for the anaerobic digestion was obtained from a preceding experiment. Because the manure for the two phases was collected at different times, it had slightly different physical properties.

#### 4.1 Laboratory experiment

The manure used had an initial dry matter (DM) content of 11.51 %. The initial DM contents of straw and oilcake were 86.3 % and 88.9 % respectively. Other characteristics of the substrates are shown in Table 1.

**Table 1: Analysis of the raw materials**

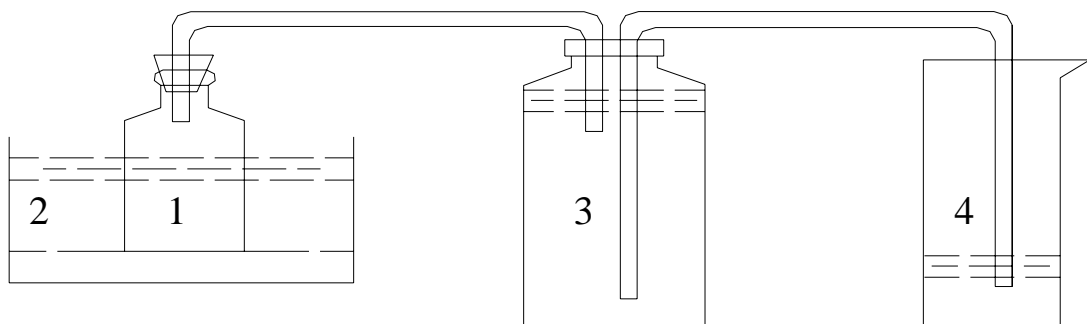
Parameters	Cattle dung for lab-scale	Wheat straw	false flax oilcake	Cattle dung for field-scale
Total Solid (%)	11.5	86.3	88.9	12.6
Volatile solid (%)	85.4	94.9	93.9	85.8
pH	6.7	na	na	7.3
C:N	13.8	80.0	7.8	13.8
Crude protein (%)	15.2	na	32.9	15.5
Crude fat (%)	2.7	na	na	2.8
Crude fibre (%)	36.2	na	na	35.8
Ammonia nitrogen (%)	0.097	na	na	0.096

Na = not analyzed, the contents of VS, crude protein, crude fat and crude fibre are given in % of DM, ammonia nitrogen is given in % of wet substance

The laboratory experiment to estimate the effects of oilcake on biogas development was conducted with three variants in two replications:

- Digester A: cattle dung and wheat straw (8 % DM)
- Digester B: cattle dung and wheat straw (8 % DM) mixed with 0.5 % of total material weight false flax oilcake (1.91 g = 0.5 % * 382.5 g)
- Digester C: cattle dung and wheat straw (8 % DM) mixed with 5 % of total material weight false flax oilcake (19.13 g = 5 % * 382.5 g)
Mixture for 8% DM: 174.99 g manure, 12.13 g wheat straw, 195.38 g water
Inoculation: 217.5 g old slurry.

The mixtures were put in 1 L glass flasks equipped with rubber stoppers and plastic tubing and maintained at 55°C throughout the experiment. Biogas was collected by water displacement in 2 l covered containers (Figure 1). Containers were replaced when they were filled with biogas. The daily biogas production was recorded by measuring the volume of displaced water. The digesters were stirred two times per day for half a minute. The samples were taken for analysis at the beginning and end of the fermentation,.



**Figure 1: Sketch of lab-scale digester and biogas collection apparatus used in the laboratory experiment (1—1 l Glass flask (Digester), 2—Thermostat water bath, 3—2 l Glass container (collecting biogas), 4—Measuring cylinder)**

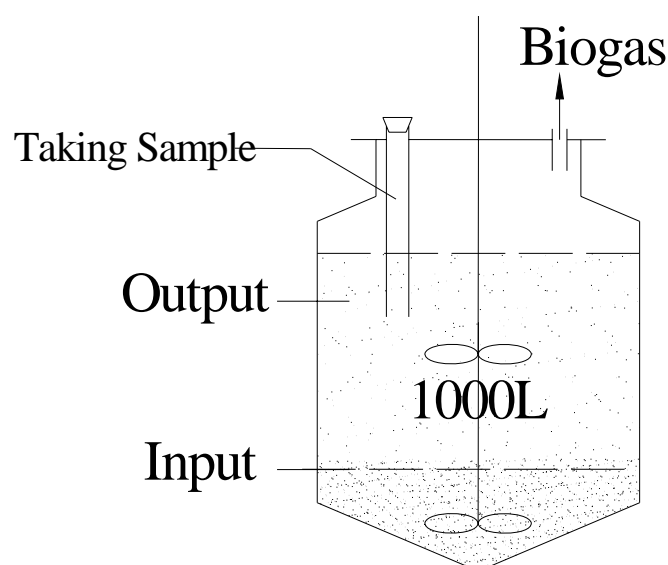
Additionally the biogas development and the methane content of two samples of the false fax oilcake, and two samples of oilcake of linseed (*Linum usitatissimum* L.) were determined from batch trials at 36 °C in the laboratory of BiogasBeratungBornim GmbH. As substrate digested cattle slurry with 2.34 % DM and 49.6 % VS (DM) was used. 1500 g slurry were mixed with 1.6 to 1.8 % of total material weight oilcake on the basis of a VS relation near to 1. 20-23 g VS oilcake and 22.39 VS digested slurry were used. In contrary to that, the VS ratio of the mixture of manure with 5 % oilcake used the described experiments in Trenthorst was much wider. Additionally non-digested material was used as a substrate like it will be the case in normal biogas production units in farms. Due to the different trial conditions analogue results from both experiments couldn't be expected.

#### 4.2 Field experiment

Two total mixing digesters were used manufactured by AFAG Company in 1986. Volume of each digester was 1000 l (Figure 2). Both digesters were operated under same conditions. In the field experiment the effects of oilcake on biogas development were tested in two variants:

- Digester I: cattle dung and wheat straw (8 % DM)
- Digester II: cattle dung and wheat straw (8 % DM) mixed with 5 % of total material weight false flax oilcake (25.5 kg = 5 % * 510 kg)
Mixture for 8 % DM: 220 kg manure, 15.24 kg wheat straw, 274.76 kg water
Inoculation: 290 kg old slurry

A PVC pipe was placed at the top of each digester and connected with a gas meter to measure biogas yield. A thermometer monitored the inside temperature in each digester. A water recycling system was used for heating. The mixing stir was controlled automatically. The temperature of both digesters was set at 55°C. Every 3 hours, the stirrers ran automatically for 10 min. The daily biogas yield was recorded by the gas meters. Substrate samples were taken for analysis every week (first week 5 days).



**Figure 2: Sketch of a biogas-digester used in the field experiment**

### 4.3 Analysis Methods

Total solid (TS), volatile solid (VS), pH, crude protein, crude fat, crude fibre and ammonia nitrogen of every sample were measured. DM was determined by oven drying at 105°C, until weight constancy, VS was measured as loss of weight on ignition in a muffle furnace at 550±50°C, until weight constancy. PH was measured by pH meter (APHA 1995). Crude protein, crude fat, crude fibre and ammonia nitrogen were tested by routine methods (VDLUFA 1976).

## 5 Results and Discussion

### 5.1 Field experiment

Both of the digesters were operated for 61 days. During the fermentation process, a total of 9,804.62 l and 23,359.87 l biogas were produced by the pure material fermentation (Digester I) and by the substrate mixed with 5 % oilcake (Digester II) respectively. The fresh weight reduction reached 3.57 % for Digester I and 8.27 % for Digester II.

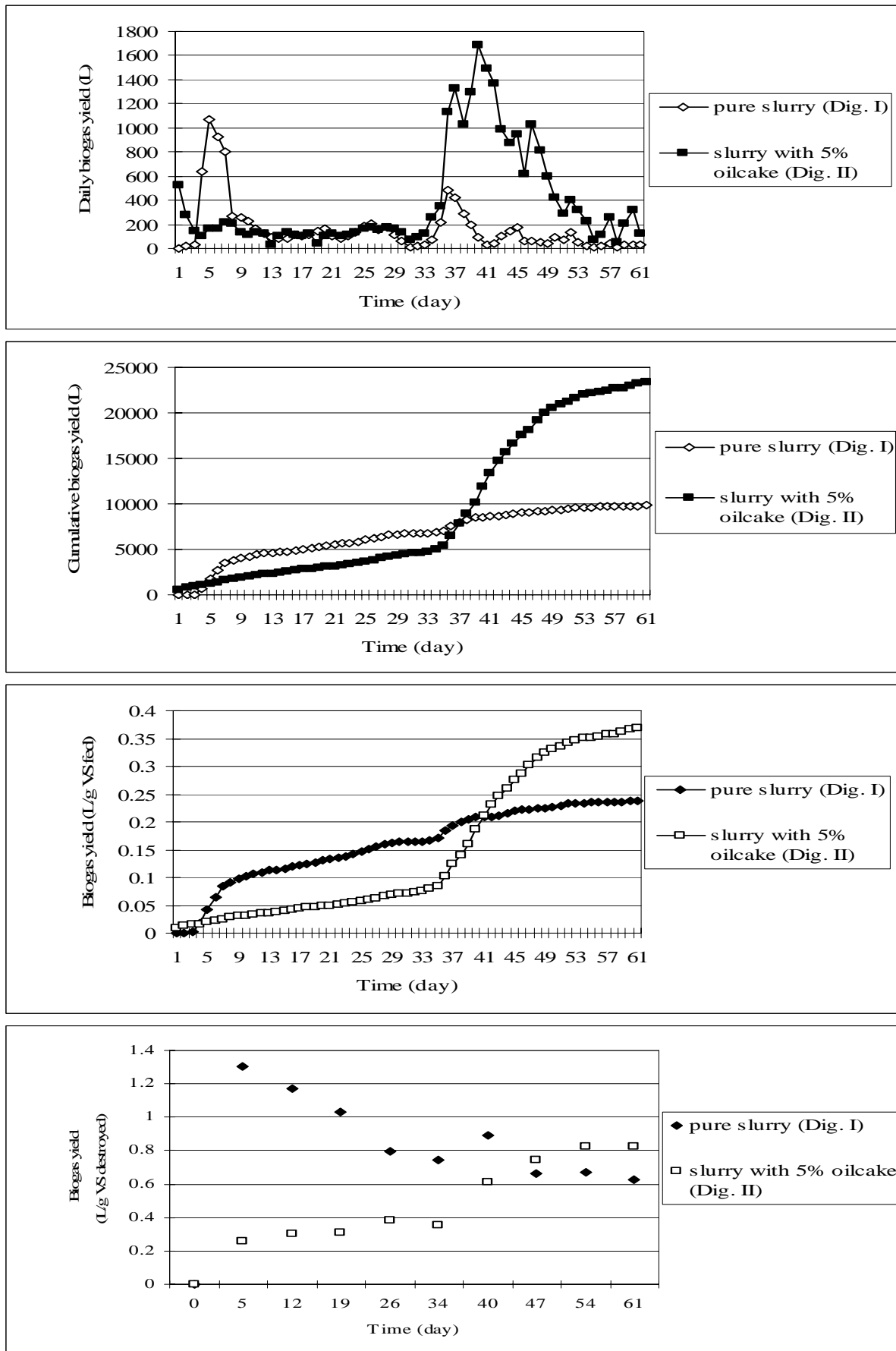
#### 5.1.1 Biogas production

Biogas production from each reactor was recorded daily. The daily and cumulative productions are shown in Figure 3. Digester I reached the gas production fastigium within 5 days after start-up, but the fastigium only maintained 1-2 days (daily yield more than 1000 l). Digester II started up sooner. 523.7 l biogas were produced on the first day. With the acid swiftly accumulating, the biogas production was restrained until the fastigium appeared at the 36th day. Compared with Digester I, the fastigium of Digester II lasted many more days, about 9-8 days, and Digester II had a higher cumulative biogas production (28.30 l kg<sup>-1</sup> wet wt fed, 0.37 l g<sup>-1</sup> VS fed) than Digester I (12.26 l kg<sup>-1</sup> wet wt fed, 0.24 l g<sup>-1</sup> VS fed). Biogas production was significantly affected by mixing in oilcake. Biogas production values of both digesters were higher than those observed by Kaiser et al. (2003) (0.19-0.22 l g<sup>-1</sup> VS fed) and Sadaka and Engler (2000) (0.2 l g<sup>-1</sup> VS fed).

From the biogas production data shown in Figure 3, it can be seen that 68.7 % biogas of Digester I was produced in the first 30 days. And the digestion process slowed after 2 weeks. Nearly 50 % of the biogas was produced in the first 2 weeks. In contrast, more than 80 % biogas of Digester II was produced in the second 30 days. Obviously the biogas production fastigium was delayed by mixing in the oilcake.

The VS conversion efficiency was also enhanced by mixing 5 % oilcake. Compared with Digester I (0.63 l g<sup>-1</sup> VS destroyed), the VS conversion efficiency of Digester II increased to 0.83 l g<sup>-1</sup> VS destroyed.

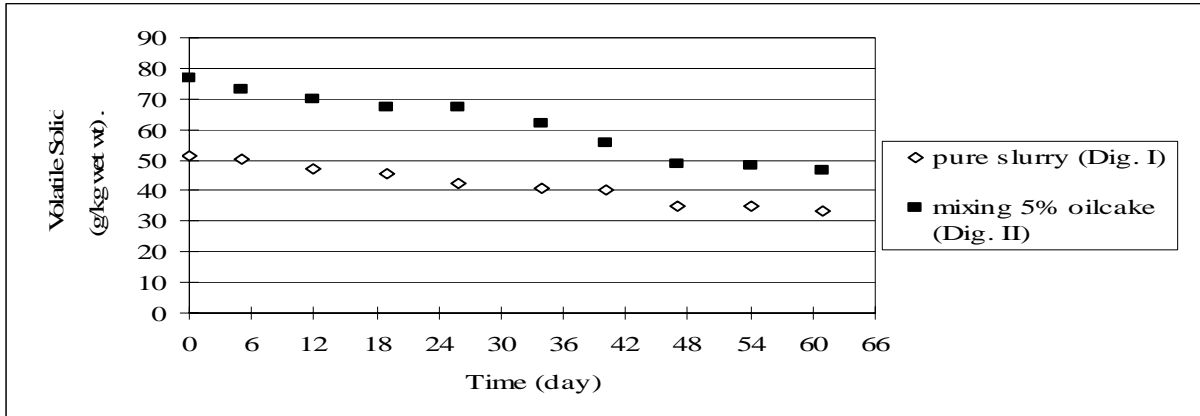
During days 31-37, there was a biogas yield fluctuation in both digesters. The reason was water loss from the heating system, which resulted in a temperature decrease down to 41.4 centigrade. As reported by several authors (Zhang and Zhang 1999, Bouallagui et al. 2003) the methane percentage of biogas ranges from 50-65 %.



**Figure 3: Daily and cumulative biogas production**

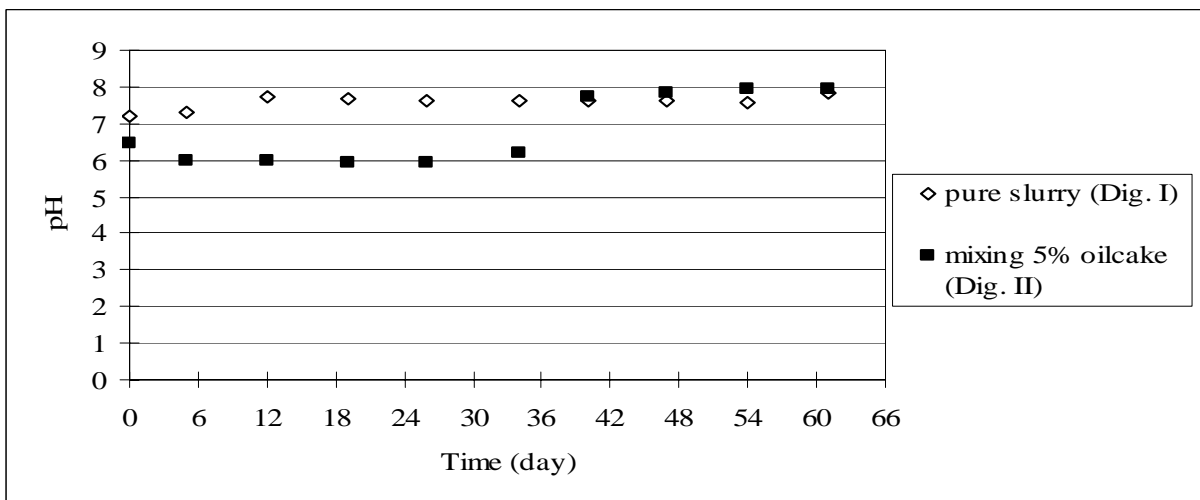
### 5.1.2 Volatile solid degradation and pH shift

Volatile solid degradations of both digesters are given in Figure 4. After 61 days, volatile solid reduction reached 33.78 % and 44.56 % for Digester I and Digester II, respectively. Reduction of VS was higher in Digester II, indicating that mixing 5 % oilcake was beneficial to the fermentation. In comparison, Kalia and Singh (2001) reported that the VS reductions of pure cattle manure and cattle dung mixed with 10 % digested slurry were 23.93 % and 36.10 %, respectively.



**Figure 4: Variation of volatile solid in both digesters**

The initial pH of Digester I was 7.2 and for Digester II 6.45. As fermentation progressed the pH of Digester I increased slowly to 7.83. During the initial phase of Digester II, the pH decreased rapidly and the conversion of substrate to biogas was inhibited due to the inhibition of methanogenic bacteria by increased volatile fatty acids (VFA). After the methanogenic bacteria adapting the environment, the fastigium of biogas production appeared and pH increased to 7.93.

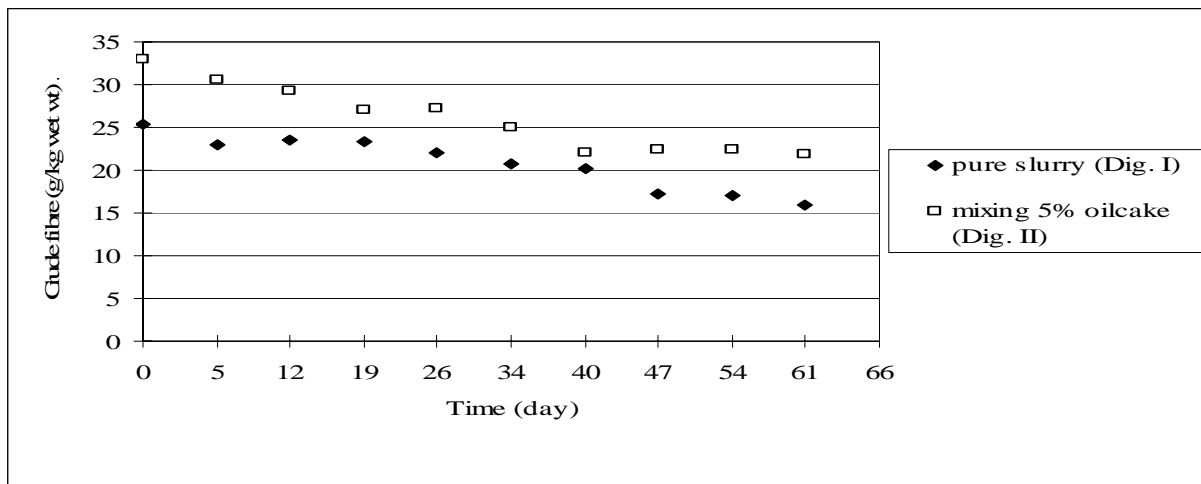


**Figure 5: Variation of pH in both digesters**

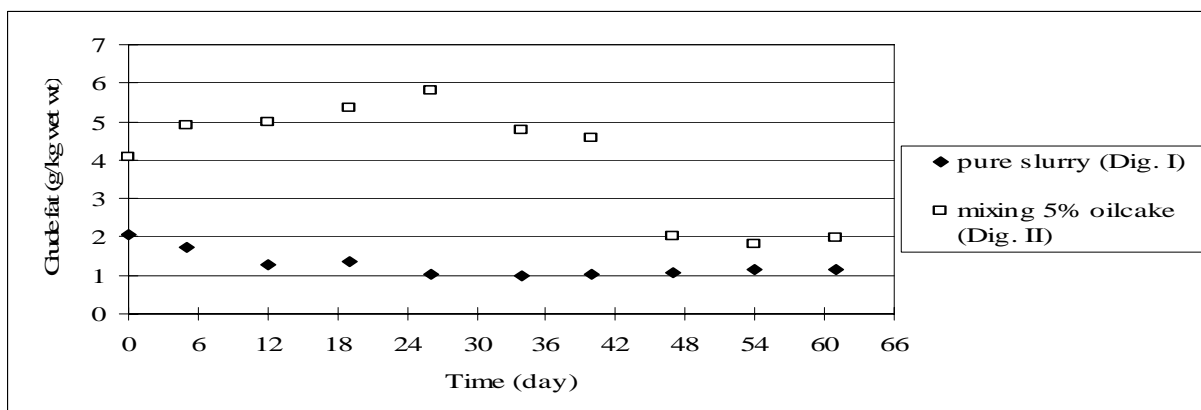
### 5.1.3 Changes of crude fibre and crude fat

During fermentation, the crude fibre contents of both digesters decreased. As shown in Figure 6, crude fibre contents decreased by 38.94 % and 39.29 % for Digester I and Digester II, respectively, owing to a combination of microbial conversion. The reduction in crude fibre content of Digester II was slightly higher than in Digester I.

With methanization, the crude fat content of Digester I was slowly decreased by 45.5 %. In contrast, the crude fat content of Digester II increased in the first 30 days, according to increasing acid content and inhibition of methane production. When the digestion process continued, the crude fat content decreased rapidly. In total the reduction of the crude fat content in Digester II was 55.7 %.



**Figure 6: Variation of crude fibre in both digesters**



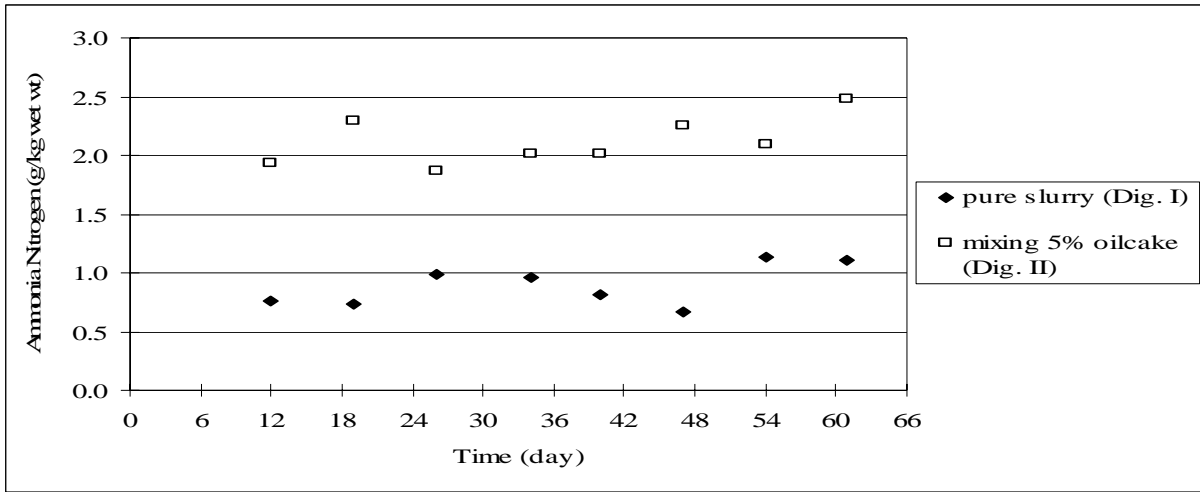
**Figure 7: Variation of crude fat in both digesters**

### 5.1.4 Ammonia nitrogen and crude protein

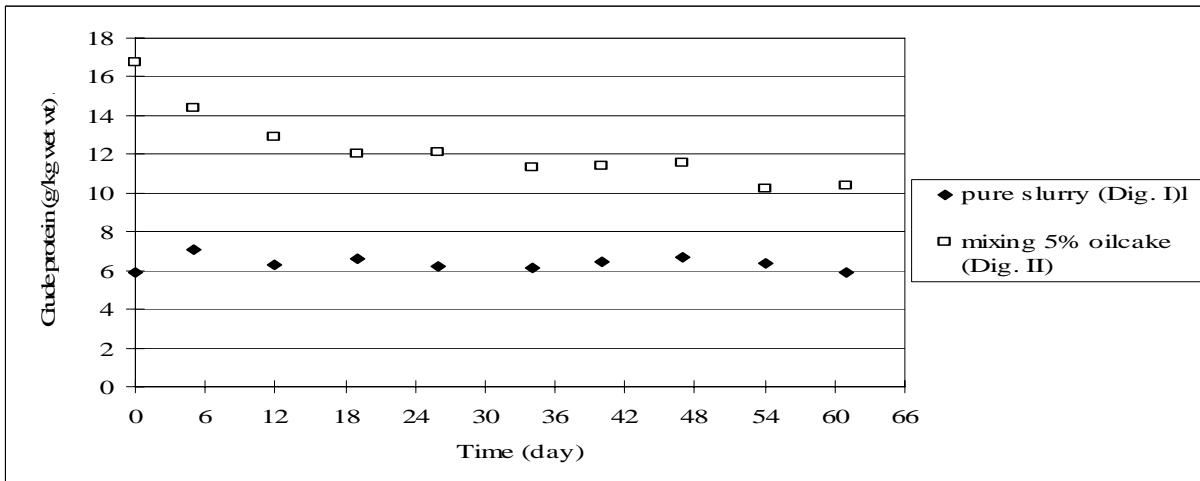
The ammonia nitrogen of both digesters was not analyzed in the first week. At  $\text{pH} > 7.0$  the ammonia nitrogen contents equal to the  $\text{NH}_3$  contents adding  $\text{H}^+$ . So the ammonia nitrogen variation was affected by pH shift and  $\text{NH}_3$  yield. The final ammonia nitrogen contents of Digester I and Digester II were  $1.11 \text{ g kg}^{-1}$  wet wt and  $2.48 \text{ g kg}^{-1}$  wet wt, respectively.



The crude protein content (N x 6.259) of Digester II decreased by 43.40 % after fermentation. In contrast, the crude protein content of Digester I changed slightly and decreased only about 3.69 %.



**Figure 8: Variation of ammonia nitrogen in both digesters**



**Figure 9: Variation of crude protein in both digesters**

## 5.2 Laboratory experiment

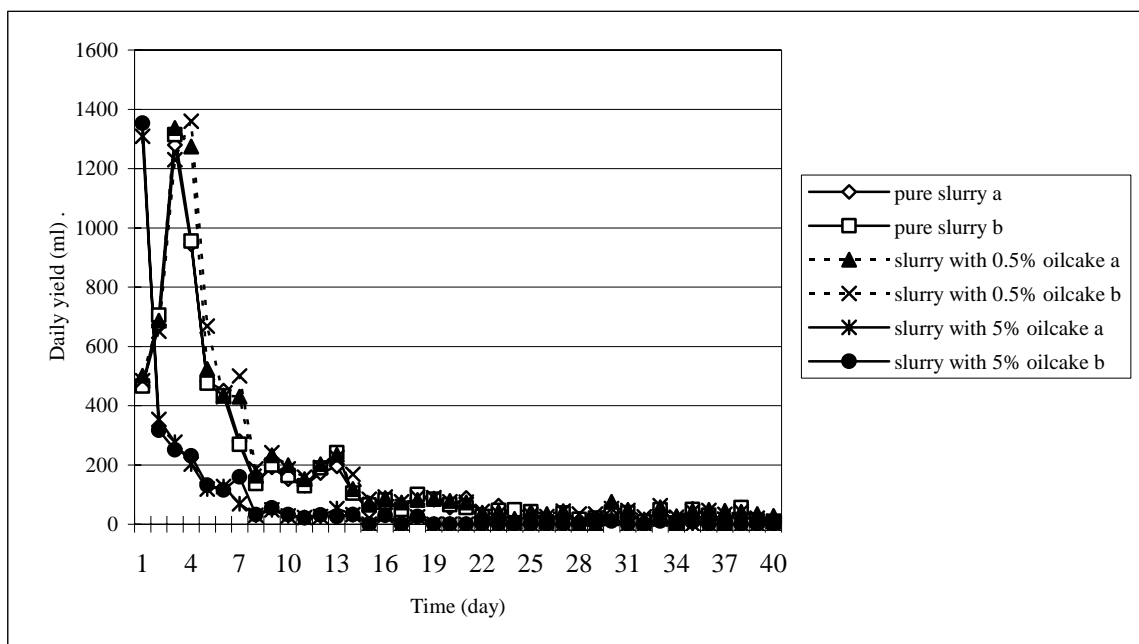
The hydraulic retention time (HRT) of the lab-scale fermentations was 40 days. And mean biogas productions of the three different levels were 6761.5 ml for pure material digester (Digester A), 7827.5 ml for mixing 0.5 % oilcake digester (Digester B) and 2848 ml for mixing 5 % oilcake digester (Digester C), respectively. After fermentation, the mean fresh weight of digesters decreased by 6.15 % for Digester A, 5.49 % for Digester B and 1.01 % for Digester C.

### 5.2.1 Biogas production

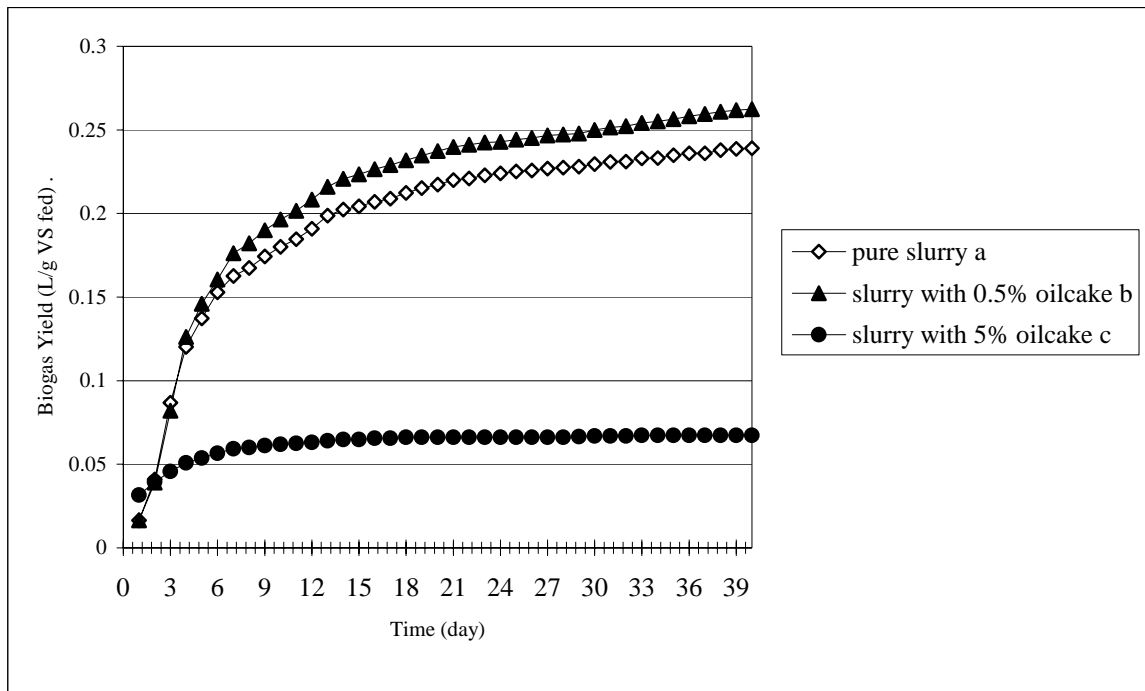
Small amounts of biogas were produced when 5 % oilcake were mixed in after the first week. After 18 days, the biogas production ceased. The highest daily biogas production appeared at the third day in the pure material digesters and at the fourth day in the digesters with 0.5 % oilcake. The biogas production was low after 2 weeks in the digesters with pure material and with 0.5 % oilcake (Figure 10).

Total mean biogas yields of Digester A, Digester B and Digester C were 0.24, 0.26 and 0.07 l g<sup>-1</sup> VS fed, respectively. The highest mean biogas yield was 0.26 l g<sup>-1</sup> VS fed of Digester B. And then 0.24 l g<sup>-1</sup> VS fed of Digester A which was as same as the result gathered from field experiment. Compared with 0.37 l g<sup>-1</sup> VS fed gained in the field experiment, biogas production of the Digester B was lower. This might be explained by the different mixing intensities and the lower concentration of oilcake. Mixing can help to break up the large clumps thereby providing better distribution of microorganisms throughout the reactor (Sadaka and Engler, 2000). Compared with mixing every 3h for 10 min in the field experiment, mixing only twice per day in the laboratory experiment was less intensive. So the biogas production was inhibited and fermentation stopped.

The biogas yields determined for the pure oil cakes in the laboratory of BiogasBeratung-Bornim ranged between 0.58-0.80 L g<sup>-1</sup> VS for false flax cake and linseed cake had biogas yields of 0.65-0.82 L g<sup>-1</sup> VS (normal liter). Measured mean methane contents of the biogas from both oilcakes ranged around 60 %.



**Figure 10: Daily biogas production for lab-scale fermentation**



**Figure 11: Mean cumulative biogas production for lab-scale fermentation**

The VS conversion efficiencies in the experiments in Trenthorst were 0.83, 0.81 and 0.38 L g<sup>-1</sup> VS destroyed of Digester A, Digester B and Digester C, respectively. The conversion efficiencies of Digester A and Digester B were similar.

### 5.2.2 Changes of elemental composition

The physico-chemical parameters of the fresh and digested slurry for the lab-scale fermentations are given in Table 3. After 40 days fermentation, volatile solid reduction decreased by 28.78 %, 32.48 % and 17.88 % for Digester A, Digester B and Digester C, respectively. The highest volatile solid reduction was 32.48 % for Digester B. The pH of Digester A and Digester B was fairly stable during the fermentation and just a little bit higher after fermentation. The pH of Digester C decreased to 5.48 and with acids accumulating, methanogenic bacteria can not survive in this environment. Fermentation of Digester C was stopped.

The crude protein contents of the three different variants decreased after fermentation. The mean crude protein contents decreased by 16.57 %, 22.25 % and 22.33 % for Digester A, Digester B and Digester C, respectively. The highest crude protein content reduction was 22.33 % for Digester C, which was the reason for the increased ammonia nitrogen content in Digester C after digestion. The mean crude fat contents of Digester A and Digester B decreased by 12.41 % and 28.31 % with the methane production. The mean crude fat contents of Digester C increased according to the acid-accumulation which bacteria can not survive to consume. After digestion, the mean crude fibre contents were decreased by 33.98 %, 33.18 % and 5.80 % for Digester A, Digester B and Digester C, respectively. Reduction of crude fibre contents in Digester A and Digester B was similar. Under laboratory controlled conditions the substrate in Digester B had a significant effect on biogas production, VS reduction and crude fat reduction, compared with Digester A. Digester C was not successful compared with the field experiment owing to lack of sufficient mixing to help bacteria consume acids.

**Table 3: Variation of elements in lab-scale fermentations**

Parameters	Pure material (Digester A)		+ 0.5 % oilcake (Digester B)		+ 5 % oilcake (Digester C)	
	Initial	Final*	Initial	Final	Initial	Final
Fresh weight (g)	600	563	602	569	619	613
Total Solid (%)	5.4	4.3	5.7	4.3	7.7	6.6
Volatile Solid (g/kg wet wt)	47.1	35.8	49.6	35.4	68.4	56.7
Crude protein (g/kg wet wt)	6.0	5.3	7.3	6.0	13.9	10.9
Crude fat (g/kg wet wt)	1.1	1.1	1.2	0.9	4.0	5.0
Crude fibre (g/kg wet wt)	21.8	15.3	21.5	15.2	22.0	21.0
Ammonia nitrogen (g/kg wet wt)	0.49	0.47	0.44	0.59	0.47	1.65
pH	7.50	7.60	7.42	7.59	6.83	5.48

\* mean values

## 6 Acknowledgements

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