Determination of kinetic constants from the co-digestion of dairy cow slurry and municipal food waste at increasing organic loading rates.

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Abstract:

The aim of this study was to investigate the performance and the kinetic constants of anaerobic mesophilic CSTR reactors run at increasing organic loading rates (OLR). The reactors were co-digesting dairy cow slurry (DCS) and municipal food waste (MFW). The supply of DCS was held constant, while the supply of MFW was increased in the four reactors: 0, 14.0, 24.5 and 32.2 % (ww). Degradation of organic matter, specific methane yield per mass unit converted organic matter, and the kinetics of the process were used to investigate the performance of the reactors. While the hydraulic retention time was decreased from 25.9 to 17.5 days, the specific methane yield increased from 0.21 to 0.44 l $CH_4 \cdot gVS^{-1}$. The relationship between the kinetic constant and the OLR was found to be linear. The efficiency of the process increased when the OLR increased in this experiment.

1. Introduction

Anaerobic digestion of dairy cow slurry (DCS) manure has several positive effects. From an environmental perspective the reduction of greenhouse gas emissions (GHG) from agriculture and the production of renewable energy are most important [1,2,3,4]. Other positive effects of anaerobic digestion are reduced numbers of pathogens and weed germs in the manure [5,6]. Due to the degradation of volatile solids (VS), the digestate has improved rheological properties compared with untreated manure [7], which simplifies the fertilization of the fields. The amount of nitrogen bound in organic matter (OM) will also be reduced. This mineralized nitrogen is more plant available and increases the speed of uptake in the plants [8]. This may also reduce the demand for chemical fertilizers.

Unfortunately, the content of VS per volume unit slurry manure is relatively low, as the substrate already has passed the digestion system of an animal. A major part of the remaining VS consists of lignocellulosic fibres, which may pass the anaerobic digester relatively undigested [9]. As a result, both the specific methane yield (SPM) (ml $CH_4 \cdot gVS^{-1}$) and the volumetric methane yield (VMY) (ml $CH_4 \cdot (l_{\text{reactor vol.}} \cdot \text{day})^{-1}$ are relatively low. Co-digestion of DCS with energy rich co-substrates has shown promising results in terms of increasing the biogas yield. The anaerobic digestion is also relatively stable as it benefits many of the positive effects of DCS [9,10,11,12].

Internal energy demand at a biogas plant is often divided in two. First there is a demand for electric energy to run pumps, valves and agitators. Second there is a demand for heat, for heating new substrates and to cower for heat losses from reactors and pipes [13]. Common for these demands are that they are dependent on the volumes of treated substrates and not the energy content of the substrates. Digestion of DCS as sole substrate has been documented to yield a very low surplus of energy when digested in cold climate [13]. Use of energy rich co-substrates could increase the energy production substantially, and the surplus energy can be sold for heating purposes. At an existing farm scaled biogas plant both manure production and reactor size are fixed, and the possibility to increase the heat production is by increasing the amounts of co-substrates. The internal thermal energy consumption is both for heating of new substrates and to cover for heat losses from the digesters and pipes. The total energy consumption is therefore dependent on the design of the plant and the climatic conditions [13, 14]. Use of co-substrates would lead to an increased surplus of energy [13]. The possibility to vary the biogas produced is by varying the amount of co-substrate. This would lead to a change in the hydraulic retention time (HRT) and the organic loading rate (OLR) of the reactor.

The aim of this study was to investigate the performance of a mesophilic CSTR reactor with a fixed daily supply of liquid dairy cow slurry (DCS) and an increasing amount of municipal food waste (MFW). This setup made it possible to study the degradation of organic matter, to find the methane yield per unit degraded organic matter, and to study the kinetics of the process in the reactors. Kinetic constants have been studied, but many of the former studies have been based on batch experiments [15, 16, 17]. The constants for various materials from batch experiments have been used in models for semi- and continuous processes, e.g. ADM 1 [18]. One of the papers that discuss kinetic constants is by Mähnert & Linke [19], but also they use batch experiments to be able to calculate the kinetic constant. Mähnert & Linke [19] concluded that this model worked well for maize ensilage, oat ensilage and cattle manure as substrates. The hypothesis is that this model is valid also for food waste and cattle manure, but one can calculate the theoretical biogas potential from the CSTR experiment, and use this rather than use the theoretical biogas potential from batch experiments.

2. Materials and methods

2.1 Experimental setup

The four reactors were supplied with 15 litres inoculum each. Two days later, the feeding of the reactors started. All reactors were fed once a day, during the whole experiment. In the adjustment period all the reactors were fed with the same amount of substrate, 86 % DCS and 14 % MFW. When all reactors gave the same quantity of biogas, the experiment started, referred to as day 1. In reactor R1, the DCS was used as sole substrate. The HRT for reactor R2 was equal to the HRT of the full-scale plant where 14% food waste was used as co-substrate. In reactors R3 and R4, 24.5% and 32.2% food waste was added, calculated from the total mass supplied. This gives an OLR of approximately two, three, four and five gram VS per litre reactor volume and day. Unlike many other experiments, both OLR and HRT varied between the reactors. This to be more comparable to farm scaled systems, where the amount of animal manure is relatively constant, while the amount of co-substrates is adjustable.

Table 1: Daily supply of substrates, as mass percent and gram per litre reactor volume and day, hydraulic retention time (HRT) and organic loading rate (OLR).

	Percentage of mass substrate supplied daily		Substrate (g L^{-1})		HRT (days)	OLR (g VS l^{-1})
Reactor	Manure	Food waste	Manure	Food waste		
R1	100	0	38.7	0	25.9	1.83
R2	86	14.0	38.7	6.6	22.1	2.99
R3	75.5	24.5	38.7	12.5	19.5	4.03
R4	67.8	32.2	38.7	18.3	17.5	5.04

2.5 Kinetic model

The modelling in this paper is based on the work of Mähnert & Linke [19]. A first order reaction model (6) is used to estimate the speed of the conversion of organic materials to biogas. The prerequisite is that when there is a fixed ratio between the feedstocks, the biogas yield will be given as a function of changes in OLR when the HRT is changed. First we assumed that the reaction can be described as a first order kinetic reaction. The reaction constant, k, is given in (1).

$$r(c) = k * c_e \qquad (1)$$

We also assume that the efficiency (η) of the process can be expressed by the decrease of organic concentration divided by the inflow concentration:

$$\eta = \frac{c_0 - c_e}{c_0} \tag{2}$$

The biogas yield can be expressed by (3) when multiplying maximum yield, y_{max}, and the efficiency, η:

$$y = y_{max} * \eta \tag{3}$$

Equations (2) and (3) gives the theoretical maximum yield (4):

$$y_{max} = y * \frac{c_0}{c_0 - c_e} \tag{4}$$

From Mähnert & Linke [19], specific gas production, y, can be calculated from $\bar{k}, c_0, y_{max}$ and OLR (4):

$$y = y_{max} * \frac{\bar{k} * c_0}{\bar{k} * c_0 + y_{max} * OLR} \tag{5}$$

Where $\bar{k}$=k*$\frac{\rho_E}{\rho_G}$

$\bar{k}$ can be calculated from:

$$\bar{k} = \frac{y}{c_e} * OLR \tag{6}$$

Nomenclatures:

η - efficiency

c_0 - VS, % of substrate

c_e - VS, % of digestate

y - specific biogas yield

y_{max} - maximum biogas yield

k - kinetic constant

$\bar{k}$ - kinetic parameter

ρ_E - density effluent

ρ_G - density biogas

The density of DCS is assumed to be 1000 g·L^{-1} [19]. The density of methane is 0.716 g·L^{-1} (273K, 1atm), and the density of carbon dioxide is 1,977 g·L^{-1} (273K, 1atm). The density of the biogas (ρ_G) from the four reactors was determined according to the ratio of methane and carbon dioxide recorded in the experiment.

The model was built by using the results from the first part of the experiment to calculate kinetic constants, while the results from the last part of the experiment were used to verify the calculated constants.

2.2 Substrates and inoculum

The DCS was collected at Tomb Agricultural Junior College in Råde, SE Norway, and used as the main substrate. The well mixed DCS was filled into 20 litre~~s~~ containers and transported to the laboratory (47km). To reduce degradation, the DCS was stored at a temperature of 4 °C until use. Samples were collected for further analyses.

The MFW in this experiment was chopped and thermally pre-treated according to the EC regulation 1069/2009, which has been implemented in Norway. The MFW was collected from Norsk Matretur AS in Lørenskog, SE Norway, and transported to the laboratory (50km), while still warm from pre-treatment. The MFW was blended well and filled into 0.5 litre~~s~~ bottles, before storing at 4 °C. Samples were collected for further analyses.

Digestate was collected from the biogas plant at Tomb Agricultural Junior College and used as inoculum. In their plant, DCS from their dairy farm was co-digested with MFW from Norsk Matretur AS. The MFW constituted 14 percent of the daily supplied mass to the reactor at the time [20]. Before inoculating the reactors the digestate was stored at approximately 20 ºC for two days.

2.3 The CSTR reactors and the monitoring equipment

Four laboratory scaled CSTR reactors were used in this experiment. Each reactor had 15 litres active reactor volume and 10 litres headspace. The CSTR reactors were constructed of a 400 mm high cylinder of casted acryl with top and bottom plate in stainless steel. These plates were provided with 32 mm ball valves for the supply of substrates and drainage of digestate. An electric heating belt around the reactors, connected to a temperature sensor in the reactor and controlled by a thermostat, heated the substrate to 37 ± 2°C. The speed of the stirring device was

60 rpm, at normal running. Before removal of digestate, the speed was increased to 180 rpm in order to ensure homogeneity in the digester.

The biogas production was measured by pressure induced peristaltic gas pumps. These were constructed at the Norwegian University of Life Sciences. The daily gas production was manually logged when the reactors were fed. For further calculations, gas temperature of 20°C and a pressure of one atmosphere has been used. The gas composition was automatically analysed by an SRI gas chromatography instrument (Model 8610 C) in average four times per day, and logged by a computer. Average methane content was then calculated on a weekly basis.

During the first three weeks, the microorganisms adapted to the substrates. After this period, the methane production was relatively constant. Calculations of average methane content and production do not include these first three weeks.

2.4 Analyses of inoculum, substrates and digestate.

The substrates and inoculum were analysed for total solids (TS) and volatile solids (VS) before the start of the experiment. The weekly samples were also analysed for TS and VS. Three replicas were collected each week from the four reactors. Weekly analyses of pH and ammonia were done with Thermo Scientific Orion Dual Star pH/ISE Benchtop, supplied with Thermo Scientific Orion 9512 ammonia electrode and WTW SenTix pH electrode.

Selected samples were analysed for pH (EN ISO 15933), TS (EN 12880), VS (EN 12979), fat, ammonium, Carbon-Nitrogen-ratio (C/N ratio), phosphorus, hydrogen, potassium, total carbon, Kjeldahl-N, protein, sulphur, volatile fatty acids (VFA). These analyses were performed by Eurofins AS, Moss, Norway.

4. Results and discussion

4.1. Methane Production

The daily total biogas production in the four reactors are shown in Figure 1. In this period the feeding was as described in Table 1. There was an adjusting time to the new loading rates of substrates in the beginning of the period, here found to be 21 days. Thereafter the biogas production was relatively stable. The 21 first days are therefore excluded from the further calculations.

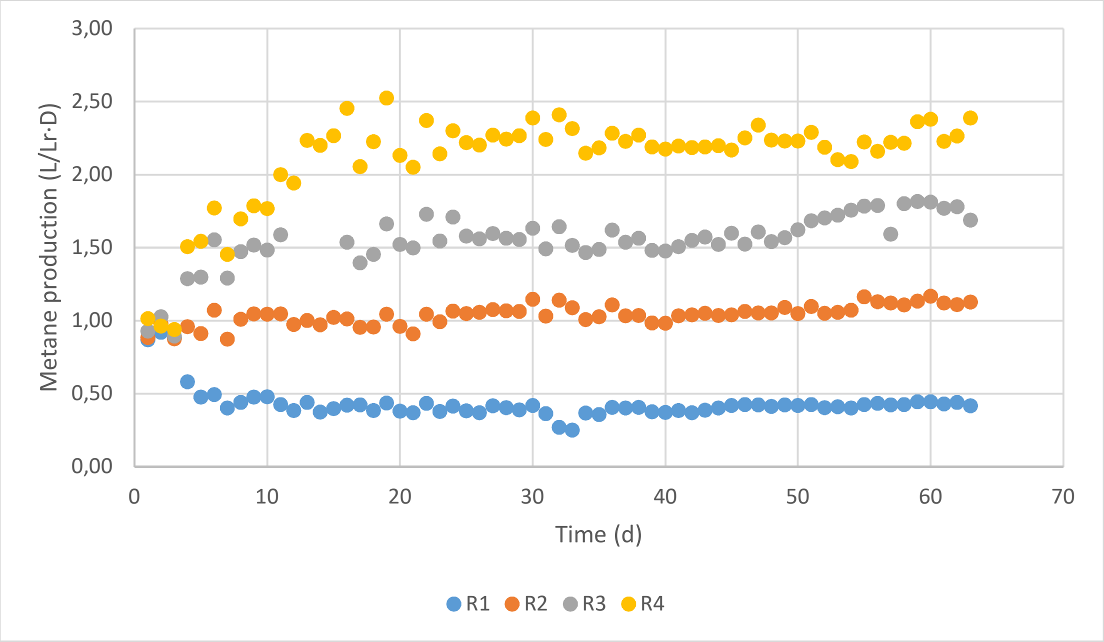

Figure 1: Methane production per litre reactor volume in the experimental period included the 21 first days. R1: 0% food waste, R2: 14% food waste, R3: 24.5% food waste, R4: 32.2% food waste.

The average methane content in the biogas was found to be higher the more MFW that were supplied to the reactors (Table 2).

Table 2: Average methane content, specific methane yield and volumetric methane production, average and standard error.

Reactor	Methane	Specific methane yield		Volumetric methane production
	(%)	[mL *g(VS)$^{-1}$]	[m^3 t^{-1} ww]	[L·(m^3·d)$^{-1}$]
R1	62.6 ± 0.7	218.4 ± 21.4	10.3 ± 1.0	399.7 ± 39.2
R2	62.8 ± 2.1	358.0 ± 15.6	23.6 ± 1.0	1070.5 ± 46.8
R3	63.3 ± 3.1	402.0 ± 26.4	31.6 ± 2.1	1620.2 ± 106.3
R4	63.7 ± 4.2	444.7 ± 15.4	39.3 ± 1.4	2241.4 ± 77.7

4.2. Analyses of digestate.

The average values of several parameters from effluent and feedstocks are reported in Table 3. TS and VS in the effluent from the four reactors was relatively stable during the experiment and between the reactors. As expected the TS was higher the higher the OLR was in the reactors. TS in the substrate mixture for the four reactors was 6.23%, 7.78%, 9.29% and 10.24% for R1, R2, R3 and R4, respectively.

Table 3 indicates that the Kjeldahl-N was higher the more MFW that was used, while the ammonium content seemed to be relatively stable. The measured values did not indicate inhibition. The VFA concentration was higher in the reactors supplied with MFW, compared to the reactor supplied with DCS as sole substrate. The levels were relatively stable throughout the experiment, and did not indicate VFA inhibition. The results also indicated that the concentration of *E.coli.* was effectively reduced when the HRT exceeded 21 days.

Table 3: Characteristics of feedstocks and digestate from the four reactors.

		R1	R2	R3	R4	DCS	MFW
TS	% of ww	5.3	5.6	6.1	6.2	6.23 ± 0.05	18.7
VS	% of TS	71.9	72.5	72.2	71.9	76.1	87.0
pH		7,75 ± 0,07	7,65 ± 0,06	7,64 ± 0,08	7,64 ± 0,07	na	4.3
Hydrogen (H)	%	4.5	4.6	4.8	4.8	na	6.6
Total carbon (C)	%	40.2	40.4	40.9	40.3	na	53.9 %
Kjeldahl-N	%	5.6	6.4	6.4	6.8	na	3.3 %

	g/100						
Ammonium	g	2.6	2.7	2.8	2.7	na	0.44
VFA	mg/l	150	246	284	230	na	4563
E.coli	MPN/g	<20	<20	220	800	> 16000	<20

4.3. Organic loading rate

The specific methane yield was expected to decrease when OLR was increased. This effect was not observed in this experiment. The specific methane production increased the higher the OLR in the reactors was (Figure 2). This was probably due to the high degradability of the MFW compared to the DCS. The specific methane production was 110% higher for the reactor with highest OLR, compared to the reactor with lowest OLR. This effect of higher specific methane production when the OLR was increased, gave a substantially increase in the methane production per reactor volume unit. The methane production per volume unit reactor was increased by 477 % when the OLR was increased from 1.83 to 5.04 g VS L^{-1} day^{-1}. At the same time the HRT was decreased from 25.3 to 17.2 days.

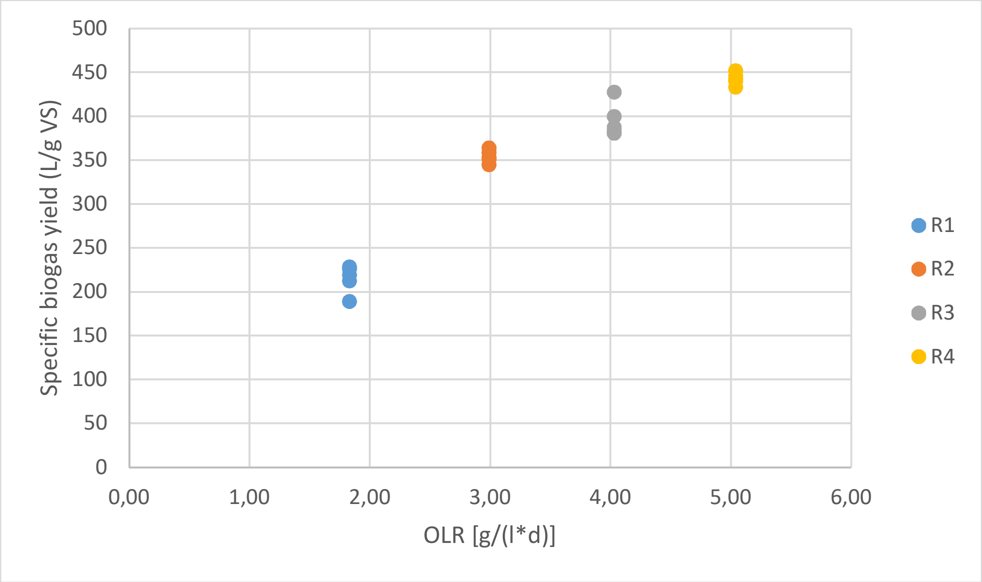

Figure 2: Specific biogas yield (average per week) versus organic loading rate in the four reactors. R1 - only cattle manure, R2 - 14.0 % food waste, R3 – 24.5 % food waste, R4 - 32.2 % food waste (ww).

Several studies on co-digestion of cattle manure together with energy-rich MFW have been conducted [10, 11, 21, 22, 23]. The results in this study are in accordance with these results.

4.4. Conversion of organic substrates

The analyses of VS in the outflow from the four reactors showed, as expected, more VS in the digestate the higher the OLR was. The averaged percentages of degradation were 28.2, 46.7, 52.6 and 55.2 for R1, R2, R3 and R4, respectively. This is comparable to the 50 % degradation found by Callaghan et al. [21] and 55% degradation found by Marañón et al. [11]. Although the outflow of organic matter was higher when the amount of co-substrate was increased, the degradation of VS per volume unit digester was also increased (Figure 3). When plotting the degradation against specific methane yields, the relationship was logarithmic with *R^2=0.958*. This gives an estimated methane yield of 0.301L CH_4 g VS^{-1}. This is lower than the figure reported by McCarty [24], 0.378L CH_4 g VS^{-1} of pure acetic acid. According to Hill [25], this could be a result of the productivity of the process. The methane percentage in the biogas was higher when the use of food waste was increased (Table 2), which could be explained by methane formation by hydrogen consuming Archaea, which would lead to a higher specific methane yield. Higher protein and lipid content in the MFW could also be a part of the explanation. If manure is excluded, the relationship is linear (R^2=0.895), and the specific methane yield would be 0.326L CH_4 g VS^{-1}, which proves this theory. The average specific methane yield of the DCS in the experiment was 0.216 ± 0.011L CH_4 g VS^{-1}.

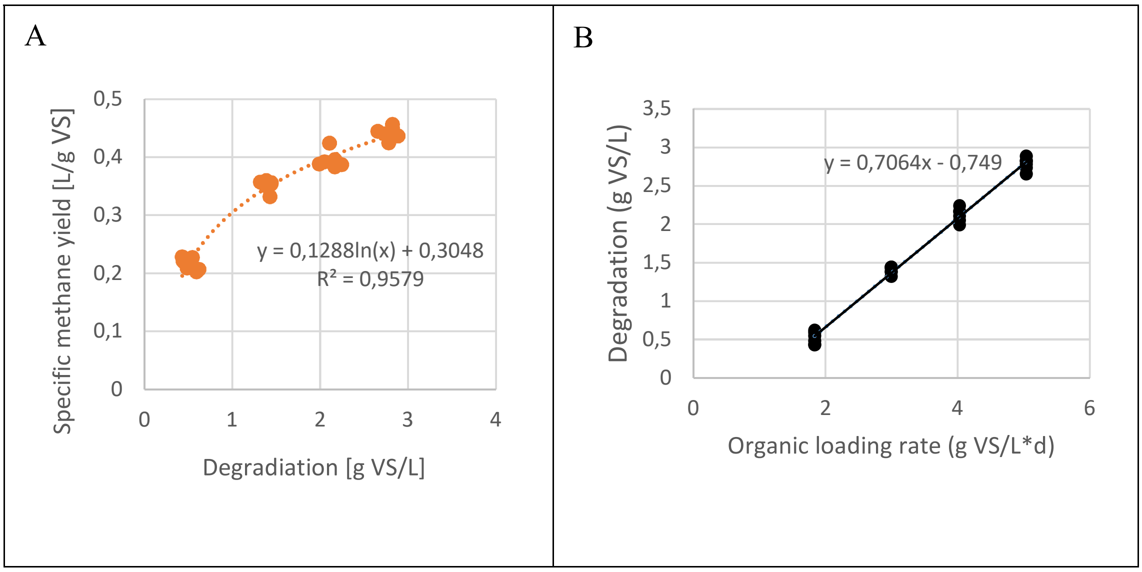

Figure 3: A) Specific methane yield vs degradation, average of weekly measures. B) Degradation vs organic loading rate (OLR).

4.5 Kinetic modelling

The model (5) is based on the parameter y_{max}, which is a theoretical value. This was determined by defining the efficiency according to:

$$\eta = \frac{y}{y+r} \qquad (7)$$

where r is the residual potential. η could also be calculated from (2).

As indicated in Table 2, the difference in the CH_4/CO_2-ratios in the biogas from the four reactors were relatively small. The same ratio was therefore selected for all the four reactors: 0.63/0.37. This gives a calculated density of the biogas of 1.183 kg/m^3.

Table 4 shows the calculated biogas production from the experimental data. y_{max} is a theoretical value, calculated from the biogas yield and the degradation rate of the process, according to equations (4) and (7).

The data from the experimental period was divided in two. Data from the first four weeks were used to estimate y_{max} and k. From these variables y for the rest of the period was predicted and compared with the measured data from the last five weeks (Table 4).

Table 4: Estimation of C_0, y_{max}, and k, predicted biogas production from (7), measured biogas production, and difference between predicted and measured biogas production.

C_0, [%]	y_{max}, [l/g]	$\bar{k}$, [1/day]	k, [1/day]	Predicted y, [l/g]	Measured y, [l/g]	Difference, [%]

0.047	1.393	21,99	0.0260	0.324	0.300	8.19
				0.342	0.336	1.59
				0.382	0.359	6.36
				0.352	0.365	-3.35
				0.366	0.380	-3.88
0.066	1.300	44,68	0.0529	0.595	0.569	4.50
				0.569	0.547	3.99
				0.590	0.577	5.16
				0.588	0.577	-1.85
				0.586	0.597	-1.91
0.079	1.230	56,22	0.0665	0.626	0.604	3.53
				0.616	0.604	2.10
				0.654	0.616	6.21
				0.669	0.675	-0.82
				0.644	0.682	-5.51
0.088	1.240	79,07	0.0935	0.698	0.706	-1.19
				0.680	0.692	-1.77
				0.704	0.700	0.59
				0.687	0.680	0.98
				0.676	0.706	-4.43

Table 4 indicates a good relationship between predicted and measured biogas production, and linear regression of the model versus measured values gave $R^2= 0.990$, $RMSE = 0.0203$. k was dependent on the initial concentration of VS, and the relationship was linear ($R^2= 0.984$) when OLR vs k was plotted (Figure 4).

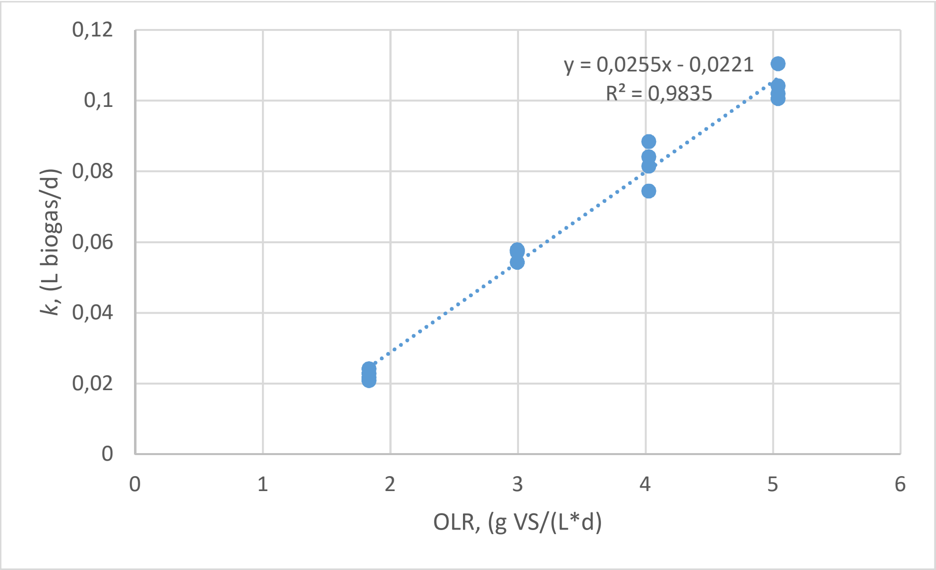

Figure 4: Kinetic constant, *k*, vs organic loading rate (OLR).

5. Conclusion

The methane production per volume unit reactor was increased by 479 % when the OLR was increased from 1.83 to 5.04 g VS L^{-1} day^{-1} by the use of MFW. This reduced the HRT from 25.3 to 17.2 days. The degradation rate and the specific methane yield per g VS was also higher for MFW compared to DCS. This resulted in a higher kinetic constant. Testing of a first order kinetic model showed very good relationship between the measured and modelled biogas production ($R^2= 0.990$). The proposed kinetic model could therefore be used to predict the biogas production. More studies should be carried out to test the model.

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