



# 2stageBiogas - Energy yield optimization of a two- stage biogas technology for the organic fraction of municipal solid waste

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Institution responsible for the project:

Section for Sustainable Biotechnology  
Aalborg University Copenhagen,  
A.C. Meyers Vænge 15, 2450 København, Denmark.  
CVR no.: 29102384

Authors:

Hinrich Uellendahl, AAU Cph  
Zeeshan Nasir, AAU Cph  
Federico Micolucci, AAU Cph

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## Preface

This is the final report of the project “2stageBiogas - Energy yield optimization of a two-stage biogas technology for the organic fraction of municipal solid waste”, which was granted by the ForskEL program (no. 10800).

The project was carried out from December 2012 to May 2015 by the Section of Sustainable Biotechnology at Aalborg University Copenhagen (SSB-AAU) in collaboration with Solum A/S.

The report comprises the main results and conclusions of the project. For more detailed information please refer to the different articles in the list of dissemination of results at the end of this report.

Copenhagen, 9 October 2015

Hinrich Uellendahl, Associate Professor,  
Section for Sustainable Biotechnology, Aalborg University Copenhagen



## Content

Preface .....	3
Content.....	4
Project overview .....	5
The work packages of the project.....	6
Brief summary of results.....	7
Detailed results and conclusions .....	9
WP1 Analysis of hydrolytic stage .....	9
WP2 Enhancing hydrolysis of OFMSW under different process conditions.....	12
WP3 Addition of enzymes.....	26
WP4 Conversion of xenobiotics .....	26
WP5 Conclusions and evaluation.....	28
Dissemination of project results .....	30
References.....	31

## Project overview

The main objective of the 2stageBiogas project was to enhance the energy yield of the AIKAN<sup>®</sup> process, a 2-stage process for the treatment of the organic fraction of municipal solid waste (OFMSW), developed by the company Solum A/S. In the AIKAN<sup>®</sup> process design the methanogenic (2<sup>nd</sup>) stage of the anaerobic digestion process is separated from the hydrolytic (1<sup>st</sup>) stage (figure 1.0), which enables pump-free filling of the screened waste into the 1<sup>st</sup> stage (waste processing module) and eliminates the risk for blocking of pumps and pipes by pumping only the percolate from the 1<sup>st</sup> stage into the 2<sup>nd</sup> stage (biogas reactor).

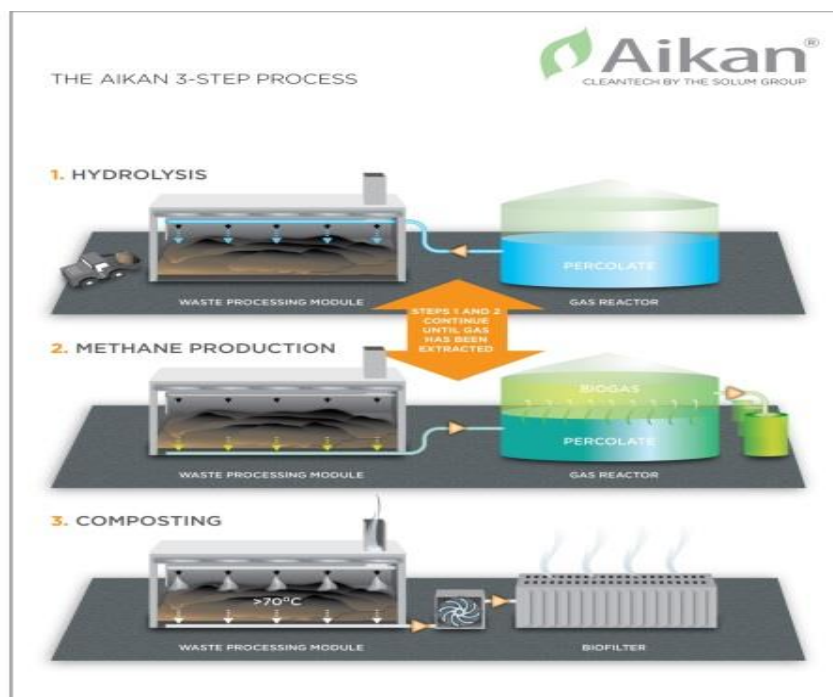


Figure 1.0. The 3 steps of the two-stage AIKAN<sup>®</sup> process

The key target of the project was to enhance the biogas yield of the AIKAN<sup>®</sup> process by at least 20%. Previous investigations revealed that the bottleneck of the whole conversion of the waste into biogas is the efficiency of the hydrolysis in the processing module (first stage) for the conversion of complex organic matter into dissolved organic matter. The 2stageBiogas project investigated both the factors that are responsible for the low performance of the hydrolysis and the means for overcoming the bottlenecks by analysis of the efficiency of the hydrolysis under standard conditions (WP1) and under different process operation conditions (WP2), including the addition of enzymes (WP3). Furthermore, the conversion of xenobiotics that could harm the use of the final compost as soil fertilizer was also analyzed (WP4).



## The work packages of the project

The project was divided into five work packages, namely the Analysis of hydrolytic stage (WP1), the Hydrolysis under different process conditions (WP2), the Addition of enzymes (WP3), the Conversion of xenobiotics (WP4) and the Evaluation (WP5).

SSB-AAU was responsible for performing work package WP1-5 in cooperation with Solum A/S. The tasks of the different work packages and the respective milestones are listed in Table 1.0.

**Table 1.0.** Work packages and milestones of the 2stageBiogas project

Work packages and tasks
<b>WP1 Analysis of hydrolytic stage</b>
WP1.1 Literature study on hydrolysis of OFMSW in 2-stage processes
WP1.2 Testing of hydrolysis of the waste delivered to the AIKAN <sup>®</sup> plant in pilot-scale percolation modules under standard conditions
WP1.3 Analysis of the hydrolysis rate in the pilot-scale modules under standard conditions by COD, TS and VS conversion, VFA production, microbial composition analysis and their activity
M1.1 Pilot-scale set-up for modeling the hydrolytic stage of the AIKAN <sup>®</sup> technology
<b>WP 2 Hydrolysis under different process conditions</b>
WP2.1 Testing of hydrolysis of the waste delivered to the AIKAN <sup>®</sup> plant in pilot-scale percolation modules under different process conditions (temperature, pH, retention time, recirculation rate, water submerge of waste)
WP2.2 Testing of hydrolysis of the waste delivered to the AIKAN <sup>®</sup> plant in pilot-scale percolation modules with addition of adapted microorganisms
WP2.3 Investigation of the influence of the AD – composting sequence on the hydrolytic conversion
M2.1 Identification of optimal process parameters and adapted microorganisms for optimization of the AIKAN <sup>®</sup> process
WP2.4 Pilot- and large-scale testing of standard and optimized conditions at the AIKAN <sup>®</sup> plant
<b>WP3 Addition of enzymes</b>
WP3.1 Lab-scale test of pretreatment of the waste by addition of enzymes in different dosage
M3.1 Identification of optimal dosage of specific enzyme blend for increasing the hydrolysis of the waste
WP3.2 Pilot- and large-scale test of pretreatment of the waste by addition of enzymes in the optimal dosage of a specific enzyme blend
<b>WP4 Conversion of xenobiotics</b>
WP4.1 Analysis of the conversion of xenobiotics during the process at different process conditions applied
M4.1 Identification of the conversion of xenobiotics at optimal conditions identified in M 2.1



### **WP5 Evaluation**

WP5.1 Calculation of the efficiency of the improved conversion process through mass balance and economic data for additional treatment and output of biogas and compost

M5.1 Identification of improvements by applying process optimization of the AIKAN® technology identified during the 2stageBiogas project

## **Brief summary of results**

### **WP1 Analysis of hydrolytic stage.**

Based on a literature study the main bottleneck for the 2-stage process is the efficiency of the hydrolysis of organic matter into soluble COD and VFA in the 1<sup>st</sup> stage, which is hindered by limitations of the mass transfer between the biomass and microorganisms. The conversion efficiency can mainly be enhanced by providing a good contact between biomass and hydrolytic organisms and by maintaining a pH of pH 5.5 - 6.5 in the 1<sup>st</sup> stage process module. Batch hydrolysis experiments with different waste/water ratios of 7.8% - 29.7% revealed that 24% of organic matter content was converted at the lowest ratio and that the VFA release was declining with increasing waste/water ratio. Pilot-scale experiments with direct recirculation of the leachate over the 1<sup>st</sup> stage without connection to the 2<sup>nd</sup> stage gave similar results with a conversion of 26% of the organic matter into VFA after 4 weeks of operation. The pH in the first stage stayed below pH 4.0 and reached 5.5 only at the end of the period. Water addition did not have a significant effect on raising the pH.

### **WP2 Hydrolysis under different process conditions**

It was shown in both batch and full-scale tests, that using effluent from the 2<sup>nd</sup> stage (biogas reactor) for percolation of the waste in the 1<sup>st</sup> stage (processing module) – as done in the full-scale process – lead eventually to inoculation of the 1<sup>st</sup> stage with methanogens. This resulted in the conversion of VFA into biogas also in the 1<sup>st</sup> stage, which could be seen by a decline of VFA release and a pH increase in the percolate, along with an increase in biogas production in the 1<sup>st</sup> phase after 2 weeks of operation.

In lab-scale 2-stage reactor tests with the same operational regime using the effluent from the 2<sup>nd</sup> stage for percolation of the waste in the 1<sup>st</sup> stage, the overall degradation efficiency measured as methane yield from the input biomass related to the theoretical maximum was only 8% for a high waste/percolate ratio of 12.5% (w/w) while 57% conversion was achieved at a low ratio of 3.1%. In full-scale an overall yield of 34% was achieved. Increasing the flow of the percolate from the 1<sup>st</sup> to the 2<sup>nd</sup> stage and of the effluent from the 2<sup>nd</sup> to the 1<sup>st</sup> stage by a factor of 4.4 – 7.0 did not lead to



a significant change in the pattern of VFA release, pH of the leachate and overall methane production.

Implementation of a pH control of the leachate to pH 5.2 through an automatic shift between using directly the leachate or the effluent from the 2<sup>nd</sup> stage for percolation resulted in a constant pH in the 1<sup>st</sup> stage of pH 5.2 for the whole operation period of 24 days with an ongoing VFA release and a conversion efficiency into biogas of 57%

### ***WP3 Addition of enzymes***

Batch tests with addition of enzymes with both cellulase and hemicellulase activity to the OFMSW resulted in an increase of the methane yield by 10-19%. The addition of enzymes in the 1<sup>st</sup> stage could therefore be an option to enhance the overall conversion efficiency. However, the additional biogas yield has to be balanced with the enzyme costs in the respective dosage.

### ***WP4 Conversion of xenobiotics***

The concentration of linear alkylbenzene sulfonates (LAS), polyaromatic hydrocarbons (PAH), di-(2-ethylhexyl)-phthalate (DEHP) and nonylphenol ethoxylates (NPE) was measured in fresh waste supplied the AIKAN<sup>®</sup> plant, in the digestate of the process after 1 week of composting and in the final compost. The concentration per kg of dry matter (DM) increased for PAH, DEHP and NPE when taken at the beginning of the composting process, showing a lower degradation of these compounds than of the overall organic matter during the biogas process phase. In the final compost the PAH and NPE concentration decreased while DEHP further increased, indicating that PAH and NPE were degraded while DEHP remained persistent during the aerobic composting phase. The concentrations of all xenobiotics were below the respective threshold limit values of 3 (PAH), 50 (DEHP) and 10 (NPE) mg/kg-DM. The concentration of LAS was in all samples much lower (< 50 mg/kg-DM) than the threshold limit value of 1300 mg/kg-DM.

### ***WP5 Evaluation***

Out of the different tested operational changes a lower waste/percolate ratio and an automatic pH control of the 1<sup>st</sup> phase appear to be the most promising measures to increase the overall energy efficiency of the 2-stage process for OFMSW. By these measures the conversion of the waste into biogas could be enhanced from 34% to almost 60%. The higher biogas production and enhanced reduction of organic matter would, furthermore, reduce the amount of aeration needed for the composting process, which would reduce the energy consumption of the plant, thereby further increasing the energy efficiency.





## Detailed results and conclusions

### WP1 Analysis of hydrolytic stage

#### 1.1 Hydrolysis of OFMSW in 2-stage processes – Literature study

The anaerobic digestion (AD) process of the organic fraction of municipal solid waste (OFMSW) has been applied in large-scale either in co-digestion with other waste biomass as so called wet and semi-dry processes (with total solids (TS) concentration below 20%) or as sole substrate in so called dry anaerobic digestion systems, operating at TS of more than 20% (Hartmann and Ahring, 2006). The dry AD systems offer the advantage of a smaller digester volume needed per volume of waste, thus requiring less heating (De Baere and Matteeuws, 2014). Furthermore, the dry AD system in a 2-stage set-up (such as the AIKAN<sup>®</sup> system) enables pump-free filling of the waste into the 1<sup>st</sup> stage process module without the need for stirring equipment and eliminates the risk for blocking of pumps and pipes by pumping only the percolate from the 1<sup>st</sup> stage to the 2<sup>nd</sup> stage biogas reactor. The main drawbacks of dry AD systems are, however, the limitation of the mass transfer between substrate and microorganisms and an inhomogeneous distribution of substrate and inocula, which leads to sub-optimal process conditions (Ahn and Smith, 2008, García-Bernet et al., 2011, Li et al., 2011). In a 2-stage reactor system, the AD pathway is divided into two stages i.e. (1) the hydrolytic and acidogenic stage followed by (2) the methanogenic stage. The first stage results in the formation of volatile fatty acids (VFAs) dissolved in the leachate of the hydrolytic reactor, which is transferred to the 2<sup>nd</sup> stage, where the VFAs are converted into biogas by methanogens (Parawira et al., 2008). For AD of solid organic matter, the hydrolysis of the complex polymers into monomers has shown to be the rate-limiting step (Ghosh and Klass, 1978, Eastman and Ferguson, 1981, Arntz et al., 1985, Noike et al., 1985). The separation of the whole conversion pathway into a two-stage digester system can have its benefit by optimizing the conditions (mainly pH, temperature) for the respective microbial populations, leading to a high overall efficiency of the process (Blonskaja, Menert et al., 2003). Based on these findings, the overall goal of the project was to identify the optimal process conditions for each stage of the 2-stage digester system to achieve a high conversion of OFMSW into biogas.

#### 1.2 Waste characterization and hydrolysis of OFMSW in the 2-stage process under standard conditions

Composition analysis of the OFMSW delivered to the AIKAN<sup>®</sup> plant (figure 2.1) revealed a food waste fraction of 60-70%, a paper fraction of 10-15% and a plastic fraction of 10-12% of the fresh weight (FW). Total solids (TS) and volatile solids (VS) content of OFMSW was 32.5% and 27.1%, respectively, and the pH of OFMSW was determined to pH 4.7. Composition analysis of OFMSW used revealed a distribution of 63% carbohydrates, 14% proteins, 7% fats and 12% lignin of the volatile solids. The theoretical biomethane potential (BMP) was determined to 410 mL/g-VS.

At large-scale the OFMSW is subjected to a mechanical pre-treatment, where (plastic) bags containing waste are opened and the coarser fractions of impurities are removed before the waste is mixed with structure material and loaded to the process module.



Figure 1.1: Sampling of OFMSW delivered to Solum's AIKAN® plant by drill sampling

The efficiency of the 2-stage system for treating OFMSW and specifically the efficiency of the hydrolytic (first) stage in the AIKAN® process was tested under standard and modified conditions in lab-scale batch tests, in pilot-scale and in large-scale.

### 1.3 Lab-scale batch tests

Hydrolysis potential batch tests (HPB) were carried out to determine VFA production of OFMSW in different waste/water ratios to evaluate the influence of different water saturation on the hydrolysis. All batch tests were performed in triplicates under mesophilic conditions ( $37^{\circ}\text{C} \pm 0.1^{\circ}\text{C}$ ) in 1 L serum bottles. The bottles were flushed with  $\text{N}_2$  and  $\text{CO}_2$  (80% / 20%) before closing to ensure anaerobic conditions). The batch tests were run for one to four weeks, depending for how long a release of VFA was observed. Samples were taken for pH, volatile fatty acids (VFAs) and hydrogen analysis. The pH gradually decreased during the first 6 days to pH 4.8 whereas the VFAs concentration increased to 4.5 g/L in the first 11 days.

In a second set-up of the batch experiment, different amounts of water were added to mimic a higher water saturation of the waste. The results in table 1.1 reveal that the VFA concentration increased with adding less water, but the total amount of VFA released from the waste decreased with lower water saturation. The highest VFA release (1.52 g) corresponded to a conversion efficiency of the total organic matter (with a VS content of 27%) into VFA of 24.1% %

Table 1.1. VFA release in batch hydrolysis setup with different amounts of water added

Set-up	OFMSW (g)	H <sub>2</sub> O (mL)	Waste/percolate ratio	VFAs (g/L)	VFA (g)
A	23.4	300.0	7.8%	5.05	1.52
B	23.4	221.0	10.6%	6.17	1.36
C	23.4	158.0	14.8%	7.55	1.19
D	23.4	78.8	29.7%	11.30	0.89

#### 1.4 Pilot-scale tests on hydrolysis in 1<sup>st</sup> stage

Pilot-scale tests (figure 1.2) were performed at Solum’s two pilot experiment facilities (PEFs) to produce baseline values of pH and VFA in the 1<sup>st</sup> stage module, where the waste was only percolated with water without the biogas reactor connected. This was done by adding 450 kg of OFMSW and 400 L of water and a direct recirculation of the percolate to the waste. Due to the relatively small volume of the pilot-scale module, no structure material was added.



Figure 1.2: Loading of OFMSW into the pilot-scale process module

The pumps of the PEF were programmed to sprinkle and percolate the waste for 3 x 5 min. per hour with recycled water. With a pumping volume of approx. 50 L per pumping sequence, the total volume of percolate was about 3600 L per day, which means the percolate was recirculated 9 times per day. The extent of the waste hydrolysis was monitored by changes in pH profiles, VFA accumulation and total COD release. Each experimental run lasted for 4 weeks until no significant further changes were detected. Samples were taken every day in the first week of the experiment and later three times a week.

The results showed that the pH of the leachate became acidic (pH 4-5) within 2 days, stayed below 4.0 for the first two weeks of the experiment, and reached 5.5 at the end of the four weeks period. Replacing parts of the leachate (25 or 50 %) with an equal amount of water before recirculation



into the first module did not have any significant impact in raising the pH of the leachate to the desired pH range of 5.5-6.5 (Weiland 2010). Measurements of the COD concentration in the percolate revealed that only 26% of the whole COD content of the waste was released in the percolate after four weeks of percolation over the first stage.

## **WP2 Enhancing hydrolysis of OFMSW under different process conditions**

Improvements on the conversion efficiencies and specifically on enhancement of the hydrolysis in the first phase of the 2-stage biogas process were tested by different modifications of the process conditions, including different ratios of waste/percolation water and by maintaining a low pH in the processing (1<sup>st</sup> stage) module through advanced control of the pumping sequence between the 1<sup>st</sup> and the 2<sup>nd</sup> stage. Due to technical problems with the operation of the pilot-scale set-up, the optimization studies were for the most conducted in lab-scale reactor set-ups, but also large-scale testing was implemented in the project.

### **2.1 Lab-scale experiments – 2-stage versus 1-stage reactor configuration**

Lab-scale reactor experiments with different percolation regimes were performed both as 2-stage and as single stage reactor set-up to evaluate the overall conversion efficiency of OFMSW in a separate hydrolysis – methanogenic system with the combined 1-stage system. This was mainly to relate the efficiency of the hydrolysis phase to the overall potential for the conversion of OFMSW into biogas. In the 2-stage set-up continuous recirculation of the effluent from the second (methanogenic) stage to the first (hydrolytic) stage was tested while in the single-stage set-up the OFMSW was directly inoculated with methanogenic microorganisms and the percolate was directly recirculated into the reactor. Each trial was operated for at least 3 weeks and the release of VFA and COD, the pH and the methane production of the system were monitored.

The 2-stage set-up consisted of two reactors with 4.5 litres capacity (Figure 2.1). In the first (hydrolytic) stage reactor, OFMSW was loaded on a sieve (mesh size 1 mm), that was fixed to the reactor walls in the middle height of the reactor. After loading of OFMSW into the reactor, the reactor was closed and 1.4 L of water was sprinkled on top of the OFMSW. The second (methanogenic) stage reactor was filled with 2.6 L of inoculum. The waste/percolate ratio was adjusted for the different reactor experiments by varying the mass of OFMSW loaded to the 1<sup>st</sup> stage reactor while keeping the amount of percolate constant. Two different waste/percolate loadings were tested by loading (a) 500 g and (b) 125 g of wet OFMSW to the first reactor, corresponding to a waste/water ratio of 12.5% and 3.1% and a substrate/inoculum (S/I) ratio based on VS of 6.5, and 2, respectively. Leachate from the 1<sup>st</sup> stage reactor was continuously pumped into the 2<sup>nd</sup> stage reactor with a flow rate of 0.4 L/h. The reactor content of the 2<sup>nd</sup> stage was used as percolate for the 1<sup>st</sup> stage reactor by pumping 0.8 L of effluent from the 2<sup>nd</sup> stage every 2 h into the 1<sup>st</sup> stage reactor. Samples of the leachate from the 1<sup>st</sup> stage and of the effluent

from the 2<sup>nd</sup> stage were analysed for pH, and VFA release. The headspaces of both reactors were connected to a volumetric gas meter for measuring the biogas production of the 2-stage system. Samples for biogas composition analysis were taken with a syringe through a sampling port connected to the gas tubes.

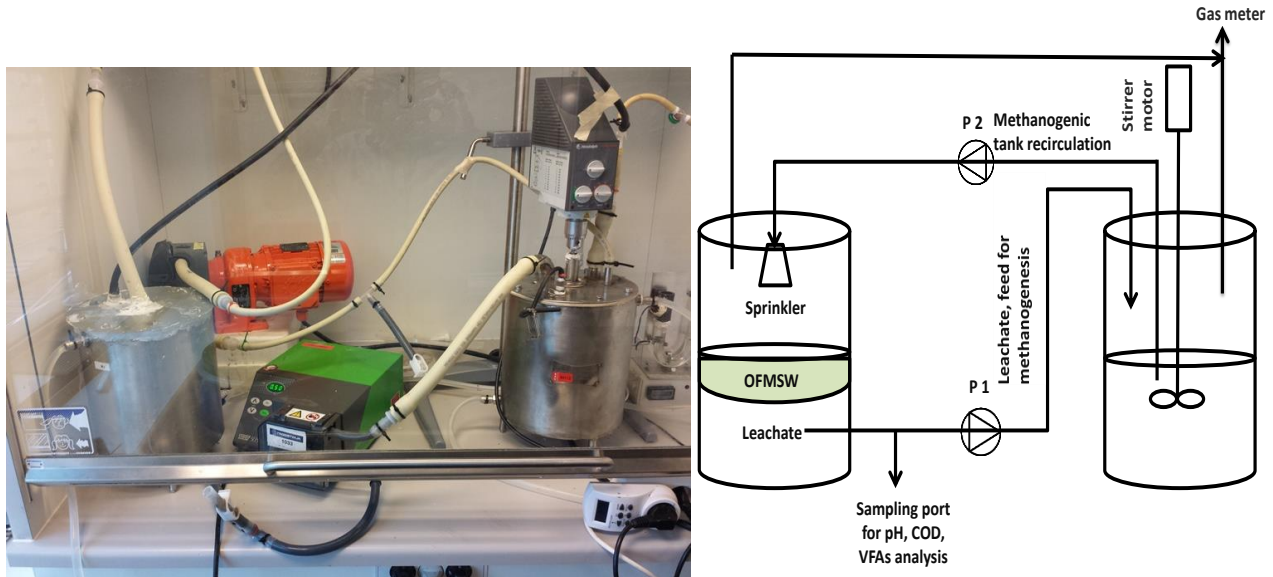


Figure 2.1: Lab-scale 2-stage set-up of OFMSW, involving recirculation and percolation between the hydrolytic 1<sup>st</sup> stage reactor (left) and the methanogenic 2<sup>nd</sup> stage reactor (right).

For the 1-stage set-up (figure 2.2), the same reactor as for the first stage of the 2-stage set-up was used, including the sieve for holding the waste. All liquid for percolation, 0.8 L water and 1.2 L inoculum, was in contrast to the 2-stage set-up filled directly together with the OFMSW into the reactor and the leachate from the reactor was percolated directly back on top of the OFMSW. Two different waste/percolate loadings were tested by loading (a) 250 g and (b) 125 g of wet OFMSW to the reactor, corresponding to a waste/water ratio of 12.5% and 6.2% and an S/I ratio of 6.5, and 4, respectively. Samples from the reactor leachate were taken for analysis of pH, and VFA release. A volumetric gas meter and a gas sampling port were connected to the reactor headspace to quantify the amount and composition of the produced biogas.

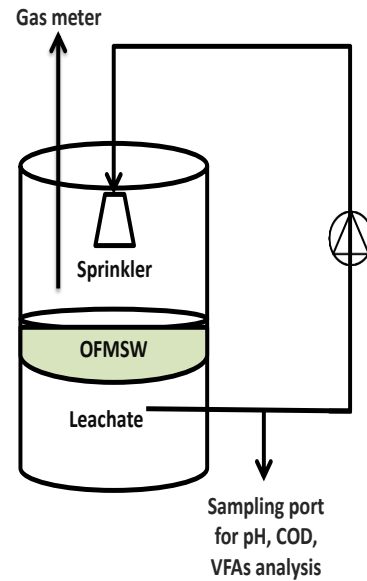


Figure 2.2. Lab-scale single stage reactor of OFMSW involving percolating and recirculation

For the 2-stage set-up, a high release of VFA was observed from the 1<sup>st</sup> stage for a high loading ( $S/I = 6.5$ ) while the methane yield was very low. In contrast, a final yield of 227 mL-CH<sub>4</sub>/g-VS was achieved in the overall system for the low load of only  $S/I = 2$ , but the VFA release from the first stage was much lower. This indicated that not only hydrolytic, but also methanogenic activity, converting the VFA into methane and CO<sub>2</sub>, occurred in the first stage and eventually the methane production of the whole system was dominated by the methane production in the first stage. In case of the high loading of the 1<sup>st</sup> stage, the hydrolytic activity in the first stage was obviously dominant and hindered the methanogens to establish in the first stage. The VFA release and conversion of these into biogas in the 2<sup>nd</sup> stage was, however, very inefficient, resulting in very low overall methane yields.

For the 1-stage set-up, the system showed also typical signs of organic overload with high VFA and low methane yields for the high waste loading of  $S/I = 6.5$ . In contrast, a stable AD process was rapidly established for a load of  $S/I = 4$  in the one-stage set-up. This revealed that the 1-stage process with higher water saturation enabled more suitable conditions for establishing both hydrolytic and methanogenic microbes. In the 2-stage set-up the hydrolysis and methanogenesis could obviously not be separated between the two reactors and the conditions for the methanogens were in both stages inferior compared to the 1-stage set-up. The overall results for the different set-ups are found in table 2.1



Table 2.1. Final methane yields achieved after 25 days of operation of the 1-stage and 2-stage reactor experiments with recirculation of percolate

Reactor configuration	OFMSW (g)	Percolate /inoculum (L)	Waste/percolate ratio	Methane yield (mL-CH <sub>4</sub> /g-VS)	% of theor. methane yield
2-stage	500	1.4/2.6	12.5%	31	8%
2-stage	125	1.4/2.6	3.1%	227	57%
1-stage	250	0.8/1.2	12.5%	29	7%
1-stage	125	0.8/1.2	6.2%	268	67%

It was concluded from these findings that for achieving a high efficiency in the 2-stage system the conditions in the first stage should be optimised to the hydrolytic bacteria (low pH) while in the second stage the process conditions should be optimal for the methanogens (high pH, sufficient retention time). Therefore, for further optimization of the 2-stage system the focus was on a controlled 2-stage reactor system with ensuring a low pH in the first stage and sufficient retention time in the second stage.

## 2.2 Full-scale test at Solum's AIKAN® plant – baseline experiment

The process scheme of the 2-stage AIKAN® process in large-scale is shown in figure 3. The waste is loaded into the 1<sup>st</sup> stage process module (PM) where the waste is irrigated with liquid from the biogas reactor tank (RT) (1) and liquid from the PM (3), which are mixed in the process tank (PT) prior to irrigation of the waste in the PM. In the PM, the complex organic matter is mainly hydrolysed to monomers and partly fermented to volatile fatty acids (VFA). The percolate drains out of the bottom of the module and is pumped back to the process tank (3) where it is mixed again with liquid from the RT (1). A portion of this mix is pumped back to the RT (4), wherein the 2<sup>nd</sup> stage (methanogenesis) takes place. The rest of this mix (2) is recycled to the process module for irrigation and percolation.

For the R&D experiment, the process module was filled with 112.3 tons of pretreated OFMSW that was mixed with 31.3 tons of inert structural material (shredded branches and roots from yard waste).

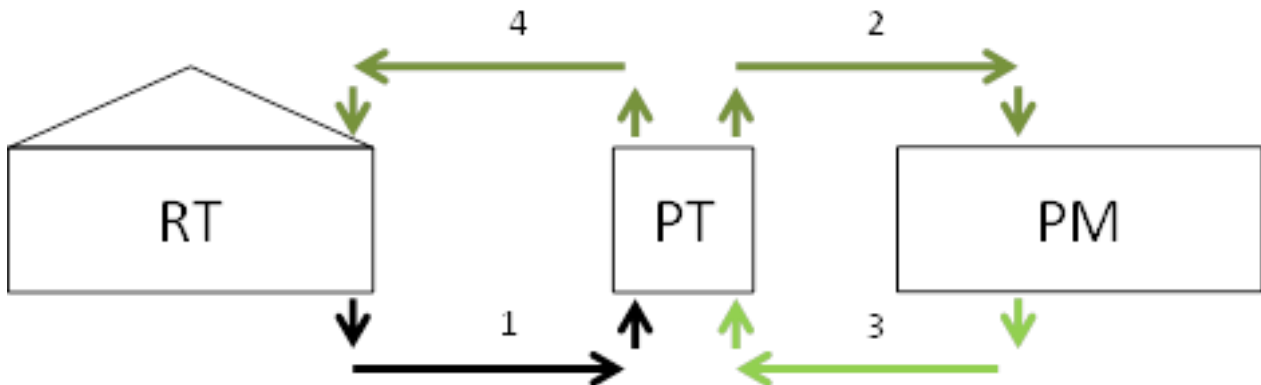


Figure 2.1. Liquid flow streams between the reactor tank (RT), process tank (PT) and process module (PM) during the Aikan biogas process. Black arrows (1) designate degasified reactor tank liquid, light green arrows (3) designate percolate loaded with energy rich organic compounds from the PM and dark green arrows (2 and 4) designate percolate mixed with reactor tank liquid.

The large-scale experiment was carried out for 32 days and samples of the leachate from the PM and of the effluent from the RT were collected 3 times a week for analysis of COD and VFA. The methane production in the PM and the RT was monitored continuously.

The percolation regimes of the experiment, which normally changes during the course of the biogas production phase, are shown in Table 2.2.

Table 2.2: The average liquid flow during different periods of the biogas production phase in the large-scale experiment. During the last period (day 20 - 28) the process module was flooded with reactor tank liquid submerging the OFMSW.

Process		Liquid flow, m <sup>3</sup> /day (average)			Total flow
Step	Flow	Day 0-4	Day 5-19	Day 20-28	Day 1 - 28
1	RT → PT	10.6	54.9	27.4	1290
2	PT → PM	31.8	40.8	32.2	1176
3	PM → PT	28.5	41.5	15	1027
4	PT → RT	7.6	56.0	15	1130

During the first 5 days the process module is mainly irrigated with recirculated leachate (31.8 m<sup>3</sup>/day) while the flow of reactor liquid to the process tank is relatively small (10.6 m<sup>3</sup>/day), see table 2.2. This procedure is changed in the period from day 5 to day 19, where a higher ratio of reactor liquid is pumped to the process tank. This may contribute considerably to the (undesirable) high pH in the process module during this period.

The organic matter, measured as chemical oxygen demand (COD) released from the processing module is highest in the start of the percolation and decreases with proceeding percolation (figure



2.2) COD in the reactor tank remains constant at about 20 g-COD/L, which is obviously particulate organic matter that is more resistant to the conversion into biogas.

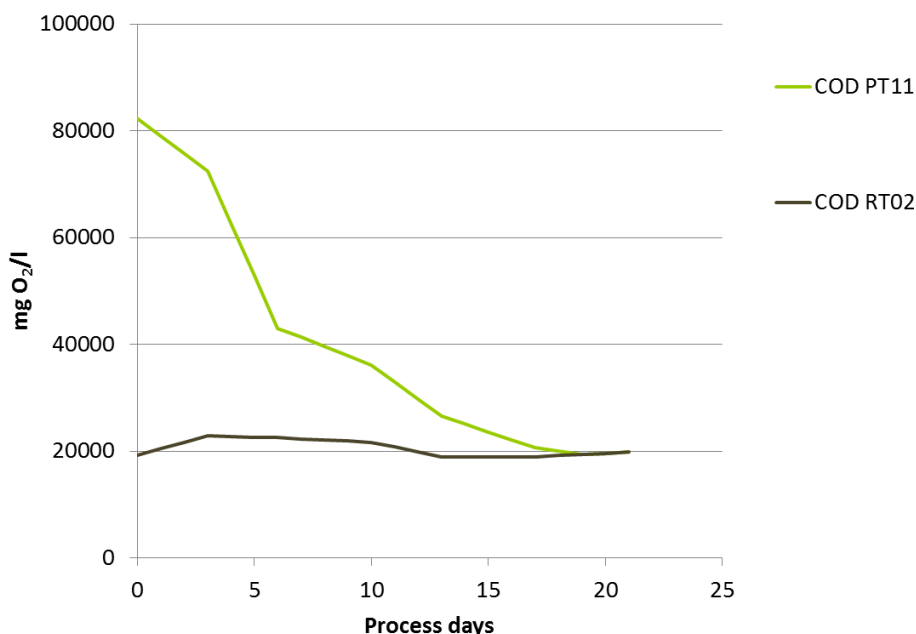


Figure 2.2. Concentration of organic matter, expressed as COD (mg O<sub>2</sub>/L) in process tank (PT11) and biogas reactor tank (RT02) during the first three weeks of the full-scale test.

Based on the flow data and the COD concentration measured during the different phases, the total COD release from the PM and the conversion of COD in the reactor tank was calculated. Figure 2.3 shows the accumulated amount of organic matter (as COD) that was loaded to the RT and the amount that returns to the PT during the first two weeks of the experiment. The difference (RT<sub>net</sub>) is the amount of organic matter measured as COD that has been converted to biogas in the RT.

The course of the biogas production shows that the main biogas production is produced in the RT, but after 14 days biogas production starts also in the PM (figure 2.4). This indicates that the PM got eventually inoculated with methanogens and not only hydrolysis, but also methanogenesis occurred in the PM. In total, 8.373 Nm<sup>3</sup> of biogas was produced during 32 days of operation, of which 4240m<sup>3</sup> was methane, revealing a methane content of 51% in the biogas. With a volatile solid (VS) content in the OFMSW of 27%, a biogas and a methane yield of 277 and 140 m<sup>3</sup> per ton volatile solids, respectively, could be calculated. These results show a conversion efficiency of about 34% of the theoretical methane yield (410 L-CH<sub>4</sub>/kg-VS) .

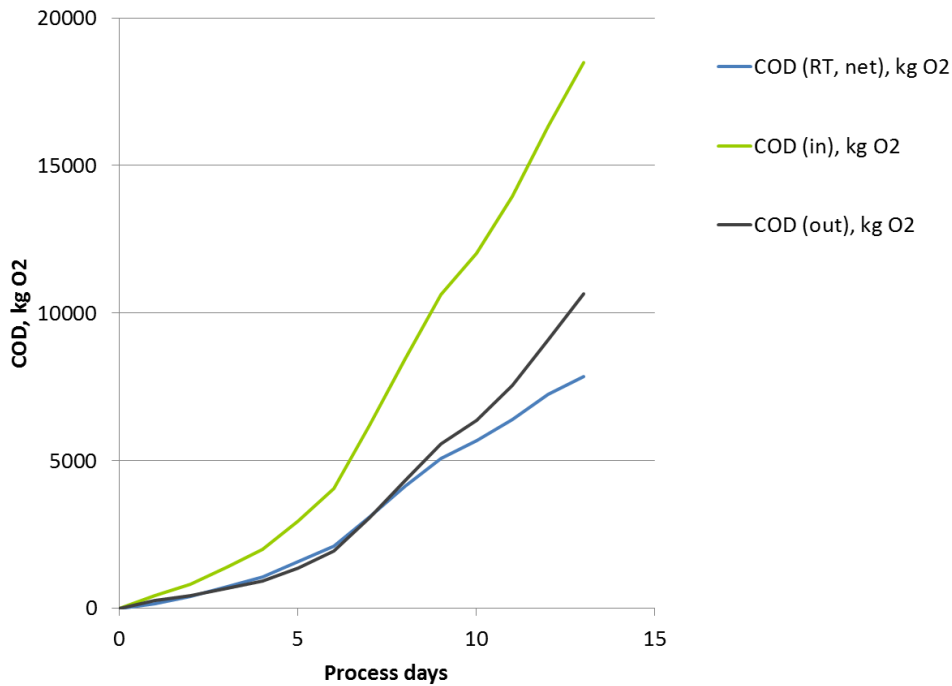


Figure 2.3. Accumulated organic matter loaded into the reactor tank (COD(in)), the amount leaving the reactor tank (COD(out)) and the COD converted into biogas as the difference (COD(RT,net)).

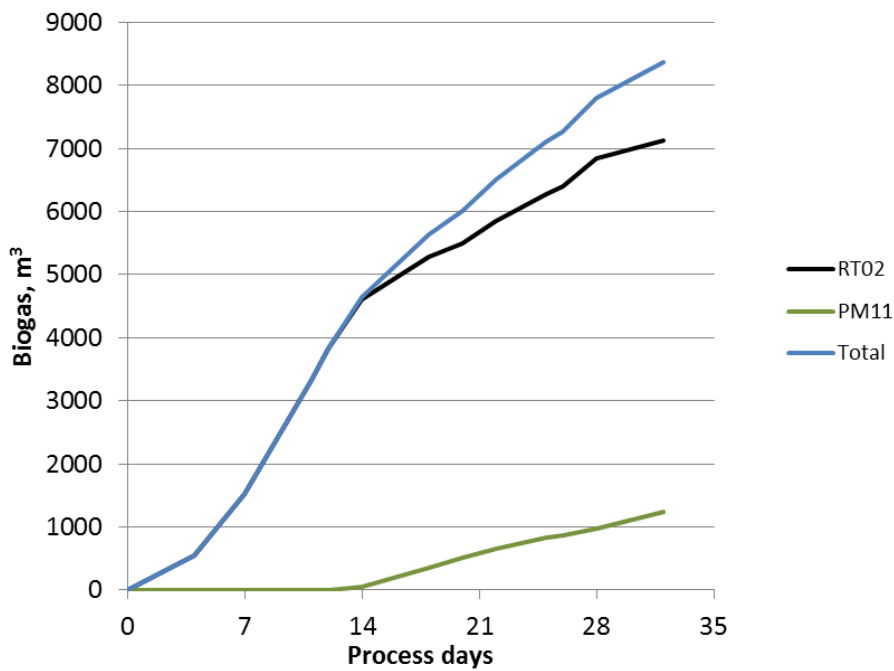


Figure 2.4. Total production of biogas and specific production in the reactor tank (RT02) and the process module (PM11).

### 2.3 Full-scale test at Solum's AIKAN® plant – different operational conditions

The efficiency of the hydrolysis process was tested in four different process modules, by monitoring pH and release of COD and VFA in the leachate of the PM. Samples of the leachate and the reactor liquid were taken immediately after starting the process and subsequently 3 times a week for operational periods of 7 - 13 days. The operation periods and respective flows between the PM and the RT are shown in table 3.3. The operational regime was very similar for all periods except the last, where the flow from PM to RT was 4.4 - 6.4 times higher and the flow from RT to PM was 5.9 - 7.0 times higher than in the other periods.

Table 2.3. Experimental set-up for the full-scale tests with OFMSW loading and flow regime between process modules (PM) and reactor tanks (RT).

Process module	Period (2014)	Process days	OFMSW (tons)	Flow (m <sup>3</sup> /day)	
				PM → RT	RT → PM
PM02	19/8 - 1/9	13	234.8	22.1	25.8
PM01	21/8 - 4/9	11	230.9	24.8	28.8
PM03	9/9 - 19/9	10	222.2	32.0	28.9
PM01	22/9 - 29/9	9	214.4	23.3	24.5
PM04	29/10-7/11	7	252.1	141	171

The results of the pH and the COD in the leachate of the process module (full circles) and from the reactor tank (open circles) for the different experiments are shown in Figure 2.5 and 2.6, respectively. The pH of the leachate from the process module rose from around pH 5 to about pH 8 during the first 10-12 days while the pH in the reactor tank was constant at pH 8.0 - 8.2 during the entire period (figure 2.5). This tendency was the same for all different experimental trials and slight differences in pH increase were not correlated to differences in the flow between the PM and the RT in the different experiments. The pH increase in the percolate from the PM was due to continuous percolation with effluent from the reactor tank with a high pH. The fact that a pH of 8 was reached after 14 days matches with the observation in the previous full-scale test that biogas production starts in the processing module after 14 days, indicating that both hydrolysis and methanogenesis were established in the PM.

Also similar to the previous full-scale experiment, the COD release from the OFMSW in the leachate from the PM was initially high (90.000 - 110.000 mg O<sub>2</sub>/l), but declined continuously during 14 days of operation (figure 2.6). The COD concentration in the leachate gradually approached the concentration of the RT after two to three weeks. This is also similar to the previous full-scale experiment and indicates that when finally methanogenesis has established in the PM, the VFA released in the PM was directly further converted to biogas and only measured in small amounts in the leachate from the PM.

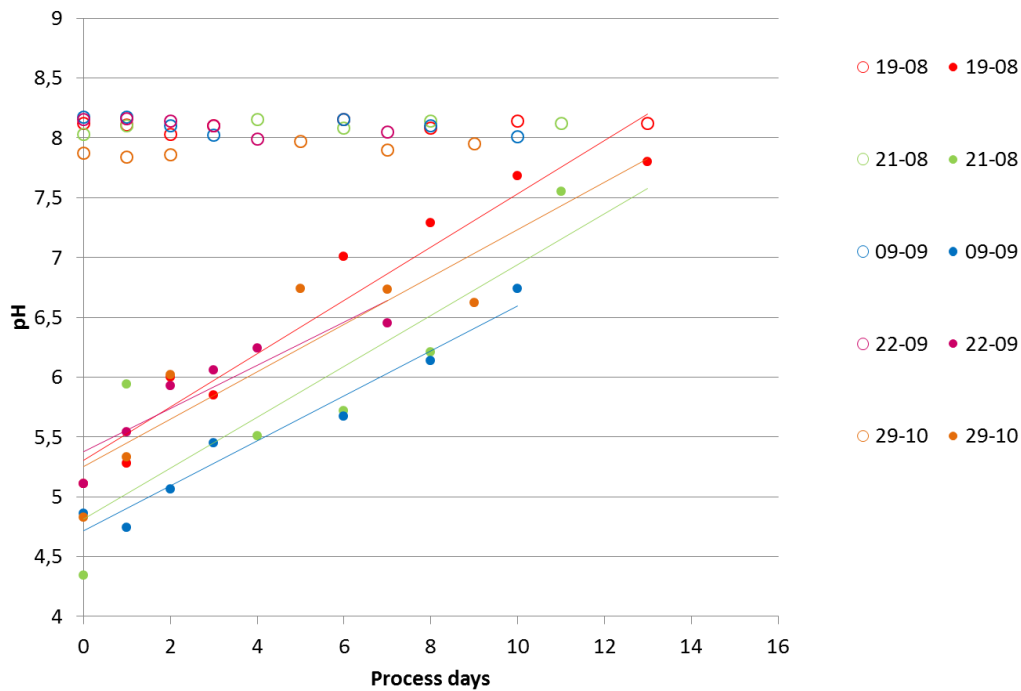


Figure 2.5. pH of the process module leachate (closed circles and regression lines) and from the reactor tank (open circles) during the first 7 - 13 days of five different process operations (table 3.3).

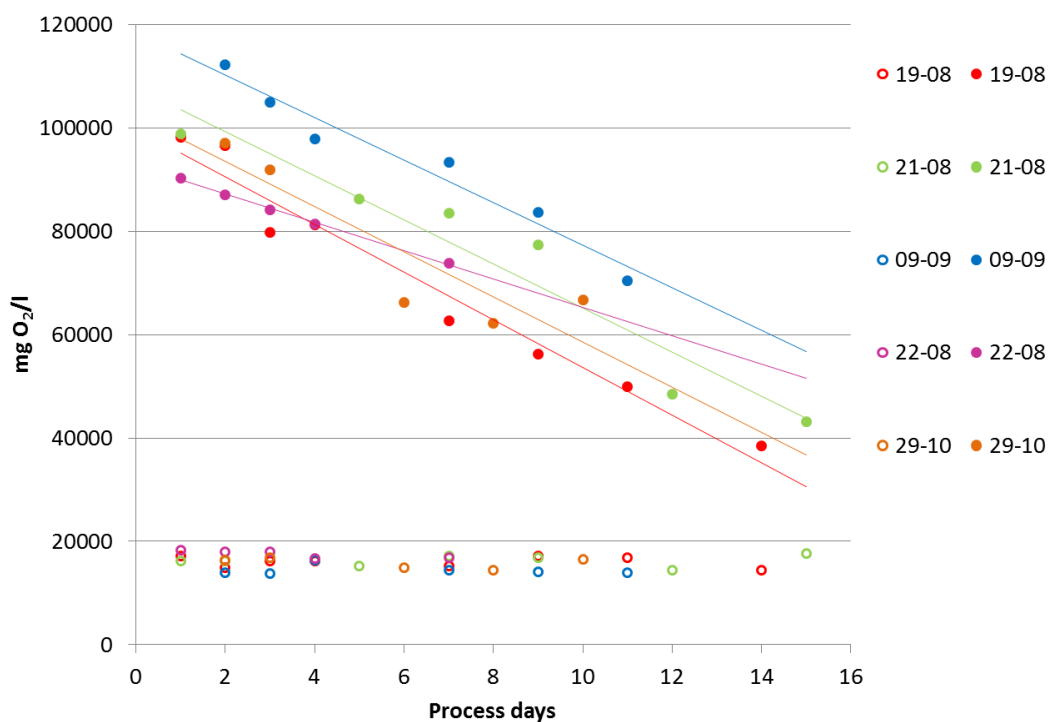


Figure 2.6. COD in the process module leachate (closed circles and regression lines) and from the reactor tank (open circles) during the first 7 - 13 days of five different process operations (table 3.3).

#### 2.4 Lab-scale experiments – Prolonging the hydrolysis in the first stage by automatic pH control

For testing an automatic pH control regime in order to prolong the hydrolysis in the first stage, the 2-stage lab-scale set-up was modified by connecting the 1<sup>st</sup> and the 2<sup>nd</sup> stage reactor with 2 pumps and 3 valves to enable switching between direct recirculation of the percolate into the 1<sup>st</sup> stage or to pump the leachate to the 2<sup>nd</sup> stage and using the effluent from the 2<sup>nd</sup> stage as percolate (figure 2.7). Pump A was used to pump the leachate from the first stage to either second stage by opening valve B and closing valve A or direct recirculation back to the first stage by opening valve A and closing valve B.

Pump B and valve C allow the passage of the digestate from the second stage to the first stage; therefore exploit the characteristics of the digestate from the 2<sup>nd</sup> stage as an alkaline buffer system to control the pH of the first phase. A control system to switch between direct recirculation of the leachate over the 1<sup>st</sup> stage or using digestate from the 2<sup>nd</sup> stage for percolation in the 1<sup>st</sup> stage was employed to regulate the pH in the first stage. A separate stirred tank (1 L volume) was inserted in the leachate recirculation line with a pH electrode-probe immersed in the leachate for on-line monitoring of the pH of the leachate. The remote control system (cRIO™ by National Instruments™) was programmed using LabVIEW™ software to create a Virtual Instrument (VI) to control the pumps and valves for different operational conditions:

- 1) Control of the pH in the first stage in the range of pH 4.5-5.5 through internal recirculation of the first phase (to decrease the pH) or by pumping digestate from the second-stage (to increase the pH).
- 2) Pumping leachate (feed) from the first phase to the second stage respecting a hydraulic retention time (HRT) in the second phase of 24 days.

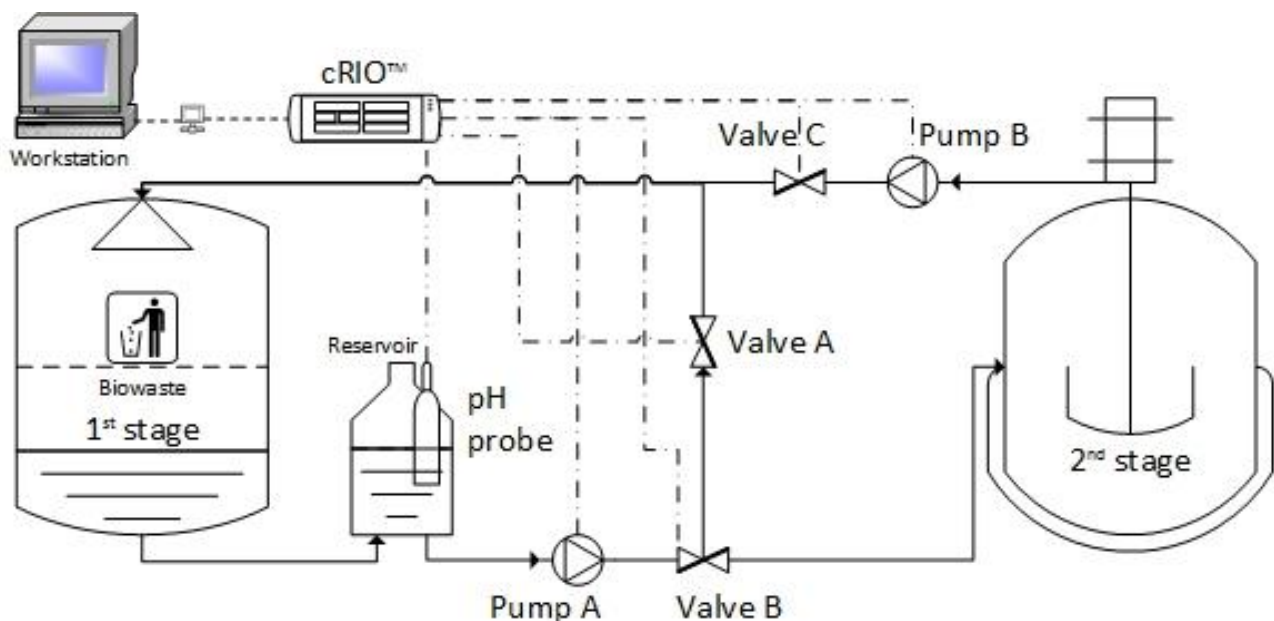
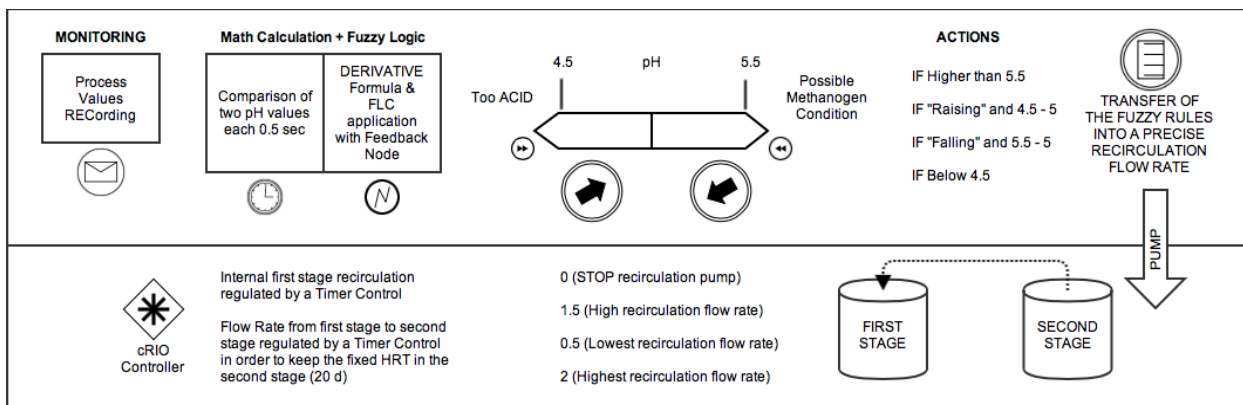


Figure 2.7. Two-phase lab-scale set-up with automatic pH control regime

Fuzzy Logic Control (FLC) was used for this process control task. A fuzzy control system is a control system based on fuzzy logic, which is a mathematical system that analyses analogue input values (e.g. pH electric signal) in terms of logical variables that take on continuous values between 0 and 1, in contrast to classical or digital logic, which operates on discrete values of either 1 or 0 (true or false). FLC was applied in four different stages (Figure 2.8). The first stage was used to record process values from the pH probe located inside the reservoir. The second stage reveals the fuzzification, to get a fuzzy set of fuzzy rules. Fuzzification means that inputs have to be converted into blurred linguistic terms, since the input parameters are too precise for human logic (e.g. the linguistic terms rising or falling for the pH). The rules of the FLC are formulated as a fuzzy set and this is the third stage after fuzzification. The numbers of the rules are determined as comparison of two pH input values per time (200 ms). The fourth stage is defuzzification, to obtain a Boolean result that activates pumps and valves depending on the flow rate required. Since the FLC provides the conclusion in linguistic terms, the result has to be converted to a number.



\* Feedback Node is analogue to a ( $Z^{-1}$ ) block in feedback control theory and digital signal processing

Figure 2.8. Principle of the Fuzzy Logic Controller (FLC) used for pH control of the 1<sup>st</sup> stage of the 2-stage lab-scale reactor system, programmed in LabVIEW™

For the 2-stage automatic control set-up in lab-scale, the first-stage reactor was loaded with 148.5 g of OFMSW and 2 L of tap water while the 2<sup>nd</sup> stage was filled with 2.5 L of inoculum (digestate from the AIKAN® full-scale process).

After a rapid decrease of the pH to pH 4.2 during the first 12 h of operation, the fuzzy logic control showed to be a very efficient tool to regulate the pH in the leachate of the first phase to an almost constant value of  $5.2 \pm 0.2$  (figure 2.9)

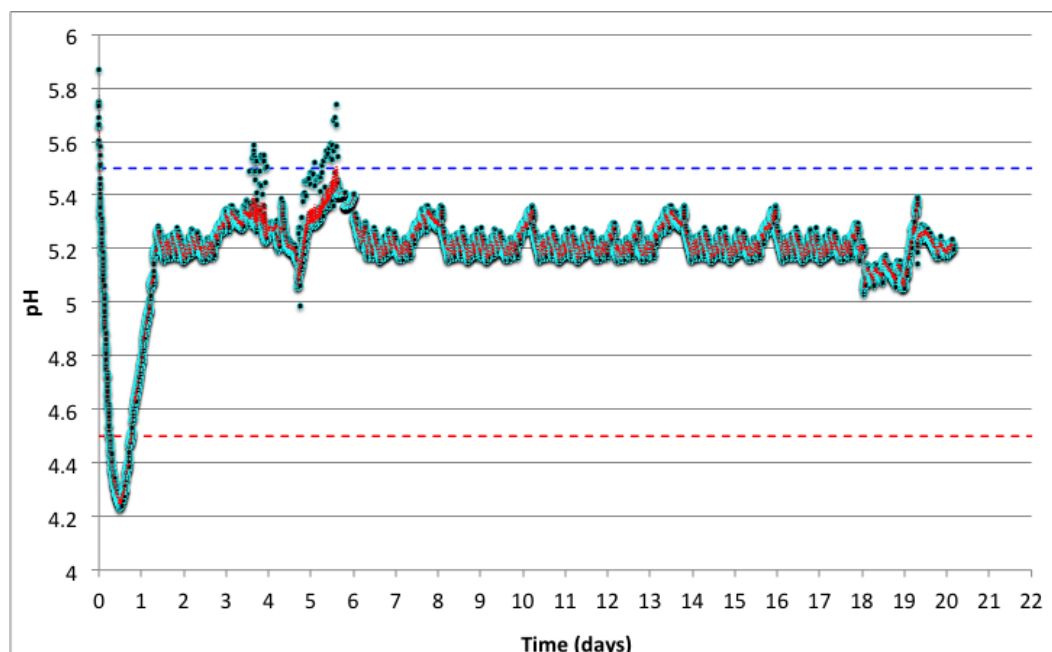


Figure 2.9. pH of the leachate of the 1<sup>st</sup> phase by applying fuzzy-logic automatic pH control of the first-stage

The release of total COD, soluble COD and VFA was steadily rising throughout the whole experiment, reaching a total tCOD, SCOD and VFA release of 30.1 g, 24.5 g and 22.7 g, respectively, after 24 days (figure 2.10). The total COD release of 30.1 g was equivalent to a COD conversion of the input COD of the waste of 71%. Soluble COD (SCOD) in the leachate was 89% of total COD and the VFA concentration was more than 90% of the SCOD and 83% of the TCOD.

The biogas production followed the release of VFA and COD in the leachate, i.e. steadily increasing during the first 12 days, stable from day 12 to day 24, and declined after day 24 (figure 2.11). In total, almost 14 L of biogas was produced with an average methane content of 65%, equivalent to an overall methane yield of 227 mL-CH<sub>4</sub>/g-VS (based on 148.5 g OFMSW with 27% of VS).

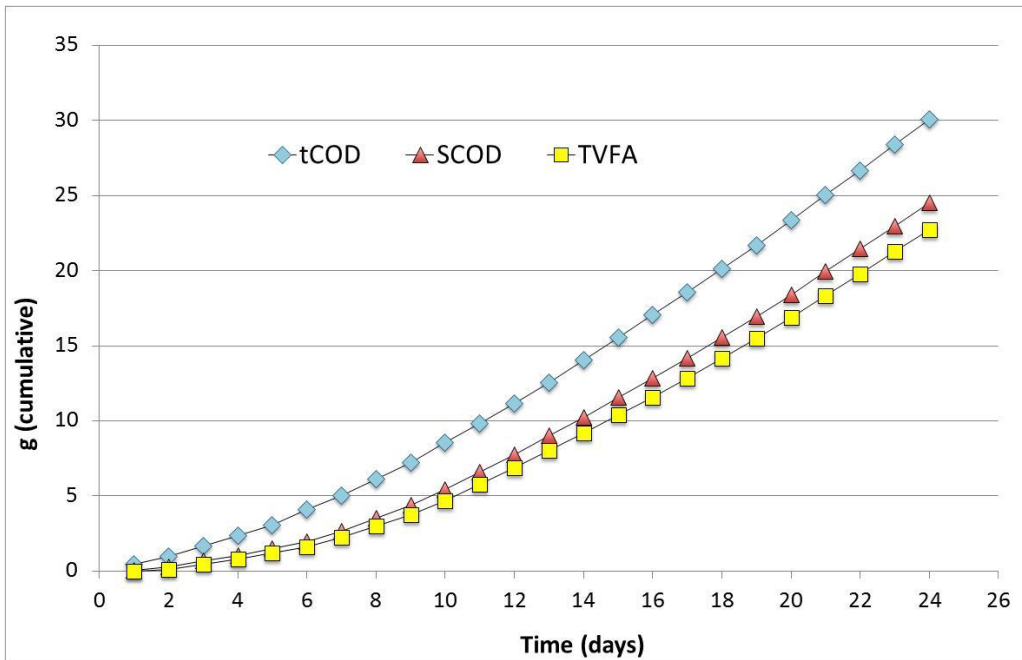


Figure 2.10. Cumulative Total COD, Soluble COD and VFA release in the leachate of the 1<sup>st</sup> stage reactor during automatic pH control

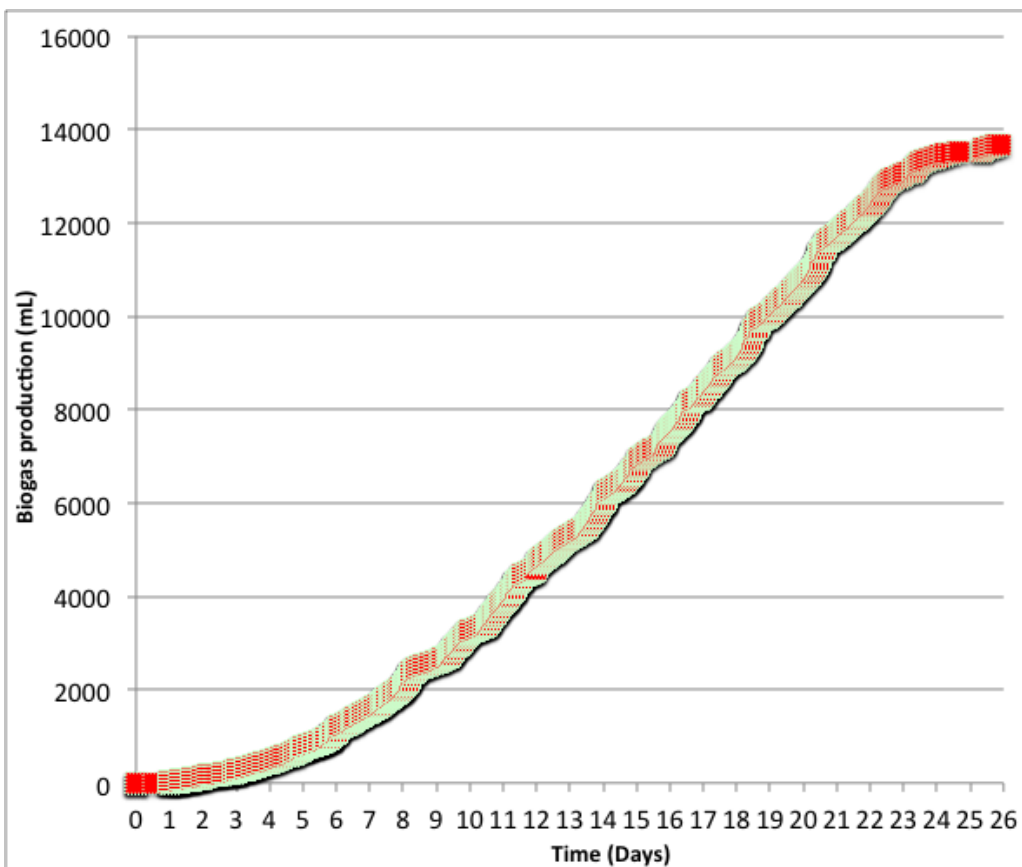


Figure 2.11. Biogas production recorded online during the automatic pH control experiment.



Analysis of the COD and VFA conversion into biogas in the 2<sup>nd</sup> phase showed that the methane yield reached more than 90% of the maximum theoretical methane yield (0.35 L-CH<sub>4</sub>/g-COD) after 8 days of operation and stayed stable with this high conversion efficiency (figure 2.12).

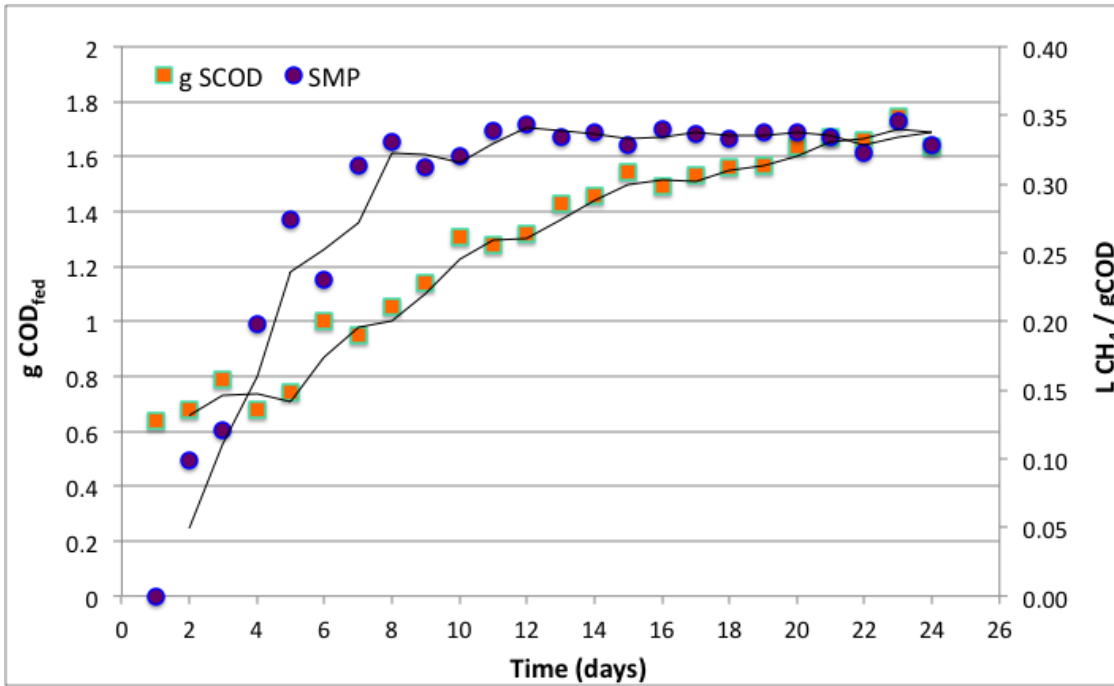


Figure 2.12. Concentration of soluble COD (SCOD) fed to the 2<sup>nd</sup> stage reactor and specific methane production (SMP) in the 2<sup>nd</sup> stage reactor during the automatic control trial.

Table 2.4. Comparison of process operation parameters with and without pH control of the 1<sup>st</sup> stage of the 2-stage reactor system

Reactor configuration	OFMSW (g)	Percolate /inoculum (L)	Waste/percolate ratio	Methane yield (mL-CH <sub>4</sub> /g-VS)	% of theor. methane yield
2-stage	500	1.4/2.6	12.5%	31	8%
2-stage	125	1.4/2.6	3.1%	227	57%
2-stage pH control	148.5	2.0/2.5	3.3%	227	57%



### WP3 Addition of enzymes

The effect of enzyme addition was studied in batch experiments with samples of OFMSW that were collected prior to the pilot-scale experiments. Different cellulases and xylanases were added to the OFMSW in different dosage for enzymatic hydrolysis at 50°C for 72 hours. After the enzymatic hydrolysis inoculum was added for anaerobic digestion and the batch vials were incubated at mesophilic temperature (37°C) for 45 days and the methane yield for the OFMSW was compared with and without addition of enzymes.

enzyme addition indicated a 10 - 19 % increase in methane yield against the controls where no enzymes were added. There was no significant difference noticed in the degradation profiles for applying the enzymatic treatment at pH 5.5 or pH 6.5.

Biomethane potential tests of OFMSW with addition of external enzyme blends with cellulase and hemicellulase activity for enhancing the hydrolysis of OFMSW resulted in an increase of 10 - 19% of the final methane yield, both for adjusting the pH at pH 6.5 and without adjustment at pH 7-8 during the enzymatic hydrolysis.

Enzyme addition could therefore be proposed to increase the process efficiency of the AIKAN® system. For deciding on the viability of the enzyme addition to increase the methane yield from OFMSW, the costs of enzymes for the used dosage have to be related to the specific increase in revenue from the sales of additional biogas.

### WP4 Conversion of xenobiotics

Compost produced from OFMSW and used as fertilizer and soil amendments in agriculture has raised concern about possible content of heavy metals and xenobiotics. Therefore, there has been also focus in the project on describing if and how xenobiotics are converted during the Aikan waste treatment process and, in particular, during the composting process.

Samples of approximately 2 kg were taken from a batch of fresh OFMSW at different time point during the Aikan processing. The first sample was taken from fresh OFMSW upon arrival at the waste treatment plant. The batch was subsequently being treated following standard operational procedure and a second sample was taken after one week of composting. Finally, a third sample was taken from the final compost product after the final screening for impurities.

The samples were analysed for their content of xenobiotics linear alkylbenzene sulfonates (LAS), polyaromatic hydrocarbons (PAH), di-(2-ethylhexyl)-phthalate (DEPH) and nonylphenol ethoxylates (NPE). The concentration of LAS are not shown, but was at all sampling points much lower (< 50 mg/ kg dry matter (DM)) than the threshold limit values of 1300 mg/ kg DM. The concentrations and dynamics of PAH, DEPH and NPE are shown in figure. 4.1.

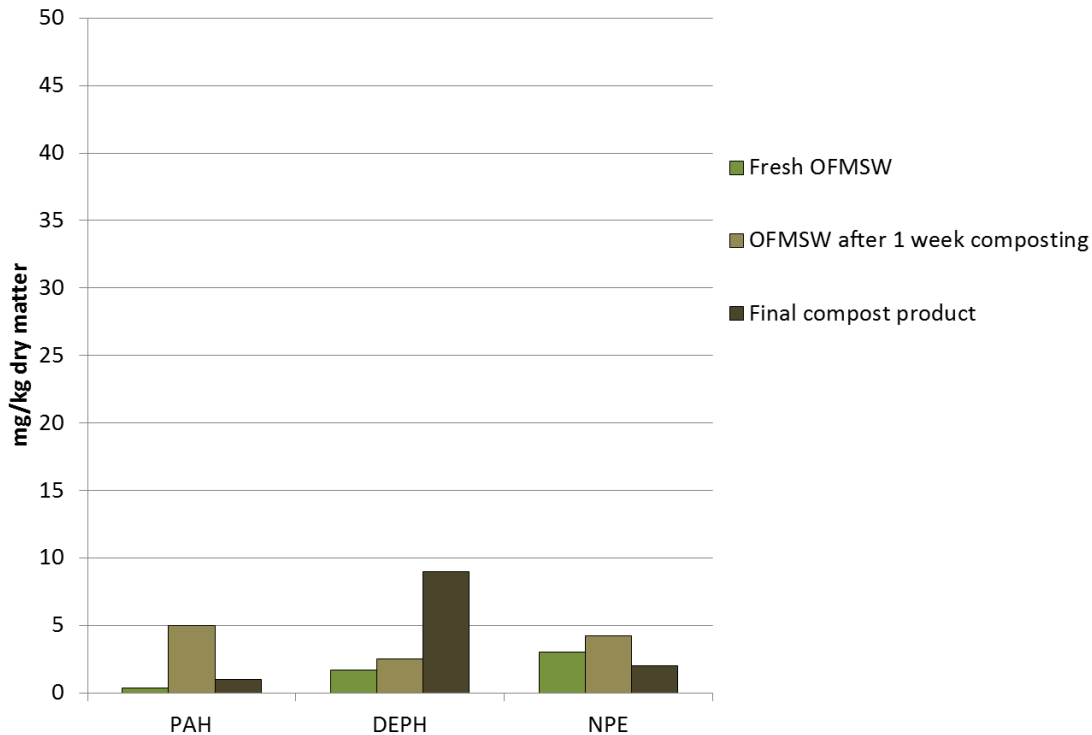


Figure4.1. Concentrations of the xenobiotics, PAH, DEPH and NPE found in Aikan compost at different stages of OFMSW processing.

All compounds show an increase in concentration after one week of composting compared to the fresh waste, indicating that the degradation of the dry matter is higher than the degradation of the measured xenobiotic compounds. In the final compost product, the concentration of PAH and NPE is much lower while the concentration of DEPH triples. This indicates that PAH and NPE are degraded during the composting process while DEHP remains persistent, at least its degradation is lower than the degradation of organic matter during composting. These findings coincide with previous findings on the fate of these xenobiotic compounds during anaerobic digestion (Hartmann and Ahring 2003). The concentrations of all compounds in the final compost product are, however, all lower than the respective threshold limit values of 3 (PAH), 50 (DEPH) and 10 (NPE) mg/kg DM.



## WP5 Conclusions and evaluation

Testing the energy yield efficiency of the 2-stage process under standard conditions showed an overall methane yield of 140 L-CH<sub>4</sub>/kg-VS for the full-scale plant and 31 L-CH<sub>4</sub>/kg-VS and 227 L-CH<sub>4</sub>/kg-VS in lab-scale for high and low loading of waste to the percolation, respectively. These values were equivalent to a conversion efficiency compared to the methane potential of 34% in full-scale and 8% and 57% in lab.-scale, respectively. It was found both in lab-scale and in full-scale, that by pumping effluent from the 2<sup>nd</sup> stage for percolation into the first stage, biogas production starts also in the 1<sup>st</sup> stage after 14 days of operation, pH has increased to above pH 7 and VFA and COD release into the leachate is considerably low. This all indicates that also the methanogenic process has established after 14 days in the 1<sup>st</sup> stage/process module. Compared to the lab-scale 1-stage reactor experiments it shows, however, that the whole conversion of the OFMSW into biogas in the 1<sup>st</sup> stage is only suboptimal and has a low efficiency especially for a high waste/percolate ratio. The overall methane yield could only be enhanced by lowering the waste/percolate ratio from 12.5% to 3.1%. Increasing the leachate recirculation by a factor of 4.4 – 7.0 in the full-scale did not significantly change the process performance as earlier found by Veeken et al. (2000).

Applying automatic pH control in the operational regime by a shift between direct recirculation in the 1<sup>st</sup> stage or using digestate of the 2<sup>nd</sup> stage for percolation the pH in the 1<sup>st</sup> stage could be kept low at pH 5.2±0.2 over the whole operation period of 25 days. In parallel, VFA and COD release in the leachate was also kept high over the whole period, indicating that high hydrolytic activity was maintained throughout the whole period and methanogenesis did not establish in the 1<sup>st</sup> stage. The COD released from hydrolysis of OFMSW in the 1<sup>st</sup> phase was converted into biogas in the 2<sup>nd</sup> phase with more than 90% efficiency. The results show that the energy efficiency of the 2-stage AIKAN<sup>®</sup> process can be increased from 34% to almost 60% by either higher water saturation in the processing module with a waste/water ratio of 3.1% or by keeping the pH in the 1<sup>st</sup> stage to pH 5.2 by applying automatic pH control and prolonging the period of direct recirculation of the leachate to the process module. The yield of methane per ton of OFMSW could be enhanced from 38 m<sup>3</sup> to 61 m<sup>3</sup> by these measures. Enzyme addition could increase the yield by 19%, i.e. 7.2 m<sup>3</sup> and 11.6 m<sup>3</sup>, respectively, but would have this effect probably only together with the other measures to secure good contact between enzymes and waste and when maintaining the pH low.

These benefits in higher methane yield have to be counterweighed to the costs for additional pumping and employing automatic pH control. However, the implementation of automatic pH control at the full-scale AIKAN<sup>®</sup> plant would mainly need installing of the software and a pH probe in the percolate tank while the existing pumps and valves should be sufficient.

Furthermore, by achieving higher conversion efficiency, the amount of residual organic matter at the end of the biogas process phase of the AIKAN<sup>®</sup> would be reduced by 21%, meaning that the energy needed for waste aeration in the following composting process could also be reduced by



21%. The final amount of compost produced would, however, probably be about the same since the organic matter that is not degraded during the biogas process under standard conditions is probably degraded during the composting process.

The analysis of xenobiotic compounds reveals that these compounds are mainly degraded during the composting process. An increased degradation of organic matter during the biogas process phase may lead to a higher concentration of the xenobiotics per g-TS. In the following composting process it can, however, be expected that the xenobiotics will be degraded in the same degree as measured during the current process operation.



## Dissemination of project results

- ✓ Nasir, Z., Kristensen, M.B., and Uellendahl, H. (2014). Enhancing the hydrolysis process of a two-stage biogas technology for the organic fraction of municipal solid waste. Biogas Science Conference, Vienna, 27-29 October 2014 (Oral presentation, under submission to journal).
- ✓ Nasir, Z., Ahring, B.K. and Uellendahl, H. (2015). Enhancing the hydrolysis process of a two-stage biogas technology for the organic fraction of municipal solid waste. (Article in preparation for international scientific journal).
- ✓ Micolucci, F., Nasir, Z. and Uellendahl, H. (2015). Automatic process control of two-phase dry anaerobic digestion of biowaste for hydrolysis optimization and biogas enhancement. (Article in preparation for international scientific journal).



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