A TOTAL CONVERTING AND BIOSAFE LIQUEFACTION COMPARTMENT FOR MELISSA

TECHNICAL NOTE: 86.1.4

Evaluation of potentially improved substrates by batch fermentation tests

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1. Introduction

This technical note presents the current state of the MAP project "A Total Converting and Biosafe Liquefaction Compartment for MELISSA" on behalf of the Laboratory for Microbial Ecology and Technology at the University of Ghent.

As agreed upon in the latest progress meeting in Hamburg (4 July 2002), the mesophilic digestion efficiency of the raw substrate was reconfirmed at various retention times in terms of carbon mass balances. A second part shortly describes the 3rd closed loop experiment and the substrate distribution of the residue to the different partners. In this respect, the returned substrates of the 2nd closed loop experiments (*Fibrobacter* residue and the sub-critical liquefaction residue) were further evaluated in terms of biogas yields at lower loading rates. A third part of this report will deal with the design and description of a biofilm methanogenic reactor for the bioconversion of soluble metabolites at low retention times (1-5 h). Initial results of this biofilm methanogenic reactor are presented in this TN for the *Fibrobacter* effluent and the thermal liquefaction residue.

2. Objectives and task description

In the latest progress meeting, all involved parties expressed their consent about the fact that the main objective of the last phase of the project was not the continuous physical coupling of the different units but the demonstration of a highly converting and biosafe liquefaction compartment for MELiSSA. To achieve these objectives, it was decided that within the terms of the current project phase, the processes will not be connected but the output samples of one process will be used as inputs of the next process ("closed loop" experiments). As a result, the data contained in this report do not agree with the tasks as described in WP 1.400 but rather focus on the further evaluation of potentially improved substrates from Partner 2 (*Fibrobacter* residue) and Partner 4 (sub-critical liquefaction residue) by means of the CSTR and the biofilm methanogenic reactor.

In Figure 1, the conceptual scheme of a total converting liquefaction compartment as designed and agreed upon in the latest progress meeting is depicted. The concept combines three technologies being methanogenesis, *Fibrobacter* liquefaction and thermal sub-critical liquefaction. Based on this concept, a 3rd closed loop experiment has been performed in which 200 g of DM residue of Partner 1 (methanogenesis) was distributed to Partner 2

(*Fibrobacter* residue). Next, the supernatant of the *Fibrobacter* unit was returned to Partner 1 for subsequent biogasification in a biofilm methanogenesis unit. The most inert fibrous fraction was then sent to Partner 4 for final liquefaction and would be recycled in the high-load methanogenic reactor of Partner 1. However, in order to evaluate the potential toxicity of both the *Fibrobacter* and the thermal liquefaction effluent, both effluents have been evaluated for their biogas potential in the biofilm reactor (section 4.2.2 and 4.3.3).

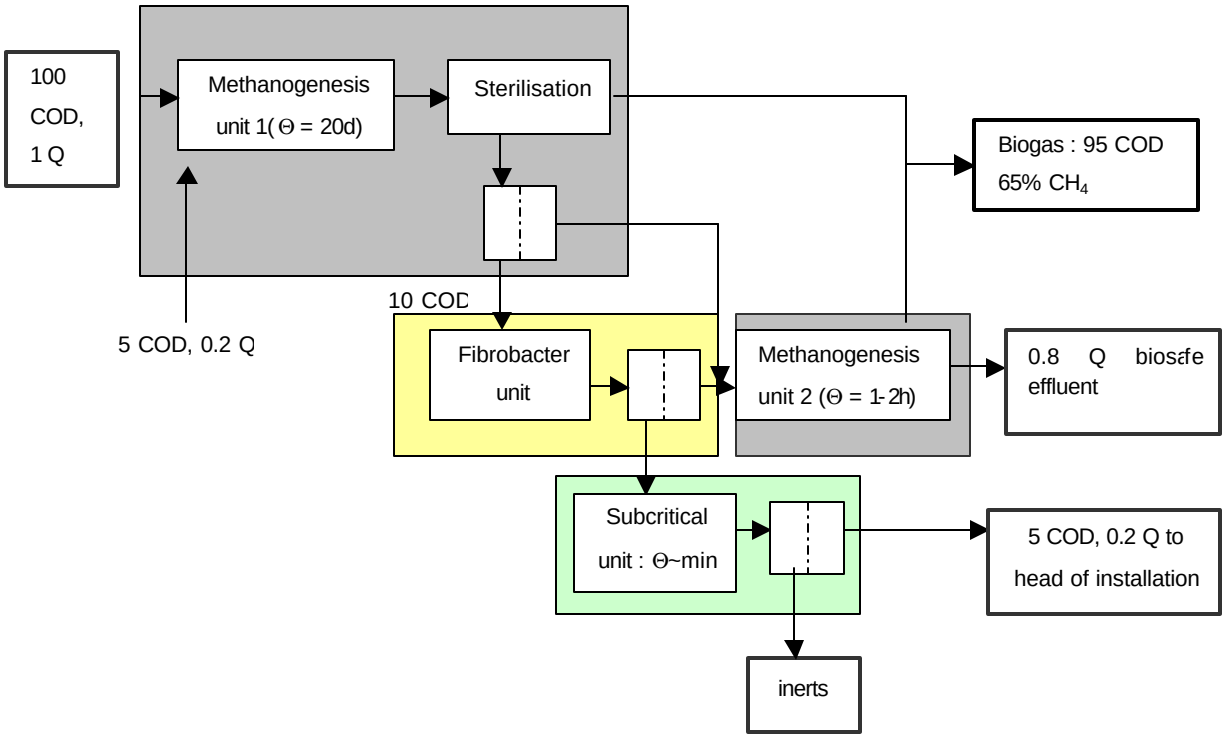


Figure 1: Conceptual scheme of a total converting and biosafe liquefaction compartment for MELiSSA

Prior to the 3rd closed loop experiment, further batch fermentation tests were performed with the residues from Partner 2 and 4 from the previous closed loop experiments.

The raw synthetic substrate was first digested at mesophilic temperature (33°C) and the fibrous residue was distributed between Partner 2 and 4 for subsequent degradation in their respective unit. Second, the digested fibers were returned to Partner 1 for a second mesophilic digestion. In this way, differences between the different substrates (and consequently between the different units) could be further evaluated.

As shown in Figure 1, the latest reported conversion efficiency of the mesophilic digester accounted for 80% of the raw substrate in to biogas at a retention time of 25 days, leaving 15-

20% of mainly undigested fibrous residue. Similar with TN3, the further liquefaction and characterisation (hydrolysis) of this residual fraction by the liquefaction units of the different partners was the main objective of this phase of the study.

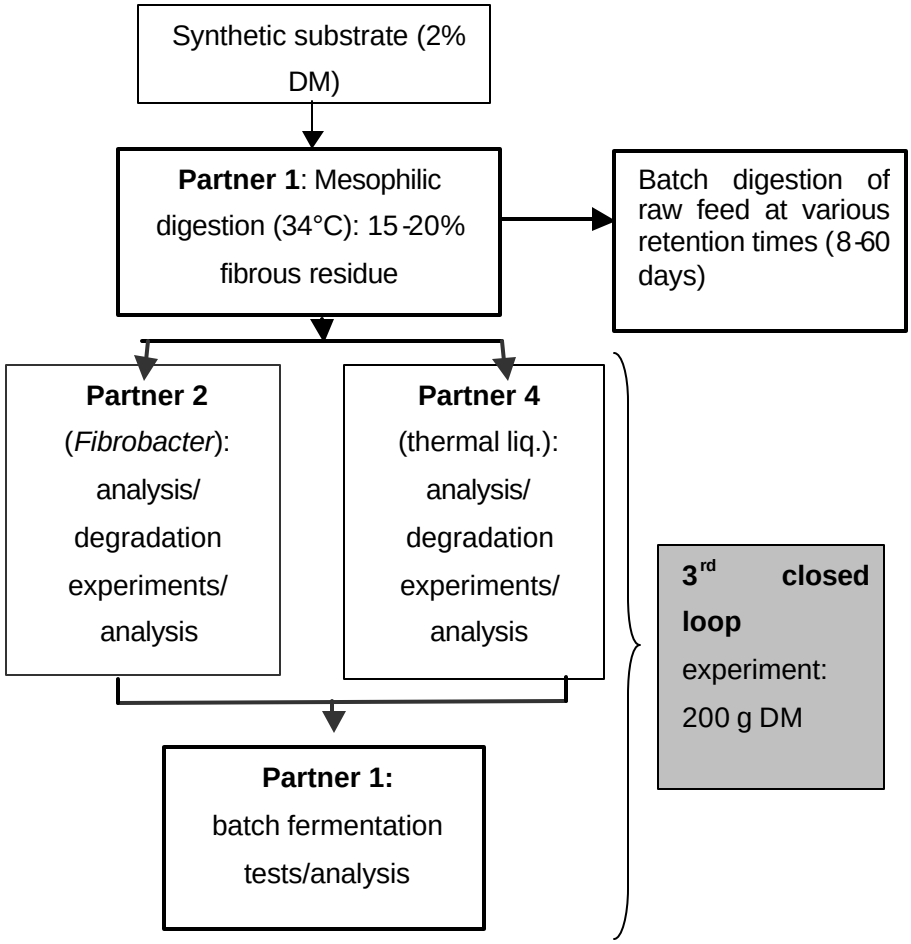


Figure 2: Performance of 3rd closed loop experiment

3. Materials and Methods

1. Experimental set-up of the high-load methanogenesis unit

3.1.1. Mesophilic digester

A 10 Liter anaerobic PVC-reactor is used for the anaerobic digestion of the defined feed. As indicated in Figure 2, the digester is maintained at a constant temperature of 34°C by placing

it in an incubator. The reactor is a CSTR-type (continuously stirred tank reactor) and is shaken two minutes/hour on a shaker platform (INNOVA shaker) at a constant 90 rpm.
The feeding of the reactor is fed-batch wise at regular time intervals. For each volume of the feed fed to the reactor, a same volume of stirred mixed liquour is withdrawn simultaneously. The biogas passes by an electronic milligascounter device (Fachhochschule Bergedorf, Hamburg -Harburg, Germany) with a resolution of 1 ml and an accuracy of 3%. The biogas composition has been monitored during the preparation and fermentation of the batch fibrous residues.

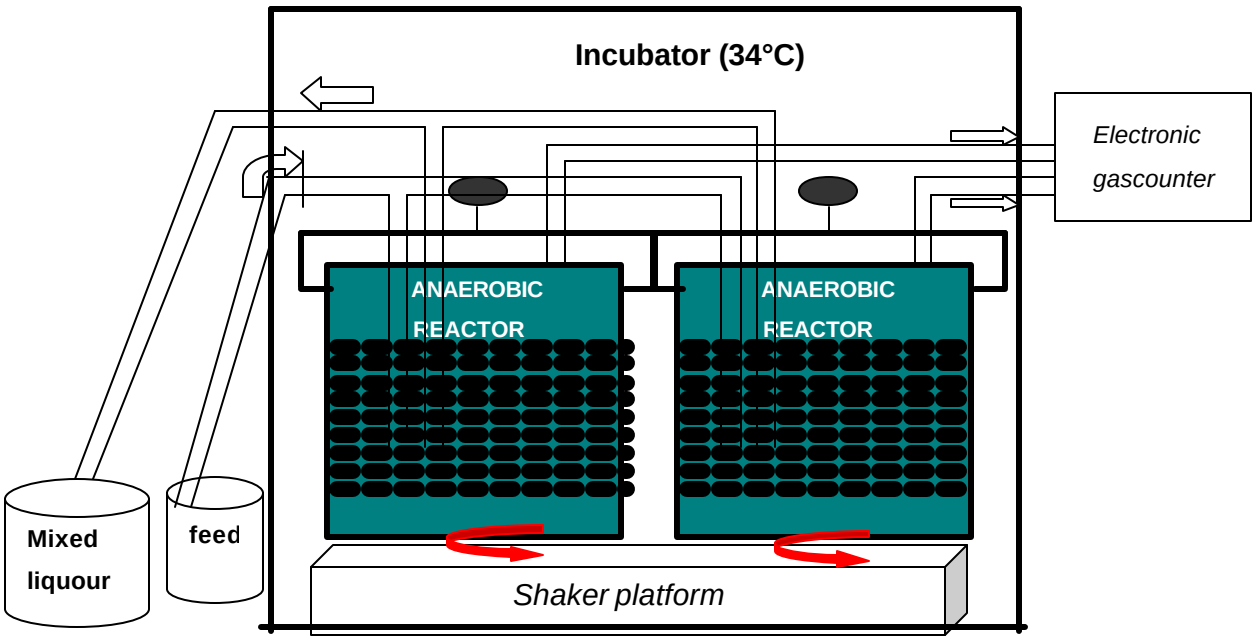


Figure 3: Scheme of the 2 mesophilic digesters for the fermentation and subsequent distribution of the synthetic substrate

The volumetric loading rate of the mesophilic digester was held at 2.17 g COD/L.day (Chemical Oxygen Demand) over a period of 2-3 months in order to obtain the necessary amount of fibrous residue (200 g DM (Dry Matter)) to distribute to Partner 2. Reactor performance was stable at the given volumetric loading rate.
The dry matter content of the synthetic feed was kept at 2% dry matter. The reactor was fed in quantities of 0.5 L feed/day. In order to maintain a hydraulic retention time of at least 15 days, the liquid reactor volume of both reactors was set at 7.5 L.

3.1.2. Batch fermentation tests

Additional fermentation tests were set up with the *Fibrobacter* residue and the thermal liquefaction residue with anaerobic sludge (mixed liquour) of the main methanogenic digesters. All experiments were performed in 2 L bottles containing a fixed amount of mixed

liquour from the two main mesophilic reactors. The volume of mixed liquour present in each batch bottle was either 400 mL (small batch tests) or 800 mL (large batch tests) depending on the organic strength of the substrate applied. The mixed liquour contained a solid phase, existing both of flocculated non-granular sludge (methanogenic bacteria) and residual fibers from previous fermentations. The liquid phase consists mainly of soluble biopolymers. The experiments were run over a period of 5 days, corresponding to hydraulic retention times varying from 15 to 60 days.

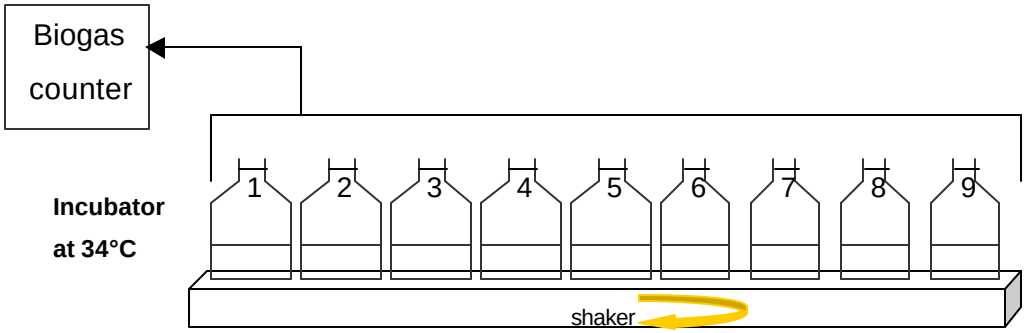


Figure 3: Experimental set-up for batch fermentation tests with raw feed, *Fibrobacter* residue and thermal liquefaction residue

Bottle 1 was used as control and therefore contained only mixed liquour from one of the main digesters. To bottles 2-9, the *Fibrobacter* residue and thermal liquefaction residue were added at various loading rates (expressed as g COD/L.d) and at various retention times (expressed as days). The residues were added only at the start of the experiment in amounts ranging from 40 mL till 150 mL, representing absolute initial COD-loading rates of 0.1 g till 2.38 g per test bottle. The volume of biogas and pH was continuously measured for each bottle. All fermentation trials were performed in duplicate or triplicate to check for the reproducibility.

Bioconversion efficiencies were calculated based on the general assumption that 1 g of COD can be transformed in 0.5 L of biogas. These yields could be confirmed by COD analysis and DM analysis.

2. Substrate composition and preparation of residue (3rd closed loop)

3.2.1. Substrate composition

The composition of the 2% DM substrate was similar to the previous TN's:

10% DM *Spirulina* (95%DM): 2.85 g/L

24% wheat straw (95%DM): 6.65 g/L

22.5% fresh cabbage (9%DM): 6.3 g/L
22.5 % soya (90%DM): 6.3 g/L
21.5 % faeces (10%DM): 6 g/L

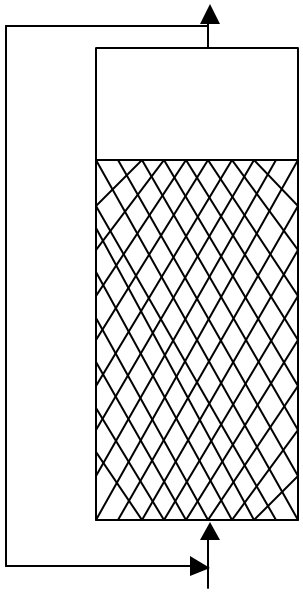
The substrate preparation method was identical to the one described in TN2.
After a first fermentation, the effluent was centrifuged at 7000 g for 15 min leaving a fibrous residue and a supernatant phase. Next, the fibrous residue was dried at 104°C to remove the incorporated water. Part of the supernatant phase was recovered.

3.2.2. Substrate preparation prior to Fibrobacter fermentation

In order to avoid contamination of the *Fibrobacter* culture, both the supernatant phase and the dried fibrous residue were sterilised by means of autoclavation. All materials were packed and distributed to Partner 2 for further liquefaction.

3. Fixed-bed biofilm methanogenic reactor

For the construction of methanogenesis unit 2 (see Figure 1), a fixed bed methanogenic reactor seems the most suitable reactor type. Fixed and fluidized bed methanogenic reactors offer significant advantages over conventional mixed reactors such as 1) less volume and space, 2) higher stability towards changes of pH, flux or substrate concentration. These type of reactors are particularly suited for the bioconversion of agricultural wastewater typically rich in carbohydrates (e.g. starch, cellulose, hemicellulose, ...). For highly biodegradable wastewater, conversion efficiencies as high as 90% have been reported.
The concept of the fixed-bed biofilm reactor is shown in Figure 4.



- *height/diameter ratio of 2 :1*
- *organic loading rates ranging from 1-20 g COD/L.day*
- *recycle mode for substrate dilution*
- *plastic rings as support material*
- *VFA' s should contribute for majority to COD-content of influent*
- *Upflow fluid velocity of 1-2 m/h*

Figure 4: Fixed bed methanogenic biofilm reactor with plastic support material

To achieve high conversion efficiencies, fatty acids should preferentially make up more than 80% of the influent COD. Otherwise, rapid growing of acidifying bacteria could easily overgrow the methanogenic biofilm and, hence, reduce the methanogenic activity. In this respect, the *Fibrobacter* liquefaction step prior to the fixed bed methanogenic reactor is expected to be very beneficial. As a matter of fact, previous results (TN3) showed already that acetate and propionate are main metabolites produced during *Fibrobacter* liquefaction.

For this specific experiment, the fixed-bed biofilm reactor had a volume of 1.5 L. The reactor was filled with 1 dm^3 of polypropylene fibres, with a specific surface of ca. 500 m^2/m^3. To initiate the biofilm formation, 1 L of tapwater and 500 mL of sludge from the CSTR was added. Subsequently the liquid was continuously recycled at an upflow velocity of 2 m/h and at daily basis 5 g COD/L.day was dosed during a period of 8 weeks. Subsequently, the excess of (free) sludge was removed from the reactor. The methanogenic activity during the experiments is thus mainly propagated by the biofilm. Only minor amounts of the CSTR-sludge couldn't be decanted, because part of the sludge was trapped within the matrix of the polypropylene fibres. The initiation of the biofilm and the experiments were performed at mesophilic temperature ranges.

After the start up period of the fixed-bed biofilm reactor, the returned effluent from the Fibrobacter digestion and the sub-critical liquefaction was added to the fixed-bed biofilm

reactor and continuously recirculated with a upflow velocity of 2 m/h. The biogas production and parameters as COD_t, COD_s, VFA and pH were followed on daily basis, during a total period of 96 h per experiment.

4. Results and discussion

4.1. Biomethanization of raw substrate at various loading rates and retention times

In a first series of experiments, batch fermentation tests were set up with raw feed at retention times varying from less than 20 days up to 65 days. For an applied initial loading of 1,85 g/L COD (with a 1,84% DM raw feed) performed in triplicate, biogas yields varying from 25% (RT = 13 d) up to 90% (RT = 65 d) were achieved based on COD and DM analysis (Figure 5). These results indicate that the retention time is a crucial factor in the conversion efficiency of the raw substrate into biogas. This in turn shows that the hydrolysis of complex biopolymers present in the waste is rate-limiting for the biomethanization of the raw waste. Although more batch tests would be required at various retention times to derive a real trend from Figure 5, it should be clear that an increase in biogas yield as a result of an increase of the retention time is most pronounced in the lower retention time range (15-35 days).

Compared to the conversion results obtained from the main CSTR digester, the conversion rates in the batch fermentation tests are lower at the same retention time. While 80% conversion for the CSTR was recorded at a retention time of 23 days, 66% of the COD influent could be converted in the small batch test at the same retention time. The lower values in the small batch tests are a result of the fact that a control was taken into consideration while this was not the case for the CSTR reactor. The control accounted for biogas production resulting from residual organic matter of previous substrate supplies and from endogenous metabolism. Consequently, biogas production from residual organic matter (or at higher RT) could not be excluded during CSTR performance.

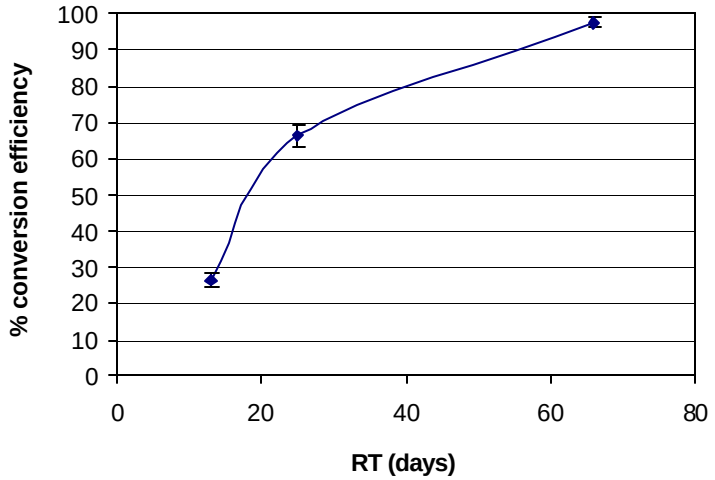


Figure 5: Conversion efficiencies for raw substrate at various retention times

From Figure 5, it can be derived that the retention time and hence, the volume of the reactor, will be a crucial parameter for the conversion efficiency reached in the methanogenic CSTR. In order to achieve conversion efficiencies higher than 90%, a trade-off should be made between the reactor volume that can be permitted and the degree of implementation of additional liquefaction/hydrolysis technologies (*Fibrobacter* and thermal liquefaction in particular). The reactor volume will therefore be an important factor to consider with regard to the optimization of the liquefaction compartment schematized in Figure 1. Alternatively, the decoupling of the hydraulic retention time from the solids retention time also permits higher methane yields at equal reactor volumes. This option will be investigated in the near future.

Figure 6 shows the biogas production at variable initial loadings. Assuming a general methanogenic activity of 0.5 L biogas production from 1 g of COD, it can be deducted that at a retention time as high as 75 days, the raw feed can nearly completely be converted into biogas at all loading rates tested. For a more conventional retention time of 30 days, on average 68% of the raw feed could be converted into biogas. At a retention time of 10-15 days, only 30-50% of the raw feed COD was transformed into methane.

These results show that in order to optimize the biogasification process, it is advisable that the hydrolysis step and biomethanization step are separated. In the CSTR as it is configured now, both hydrolysis (liquefaction) and biomethanization of the raw substrate occur in the same reactor. If biogas yields as high as 90% need to be obtained, this will in many cases lead to unaffordable high reactor volumes due to the high retention times needed. Beside the implementation of additional liquefaction technologies, one could apply a two-phase

methanogenic digester in which the hydrolysis and methanization step are separated. In this respect, the potential use of thermophiles or extreme thermophiles as hydrolysis step could have a highly beneficial effect.

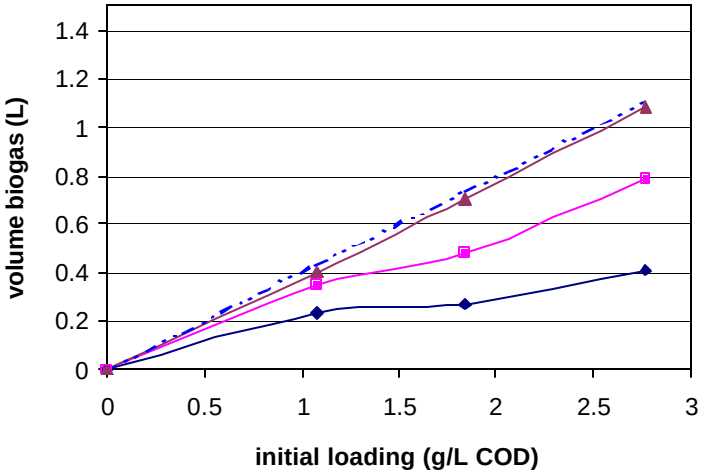


Figure 6: Biogas production at various initial loadings and retention times
Key: ? = 5 d (average RT = 75 d), ¦ = 2.5 d (average RT = 30 d), ? = 1 d (average RT = 15 d), dotted line: maximum theoretical biogas volume (1 g COD ~ 0.5 L biogas)

Additional batch fermentation tests clarified the results with regard to the bioconversion potential of the *Fibrobacter* residue and the thermal liquefaction residue. Initial loading rates between 2-6 g/L COD were applied for the *Fibrobacter* residue while an initial loading rate of 0,2-0,75 g/L COD was applied for the thermal liquefaction effluent.

As can be derived from Figure 7, about 30% of the COD of the *Fibrobacter* residue could be converted into biogas (tested in triplicate) from a retention time on of 20 days. This value corresponds fairly well with the results obtained in TN3 (30-40% conversion).

Conversion efficiencies for the sub-critical residue were however higher compared to the data given in TN3. While a conversion efficiency of only 35% could be reached in the previous experiments (TN3), a biogas yield as high as 60% was obtained in the current experiments at a retention time of 40 days. Clearly, the retention time seemed to play a much more important role compared to the *Fibrobacter* effluent. This was somehow to be expected because of the higher complexity of the medium and the potential risk of inhibitory substances to be present.

Due to the low amount of liquid left of the 2rd closed loop experiment, only few experiments could be performed with the sub-critical effluent. Therefore, it should be remarked that these results are not substantial enough to derive a final conversion figure for the thermal

liquefaction residue. It is therefore recommendable that a continuous fermentation experiment of at least a few weeks should be performed with this type of residue in the future.

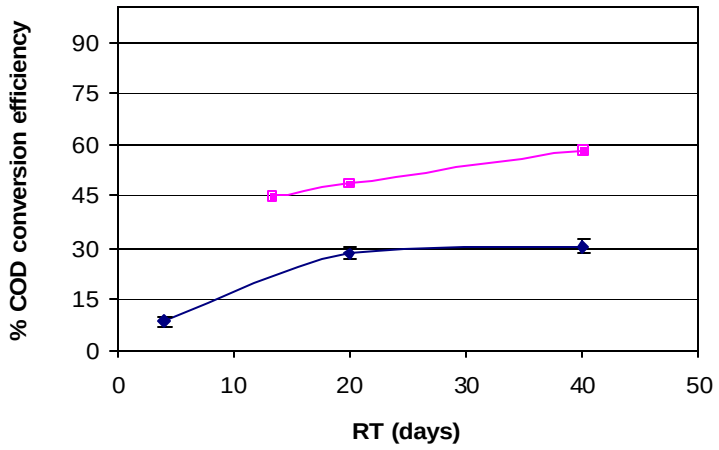


Figure 7: Conversion efficiencies for the *Fibrobacter* effluent (?) and the sub-critical liquefaction effluent (¦)

Based on the results displayed in Figure 5, 6 and 7, the overall conversion efficiencies were calculated for the sequential cycle methanogenesis/*Fibrobacter*/methanogenesis and the cycle methanogenesis/thermal liquefaction/methanogenesis (Figure 8).

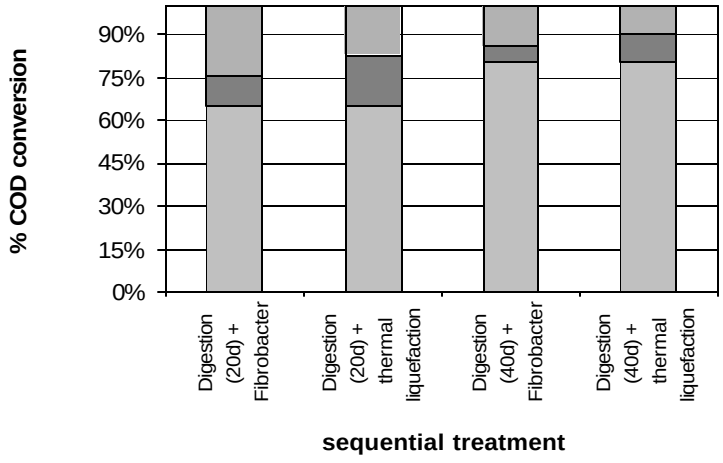


Figure 8: Overall bioconversion of the *Fibrobacter* and the thermal liquefaction sequential treatment for RT = 20 d and RT = 40 d. Key: light grey: methanogenesis of raw substrate; dark grey: methanogenesis after liquefaction treatment; stripes: inert residue after sequential treatment

It can then be derived from Figure 8 that at least 75% of the influent can be converted by applying the sequential treatment methanogenesis/*Fibrobacter*/methanogenesis at a retention time of 20 days for the CSTR digester. By applying higher retention times in the methanogenesis unit (40 days), at least 86% of the influent COD can be converted into biogas. By coupling the thermal liquefaction unit with the methanogenesis unit (methanogenesis/thermal liquefaction/methanogenesis), at least 90% of the influent COD is effectively used for biogas production.

4.2. 3rd closed loop experiment

4.2.1. Digestion of raw substrate by methanogenesis

The raw feed was digested at a theoretical retention time of 20 days. However, as already remarked, the real retention time was higher due to the digestion of residual fibrous matter from previous feeding. As a whole, it can be estimated that the real conversion efficiency of the CSTR is in the order of 70-75% at a RT of 20 days, since it was found that there is a solid build-up of on average 10% of the influent COD due to insufficient mixing in the CSTR.

4.2.2. Digestion of returned Fibrobacter effluent (supernatant)

During the experiment, 1.2 L effluent from the returned *Fibrobacter* effluent could be used. Because the reactor volume was 1.5 L, one was obliged to add 0.3 L of tap water in order to make recycling possible. Consequently, the Fibrobacter effluent used in the biofilm experiments made 80% out of the normal *Fibrobacter* effluent.

For a period of 96 h, the liquid was recycled over the fixed-bed biofilm reactor. Parameters such as COD (both total and soluble), VFA, pH and biogas production were measured on daily basis. The results are shown in Table 1 (general parameters), Table 2 (different compounds of the VFA) and Figure 9.

Table 1. General parameters during 96 h recycling of the Fibrobacter effluent over the fixed-bed biofilm reactor

Time (h)	0	24	48	72	96
COD_t (ppm)	16298	13450	9947	7853	6478
COD_s (ppm)	15260	12459	9414	7689	6124
VFA (ppm)	4910	3891	2097	1178	971
pH	6.77	7.25	7.55	7.59	7.67
gas (L)	0	1.45	3.20	4.25	4.95

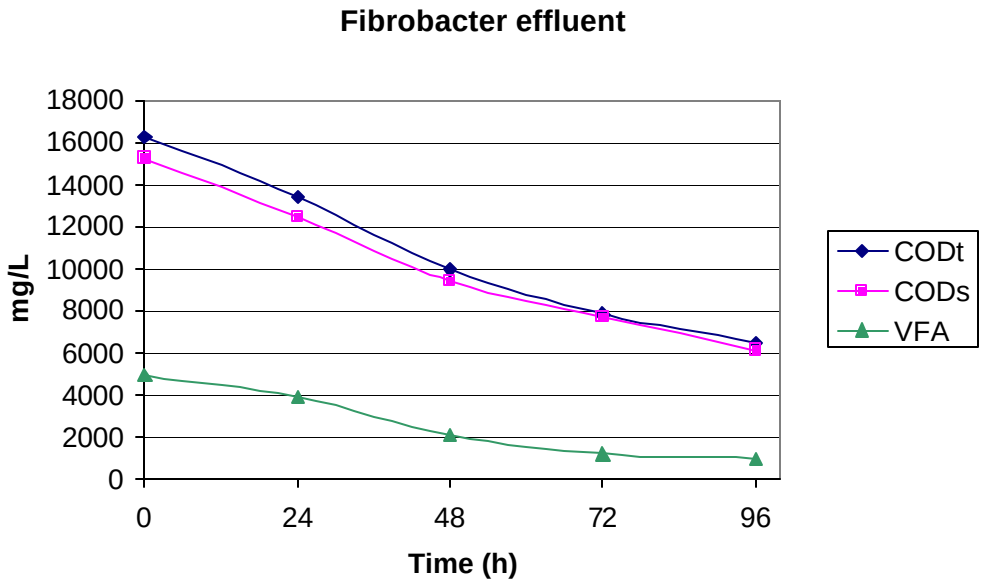


Figure 9. Evolution of the COD_t, COD_s and VFA during the 96 h recycling of the Fibrobacter effluent over the fixed-bed biofilm reactor

As one can notice from Table 1, the *Fibrobacter* effluent is still very rich in both COD and VFA. Moreover, both the residual COD and VFA can be easily digested by an fixed-bed biofilm reactor. Within a period of 96 h, 60% of the COD_t could be digested. If one has a closer look to the diversity of the VFA, it can be noticed that the lower VFA's (acetate, propionate and butyric acid) are nearly completely be converted into biogas. The concentration of higher VFA's remain more or less at the same level or slightly increase. Furthermore, from Table 1 it can also be seen that per gram of COD converted, about 0.5 L biogas is produced. This value is noted in literature as high efficiency. Finaly, from Figure 9,

one can assume that even more COD can be converted if the effluent would be recycled for a more prolonged period over the fixed-bed biofilm reactor.

Table 2. Different compounds of VFA during the 96 h recycling of the Fibrobacter effluent over the fixed-bed biofilm reactor

Time (h)	0	24	48	72	96
acetate	2205	1285	54	30	21
propionate	1258	1154	560	168	15
isobutyric acid	186	195	220	228	233
butyric acid	1012	994	981	654	405
isovaleric acid	203	217	231	235	228
valeric acid	33	33	36	46	52
isocapric acid	14	14	14	13	14
capric acid	0	0	2	2	3
Total (ppm)	**4910**	**3891**	**2097**	**1178**	**971**

4.2.3. Digestion of returned sub-critical effluent (mixed liquour)

An analogue experiment was performed with the effluent from the sub-critical liquefaction. The results are shown in Table 3 (general parameters), Table 4 (details of the VFA) and in Figure 10. However, one should notice that only 0.9 L of effluent could be used during the experiment. As a consequence, one was obliged to make a 60% dilution in order to obtain the required 1.5 L volume.

Table 3. General parameters during 96 h recycling of the effluent of the sub-critical liquefaction over the fixed-bed biofilm reactor

Time (h)	0	24	48	72	96
CODt (ppm)	2590	1961	1658	1475	1274
CODs (ppm)	1810	1588	1379	1221	1087
VFA (ppm)	87	5	0	0	0
pH	6.80	7.19	6.93	6.85	6.89
gas (L)	0	0.30	0.45	0.55	0.65

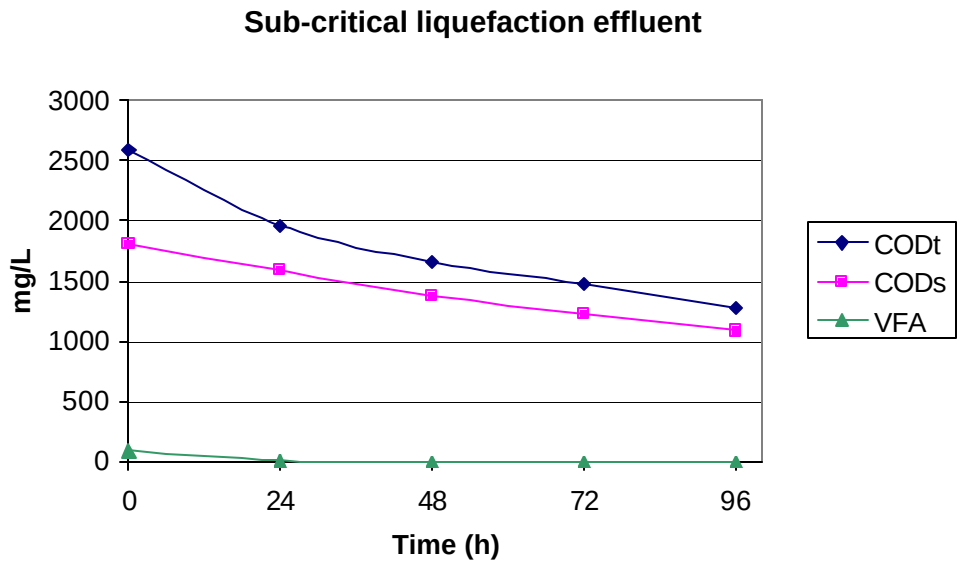


Figure 10. Evolution of the CODt, CODs and VFA during the 96 h recycling of the effluent of the sub-critical liquefaction over the fixed-bed biofilm reactor

As can be seen from Table 3, the effluent after sub-critical liquefaction contains a considerable lower amount of COD and is very poor in VFA. The efficiency of the fixed-bed biofilm reactor is also slighty less compared to the *Fibrobacter* effluent: after a 96 h period, 50% of the COD_t can be converted into biogas, whereas about 60% could be reached with the *Fibrobacter* effluent. On the other hand, one can see that also here a volume of 0.5 L of biogas is produced for every gram of COD_t that is removed. It can be assumed from Figure 10 that a more prolonged period of recycling will result in a further conversion of the COD into biogas.

Table 4. Different compounds of VFA during the 96 h recycling of the effluent of the subcritical liquefaction over the fixed-bed biofilm reactor

Time (h)	0	24	48	72	96
acetate	20	0	0	0	0
propionate	4	0	0	0	0
isobutyric acid	19	3	0	0	0
butyric acid	20	0	0	0	0
isovaleric acid	21	2	0	0	0
valeric acid	3	0	0	0	0

isocapric acid	0	0	0	0	0
capric acid	0	0	0	0	0
total (ppm)	**87**	**5**	**0**	**0**	**0**

5. Conclusions

- The applied retention time for the CSTR methanogenic reactor is a crucial factor in the optimization of the conversion efficiency of the raw substrate into biogas. Net-conversion efficiencies vary from 30% up to 90% at various loading rates for a retention time of 15 days and 65 days respectively.
- Further optimization of the CSTR methanogenic reactor can lead to very high conversion efficiencies (> 90%). Technical potentialities are the use of (hyper-) thermophilic organisms as a pre-hydrolysis step, the use of 2-stage methanogenic reactor and/or the decoupling of the solid retention time from the hydraulic retention time. In all cases, the separation of the hydrolysis step from the methanogenic step in the reactor itself seems to be preferable.
- From batch fermentation tests, it could be confirmed that the biogas yields for the fermented fibrous residue processed by the rumen bacterium *Fibrobacter* are in the order of 30% for triplicate tests. Fermentation yields using the thermal liquefaction residue were in the order of 50-60%. These values were found to be significantly higher to the ones found in previous experiments (40% in TN3). In this respect, it should be remarked that the loading rates applied were significantly lower in the current experiments. A long-term fermentation test with the sub-critical residue should confirm this.
- It was stated that by coupling the thermal liquefaction unit with the methanogenesis unit (methanogenesis/thermal liquefaction/methanogenesis), at least 85-90% of the influent COD is effectively used for biogas production. Depending on the retention time applied, the sequential treatment methanogenesis/*Fibrobacter*/methanogenesis was able to convert 75-86% of the raw substrate.
- Both the Fibrobacter effluent as the sub-critical liquefaction effluent from the 3rd closed loop experiment could be easily further digested by means of a fixed film reactor. From the first tests (no duplicates), one can already make an estimation of the efficiency and thus reactor design (reactor volume and retention times). Further optimization with proper dimensions will be performed in in the near future to optimize the methanogenic biofilm reactor.

6. Outlook

- A long fermentation experiment (at least 1 month) with the subcritical liquefaction effluent is needed to determine in more detail (duplicate and triplicate tests) the bioconversion of the thermal liquefaction effluent. This way, a methanogenic consortium can be cultured, specifically conditioned (adapted) towards the sub-critical liquefaction effluent.
- Further optimization of the methanogenic biofilm reactor
- Writing of the final report

6. References

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