EPAS n.v.

Eco Process Assistance

IIC-Universiteit Gent
Technologiepark 3 - 9052 Gent (Zwijnaarde)

Tel. (09) 241.56.18
Fax (09) 221.82.18

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TECHNICAL NOTE 56.1

Coupling of compartment 1 to a microfiltration membrane

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Ing. Veronik Hermans
ir. Dries Demey

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1. Introduction

The liquefying compartment is responsible for the biodegradation of human faecal material and other waste generated by the crew. The current biodegradation efficiency of this compartment is between 55 and 70% depending on the process conditions. Since the first compartment is not working continuously for the moment and the biodegradation efficiency in not 100%, a method to separate the non biodegraded organic matter from the produced ammonium and VFA needs to be found. In this technical note, the results of preliminary tests using a microfiltration unit to filter the MELiSSA supernatant (after a centrifugation) and the MELiSSA effluent itself are represented. The results of the coupling of the microfiltration unit to the MELiSSA demonstration reactor are also included in this technical note.

2. Preliminary microfiltration tests

2.1 Introduction

A membrane is any material which forms a thin wall (0.05 mm to 2 mm) and is capable of putting up a selective resistance to the transfer of different constituents of a fluid, thus allowing the separation of some of the elements (suspension, solutes or solvents) making up this fluid. With filtration membranes, water is the preferred transfer phase under the effect of a pressure gradient. These membranes are classified according to the size of their pores. The classification is represented in Figure 2-1.

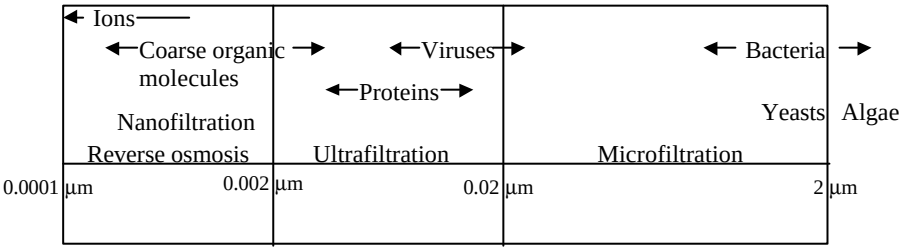


Figure 2-1 Types of membrane filtration

There are two kinds of filtration techniques, dead end and tangential filtration. In the dead end filtration, the water is forced through the membrane and the retained particles build up in the form of a filter cake, which causes a reduction in the specific flow. In tangential filtration, the membrane is designed in such a way as to allow part of the inflow to be used as a circulation flow across the active side of the membrane. This limits the build-up of cake by continuously carrying away the substance discharged out of the system. In Figure 2-2 both type of filtration are represented.

A few parameters are noted below and are important for microfiltration:

- Flow through speed (V): the higher the speed, the higher the turbulence at the membrane, and therefore the limited the clogging of the membrane
- Transmembrane pressure (TMP): the driving force for filtration of the liquid through the membrane. The higher the pressure, the higher the driving force, the higher the flux
- The matter to be retained: the more suspended solids there are in the liquid, the greater the cohesion of these solids and the more liable they are to proliferate (algae, bacteria)
- Temperature (T): The higher the temperature, the lower the viscosity, the higher the flux.

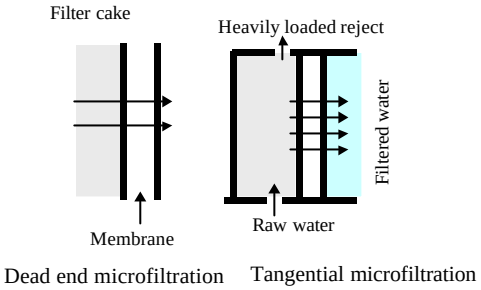


Figure 2-2 Microfiltration modes

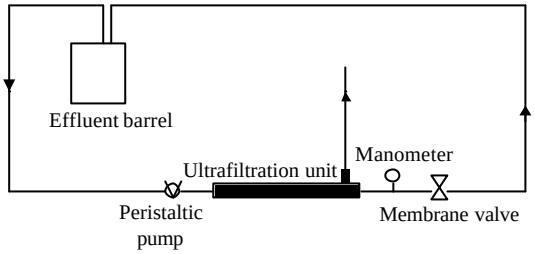
2.2 Set-up of microfiltration test

Based on the results of the tests, VITO performed last year, a WFF4385 membrane was selected. These membranes are highly efficient hydrophilic tubular polyvinylidene fluoride membranes, cast on a polyester carrier, for use in a variety of filtration processes in food and non-food applications. The hydrophilic properties of these membranes ensure a high performance and a good antifouling behaviour. The physical and chemical properties and the performance data are represented in Table 2-1.

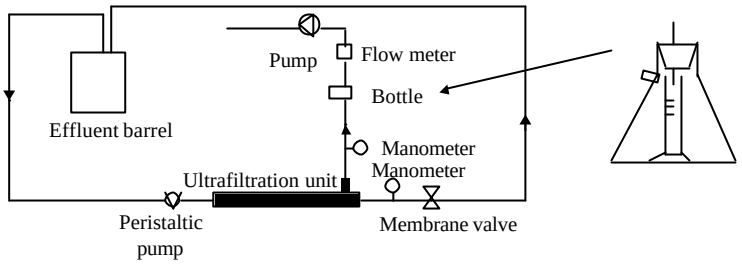
Table 2-1 Physical and chemical properties and performance data of the WWF 4385 membrane

WFF4385 **Physical and chemical properties**	
Membrane material	polyvinylidene fluoride
Structure	asymmetric/microporous
Membrane carrier	composite polyester fabric
Geometry	tubular
Performance data	
Initial flux (l/m^2.h)	>1000
Transmembrane pressure (bar)	-1..3
Mean pore size (nm)	30
pH	2-10
Chlorine exposure (ppm.h)	250000
Temperature (°C)	1-70
Hydraulic diameter (mm)	5.2

Two types of set-ups were tested. In the first set-up overpressure was obtained in the retentate tubing. With this overpressure part of the liquid was forced through the membrane. In the second set-up, next to overpressure in the retentate tubing, underpressure was created in the permeate tubing. The set-ups are shown in Figure 2-3. In Figure 2-4 a picture of the set-up is represented.



Set-up 1: overpressure



Set-up 2: overpressure and underpressure

Figure 2-3 Experimental set-ups

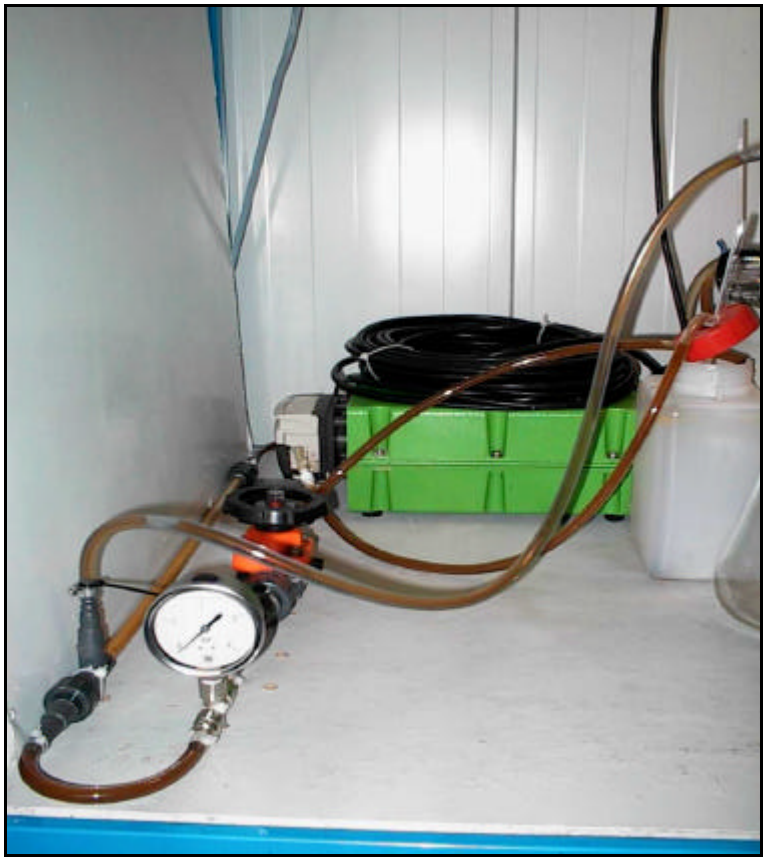


Figure 2-4 Picture of experimental set-up

2.3 Preliminary tests with water

2.3.1 Introduction

First some tests were performed with water and overpressure in the retentate tubing to have an idea about the flux. The flow through speed (V) varied between 5.5 l/h and 14 l/h. The pressure after the membrane varied between 0.4 bar and 1.8 bar.

2.3.2 Results

The flux evolution of four different tests is shown in Figure 2-5. The different parameters for the four different tests are explained in Table 2-2. In test 1 the flux decreased until a more or less stable value of 400 l/m²*h was reached. In test 2,3 and 4 the flux is around 200-250 l/m²*h during the whole experiment.

Table 2-2 Parameters of different tests

Test	Parameter
Test 1	Flow through speed (V): 10.8 l/h P_{out} (begin): 0.8 bar
Test 2	Flow through speed (V): 10.8 l/h P_{out} (begin): 0.4 bar
Test 3	Flow through speed (V): 5.5 l/h P_{out} (begin): 0.4 bar
Test 4	Flow through speed (V): 13.9 l/h P_{out} (begin): 0.4 bar

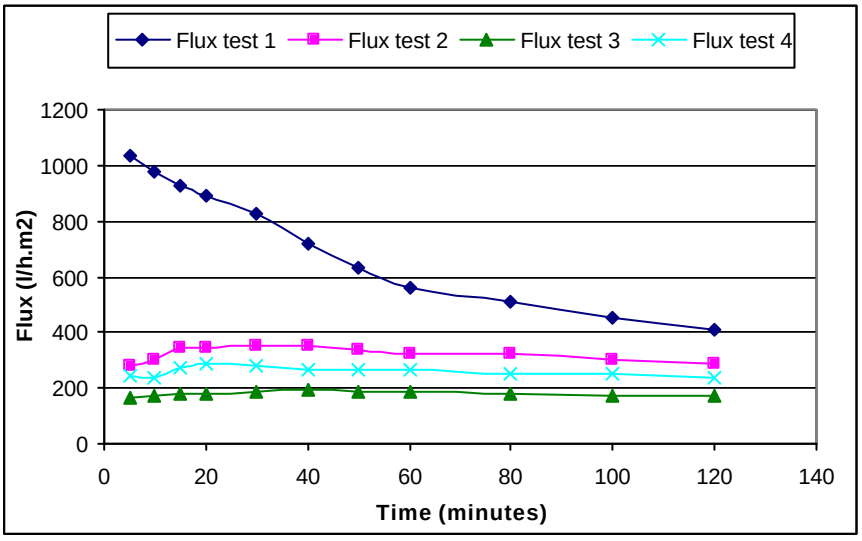


Figure 2-5 Flux evolution of different tests

The pressure evolution is represented in Figure 2-6. The pressure increased for all the experiments until a stable value was reached. This stable value was dependent on the begin pressure. In test 1 the begin overpressure was higher than in the other tests, therefore the final pressure is about 1.5 bar instead of the ± 1 bar in the other tests.

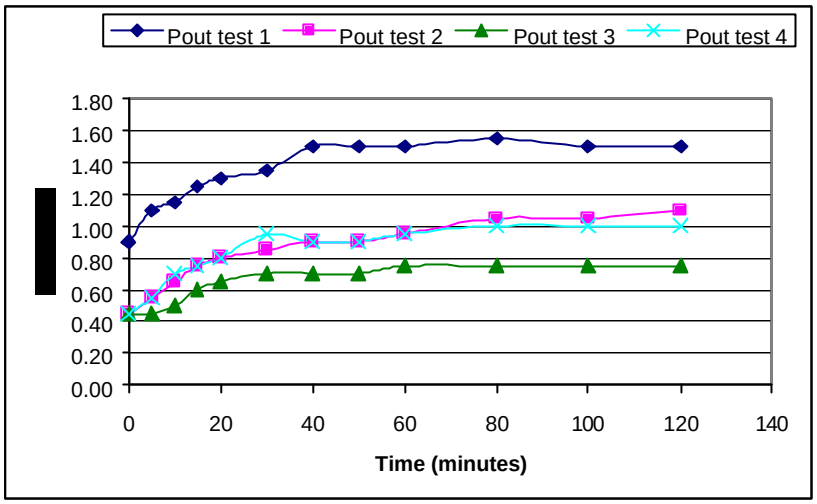


Figure 2-6 Pressure evolutions of different tests

2.4 Preliminary tests with MELiSSA supernatant

2.4.1 Introduction

In this series of tests the MELiSSA effluent was first centrifuged at a speed of 3000 rpm during 15 minutes. Afterwards the MELiSSA supernatant was filtered using the tubular microfiltration membrane. The tests were performed with a flow through speed (V) of about 10.8 l/h. The overpressure varied between 0 and 1.5 bar. Some tests were carried out with overpressure in the retentate and /or underpressure in the permeate tubing.

2.4.2 Results

The flux evolution of 6 different tests is represented in Figure 2-7. The differences between the six tests are gathered in Table 2-4.

The flux in all the different tests stabilised at a value of around 8 l/m²*h. There was no major difference between the test with underpressure or without underpressure. In Figure 2-8 the overpressure evolution is represented. In all 4 tests, the pressure stabilised.

In test 5 some analyses were performed in order to check the performance of the membrane. In Table 2-3 the results are shown. The membrane separates most of the ammonium and VFA.

Table 2-3 Performance of membrane

Test 5	**pH**	**N-tot** (mg/l)	**NH_4-N** (mg/l)	**VFA** (mg/l)	**DM** (g/l)	**ASH** (g/l)
Supernatant (feed)	7.29	865	580	1786	6.16	2.11
Permeate	7.92	660	495	1411	3.38	1.76

Table 2-4 Parameters of the different tests

Test	Parameters
Test 5	Flow through speed: 10.8 l/h P_{out} (begin): 1.55 bar $P_{underpressure}$: no
Test 6	Flow through speed: 10.8 l/h P_{out} (begin): 0 bar $P_{underpressure}$: yes
Test 7	Flow through speed: 10.8 l/h P_{out} (begin): 1.0 bar $P_{underpressure}$: yes
Test 8	Flow through speed: 10.8 l/h P_{out} (begin): 0.bar $P_{underpressure}$: yes
Test 9	Flow through speed: 10.8 l/h P_{out} (begin): 1.0 bar $P_{underpressure}$: yes
Test 10	Flow through speed: 10.8 l/h P_{out} (begin): 1.0 bar $P_{underpressure}$: no

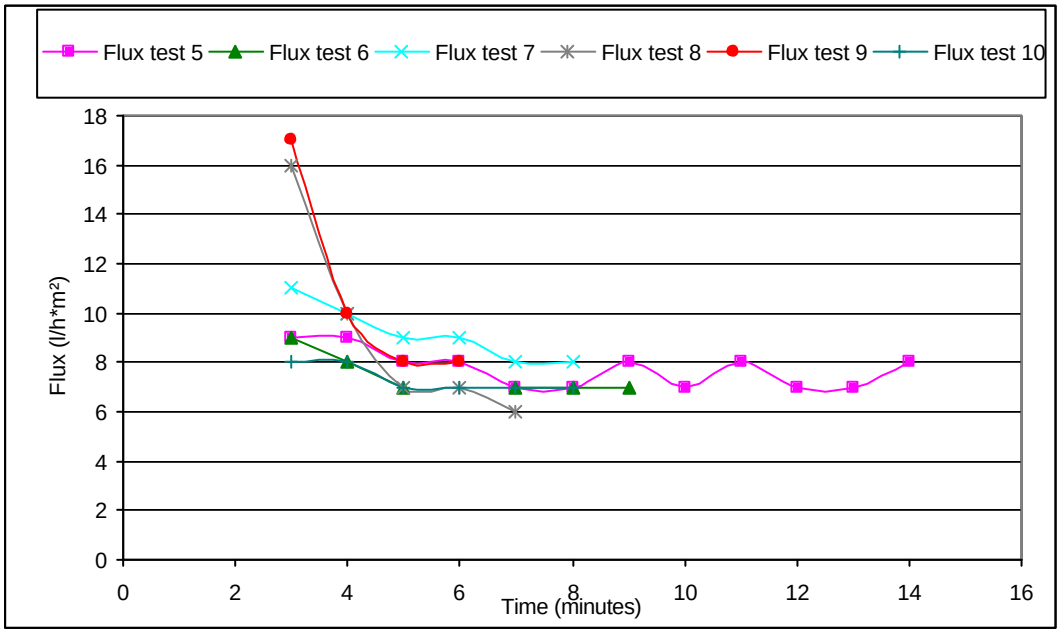


Figure 2-7 Flux evolutions of different tests

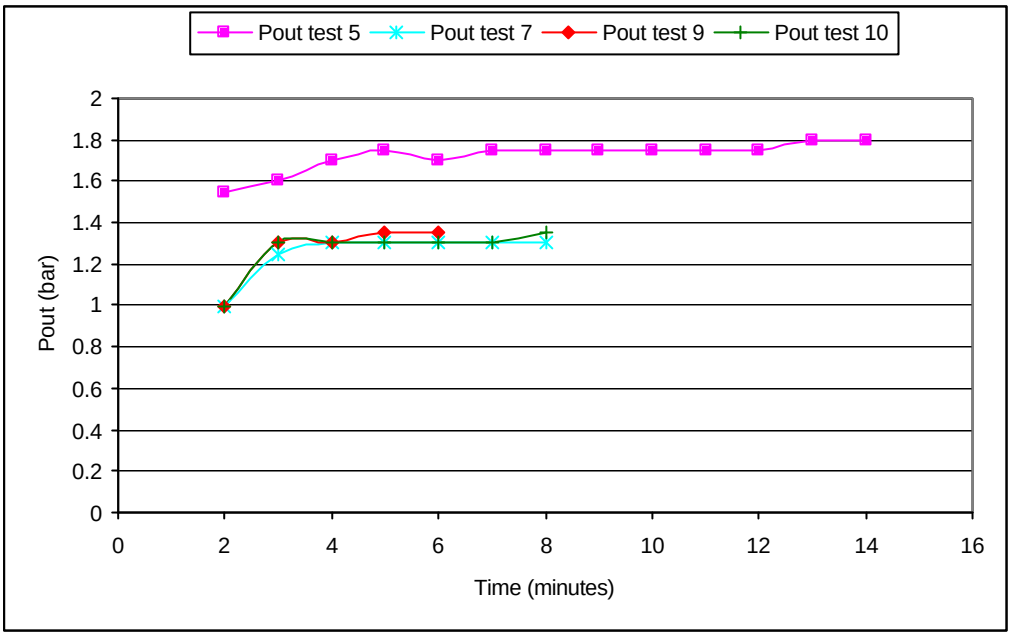


Figure 2-8 Overpressure evolution in the different tests

2.5 Preliminary tests with MELiSSA effluent

2.5.1 Introduction

In this series of tests the MELiSSA effluent was send through the membrane, without any pretreatment (like centrifuge). The flow through speed was for test 11 and test 12, 10.8 l/h and for test 13, 24 l/h. The begin overpressure in the retentate tubing was between 0.7 bar and 1 bar. In test 11 underpressure in the permeate was obtained.

2.5.2 Results

In Table 2-5 the different parameters of the different tests are shown. In Figure 2-9 the results of the flux are represented. The flux in test 11 and test 12 is very similar and oscillated around 6 l/h*m². In test 13 the begin flux is around 10 l/h*m² and stabilised at a value of 9 l/h*m². In Figure 2-10 the pressure is shown. The pressure during the three tests is not stable and sometimes high values of around 2 bar were noticed. At that moment the valve after the membrane unit (Figure 2-3) was opened in order to manipulate the pressure and to obtain again a pressure of around 1 bar .

Table 2-5 Parameters of the different tests

Test	Parameters
Test 11	Flow through speed: 10.8 l/h P_{out} (begin): 1 bar $P_{underpressure}$: yes
Test 12	Flow through speed: 10.8 l/h P_{out} (begin): 1 bar $P_{underpressure}$: no
Test 13	Flow through speed: 24 l/h P_{out} (begin): 0.7 bar $P_{underpressure}$: no

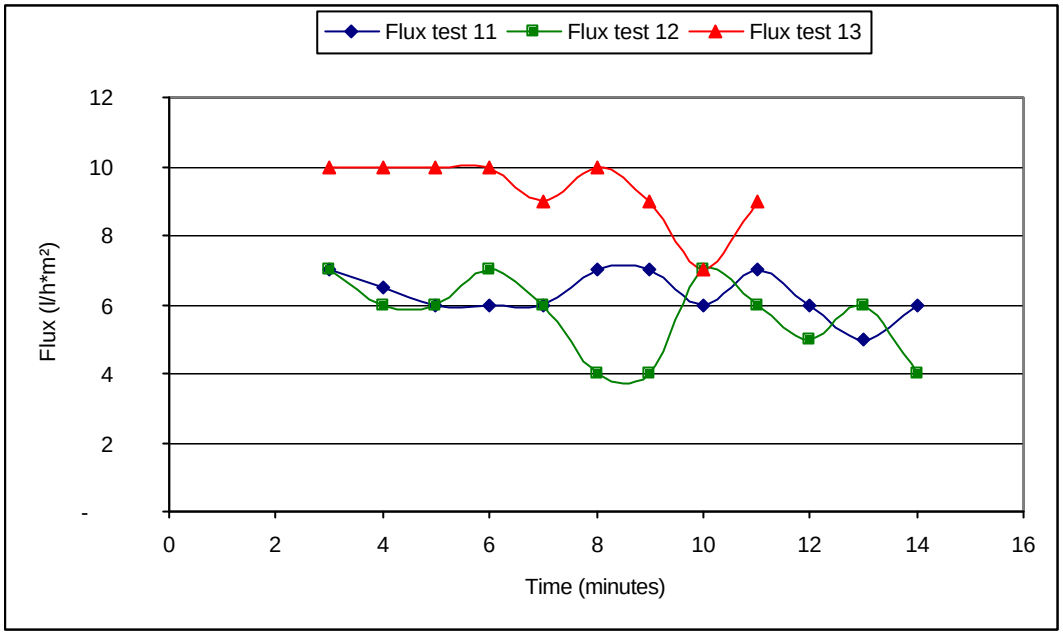


Figure 2-9 Flux evolutions in different tests

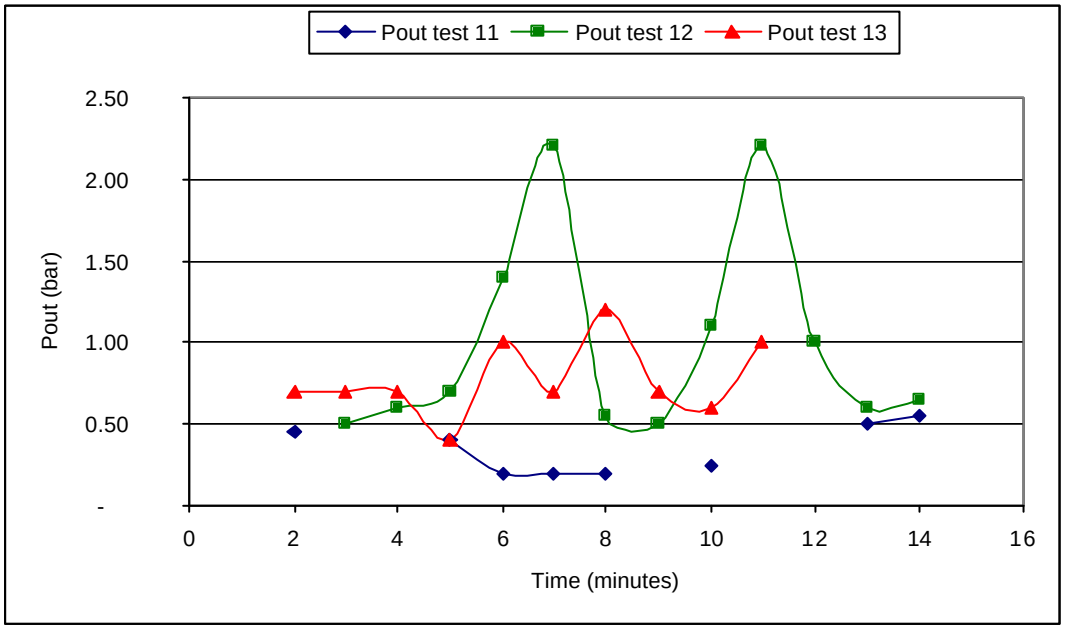


Figure 2-10 Overpressure evolutions in different tests

The membrane performance was calculated for test 12 and is represented in Table 2-6.

Table 2-6 Membrane performances

Test 13	**pH**	**N-tot** (mg/l)	**NH_4-N** (mg/l)	**VFA** (mg/l)	**DM** (g/l)	**ASH** (g/l)	**OM (g/l)**
MELiSSA effluent	6.9	1245	590	3100	15.8	2.9	12.9
Permeate	7.3	735	590	3033	4.0	2.1	1.9

2.6 Conclusion

The membrane can not retain the dissolved organic matter. A high amount of ammonium and VFA are separated from the non-biodegraded organic matter and are present in the permeate.

With the microfiltration membrane a flux of about 6 l/m²*h was reached. This flux corresponds with the flux found in literature for anaerobic biomembrane reactors (Stephenson et al., 2000).

3. Reactor 1

3.1 Introduction

After the preliminary tests the microfiltration membrane was coupled to the anaerobic demonstration reactor.

3.2 Set-up

The demonstration reactor had a wet volume of 1.6 litres and a temperature of 55°C. The reactor was set at a pH of 6 - 6.5 in order to inhibit the methane production and was continuously stirred with a magnetic stirrer. The reactor was fed with faecal material collected from 8 different persons between age 24 and 40. The characteristics of the faecal material are represented in Table 3-1. Three times a week 150 ml of waste material was fed into the reactor. From day 0 until day 182, 150 ml of sample was taken from the reactor. From day 182 until day 322, 50 ml from the reactor and 100 ml of permeate was sampled. The sampling method is presented in Figure 3-1. After every feeding, the reactor was flushed with N_2-gas during 2 minutes to obtain anaerobic conditions. In Figure 3-2 the reactor set-up is shown.

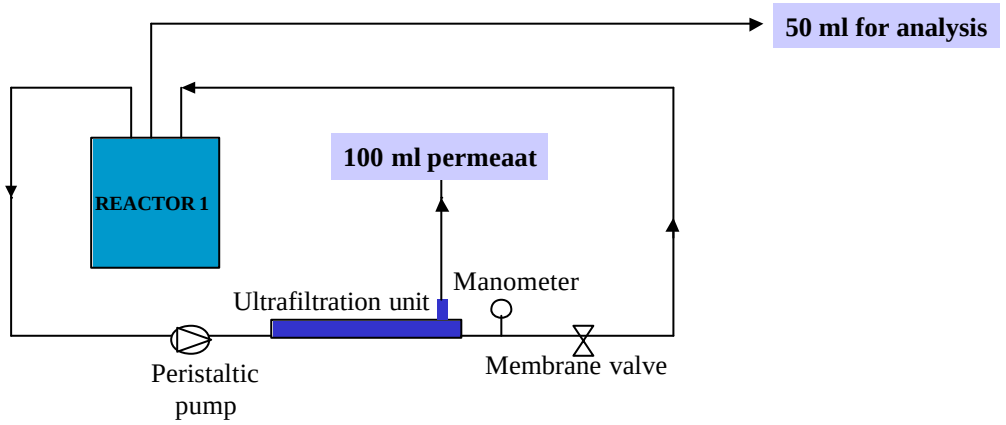


Figure 3-1 Sampling methode

Figure 3-2 Reactor set-up

Table 3-1 Characteristics of the faecal material

Parameter	Unit	Mean value
pH		7
Dry matter	g/l	25
Ash	g/l	3.3
Total nitrogen	mg/l	1093
Ammonium nitrogen	mg/l	70
VFA	mg/l	844
Acetic acid		499
Propionic acid		144
Iso butyric acid		14
Butyric acid		110
Iso valeric acid		23
Valeric acid		32
Iso caproic acid		5
Caproic acid		18

The produced biogas was measured using a gas column that was connected to the demonstration reactor. The gas column contained a coloured solution at pH 3. This low pH was used to prevent the CO_2 being absorbed by the liquid in the gas column. Every time before feeding the reactor, the produced biogas was read.

The parameters analysed on the waste mixture and effluent of the reactor are pH, conductivity (EC), dry matter, organic matter, ammonium-N, total-N and volatile fatty acids. The biogas composition was frequently measured with an Infrared gas analyser. With these results, the conversion efficiencies could be calculated. The permeate was analysed on DW, Ash, VFA and ammonium in order to evaluate the performance of the membrane. The amount of bacteria in the permeate was measured by plating a small amount of permeate (different dilutions) on solid culture media. After one week the bacteria were counted.

With this method the permeability of the membrane for the bacteria present in the anaerobic reactor could be assessed.

3.3 Results

3.3.1 pH and EC

The pH fluctuated around 6 in order to inhibit the growth of the methanogens and therefore to prevent methane production. The EC was measured to have an idea about the dissolved salts present in the reactor. The value fluctuated between 5 and 8 mS/cm, which is normal for anaerobic reactors. During the Christmas period the reactor was fed with a mixture of gelatine and starch. This caused an increase in EC of 5mS/cm. After this period, when the reactor was again fed with faecal material, the EC decreased until a stable value of around 8 ms/cm was reached.

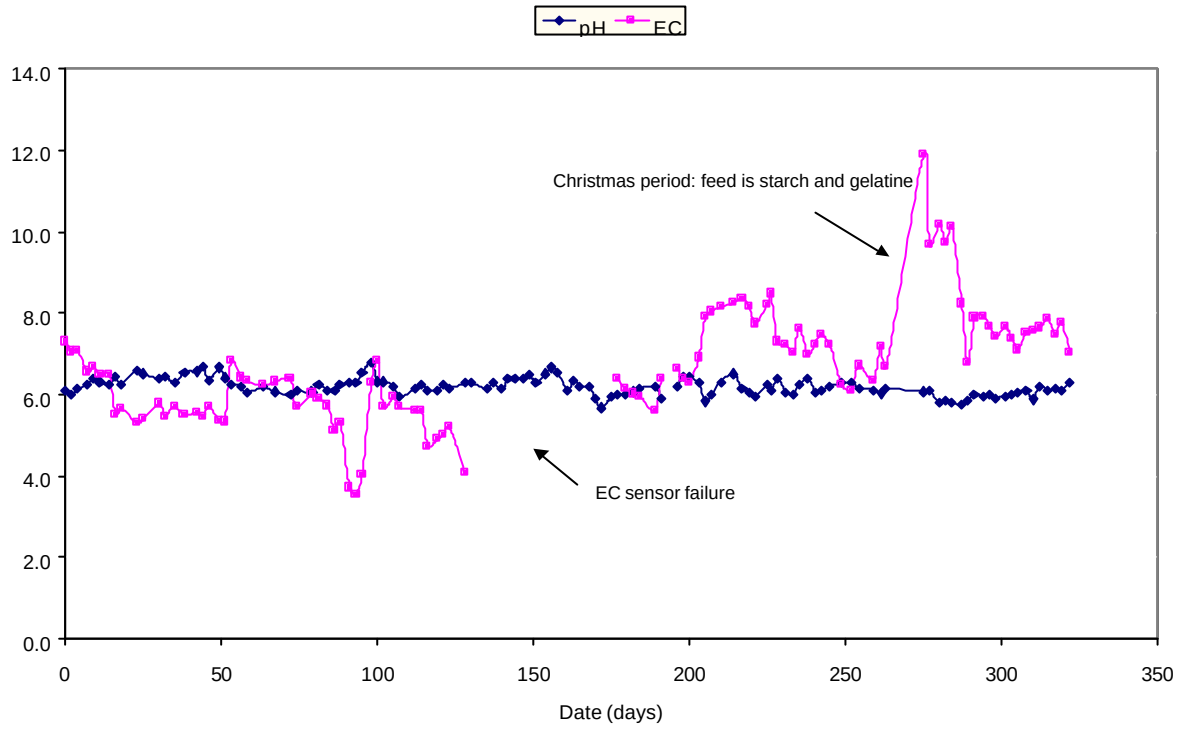


Figure 3-3 pH and EC in reactor

3.3.2 Dry matter and organic matter

It was difficult to take a homogenous sample because the reactor contained a high amount of particulate material. This particulate material is visual perceptible. High fluctuations in dry matter and organic matter concentrations were noticed. This phenomenon was also noticeable last year, but now the amount of particulate material is increased, and the fluctuations are more pronounced, which can be seen in Figure 3-4.

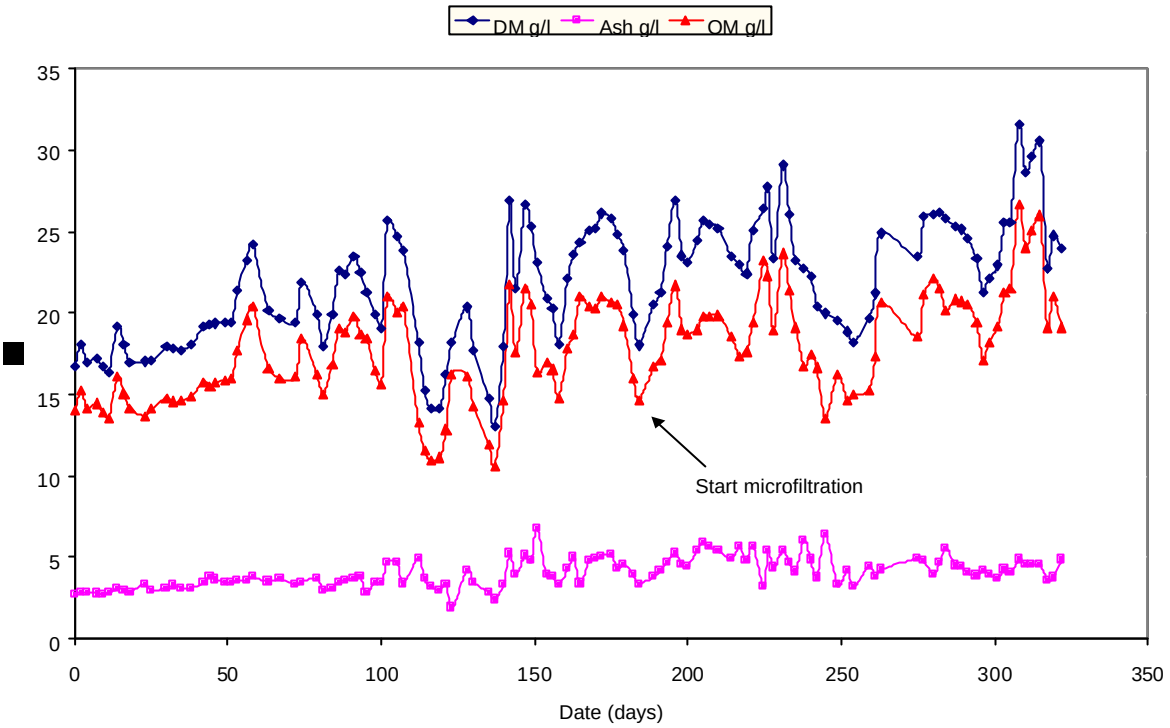


Figure 3-4 Dry matter, organic matter in reactor

3.3.3 NH_4-N and N-org

The increase of N-org at day 136 was due to a different feed. This faecal material contained a higher N-tot percentage than the previous feed. At day 228 the N-org concentration decreased due to a lower N-tot concentration in the feed. During the Christmas period gelatine and starch were fed into the reactor. The increase of NH_4-N was due to an easy conversion of gelatine into NH_4-N. The amount of gelatine fed into the reactor during Christmas was probably to high because not all N-tot was converted into ammonia. After the Christmas period the NH4-N concentration decreased and reached a stable value of around 800 mg/l. The fluctuations in N-org are due to the difficulty to take a homogenous sample since particles floated in the solution.

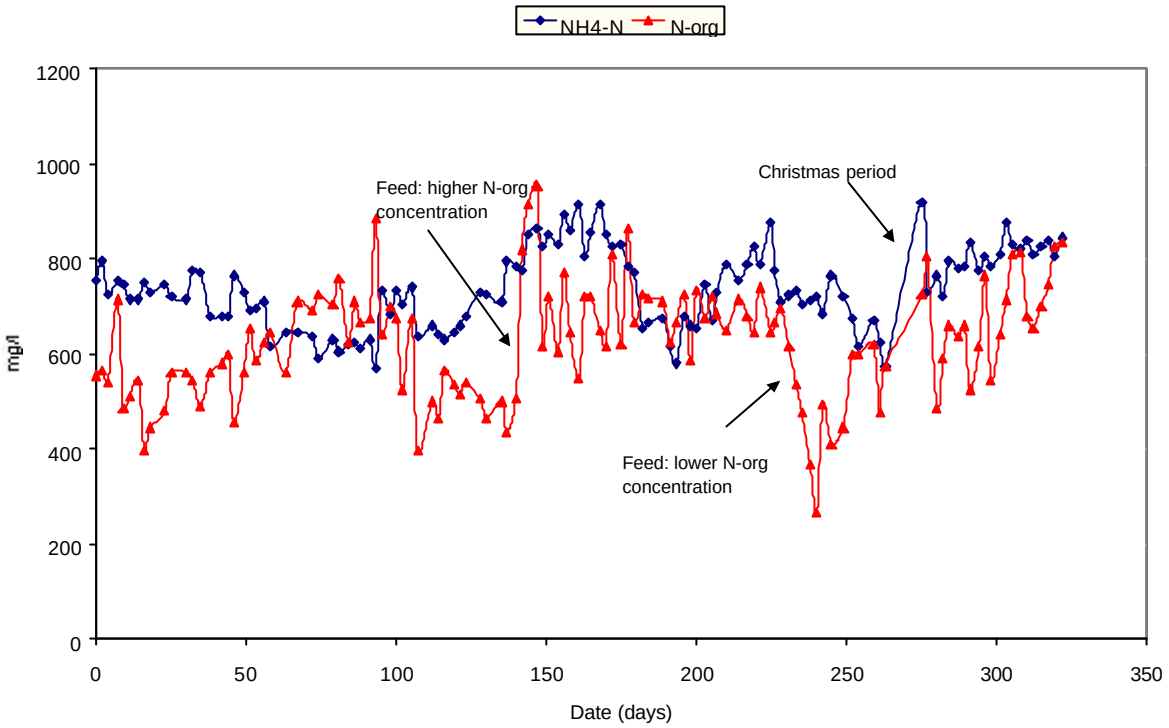


Figure 3-5 NH_4-N and N-org in reactor

3.3.4 Volatile fatty acids

In Figure 3-6 the volatile fatty acids concentration and composition are represented. The VFA concentration fluctuated around 4000 mg/l. The small fluctuations are due to different VFA concentrations in the different feeds. At day 263 the reactor was fed with gelatine and starch (Christmas period). The increase of VFA at that time was due to the fact that starch is good degradable by the autochtonous bacteria of faecal material. The majority of produced VFA was acetic acid due to the inhibition of the methanogenesis.

3.3.5 Biogas production

The cumulative biogas production is represented in Figure 3-7. At low pH, the methanogens were inhibited and only CO_2 was produced. The gas production during the Christmas period was not measured.

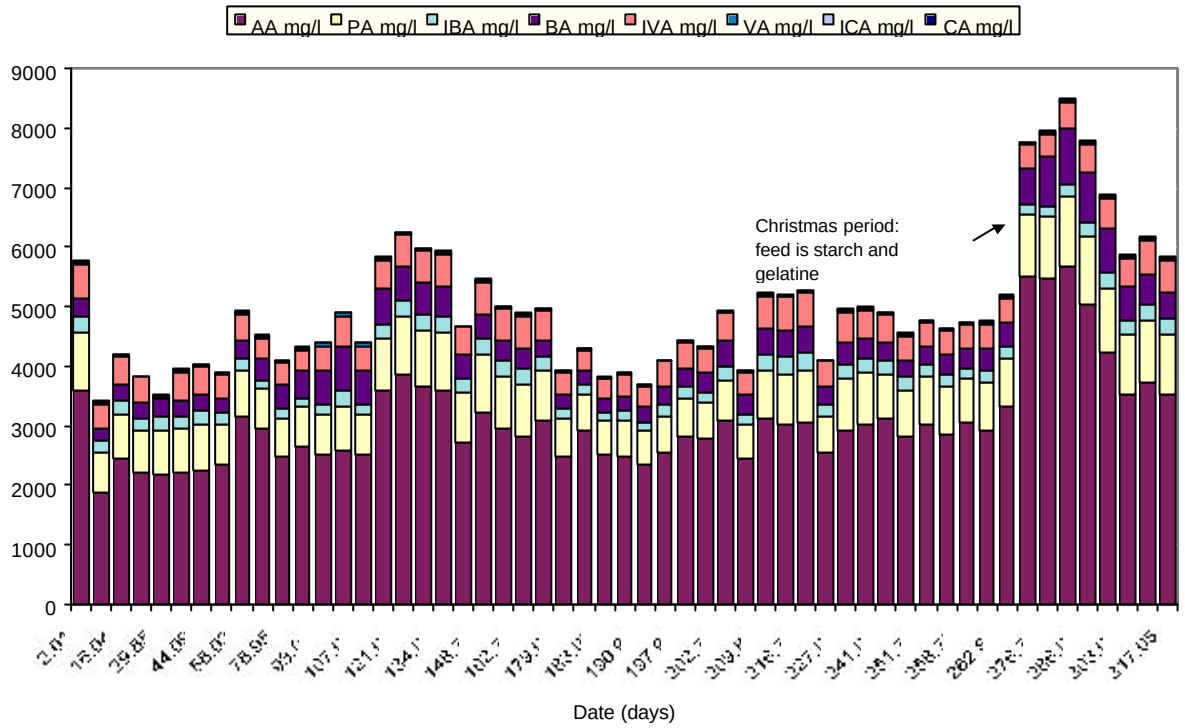


Figure 3-6 VFA concentration and composition in reactor

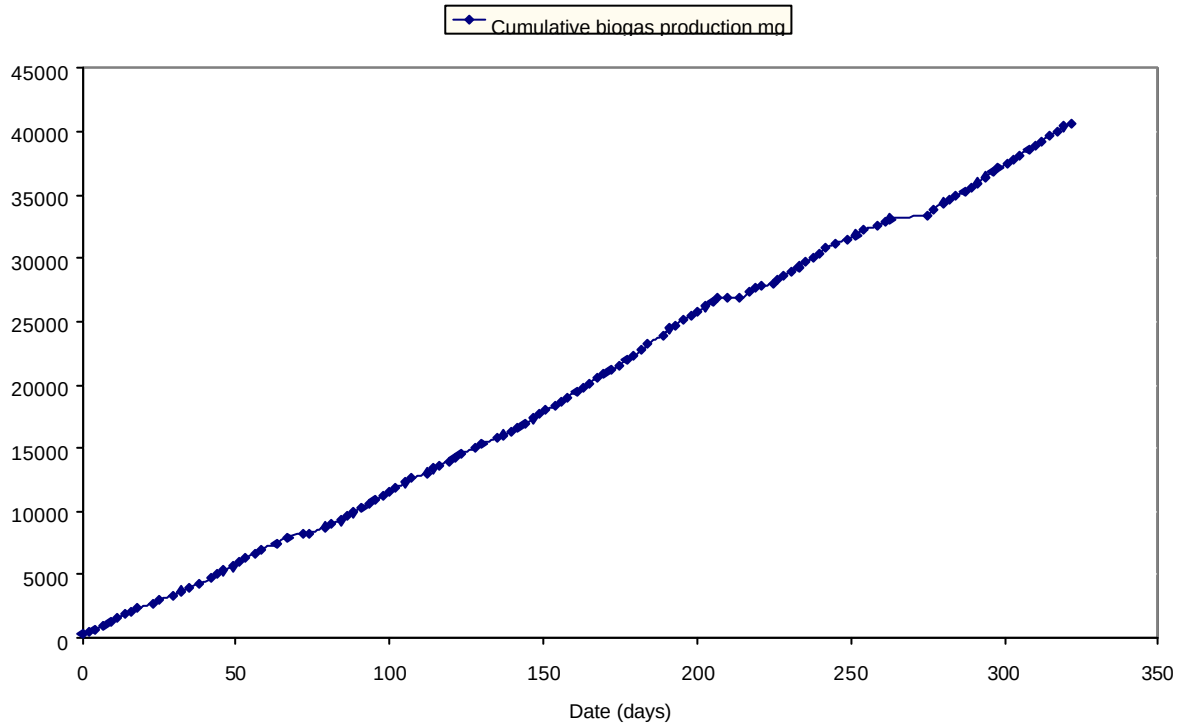


Figure 3-7 Cumulative CO_2 production

3.3.6 Conversion efficiencies

The conversion efficiencies of the waste material fed to the reactor were calculated taking into account some boundary conditions. In the case of total conversion efficiency, it is assumed that during the anaerobic degradation polymers were converted into VFA and biogas (methane and carbon dioxide). Since the reactor was operated at pH 6-6.5 no methane was measured in the biogas. Besides the total conversion efficiency, the OM conversion efficiency was calculated using the organic matter fed to the reactor and the organic matter removed from the reactor. The total conversion efficiency increased at day 182 with 5% until a value of 30% was reached. From this day on the microfiltration unit was inserted. The OM conversion efficiency increased with 10 % after the insertion of the microfiltration unit and reached a value of 37%. It is possible that besides VFA and biogas other compounds were produced, like lactate and alcoholic components. The protein conversion efficiency is stable during the entire period and is about 60%. Fibres are converted for about 14%.

Figure 3-8 Conversion efficiencies

3.3.7 Membrane performances

During the experiment the membrane performance was tested. This was performed by measuring dry weight, Ash, ammonium and VFA in the permeate. In Figure 3-9 the organic matter in the reactor is compared with the organic matter content in the permeate. Not all of the organic matter is retained by the membrane. Most of the VFA and ammonium can be found in the permeate (Figure 3-10 and Figure 3-11).

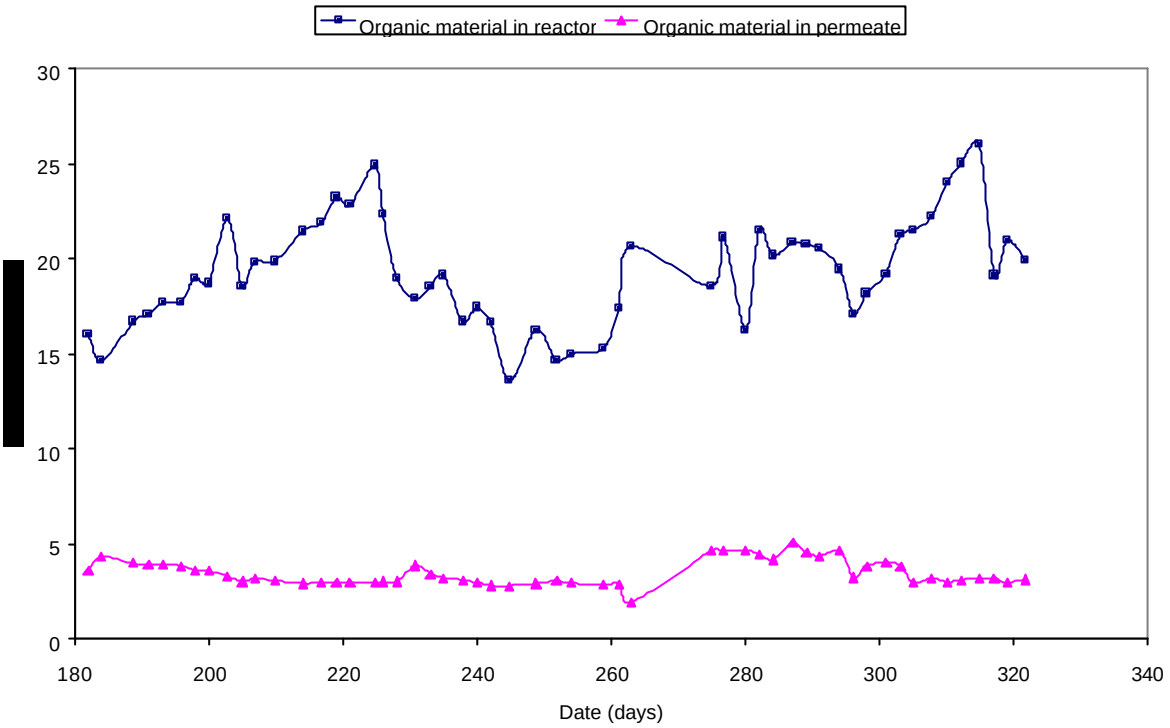


Figure 3-9 Organic matter content in reactor and permeate

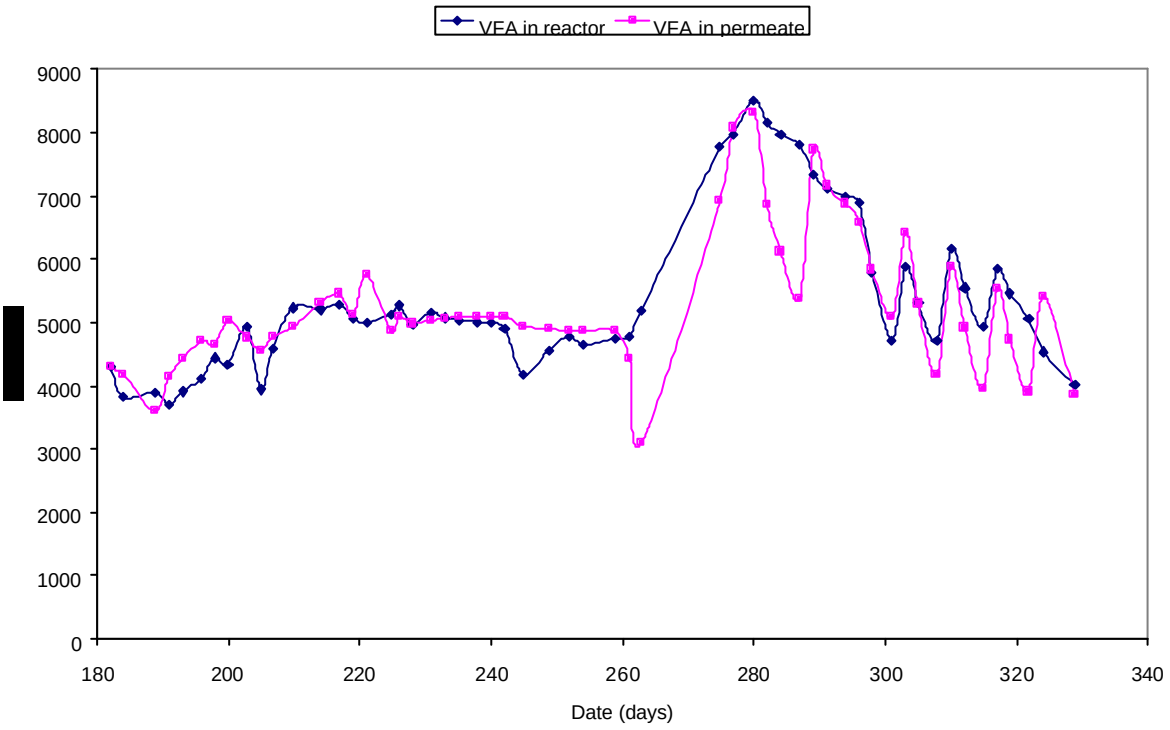


Figure 3-10 VFA content in reactor and permeate

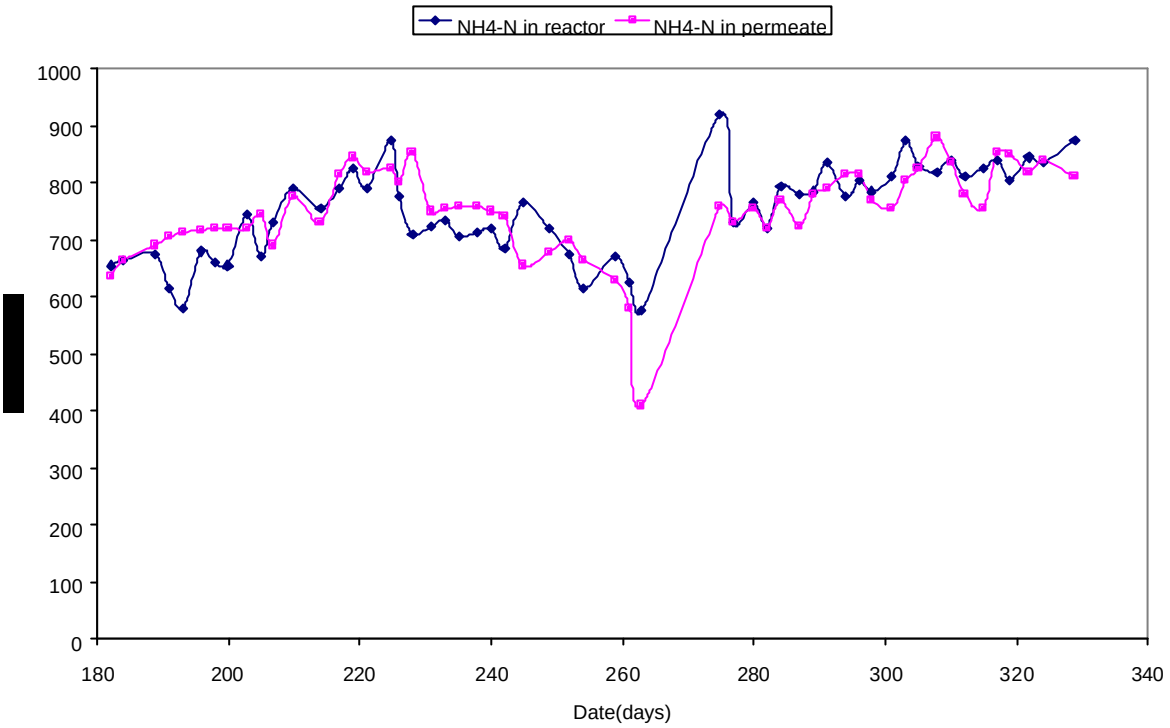


Figure 3-11 Ammonium content in reactor and permeate

As a preliminary test, the amount of bacteria in the permeate was measured by plating a small amount of permeate (different dilutions) on solid culture media at 55°C. After one week the bacteria were counted. With this method the permeability of the membrane for the bacteria present in the anaerobic reactor was assessed. From the results of the test could be concluded that no bacteria were in the permeate. These results need to be confirmed with more specialised investigations like DNA tests, before final conclusion regarding the efficiency of the membrane on bacteria can be made.

4. Conclusions

The conversion efficiencies of faecal material was investigated using a thermophilic demonstration reactor with autochtonous bacteria of faecal material. The reactor was operated at pH around 6 to avoid methane production, because it is of no use for the further loop. A total conversion efficiency of 30% was found. Proteins were converted for 60% and fibres for 14%. The biodegradation efficiency based on the calculation with organic matter analysis reached a value of 37%. This is higher than the conversion efficiency due to the fact that besides VFA and biogas, other compounds like lactate and alcoholic components can be produced. It can be concluded that the total conversion efficiency and the biodegradation efficiency increased after the introduction of the microfiltration unit. The microfiltration unit did not retain the dissolved organic matter, but most of the VFA and ammonium could be found in the permeate. In the permeate no bacteria were noticed. This result needs to be confirmed with DNA tests, to make sure that all bacteria were retained by the membrane.

The data will be used for the validation of the model of the anaerobic thermophilic reactor.

In future, compartment I and II will be coupled, using a microfiltration membrane.

5. References

Stephenson, T., Judd, S., Jefferson, B. and Brindle, K. (2000). Membrane bioreactors for wastewater treatment. IWA Publishing