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<u>Filtration: a literature review</u>

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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. This anaerobic compartment is operated at thermophilic conditions. The end products of this compartment are CO_2, VFA and ammonia. The production of methane, formed under anaerobic conditions, is inhibited, because it is of no use in the further compartments. CO_2 is supplied to the photosynthetic compartment, where *Spirulina platensis* and higher plants convert this CO_2 into oxygen. The volatile fatty acids and the ammonia are fed into the second phototrofic anoxygenic compartment, containing *Rhodospirillum rubrum*. Up to now, the volatile fatty acids and the ammonium are separated from the liquefying reactor content by centrifugation at a speed of 3000 rpm. The supernatant, separated from the cake, contains 96% of the total volatile fatty acids and 79% of the total ammonia, but also 40% of total dry weight is present in the supernatant. This must be avoided, therefore better separation techniques need to be found.

In this technical note, a few techniques will be investigated.

2. Separation techniques

2.1 Introduction

In this chapter a few separation techniques were separately described. Most of the techniques mentioned were used for the separation of animal manure.

2.2 Sedimentation and screening

Sedimentation and screening are the most commonly used techniques for animal manure treatment. Sedimentation is most effective for treating dilute wastewaters, such as flushed manure.

Settling of solids simply takes advantage of gravity to separate the solids from the liquids. A detention time as short as 30 minutes can be used to settle out the solids. The liquid can be removed from the top by means of a pump. This method can remove up to 50% of the solids (Hermanson, 1993). Coagulating agents such as ferric chloride, lime, alum and organic polymers can greatly improve the dewatering characteristics of manure. These chemicals bring the solids in manure together so they settle more rapidly. With chemical precipitation , it is possible to remove 80 to 90 % of the suspended solids(Metcalf & Eddy,1991).

Moore et al. (1975) measured sedimentation efficiency with time for manure slurries from several livestock species and reported that over 60% of TS (Total solids) from a dairy slurry can be removed in the first 10 minutes of settling.

Safley and Owens (1986) sedimented poultry slurry of 5 to 6% TS and observed a 57% reduction in TS. They found that with poultry manure above 7% TS, little or no settling occurred because the product remained a homogeneous mixture.

Screening solids from dairy manures effectively removes larger particles from flushed manure. However, the 24% of TS removed with screens of 2 mm or larger probably is a maximum to expect from screens on dairy farms (Powers et al., 1995).

Pain et al. (1978) evaluated the use of a vibrating screen for dairy and swine waste slurries containing up to 12% TS and found that the screens were ineffective above 8% TS, because the slurry accumulated on top of the screen.

2.3 Chemical treatment

Several studies have been reported in the literature on the chemical treatment of animal manure for solid-liquid separation.

Coagulation and flocculation of particles in wastewater by means of chemical addition are physic-chemical processes. Coagulation is a process of aggregating suspended particles to form settable flocs through additions of electrolytes or organic polymers. The electrolytes are multivalent cations or inorganic salts of such metals as iron, aluminum and calcium. These metal ions react with hydroxyl ions and/or carbonate ions in the wastewater to form settable flocs so that indigenous suspended particles can be made to settle together with these newly formed flocs by sweeping actions (Tchobanoglous et al., 1991). The most commonly metal salts are ferric chloride, aluminum sulfate and lime.

Flocculation is a process of agglomerating coagulated particles into large, rapidly settling flocs. It is effected through particle-particle interaction under appropriate electrolytic environments or through attachment to long chain polymer molecules. Polymers are complex molecules and are characterised by their molecular weight and charge density. Generally, the higher molecular weight and the longer the molecular chain, the more effective the polymer is for flocculating particles. Next to synthetic polymers, natural polymers can be used. These natural polymers include modified starches and chitosan (a product made from fish shells). Most of the synthetic polymers are the derivatives of polyacrilamide (PAM).

Powers et al. (1995) tested four chemicals on dairy manure slurries with 0.5%, 1% and 1.5% TS. The TS removal after the treatment with chemicals was increased by 18 to 30% in comparison with no use of the chemicals.

Hanna et al. (1985) treated flushed swine manure (1% TS) with nine chemicals among which $MgCl_2$, $Al_2(SO_4)_3$ (1000ppm), $Ca(OH)_2$ (1000 ppm). Their results showed that the volatile solids removal was increased by 8 to 13% with the use of different chemicals as compared with no use of chemicals.

Different separation processes may have different requirements for the size and density of particle flocs to be formed (Zhang et al., 1998). For example, if sedimentation or centrifugation is to be used as the separation method following the chemical treatment, heavy and dense particles will be desired, but if screening is used, large and tight flocs will be desired. Zhang et al. tested $FeCl_3$ for different TS levels of two different manures (swine manure and dairy manure). The $FeCl_3$ dosage was 100 to 1500 mg/l for swine manure and 250 to 1750 mg/l for dairy manure. Also 5 cationic polyacrilamide polymers and two metal salts were investigated. There is an optimum dosage of $FeCl_3$ for each TS level what can be observed in Figure 2-1. At the optimum dosage, the coagulated particles had the highest density and settled the fastest. Beyond the optimum dosage, however, the particles became larger and fluffy, resulting in a reduced settling rate.

For a given TS level, an increase in the polymer dosage had a positive impact on the solids removal until the polymer dosage reached a certain level. Beyond this level, further increase of polymer dosage would have little more impact (Figure 2-2, Figure 2-3).

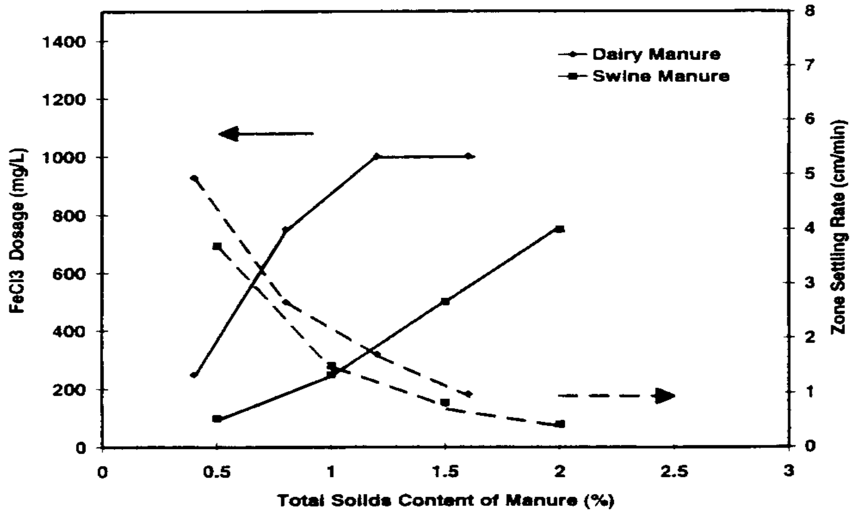

Figure 2-1 Optimum $FeCl_3$ dosage and zone settling rate for different total solids contents of swine and dairy manure

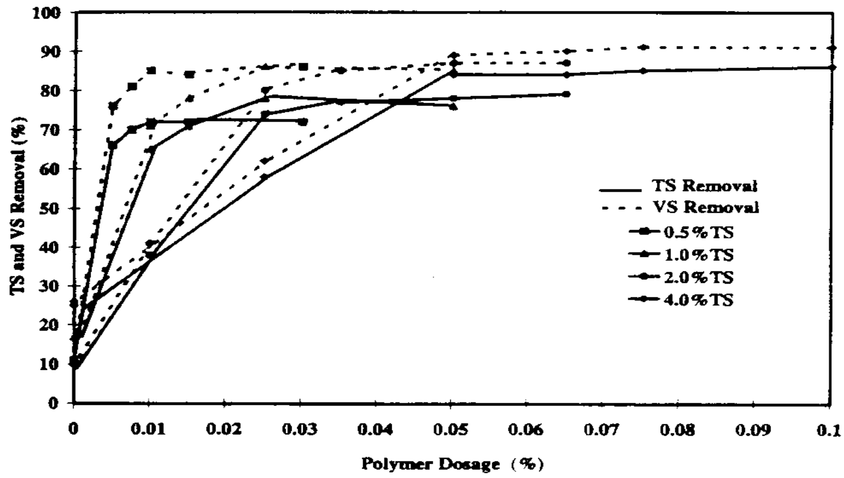

Figure 2-2 Effect of polymer dosage on the solids removal for swine manure

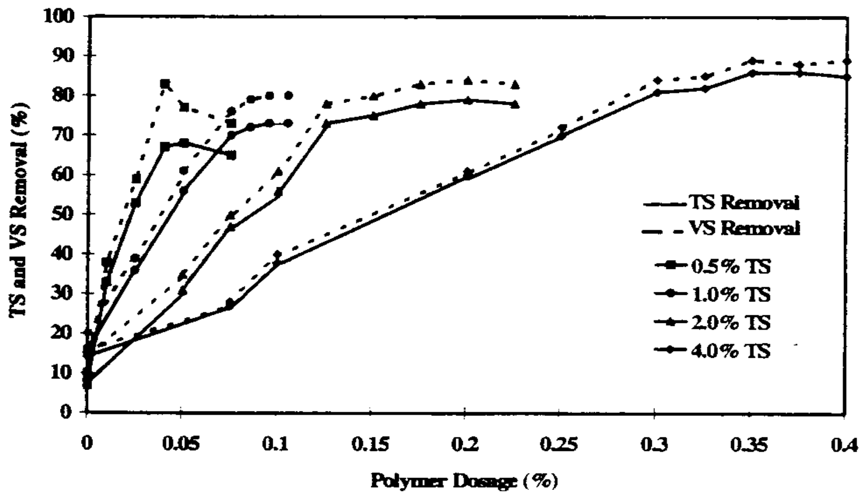

Figure 2-3 Effect of polymer dosage on the solids removal from dairy manure

These studies confirm the positive effects of chemical treatment on improving the solid removal from animal manure.

This method can not be applied within the MELiSSA concept, since the use of chemical products in the MELiSSA loop must be avoided, as the compounds can not be produced within the loop and have to be taken from earth.

2.4 Centrifugation

Centrifugation is a separation process which uses the action of centrifugal force to promote accelerated settling of particles in a solid-liquid mixture. The centrifugation of the MELiSSA effluent, obtained after the biodegradation of human faecal material, was tested and the results were represented in TN43.2. It could be concluded that a centrifugation of biodegraded faecal material at 3000 rpm resulted in a centrifugate containing still 42% of the total dry weight. Taking into account these results it can be proposed to apply centrifugation as a first step of a series of separation techniques, but it can not be applied as a single separation technique to obtain a centrifugate without solids. Increasing the speed of the centrifuge will result in an increase in solid separation, but still this method needs to be combined with other separation techniques.

2.5 Filtration

2.5.1 Introduction

Filtration is by far the most widely used method in the treatment of sludge. Filtration is a separation process that consists in passing a solid-liquid mixture through a porous material (filter) which retains the solids and allows the liquid to pass through. Clogging of filters are of frequent occurrence. The clogging rate depends on:

- 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)
- The filtration rate: the higher the rate, the higher the turbulence at the membrane, and therefore the limited the clogging of the membrane
- Temperature: the higher the temperature, the lower the viscosity, the higher the flux.

- The characteristics of the filter material: size of the pores, uniform particle size, roughness, shape of the material

- Type of filtration (Figure 2-4): dead end or 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.

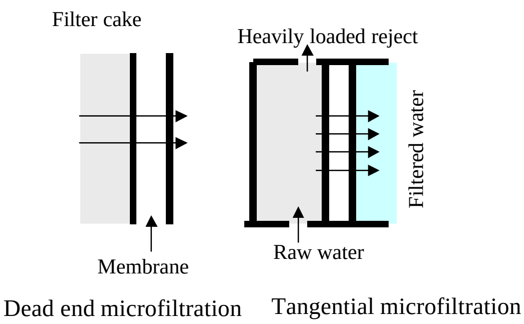

Figure 2-4 Microfiltration modes

Raspoet et al. investigated the separation of *Spirulina* from the liquid. From this investigation could be concluded that a filter press was the most efficient method. The concept is represented in Figure 2-5.

Spirulina enters the press cylinder . The sucker presses the medium through the filter. *Spirulina* remains on the filter and the filtrate flows through the filter.

A metal Miltipor microfilter was taken because with this filter, unlike other filters, the *Spirulina* remained on top of the filter and could be easily removed. These filters have a more accurate filtration, less pressure drop, longer life time and easy to clean by back pressure.

The filter press can be tested with MELiSSA effluent. The cake on top of the filter can be recycled to the thermophilic demonstration reactor, so that a higher retention time and maybe a better biodegradation efficiency will be obtained.

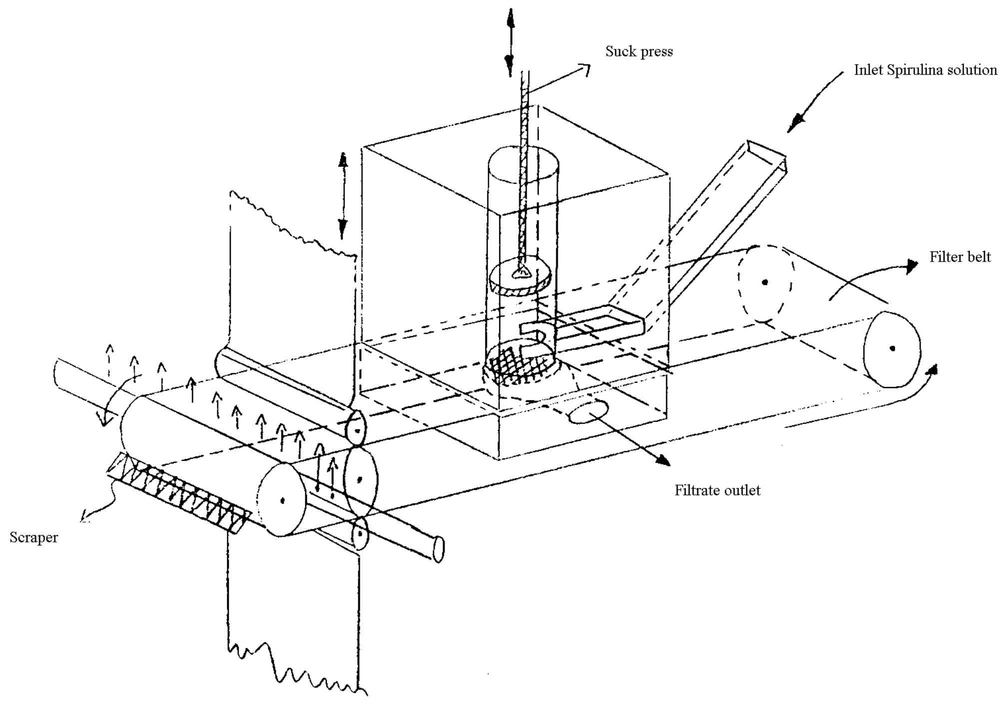

Figure 2-5 Concept of the filter press

2.5.2 Separation by membranes

2.5.2.1 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. This classification is represented in Figure 2-6.

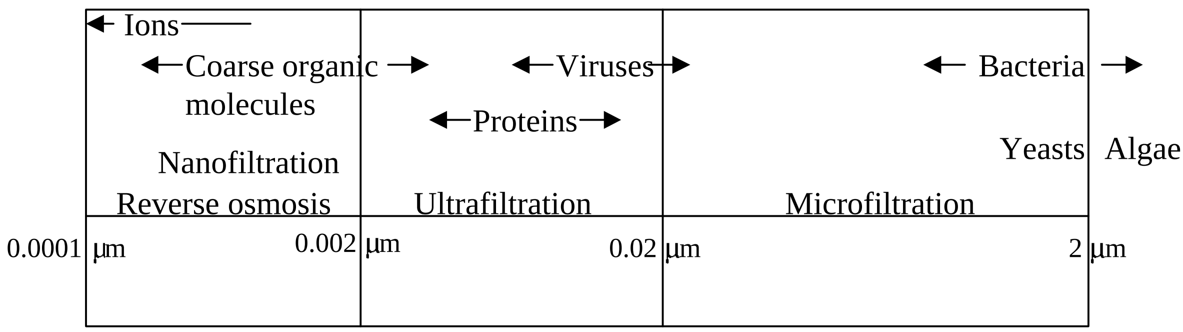

Figure 2-6 Types of membrane filtration

2.5.2.2 Membrane separation of raw and anaerobically digested pig manure by reverse osmosis

Many farms in Norway have a disproportionate number of animals compared to their arable land. A biogas plant, a reverse osmosis (RO) membrane plant and a composting plant were built to convert the manure in useful energy, clean water and odourless organic fertiliser.

The objectives of the RO studies were to determine whether membrane separation of anaerobically digested pig manure was a suitable polishing technique for the effluent permeate as well a volume reduction device for the concentrate.

The digested manure was chemically conditioned by adding a cationic polymer prior to mechanical dewatering through a screw press. The screw press was made of galvanised and stainless steel. The thick fraction after dewatering was approximately 25% solids, whereas the liquid fraction was 1% solids.

A schematic overview of the manure processing pilot plant is represented in Figure 2-7.

The reverse osmosis module consisted of 18 tubular polyamide membranes each with 12.5 inside diameter and an individual length of 1219 mm. The total membrane area available for separation was 0.861 m^2. The following membrane operating parameters were: P<7 MPa, T<70°C and 3<pH<11.

Chemical cleaning of the pilot plant consisted of circulating a 0.3% HNO_3 solution at 40°C for 45 minutes through the RO pant. This procedure was followed by an alkaline detergent (Ultrasil 11) also at 40°C for 45 minutes.

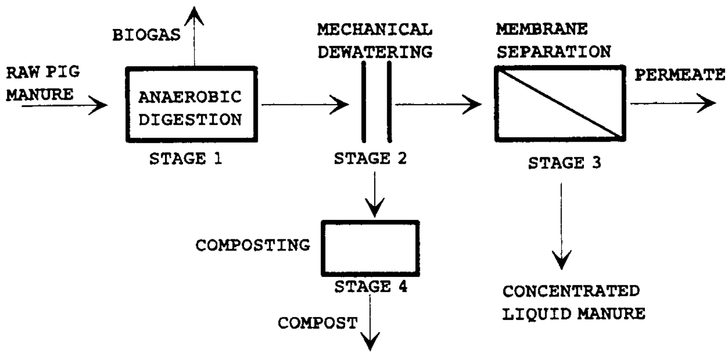

Figure 2-7 Diagram of the pig manure pilot processing plant

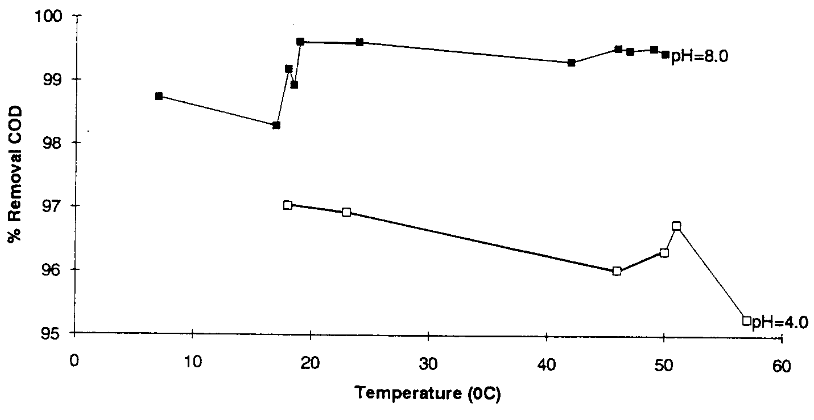

Figure 2-8 Organic removal measured as function of feed temperature and pH

Organic separation is expressed as % COD removal and is above 98% at pH 8 and around 96% at pH 4. This slight decrease in separation performance with decreasing pH is likely due to some solubilisation of organics at pH 4 compared with pH 8. There is no change in removal efficiency of COD with increasing temperature for any pH value.

It can be concluded that membrane separation by reverse osmosis is a good method for polishing the effluent from dewatered pig manure. The anaerobically digested pig manure was concentrated 8 times by reverse osmosis with an appreciable flux and acceptable quality of the permeate.

2.5.2.3 Ultra- and Microfiltration of MELiSSA cake

2.5.2.3.1 Introduction

For this test MELiSS A effluent was collected and stored in the freezer. After the centrifugation, at 3000 rpm and during 15 minutes, the MELiSSA supernatant, containing still 40% of the total dry weight, was filtered using three different tubular ultrafiltration membranes. During the tests, the flux was followed up in order to notice any clogging. In Figure 2-9 the set-up of the test is represented. During the test, the concentrate and permeate was recycled continuously and added to the MELiSSA supernatant in order to obtain a high recirculation flow to avoid immediate clogging of the membrane.

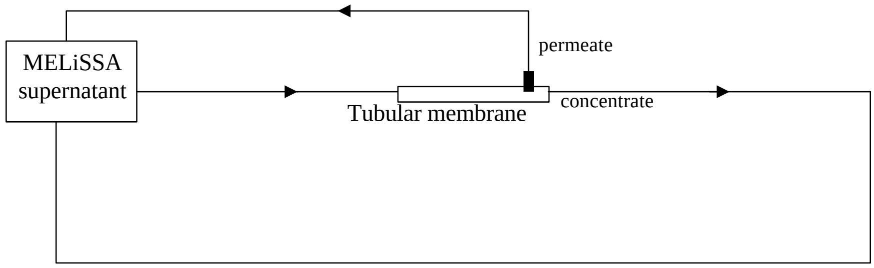

Figure 2-9 Simplified representation of test set-up

2.5.2.3.2 Tested membranes

Three membranes were tested by VITO : WFA4125, WFB4125 and WFF4385. More details of these three membranes are described in Table 2-1.

Table 2-1 Characteristics of the filtration membranes

Membrane	***Filtration***	***Composition***	***MWCO (Dalton)***	***pore size (nm)***
WFA4125	ultafiltration	polyacrilonitrile	100 000	-
WFB4125	ultrafiltration	polysulfon	50 000	-
WFF4385	microfiltration	polyvinylidene fluoride	-	30

MWCO: molecular weight cut off (size of the restrained molecules)

The used membranes were tubular with an internal diameter of 5 mm and a surface of 0.0082 m^2, wich is very small and suitable within the MELiSSA concept. To obtain a flow through speed of V= 4.1 m/s, the recirculation flow was Q=290 L/h. The transmembrane pressure (TMP) varied between 37 and 200 kPa.

2.5.2.3.3 Results

The results of the different membranes are represented in Table 2-2. In 245 minutes, the flux decreased from 74 until 40 L/m^2*h when using the WFA4125 membrane. The WFB4125 membrane was only tested for about half an hour. During this half an hour, the flux was 60 L/m^2*h.

The WWF4385 membrane had the best result. Although the flow through speed in this test was reasonable high (4m/s), a flux of 60 $L/(m^2$*h) was achieved at a restricted TMP (about 40 kPa) and a restricted temperature (about 25°C).

The WWF4385 membrane is developed to produce reasonable fluxes with a limited flow through speed and TMP.

Table 2-2 Fluxes when using different membranes

Membrane	*Filtration time (min)*	*TMP (kPa)*	*V (m/s)*	*T (°C)*	*Flux (L/m^2*h)*
WFA4125	0	100	4.1	8.5	74
	10	100	4.1	8.5	40
	30	100	4.1	10.5	32
	75	100	4.1	16	40
	200	182	4.1	22	63
	205	100	4.1	22	42
	245	100	4.1	22	42
WFF4385	265	100	4.1	22	88
	300	200	4.1	22	74
	315	100	4.1	24	76
WFB4125	330	100	4.1	24	60
	345	100	4.1	24	59
	365	100	4.1	24	58
WFF4385	385	100	4.1	24	84
	390	37	4.1	24	74
	1305	37	4.1	26.5	60

TMP: trans membrane pressure

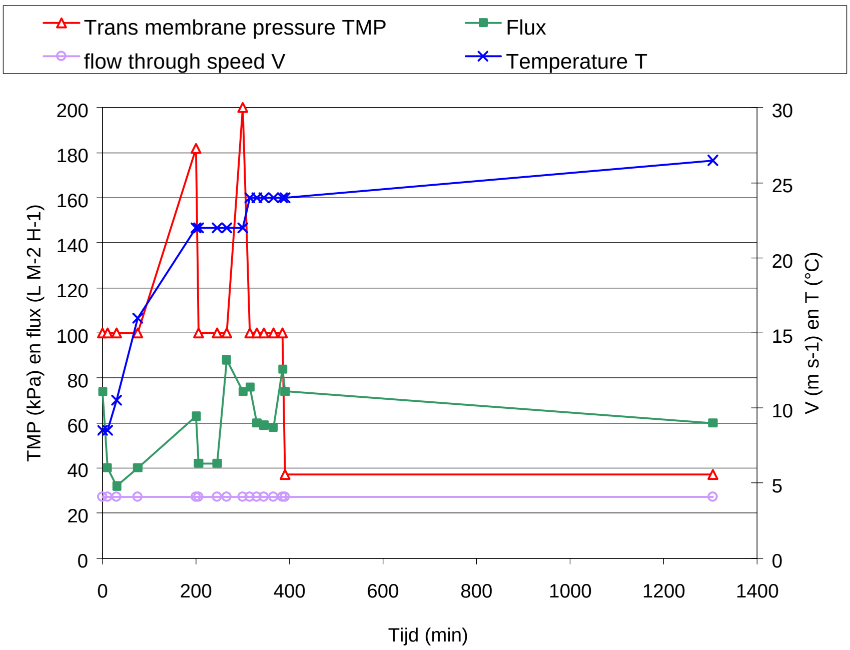

Figure 2-10 TMP, flux and V with different membranes and MELiSSA supernatant

In future experiments membrane WFF4385 will be further tested with effluent from the anaerobic MELiSSA demonstration reactor. First preliminary tests will be started up without a pre centrifugation of the MELiSSA effluent. The MELiSSA effluent will be collected for a certain period of time and then filtrated using the membrane WFF4385. The dry weight, volatile fatty acids, ammonia and the decrease in flux will be closely followed up. To obtain a high recirculation flow, permeate and concentrate will be collected and recycled to the MELiSSA effluent tank. In a second test compartment 1, the liquefying compartment, and compartment 2, the photoheterotrophic compartment, will be connected with each other. The effluent from the thermophilic anaerobic reactor will be collected and introduced in a tubular membrane. The volatile fatty acids and ammonia will be separated from the non biodegraded organic matter. The permeate containing the VFA and ammonia will be fed into the photoheterotrophic compartment and the concentrate containing the non biodegraded organic matter, will be recycled to the anaerobic compartment. In order to investigate the efficiency of the membrane, several parameters under which volatile fatty acids, ammonia, dry weight etc. will be measured. In Figure 2-11 a schematic overview of the connection between the two compartments is represented.

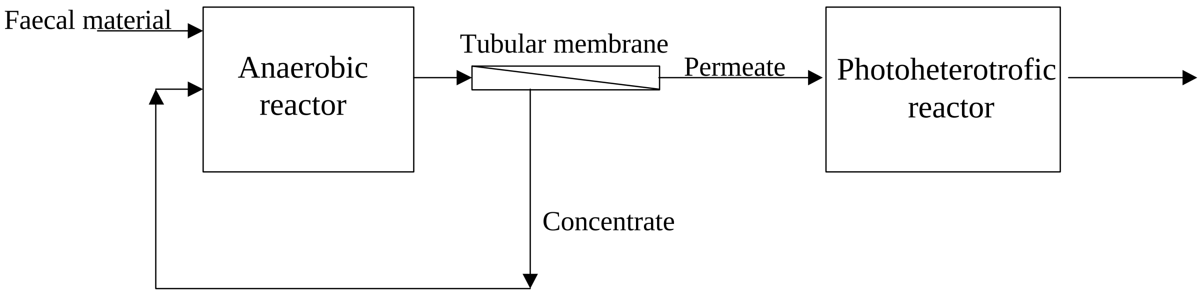

Figure 2-11 Schematic overview of the connection of two compartments

3. Conclusions

The most widely used method for the separation of solids from the liquid of animal manure is sedimentation and screening, with the addition of coagulants and flocculants. This method is not suitable for the MELiSSA cycle, since external chemicals are necessary.

42% of the solids remained in the centrifugate after centrifugation at a speed of 3000 rpm. Centrifugation can be used as a first step of a series of separation techniques but not as a single separation technique.

The separation by membranes is a good technique to separate manure. Reverse osmosis was tested with animal manure after an anaerobic digestion and a mechanical dewatering. The anaerobically digested pig manure was concentrated 8 times. The screw press for dewatering the digested manure is probably too large and can only be used when high amounts of manure is available. In case of the MELiSSA concept, using a screw press is excluded. An Ultrafiltration and microfiltration technique was performed with the MELiSSA supernatant, using three different membranes. Based on the fluxes, it could be concluded that the WWF4385 membrane was the most suitable membrane. This membrane needs to be further investigated with MELiSSA effluent. With these results a set-up will be made and tested in order to connect the anaerobic compartment and the photoherotrophic compartment.

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