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The filtration characteristics of anaerobic digester effluents employing cross flow ceramic membrane microfiltration for nutrient recovery

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Abstract

In the present study, a monolithic alumina coated microfiltration ceramic membrane was used for solid particulates removal and nutrients recovery from anaerobic digester complex effluent streams. The aim was to test the effect of the cake layer developed by the solids, on the surface of the membrane channels, to the filterability of these materials. The solids content ranged between 2.6 g/L to 15.1 g/L. During practical application, two processing techniques targeting the enhanced recovery of the materials of interest including ammonia, phosphate, calcium bicarbonate and volatile fatty acids, namely dewatering and diafiltration, were used. These had an immediate effect on the solids content (PDS 13 μ m to 3.97 μ m) enhancing the filterability of the effluents. Their processability was evaluated in terms of flux, cross flow velocity, membrane resistance and cake resistance. Important findings of this study is the nonalignment of the flux rates to the cake resistance, explained by the formation of a compressible, permeable cake layer that allowed the continuous operation of the system, under constant low pressure conditions (TMP 15 psi). Permeate flux remained constant to 120 L/m² h when applying diafiltration, while when dewatering process is used the permeate flux remained constant at 115.4 L/m² h.

Keywords: sludge; ceramic filter; cake resistance; flux; cross flow filtration

Highlights

- Digested effluents filterability was tested by a ceramic MF system of processing volume 140 L/m² h.
- Pretreatment scheme reduced TS by 20.75%, 48.58% coarse particles (PDS 27.17 to 13.97 μ m).
- Diafiltration and dewatering schemes were tested for fluids processability at TMP 15 psi.
- A compressible permeable cake layer was formed allowing continuous operation in DF
- Enhanced filterability of fluids and varying concentration in nutrients is found using DF

1. Introduction

A key operation in the sustainable use of materials is the removal and recycling of nutrients and energy from the anaerobic digesters [1-3].

In the quest for substitute fossil fuel alternatives sources, the development of anaerobic digesters for biogas production used for combined heat and power (CHP) has been proposed [4, 5, 44]. Being relatively easy to be constructed, enhancing local and national economies by supporting small and medium sized companies [6] and relying on a well known and widely investigated process of anaerobic fermentation [7], the use of anaerobic digesters has seen rapid expansion throughout western Europe and United States [8]. In the Western economies the production via anaerobic digestion of biogas for power generation represents the 1.2% of the annual production of electricity and nearly 10% of renewable energy, with an installed power close to 1500MW [9].

However, as the raw materials used as substrate during the digestion are mainly animal, crop and food wastes, the waste effluents that are generated from the process can be potentially hazardous to human health and environment having high concentration of freely available nutrients. The current treatment predominantly involves land spreading which can potentially cause eutrophication and land toxicity due to excessive phosphate and ammonia application [10]. There are also human health concerns due to land related pathogenicity [11, 12] contained in the raw materials.

These concerns have highlighted the problems of sludge disposal. At the same time the value of nutrients is increasing due to high manufacturing costs or reduced availability such that recovery is of vital importance and is becoming economically viable [13]. For example, ammonia and phosphate are becoming more costly, since ammonia synthesis has an inherently large carbon footprint and consequently is heavily dependant on fossil fuel prices, and phosphate production is already thought to have peaked, hence the need for an effective treatment method has emerged [14]. Mechanical separation and recovery of nutrients from waste, using membrane processes, has been proposed and applied to many simple, well defined waste systems, e.g. recovery of cells metabolites and water [15].

Microfiltration pore size ranging from 0.1 μm to 1 μm allows smaller particles such as aqueous salts, small macromolecules, carbohydrates, proteins, metal ions and other inorganic and organic molecules to pass through formulating a sterile permeate effluent [16]. With larger particles removed these permeates can be further processed to recover useful nutrients as well as metal ions, or even used as substrates for microbial growth, which is an emerging option in the constantly evolving field of biotechnology [17]. Several beneficial features lay in membrane processing, [18-20] including reuse and more economical disposal of waste [21,22], formulation of sterile streams [23] low pressure operation, ease of in-situ separation in addition to simple scale-up using commercial modules. These processes have shown treatment feasibility for several types of aqueous waste streams [24]. Research has been focused on treatment of municipal, domestic and sewage sludge [10, 11, 19, 20, 23, 24].

The main problem that develops is membrane fouling which needs to be avoided and may require frequent cleaning of the membrane to manage the process effectively [21]. Several researchers [25, 26] have investigated the filterability of sludge types in relation to cake formation due to solids deposition on the membrane surface [27, 28]. Cake formation results in increased membrane resistance and decreased flux i.e. limited cost effective recovery of the materials of interest through decreased membrane productivity and increased energy consumption [29]. These processes involve the use of synthetic membranes such as polysulfone or polypropylene or inorganic, ceramic membranes [30]. Ceramic membranes have been widely applied in the industry, although due to their high cost compared to their polymeric counterparts, their application has been limited in the field of food, beverage and pharmaceutical industry [47, 48]. However, their exceptional advantages, chemical and thermal stability as well as robust structural stability have attracted interest to their potential use in the treatment of waste streams [49]. Ceramic filters, either monolithic or tubular have been proven effective for the separation of various colloidal effluents of micron and sub-micron suspended particles [50]. Monolithic membranes have numerous parallel channels arranged in the axial direction, with the inner surface of the channels acting as filters offering a large membrane area suitable for processing significant amounts of effluent [38, 41, 51]. Ceramic filters are fabricated using alumina, zirconia or zeolite, materials that withstand extreme pH, pressure conditions and high flux rates [47].

These characteristics facilitate effective cleaning with acidic or alkali solutions, indicating ceramic membranes as ideal candidates for processing complex effluent streams of sludge nature [49]. Ceramic membrane configuration, does allow the deposition of particles in the inner side of the channels, forming a cake, which may hinder the permeate flux. Moreover, experimental investigation of waste stream has been limited to small scale [31, 32], where the fluid and membrane arrangements offer limited information on the applicability of these methods and processing techniques on the

industrial scale.

Therefore, this work reports the filtration of spent agricultural sludge through a ceramic membrane filter in a pilot scale arrangement. Its filterability has been evaluated in terms of flux, membrane resistance and cake resistance, using various operating conditions. Attempts have been made to correlate the solids contents and characteristics with the filterability of sludge using different treatment schemes. These correlations, when applied with the universal model for flux prediction, can be used to predict the filtration behavior of these complex systems, as well as to identify the necessary processing time needed to extract the necessary amounts of nutrients.

2. Materials and Methods

2.1. Materials

Waste effluent (agricultural wastewater derived from agricultural digested sludge namely mixed waste of cattle slurry, vegetable waste and silage) stream samples taken off the output line of a sedimentation tank, before any treatment.) were collected from Farm Renewable Environmental Energy Limited (Fre), Wrexham, United Kingdom. These were samples were taken of the output line of the anaerobic digester used for manure production before passing through the automatic coarse particle separator (>5mm) and collected in 25L capacity plastic jerry cans.

2.2. Experimental

2.2.1. Effluents Pre-Treatment Schemes

The effluents were considered to be rich in solids, mostly comprised of large particles i.e. straw, stones. These had to be removed for the successful filtration of the effluents. A pre-treatment scheme was developed to address this problem, combining dilution with tap water (50% v/v), thorough mixing for one hour using a rod, twenty-four hour sedimentation in a settling tank and treatment of the supernatant with a series of coarse filters varying in pore size between 1.045 mm to 0.5 mm [58].

2.2.2. Filtration Unit Design

The waste was processed through a cross-flow microfiltration unit (Fig.1). The unit consisted of a 100 L stainless steel vessel linked via 5 m of 1 inch stainless steel piping arranged in two fluid loops each driven by a centrifugal pump, Kennet -12-2 (Stuart Turner, UK). Waste was passed from the tank into the first pump loop which pressurised the system against a diaphragm valve (Axium Process, Hendy, Wales, UK) on the return side, which could be adjusted to control the pressure. Within this loop a second pump circuit feeding the membrane (Membralox ceramic (α -Al₂O₃) monolith microfiltration

(pore size 0.2 μ m) , able to withstand a pH range between 0-14, fitted in stainless steel, commercially available by Pall (Portsmouth, UK)) and water cooled heat exchanger Axium Process, Hendy, Wales, UK) enabled high flow rate around the loop. There was very little pressure dropping in this loop and as such high fluid velocity over the membrane surface was achieved, which could be kept constant over a range pressures. The membrane comprised of 19 channels, of 3.70 mm diameter each and length of 1016 mm. The effective membrane area was determined as 0.22m². All the parts of the unit were connected with stainless steel heavy duty clamps and sealed with 1.5 inches clamp lipped solid PTFE seals, provided by Axium Process, Hendy, Wales, UK.

2.2.3. Membrane Characterisation

Membrane characterisation studies using tap water were carried out to determine the membrane resistance and the influence of pressure during the operation of the system. The permeability of tap water was measured in order to analyze the behaviour of the system, using a graduated cylinder and a stopwatch. The membrane resistance was calculated after every run from flux measurements as a function of transmembrane pressure. It was calculated from the slope of the steady state membrane flux over the trans membrane pressure, at 25 °C, all the calculations used the value 8.90*10⁻⁴ Pa s⁻¹ for the viscosity of water.

2.2.4. Processing Schemes

The processing of sludge was carried out using two schemes (Fig.2 a, b): dewatering, where the filtration characteristics of the sludge are a function of its concentration and diafiltration, where the filtration characteristics were studied as a function of dilution of the liquid in the sludge. These procedures are described in detail below.

Diafiltration: The purpose of diafiltration was to investigate the effects of removing the soluble components of the sludge. The batch process involved 4 sequential washes which consisted of first concentration and then dilution of the sludge with fresh tap water. Initially 30 L of the pre-treated sludge were collected and placed in the feed vessel and then concentrated to 20 litres, the permeate was then discarded. In the concentrated sludge, 20 litres in the vessel, 10 L of tap water were added and then processed by the unit, to collect 10 L of permeate. This was repeated three more times. The permeate flow rate was manually recorded using a graduated vessel where the permeate fluid was collected. The difference in volume was recorded per minute using a stopwatch (Casio electronics, UK); on a two decimal points precision electronic scale (OHAUS I-10)

Dewatering: 30 L of the pre-treated sludge were placed in the feed vessel and filtered. 10 L of permeate were collected. The concentrated sludge, 20 litres in the vessel, filtered through the sludge

filtration unit. 5 L of permeate were collected. The remaining 15L in the feed vessel concentrated sludge, were filtered again through unit and 5L of permeate were collected. Finally, the remaining 10L of concentrated sludge was filtered through the unit. The difference in volume was recorded as described on diafiltration section.

2.2.5. Analysis of dry matter content and physicochemical characteristics

Total solids (TS, g/L), total suspended solids (TSS, g/L), total dissolved solids (TDS), alkalinity, and optical density were determined according to APHA, 1998. Particle size distribution (PSD) of the sludge samples was determined by light scattering technique using Mastersizer 2000 (Malvern, UK), the zeta potential was determined by the Zetasizer (Malvern, UK) the conductivity and salinity of the samples were measured used a conductivity meter (Russell systems, UK) calibrated with a standard solution of 0.1M of KCl. Butyric and acetic acid were determined using head space gas chromatography [56], nitrogen measured as ammonia (NH₃-N) and phosphorous (PO₄-P) using the phenate and vanadomolybdo-phosphoric acid colorimetric methods respectively as described by APHA, 1998. A spectrophotometer UV-Visible UNICAM UV300 dual beam was used for both methods. Each parameter was triplicated to obtain the average data (standard deviation of mean <5%, standard error <7%) offering highly significant results. When necessary, samples were diluted with deionised water to fit within the calibration range.

2.3. Theoretical

2.3.1. Determination of the Filtration Parameters

For the determination of flux and other parameters the following equation [33-36] was used

Flux (J) in the system was determined as

$$J = \left(\frac{\Delta P * \Pi}{(R_m + R_c) * \mu} \right)$$

[1]

Transmembrane pressure (ΔP) was defined as

$$\Delta P = \text{TMP} = \left(\frac{P_{\text{inl}} + P_{\text{out}}}{2} \right) - P_{\text{permeate}}$$

[2]

The permeate flux was defined as

$$J_{\text{permeate}} = \left(\frac{Q_f}{A_m} \right) = \left(\frac{dV}{dt} \right) \left(\frac{1}{A_m} \right)$$

[3]

The total membrane resistance was also calculated by

$$R_T = (R_m + R_c)$$

[4]

where the membrane resistance was defined by Darcy's law as

$$R_m = \frac{\Delta P}{J * \mu}$$

[5]

that for the calculation of the cake resistance becomes

$$R_c = \left(\frac{\Delta P}{J * \mu} \right) - R_m$$

[6]

where the R_m equals to the R_m of water under the same operating conditions.

For cross flow velocity the following equation was used

$$U = \left[\frac{(Q_f)}{(\pi * r^2) * n} \right]$$

[7]

3. Results and Discussion

3.1. Physical Characteristics of Agricultural Waste Effluent Streams

Twenty five liter (25 L) sludge samples were taken from the anaerobic digester without any on site processing. These materials required some pretreatment to allow the sludge to be easily handled within the filtration unit [37]. The pretreatment scheme was a combination of dilution and sedimentation, this enhanced the removal of larger particulates of the anaerobically digested effluents (<100 μm) and facilitated their filterability through the ceramic filter. Dilution allowed the disengagement of the chemicals and nutrients bound in the solids, facilitating their recovery ensuring the settled, undisturbed and economical, due to operating conditions being in low pressures of the system. Reduction to the total solids content by 20.75% (15.13 g/L to 11.99 g/L) was observed; in color by 12.5% (0.86 to 0.70 at 580nm) while the mean particle size dropped by 48.58% (27.17 μm to 13.97 μm)[56]. Significant reduction was observed in the TSS content, 58.75%, thus making the effluent to be filtered a simpler material to be processed (Table 1). In addition to the successful removal of large particulate matter, it was also possible to recover some important nutrients in the supernatant fluid that are normally loosely associated with the solids. These successfully recovered materials of interest can be formulated, through further processing with membrane technology i.e. UF, NF, and RO into effluents suitable for use as biofertilisers or as nutrient media for microbial fermentations, so to produce biofuels and chemicals.

3.2. Characterization Study of the Ceramic Filter

The permeability of water through the membrane was measured to analyze the behavior of the unit. The flux values and cross-flow velocity linearly increased with increasing pressure. For water the flux increased from 148 to 539 $\text{L}/\text{m}^2 \text{ h}$ with an increase in outlet pressure from 0 to 20 psi, thus cross flow velocity increased from 3.05 m/s to 10.89 m/s. The membrane permeability (L) was defined by the slope of the linear functions using the plots of the flux over the TMP. It is characteristic to the unfouled membrane and was calculated as 18.5 m. The system is designed and developed to operate efficiently into various pressure conditions, allowing high productivity. High flux and cross flow velocity can be achieved into low pressure conditions, enabling continuous operation of the system with limited obstructions. Operating the system into low pressure conditions does enable the development of a cost effective, due to the controlled energy consumption, scalable mechanical treatment of agricultural wastewater, in the context of recovery of valuable nutrients.

3.3. Filtration Characteristics of Sludge using Dewatering or Diafiltration Strategy

The effluents were filtered in the dual loop microfiltration system under constant temperature and pressure control, with one centrifugal pump being used in a recirculation loop to maintain high constant fluid velocity across the membrane while the second pump introduced the fluid and pressurized the system, establishing a cross-flow microfiltration system. Two schemes were applied for its treatment, namely, diafiltration and dewatering.

3.3.1. Filtration characteristics using Diafiltration Strategy

The filterability of the digested effluents, using diafiltration (Fig.2a), was evaluated in terms of flux, total membrane resistance and cross flow velocity. The cross flow velocity remained within a range of 2.32 m/s to 3.22 m/s through the processing of the effluents at 15 psi TMP (Table 1), while flux varied between $3.17 \times 10^{-5} \text{ m}^3/\text{m}^2 \text{ h}$ to $3.55 \times 10^{-5} \text{ m}^3/\text{m}^2 \text{ h}$ (Fig. 3). Over the course of the filtration, the total membrane resistance gradually increased, 4.07×10^{12} to 4.67×10^{12} , due to the continuous deposition of matter on the membrane channels, since particulates larger than the membrane pore size ($>0.2 \mu\text{m}$) are retained. A cake was formed on the inner surface of the membrane channels, reflected by the development of the cake resistance at each washing step, varying between 1.19×10^{12} and 1.85×10^{12} . The leaching process has an effect on the composition of the digested fluids in the feed, with a mean size drop of particulates from $13.97 \mu\text{m}$ to $3.97 \mu\text{m}$. This is further reflected by the decreased amount of particles in the feed at each step of the process with TS from 11.9 to 2.6 g/L, TSS varying between 252.6 mg/L to 174.54 mg/L and TDS from 7743 mg/L to 943.5 mg/L (Table 1).

Consequently, the effect of the cake resistance is minimized; the fluids are transferred across the membrane, leaving the flux relatively unaffected. The cake is presumably permeable due to the diafiltration pattern followed that allows its continuous leaching, altering significantly the chemical properties of the digested effluents. The changing content of ions, due to the hydrolysis of the ionic bonds is shown by the gradual reduction of conductivity ($9.11 \text{ mS}/\text{cm}^{-1}$ to $1.11 \text{ mS}/\text{cm}^{-1}$) zeta potential (-30.06 mV to -23.25 mV) and alkalinity ($5000 \text{ mg CaCO}_3/\text{L}$ to $1250 \text{ mg CaCO}_3/\text{L}$), positively influences the filterability of the digested fluids. This is done by consisting the particles less absorbent to the membrane surface as well as soluble in water allowing the continuous filtration of sludge in low pressure operation. This benefits greatly the operation of the system into long term since interruptions due to cleaning of the system with expensive chemical agents or back flushing are avoided. Color of the digested effluents, was successfully removed (OD from 0.70 to 0.08, 88.57% total reduction) through the four leaching stages of this process (Table 1). Consequently the process treats effectively the organic matter content in the digested effluents, since color is commonly caused by organic decomposition products from vegetation or a result of impurities of minerals such as iron and manganese.

Microfiltration is a pressure driven process. Consequently, changes in pressure differential are

considered to have an impact on the filterability of the fluids. The effect of a range of TMP on the diafiltration pattern varying between 10 to 27 psi was tested (Fig.4). It was found that the system can be successfully operated at higher pressures; however there are limited variations in the flux between the different concentrations of filtered effluents, making the operation of the system in a low pressure differential (TMP 15 psi) preferable as being more economic in terms of energy consumption.

3.3.2. Filtration characteristics using Dewatering Strategy

The application of the dewatering scheme (Fig.2b) into continuous filtration of the treated digested sludge has a different effect of the processability of the digested fluids. The cross flow velocity varied slightly from 2.05 m/s to 2.16 m/s, flux was slightly reduced (3.73×10^{-5} to 3.69×10^{-5}) with simultaneous increase of total membrane resistance (3.96×10^{12} to 4.61×10^{12}) (Table 2). This is due to the continuous deposition of solids on the membrane surface, a phenomenon reflected by the rising cake resistance, from 1.21×10^{12} to 4.61×10^{12} during the four dewatering steps. Similar pattern of flux decline was observed when a varying set of TMP (10 to 27 psi) values was applied, with flux declining at every dewatering step (Fig.6).

The flux did remain elevated (Fig.5), throughout the process suggesting that even with continuous dewatering, the system can still handle sludge. This is due to the nature of the sludge as well as to the pretreatment scheme, which allowed the elimination of coarse particles.

Dewatering strategy did not strongly influence the physical properties of the processed fluids, with slight deviations being found in TS, from 10.4 g/L to 14.9 g/L, TDS from 7658.50 mg/L to 7072 mg/L and mean particle size of 13.49 μm to 12.89 μm (Table 2). TSS though changed significantly from 258.00 mg/L to 511.33 mg/L, since the cross flow arrangement of the system allowed continuous leaching of the solids deposited on the membranes channels, this resulted in higher content of particulates in the feed vessel. Chemical properties of the processed fluids were relatively constant, pH was increased from 8.34 to 8.45 while a decrease in alkalinity from 6875 mg CaCO_3/L to 5000 mg CaCO_3/L , conductivity (9.01 mS/cm^{-1} to 8.32 mS/cm^{-1}) and zeta potential (-30.06 to -27.50 mV) was observed.

These values regarding processability (Table 1, 2) do show that both filtration processes namely diafiltration and dewatering could be carried out successfully on these complex streams. With both strategies, the cross flow velocity of the system did remain high throughout the process allowing continuous filtration of the feed fluid, using the developed configuration. Such a finding does indicate the potential of the processing membrane system in terms of scalability. Often phenomena of drag, low flow velocity and fouling do occur when such systems are being built in industrial scale, as shown by limited studies that were conducted to simulate large scale conditions, especially in terms of

mechanical structure and configuration [39-42]. In this case, the configuration of the system can be easily extended to large scale with minimal problems.

3.3.3. Cost Estimation

The wide adaptation of these waste processing schemes is strongly correlated with the cost efficiency of these applications when compared to the conventional methods of waste treatment and production of chemicals. Estimating the cost of these processes though is rather complicated as several factors have to be taken into consideration, such as capital cost related to manufacturing and maintenance of the system and relevant equipment, labor costs, energy consumption and transportation of waste [57].

Preliminary energy cost studies in un-optimized MF systems have indicated that the energy cost per cubic meter (m^3) of sludge (11.9 g/l dry solids containing 56.31 mmols L^{-1}N , 1.31 mmols L^{-1} of P and 22.11 acetic acid and 16.71 butyric acid mmols L^{-1} VFA) processed is £0.91-1.02 kWh (using potable water £0.75 m^3). However, when this methodology are applied industrially, potable water usage can be replaced with rainwater, minimizing significantly the cost (£0.91-£0.16). Recovery per kg of ammonia, phosphate and VFA was calculated as £1.4. Further details regarding the cost of recovery of the materials of interest have been reported elsewhere [57].

3.4. Discussion

Filtration treatment of the waste effluents has been proposed throughout the literature [45] and has often been applied in the industry [46]. Having successfully removed a large part of the solids due to the pre-treatment scheme, the effluents were filtered through a cross filtration unit equipped with a ceramic membrane. Nevertheless, when using diafiltration strategy in a varying range of TMP, cake resistance was considerably reduced when compared to the cake resistance occurring during dewatering strategy (Fig.7). At the final sequential step in either dewatering or diafiltration, the highest cake resistance occurred, due to the formation of a compressible cake. However, this was easily permeable in the case of diafiltration, since the flux remains elevated through the range of TMP by the retention of particles by the membrane. Diafiltration strategy allows the successful continuous operation of the system in lower transmembrane pressures.

In both process, there is a strong dependence of the system on the TSS (Fig.8) since when high concentration of TSS were found even in high TMP the cake resistance increased (Fig.9), resulting in lower flux and consequently lower productivity. The cake resistance can be correlated also with the size of the solids and the ionic properties of the digested fluids reflected by the zeta potential (Fig.10). Dewatering treatment is proven to contribute to elevated cake resistance, as the particle size and zeta potential remains almost unchanged while TSS concentration becomes higher. This might cause the

formation of an insoluble irreversible cake on the membrane layer, resulting into the inability of the system to process the fluids. Therefore, diafiltration is proven beneficial and cost effective; treating the commonly faced problem of formation of insoluble salts deposits on the membrane surface.

These treatments can potentially ensure the formulation of microbial particle free effluents, safe for disposal in the landfills. Animal waste can cause health hazards related to microbial load as well as toxic compounds that can be potentially dangerous to human health. Membrane filtration offers a viable alternative to the current techniques for waste management.

Having therefore, successfully valorized the effluents by removing coarse particles, indigenous microbial/viral load, toxic substances and colorants, the produced effluents can be used as source of nutrients, organics and salts that when precisely formulated, can serve as fertiliser and growth medium for microbial production of platform chemicals and biofuels. These effluents, if used as nutrient media, are potentially highly profitable, especially when compared to the traditional synthetic media or that derived from food sources such as crops. Filtration allows manipulation of the nutrient content, since it can be combined with leaching and acidification using microfiltration or selective separation and concentration using subsequent nanofiltration and reverse osmosis processes. This approach has several advantages such as: recycled materials that will substitute for newly synthesized or mined materials; the reduction in the volume and concentration of waste will reduce demand and costs in waste treatment plants; creation of valuable streams such as formulated of nutrient streams for application in agriculture and bioprocessing [58].

Within this context when diafiltration is applied, effluents are produced of different ratios of nutrient content including ammonia, phosphate, acetic and butyric acid (Table 3). Each washing step reduces the amount of nutrients in the effluents, gradually depleting the digested sludge and making it safe for disposal in the environment. The depleted sludge, having a small amount of phosphate and ammonia can be recycled by being placed back in the processing system. The processing time needed for each step is lower than the time needed for dewatering (Table 3), the economic operation of the system due to elevated flux and cross flow velocity, make diafiltration a highly effective system in terms of productivity and fluids processability. Furthermore, the composition of these effluents can be modified accordingly to address specific nutritional needs of industrially relevant microorganisms, this can be potentially highly profitable, especially when compared to the traditional synthetic media or that derived from food sources such as crops.

On the other hand, dewatering is proven to be uneconomical, since longer time for processing is needed, as well as interruptions due to cleaning and maintenance as when the system is operated continuously this results in formation of cake. In terms of nutrient production, the concentration of substances of interest in the effluents remains constant, allowing limited manipulation and benefiting

only in volume reduction and nutrient depletion.

4. Conclusions

These results suggested that complex effluent streams after pre-treatment and screening to remove the large particles can be filtered.

- The filterability of sludge was tested on a pilot scale unit, equipped with a ceramic membrane, capable of processing up to 140 L/m² h volume.
- The pre-treatment scheme applied had a significant effect on the filterability of sludge, reducing by 20.75% total solids and by 48.58% coarse particles (PDS 27.17 to 13.97 μm)
- Diafiltration had an immediate effect on the solids content (PDS 13μm to 3.97μm), colour (0.70 to 0.08 nm) and conductivity (9.11 to 1.10 mS/cm⁻¹).
- Independence of the flux rates to the cake resistance was found for both treatments, explained by the formation of a compressible permeable cake layer that allowed the continuous operation of the system, under constant low pressure conditions (TMP 15 psi).

Filtration processes, cross- flow microfiltration and diafiltration, could be carried out successfully on sludge to produce clear, sterile, particle free solutions. Membrane processing can establish an alternative to the current disposal techniques, allowing the formulation of a valorized waste effluents, that can be further processed for the recovery of valuable nutrients. These effluents are suitable to be used as nutrient media for industrially relevant fermentations , for example for the production of bioethanol or as fertilisers. Using the general filtration model, correlating the membrane resistance with the sludge properties, can be further applied to forecast the behavior of other waste effluent.

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Nomenclature

J	Flux ($\text{m}^3/\text{m}^2 \text{ s}$)
ΔP	Pressure differential (psi)
Π	Osmotic pressure
R_m	Membrane Resistance (m^{-1})
R_c	Cake Resistance (m^{-1})
μ_{water}	Viscosity _{water} ($\text{N m}^2/\text{s}$)
TMP	Transmembrane pressure (psi)
P_{inl}	Pressure inlet (psi)
P_{out}	Pressure outlet (psi)
P_{permeate}	Pressure of permeate (psi)
dV	Volume differential (L)
dt	Time differential (s)
A_m	Membrane area (m^2)
Q_f	Flow rate (L/h)
R_t	Total Resistance (m^{-1})
J_{permeate}	Permeate Flux ($\text{m}^3/\text{m}^2 \text{ s}$)
U	Velocity (m/s)
n	Number of channels in the membrane
r	Radius of the circular membrane channel (m^2)
π	mathematical constant (3.14159)
A_m	Cross-sectional membrane area (m^2)
TS	Total solids (g/L)
TSS	Total suspended solids (mg/L)
TDS	Total dissolved solids (mg/L)

References

- [1] A. A. Dimakis, M. Biberacher, J. Dominguez, G. Fiorese, S. Gadocha, E. Gnansounou, G. Guariso, A. Kartalidis, L. Panichelli, I. Pinedo, M. Robba, Methods and tools to evaluate the availability of renewable energy sources, *Renewable Sustainable Energy Reviews*. 15 (2011) 1182-1200.
- [2] M. Jefferson, Accelerating the transition to sustainable energy systems, *Energy Pol.* 36 (2008) 4116-4125.
- [3] P.A. Østergaard, Comparing electricity, heat and biogas storages' impacts on renewable energy integration, *Energy* 37 (2012) 255-262.
- [4] L. Appels, J. Baeyens, J. Degrève, R. Dewil, Principles and potential of the anaerobic

- digestion of waste-activated sludge, *Progr. Energy Comb. Sci.* 34 (2008) 755-781.
- [5] E.A. Salminen, J. A. Rintala, Semi-continuous anaerobic digestion of solid poultry slaughterhouse waste: effect of hydraulic retention time and loading, *Water Res.*36 (2002) 3175-3182.
- [6] E.A. Salminen, J.A. Rintala, Anaerobic digestion of organic solid poultry slaughterhouse waste – a review, *Bioresource Technology* 83 (2002) 13-26.
- [7] T. Amon, B.Amon, V.Kryvoruchko, A. Machmüller, K. Hopfner-Sixt, V. Bodiroza, R. Hrbek, J. Friedel, E. Pötsch, H. Wagentristl, M.Schreiner, W. Zollitsch , Methane production through anaerobic digestion of various energy crops grown in sustainable crop rotations, *Bioresource Technology* 98 (2007) 3204-3212.
- [8] A. Bauer, H. Mayr, K. Hopfner-Sixt, T. Amon, Detailed monitoring of two biogas plants and mechanical solid–liquid separation of fermentation residues, *J. Biotech.* 142 (2009) 56-63.
- [9] D. Antoni, V.V. Zverlov, W. H. Schwarz, Biofuels from microbes *Applied Microbiology Biotechnology* 77 (2007) 23–35.
- [10] W. Fuchs H. Binder, G. Mavrias, R. Braun, Anaerobic treatment of wastewater with high organic content using a stirred tank reactor coupled with a membrane filtration unit, *Water Research* 37 (2003) 902–908.
- [11] J. Cho, K.-G. Song, K.-H. Ahn, The activated sludge and microbial substances influences on membrane fouling in submerged membrane bioreactor:unstirred batch cell test, *Desalination* 183 (2005) 425-429.
- [12] S.B. Sadr Ghayeni, P.J. Beatson, A.J. Fane, R.P. Schneider, Bacterial passage through microfiltration membranes in wastewater applications, *Journal of Membrane Science* 153 (1999) 71-82.
- [13] S.B. Sadr Ghayeni, S.S. Madaeni, A.G. Fane, R.P. Scheider, Aspects of microfiltration and reverse osmosis in municipal wastewater reuse, *Desalination* 106 (1996) 25-19.
- [14] I.S. Jung, R.W. Lovitt, Leaching techniques to remove metals and potentially hazardous nutrients from trout farm sludge, *Water Research* 45 (2011) 5977-5986.
- [15] R. van Reis, A. Zydney, Bioprocess membrane technology, *J. Mem. Sci.* 297 (2007) 16-50.
- [16] J. M. Coulson, J. F. Richardson, *Chemical Engineering, Particle Technology and Separation Processes*, third ed., Butterworth-Heinemann, Oxford, 1991.
- [17] I.S. Jung, R.W. Lovitt, Integrated production of long chain polysaturated fatty acids (PUFA)-rich *Schizochytrium*, biomass using a nutrient supplemented marine aquaculture wastewater, *Aquaculture Engineering* 43 (2010) 51-61.
- [18] H. Fang, X. Shi, Pore fouling of microfiltration membranes by activated sludge, *Journal of Membrane Science* 264 (2005) 161-166.
- [19] A.L. Lim, R. Bai, Membrane fouling and cleaning in microfiltration of activated sludge wastewater, *Journal of Membrane Science* 216 (2003) 279-290.

- [20] Y. Shimizu, K. Uryu, Y.-I. Okuno, S. Ohtubo, A. Watanabe, Effect of particle size distributions of activated sludges on cross-flow microfiltration flux for submerged membranes, *Journal of Fermentation and Bioengineering* 83 (1997) 583-589.
- [21] F.J. Benitez, J.A. Acero, A.I. Leal, Application of microfiltration and ultrafiltration processes to cork processing wastewaters and assessment of membrane fouling, *Separation and Purification Technology* 50 (2006) 354-364.
- [22] G.D. M. Masson, Membrane filtration and reverse osmosis purification of sewage : Secondary effluent for re-use at Eraring Power Station, *Desalination* 106 (1996) 11-15.
- [23] R. Bai, H.F. Leow, Microfiltration of activated sludge wastewater-the effect of system operation parameters, *Separation and Purification Technology* 29 (2002) 189-198.
- [24] L. Chu, S.Li, Filtration capability and operational characteristics of dynamic membrane bioreactor and municipal wastewater treatment, *Separation and Purification Technology* 51 (2006) 173-179.
- [25] L.Seminario, R. Rozas, R. Borquez, P.G. Toledo, Pore blocking and permeability reduction in cross-flow microfiltration, *Journal of Membrane Science* 206 (2002) 121-142.
- [26] H. Yukseler, I. Tosun, U. Yetis, A new approach in assessing slurry filterability, *Journal Membrane Science*. 303 (2007) 72-79.
- [27] H.J. Lin, K. Xie, B. Mahedran, D.M. Bagley, K.T. Leung, S.N. Liss, B.Q. Liao, Factors affecting sludge cake formation in submerged anaerobic membrane bioreactor, *Journal of Membrane Science* 361 (2010) 126-134.
- [28] W. Lee, S. Kang, H. Shin, Sludge characteristics and their contribution to microfiltration in submerged membrane bioreactors, *Journal Membrane Science*. 216 (2003) 217-227.
- [29] Y. Xu, T. Dabros, J. Kan, Filterability of oil sands tailings, *Process Safety Environmental Protection* 86 (2008) 268-276.
- [30] N. Dizge, G. Soydemir, A. Karagunduz, B. Keskinler, Influence of type and pore size of membranes on cross flow microfiltration of biological suspension, *Journal of Membrane Science* 366 (2011) 278-285.
- [31] P. Krzeminski, A. Iglesias-Obelleiro, G.Madebo, J.M. Garrido, J.H.J.M. van der Graaf, Impact of temperature on raw wastewater composition and activated sludge filterability in full-scale MBR systems for municipal sewage treatment, *Journal of Membrane Science* (2012).
- [32] R.W. Field, D. Wu, J.A. Howell, B.B. Gupta, Critical flux concept for microfiltration fouling, *Journal of Membrane Science* 100 (1995) 259-272.
- [33] M.Mulder, *Basic Principles of Membrane Technology*, second ed., Springer, Dordrecht, 1996.
- [34] S. D. Lin, C. C. Lee, *Water and Wastewater Calculations Manual*, second ed., McGraw-Hill Chichester, 2007.
- [35] N. Kaushik, *Membrane Separation Processes*, first ed., PHI Learning, India, 2008
- [36] R. Baker, *Membrane Technology and Applications*, first ed., McGraw-Hill, Chichester, 2000.

- [37] Metcalf, Eddy, G. Tchobanoglous, F. L. Burton, H. D. Stensel, Wastewater Engineering: Treatment and Reuse, fourth ed., Mac-Graw Hill, Chichester, 2002.
- [38] C. Reith, B. Birkenhead, Membranes enabling the affordable and cost effective reuse of wastewater as an alternative water source, *Desalination* (1998) 203-210.
- [39] D. Jeison, J.B. van Lier, Cake formation and consolidation: Main factors governing the applicable flux in anaerobic submerged membrane bioreactors (AnSMBR) treating acidified wastewaters, *Separation and Purification Technology* 56 (2007) 71-78.
- [40] D. Jeison, J.B. van Lier, Cake layer formation in anaerobic submerged membrane bioreactors (AnSMBR) for wastewater treatment, *J. Mem. Sci.* 284 (2006) 227-236.
- [41] E. van Houtte, J. Verbauwhe, F. Vanlerberghe, S. Demunter, J. Cabooter, Treating different types of raw water with micro- and ultrafiltration for further desalination using reverse osmosis, *Desalination*. 117 (1998) 49-60.
- [42] F. Meng, H. Zhang, F. Yang, S. Zhang, Y. Li, X. Zhang, Identification of activated sludge properties affecting membrane fouling in submerged membrane bioreactors, *Separation and Purification Technology*. 51 (2006) 95-103.
- [43] J. Ho, S. Sung, Effects of solids concentrations and cross-flow hydrodynamics on microfiltration of anaerobic sludge, *Journal Membrane Science*. 345 (2009) 142-147.
- [44] V. Nallathambi Gunaseelan, Anaerobic digestion of biomass for methane production: a review, *Biomistry and Bioengineering* 13 (1997) 83-114.
- [45] J.A. Müller, Prospects and problems of sludge pre-treatment processes, *Water Science & Technology* 44 (2001) 121-128.
- [46] W. Nah, Y. W. Kang, K.-Y. Hwang, W.-K. Song, Mechanical pretreatment of waste activated sludge for anaerobic digestion process, *Water Research* 34 (2000) 2362-2368.
- [47] K. Araki, H. Sakai, Ceramic membrane development in NGK, *IOP Conference Series: Materials Science Engineering* (2011).
- [48] K. Parameshwaran, A.G. Fane, B.D. Cho, K.J. Kim, Analysis of microfiltration performance with constant flux processing of secondary effluent, *Water Research*. 35 (2001) 4349-4358.
- [49] R. J. Ciora, P.K.T. Liu, Ceramic membranes for environmental related applications, *Fluid Part. Separation Journal* 15 (2003) 51-60.
- [50] I.-J. Kang, S.-H. Yoon, C.-H. Lee, Comparison of the filtration characteristics of organic and inorganic membranes in a membrane-coupled anaerobic bioreactor, *Water Research*. 36 (2002) 1803-1813.
- [51] F. Waeger, T. Delhaye, W. Fuchs, The use of ceramic microfiltration and ultrafiltration membranes for particle removal from anaerobic digester effluents, *Separation and Purification Technology*. 73 (2010) 271-278.
- [52] J.-O. Kim, S.-K. Kim, R.-H. Kim, Filtration performance of ceramic membrane for the recovery of volatile fatty acids from liquid organic sludge, *Desalination* 172 (2005) 119-127.

- [53] L. Fan, T. Nguyen, F.A. Roddick, J.L. Harris, Low-pressure membrane filtration of secondary effluent in water reuse: pre-treatment for fouling reduction, *Journal of Membrane Science* 320 (2008) 135-142.
- [54] A. Spagni, S. Casu, N.A. Crispino, R. Farina, D. Mattioli, Filterability in a submerged anaerobic membrane bioreactor, *Desalination* 250 (2010) 787-792.
- [55] E. Iritani, N. Katagiri, T. Sengoku, K.M. Yoo, K. Kawasaki, A. Matsuda, Flux decline behaviors in dead-end microfiltration of activated sludge and its supernatant, *Journal of Membrane Science* 300 (2007) 36-44.
- [56] M.P. Zacharof and R. W. Lovitt, Recovery of volatile fatty acids (VFA) from complex waste effluents using membranes. *Water Science and Technology* 2013 (**Article In Press**) DOI:10.2166/wst.2013.717
- [57] M.L. Gerardo, M.P. Zacharof, R. W. Lovitt, Strategies for the recovery of nutrients and metals from anaerobically digested dairy farm sludge using cross-flow microfiltration. *Water Research* (2013) 47, 4833-4842
- [58] M.P. Zacharof and R. W. Lovitt, Complex effluent streams as a potential source of volatile fatty acids. *Waste and Biomass Valorisation* (2013) 4, 577-581

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