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Biogas Production from Concentrated Municipal Sewage by Forward Osmosis, Micro and Ultrafiltration

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Abstract: Direct application of anaerobic digestion to sewage treatment is normally only possible under tropical weather conditions. This is the result of its diluted nature and temperatures far from those suitable for anaerobic conversion of organic matter. Then, direct application of anaerobic treatment to sewage would require changing temperature, concentration, or both. Modification of sewage temperature would require much more energy than contained in the organic matter. Then, the feasible alternative seems to be the application of a pre-concentration step that may be accomplished by membrane filtration. This research studied the pre-concentration of municipal sewage as a potential strategy to enable the direct anaerobic conversion of organic matter. Three different membrane processes were tested: microfiltration, ultrafiltration and forward osmosis. The methane potential of the concentrates was determined. Results show that biogas production from the FO-concentrate was higher, most likely because of a higher rejection. However, salt increase due to rejection and reverse flux of ions from the draw solution may affect anaerobic digestion performance.

Keywords: sewage; forward osmosis; microfiltration; ultrafiltration; concentration; biogas

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1. Introduction

Anaerobic digestion is nowadays an established environmental technology, providing a reliable and affordable treatment alternative for a wide variety of wastewaters. However, direct application of anaerobic digestion to sewage treatment is normally only possible under tropical weather conditions as a result of wastewater temperature and a low organic matter concentration. Then, sewage treatment is generally conducted by aerobic processes. The traditional aerobic treatment is an effective technology for reducing the organic matter concentration of sewage. However, it implies an extensive use of energy. Indeed, aeration represents the most relevant operational cost for aerobic sewage treatment.

Figure 1a presents a theoretical chemical oxygen demand (COD) balance for what could be considered a traditional sewage treatment facility: aerobic treatment by activated sludge, combined with anaerobic digestion of secondary sludge. It is clear that only a small fraction of the chemical energy contained in sewage may be potentially recovered as methane in such a process. Data provided by this figure include theoretical methane yields, so practical energy recovery is normally much lower. A process based on the direct application of anaerobic digestion, followed by an aerobic post-treatment, would greatly increase the potential energy recovery in the form of methane, as is clear when comparing Figure 1a,b.

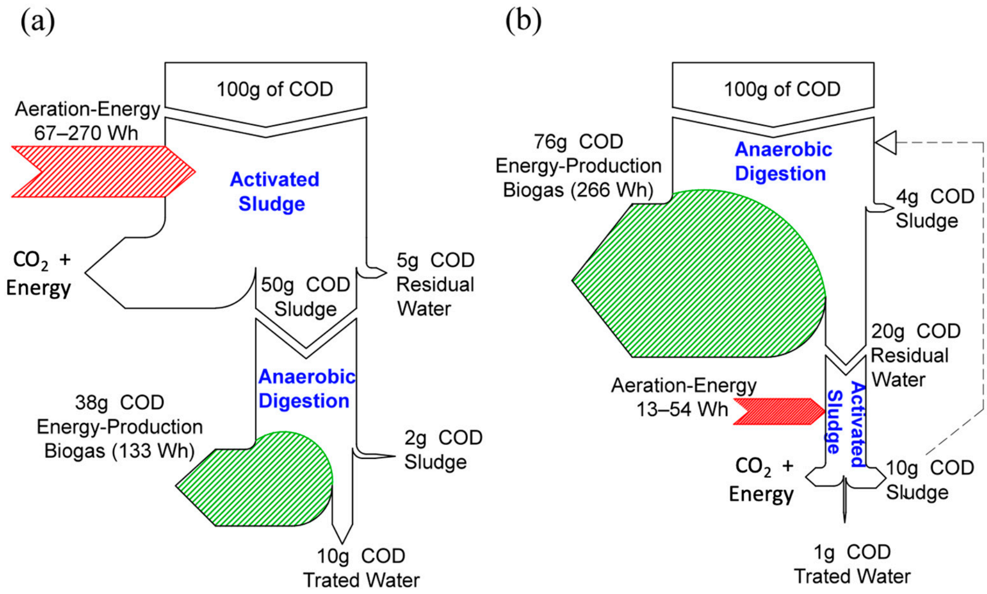

Figure 1. Theoretical COD balance of potential sewage treatment processes: (**a**) aerobic–anaerobic and (**b**) anaerobic–aerobic configuration. Balance considers a theoretical methane production of 0.35 L CH_4 $gCOD^{-1}$, and a methane (lower) heating value of 10 Wh L^{-1}.

The direct application of anaerobic treatment to sewage would require the modification of temperature and/or concentration since both factors are known to determine the performance of anaerobic digestion. The increase in temperature seems an unlikely approach since it would require much more energy than that contained in the organic matter of sewage. Then, the feasible alternative seems to be the application of a pre-concentration step. Water freezing has been reported as a tool for wastewater concentration. By crystallizing water into ice, pollutants can be concentrated in the remaining liquid [1–3]. However, this approach may only be an alternative in geographical zones presenting very low ambient temperatures. Water evaporation is another alternative [4,5]. However, the elevated enthalpy of the evaporation of water would demand high energetic requirements for diluted wastewaters, such as sewage. Moreover, it may induce emissions of volatile substances, such as volatile organic compounds (VOC). Membrane processes are more likely to represent an alternative for sewage concentration. Indeed, the application of membrane technology to wastewater treatment is not new; the application of membrane bioreactors is a clear example [6,7]. The development of membrane processes for environmental applications during the last 3 decades has produced relevant improvements in membrane manufacture and has steadily decreased their costs. Moreover, a series of emerging membrane technologies has arisen as alternatives for water reclamation [8]. As a result, membrane separation techniques are nowadays applied in fields where their use may have been considered unlikely not so long ago, such as the treatment of municipal and industrial wastes [9–11]. The potential advantages of membrane processes, when compared with other separation techniques, are: low energy consumption, environmental friendliness, and high quality permeate.

The concentration of wastewater by membranes, which has normally been referred to as direct membrane filtration (DMF), has been already reported in literature. Indeed, recently, Hube et al. [12] reviewed the use of direct membrane filtration for wastewater treatment, identifying DMF as a tool for resource recovery from wastewaters. In general, moderate to high levels of organic matter retention have been reported. Kimura et al. [13] reported 75% of organic matter recovery using a microfiltration membrane of 0.1 µm of pore size. Gong et al. [14] reported similar levels of recovery, using a pilot-scale hollow-fiber PVDF (polyvinylidene fluoride) membrane with 0.02 µm pore diameter. Similar performance was reported by Lateef et al. [15] when working with a microfiltration (MF) concentration unit. On the other hand, Ravazzini et al. [16] only achieved up to 40% retention of chemical oxygen demand (COD) using ultrafiltration (UF). Differences in performance are expected, because of different sewage characteristics, since COD removal will largely depend on the contribution of solids, colloids and solutes to total COD.

Although MF and UF can retain part of the COD, they are ineffective in retaining nitrogen and phosphorus since a large part of these nutrients are in sewage as soluble compounds [17]. An efficient sewage concentration would need a process enabling retention of all pollutants contained in sewage, including solids, colloids and soluble material. Forward osmosis (FO) is a concentration process based on the natural phenomenon of osmosis. It has gathered increasing attention during the last decade as an alternative for traditional pressure-driven membrane processes, such as reverse osmosis (RO) or nanofiltration (NF). Recently, it has also been proposed as an alternative for sewage treatment or pre-concentration [18]. Osmosis is a natural process that promotes the transport of water between two fluids presenting different osmotic potentials, separated by a selective membrane: the feed and a draw solution. Then, as a result of water migration, the solution presenting higher osmotic potential is diluted, and the solution of lower osmotic potential is concentrated. A secondary process is also required in order to re-concentrate the diluted draw solution so that it can be used in a closed-loop cycle. Although first developments dealing with FO were based on the use of RO membranes, membranes specially developed for FO processes are now readily available in the market [10,19]. The FO-based concentration of sewage has been already reported [20–22]. However, few reports include assays to directly determine the methane potential of the produced concentrates. Moreover, reported research does not usually involve the testing of different membrane technologies with the same sewage. Sewage properties can change depending on the particular characteristics of the community it comes from, making performance comparison between different reports difficult.

This research studied the performance of MF, UF and FO processes for pre-concentration of municipal sewage. Biogenic methane potential tests (BMP) were used in order to determine the potential energy that could be recovered from the concentrated sewage.

2. Materials and Methods

2.1. Sewage

Experiments were conducted with real pre-settled sewage from the city of Temuco, Chile. Sewage was collected at the city's sewage treatment plant from the effluent of the primary settler. After collection, municipal sewage was stored at 4 °C. Pre-concentration processes started within the first 24 h following sewage collection. Pre-settled sewage presented COD concentration, total solids (TS) and volatile solids (VS) of 0.25, 0.56 and 0.34 g L^{-1}, respectively.

2.2. Sewage Concentration Setup

Sewage was concentrated by 3 membrane processes: MF, UF and FO. MF concentration was performed using a tubular ceramic membrane with a pore diameter of 0.2 µm. The membrane tube was 60 cm long and had an internal diameter of 0.5 cm. UF was carried out using a tubular polymeric membrane of 30 nm pore diameter. The membrane tube was 70 cm long and had an internal diameter of 0.8 cm. In both cases, filtration modules contained a sole membrane tube. Table 1 presents the general characteristics of membranes used in this study. Before filtration experiments, MF and UF membranes were subjected to 30 min of oxidative cleaning using NaOCl, providing a free chlorine concentration of 500 mg L^{-1}. During MF and UF operation, transmembrane pressure (TMP) was measured online by means of a pressure transducer. Permeate was collected by means of a peristaltic pump, which provided the required TMP. During MF and UF concentration processes, the flux was automatically controlled in order to keep it nearby the critical permeate flux. A tool previously reported was used for that purpose [23]. Both membranes were operated in cross-flow mode, according to the setup shown in Figure 2a. Initial membrane resistances (measured with clean water) were 9.4×10^{10} and 2.7×10^{11} m^{-1} for MF and UF, respectively.

The FO concentration process was conducted using cellulose triacetate with an embedded polyester screen flat-sheet membrane (CTA) with 0.03 m^2 of membrane area. A new membrane was used during this assay. The membrane was used with the active layer facing the feed side (sewage). The system was operated in cross-flow mode, and no spacers were used. A 0.6 M solution of NaCl was used as a draw solution. Figure 2b presents a schematic representation of the FO filtration setup.

Initial sewage volumes were between 6 and 7 L for the tested membrane processes. The fed (sewage) was kept refrigerated during the concentration process. Once the concentration process was finished, water was flushed through the setup as a way to remove organics that may have remained in the system as fouling layers.

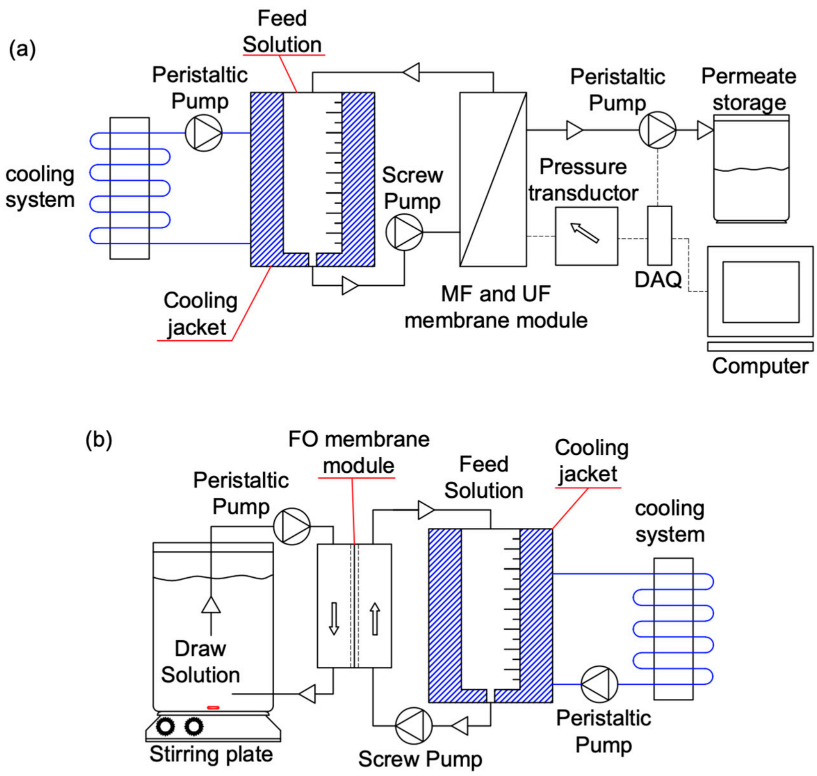

Figure 2. Filtration setups used in this study. (**a**) Micro and ultrafiltration. (**b**) Forward osmosis setup.

Table 1. General characteristics of the membranes used in this study.

	MF	UF	FO
Provider	Atech Innovations, Germany	X-Flow Norit, The Netherlands	Hydration Technology Innovations, USA
Material	Ceramic aluminum oxide (Al_2O_3)	Polymeric	Cellulose triacetate with embedded polyester screen flat-sheet membrane (CTA)
Pore size	0.2 µm	20 nm	-
Configuration	Tubular, inside/out	Tubular, inside/out	Flat sheet
Filtration area	0.0094 m^2	0.0176 m^2	0.03 m^2

2.3. Biogenic Methane Potential

BMP assays were conducted to determine the energetic potential of concentrated sewage through MF, UF and FO processes. Assays were conducted in serum bottles of 120 and 80 mL of total and reaction volume, respectively. Granular sludge from a full-scale UASB reactor treating brewery wastewater was used as inoculum. Substrate (concentrated sewage) was supplemented with 200 mg L^{-1} yeast extract, 65 mg L^{-1} NH_4Cl, 18.5 mg L^{-1} KH_2PO_4, 4 mg L^{-1} $CaCl_2 \cdot 2H_2O$ and 5.7 mg L^{-1} $MgSO_4 \cdot 7H_2O$. Sodium bicarbonate was also added to provide buffer capacity (5 g L^{-1}). Reactor bottles were flashed for a duration of 30 s with a mixture of N_2/CO_2 (70/30% respectively) in order to displace the oxygen present in the headspace and then were incubated at 35 °C. Methane production was determined following pressure increase and gas composition in the headspace of the serum bottles. Pressure was determined with a pressure transducer (Cole Parmer, model 200). Biogas composition was measured by gas chromatography with a thermal conductivity detector (Clarus 580, Perkin Elmer, Waltham, MA, USA). BMP is presented, and the volume of produced methane (in standard conditions) per mass of VS is present in the concentrated sewage. Assays were conducted in triplicate, and blank tests without substrate (concentrated sewage) were included to determine endogenous methane production.

A modified Gompertz model [24] was used to analyze and compare data provided by BMP tests. The model is given by the following expression:

$$P = P_m \cdot exp\left(-exp\left(\frac{R_m \cdot e}{P_m}(\lambda - t) + 1\right)\right)$$

where P is the produced methane at time t ($mLCH_4$ gVS^{-1}), P_m is maximum methane production ($mLCH_4$ gVS^{-1}), R_m is the methane production rate ($mLCH_4$ gVS^{-1} d^{-1}) and λ is the lag time (d). Calculation of parameters was achieved through a non-linear regression of experimental data. The coefficient of determination (R^2) was computed to evaluate the goodness of fit of experimental data to the model.

2.4. Specific Methanogen Activity (SMA)

During FO concentration, reverse salt flux was observed from the draw solution to the feed. Specific methanogenic activity (SMA) tests were carried out to determine the influence of NaCl concentration on the activity of anaerobic sludge used for BMP tests. Five different NaCl concentrations were tested: 0, 2.5, 5, 7.5, and 10 g L^{-1}. The SMA assays were carried out in 120 mL serum bottles containing 50 mL of media. Biomass concentration was 1 gVS L^{-1}. A solution of acetic acid was used as a substrate, which was previously neutralized to pH 7 with sodium hydroxide. The composition of the culture media and the procedure to determine the methane production were the same already described for BMP tests. SMA was evaluated, determining the maximum production of methane observed during each assay. Assays were conducted in triplicate.

2.5. Analyses

COD concentration, total solids (TS) and volatile solids (VS) were measured according to Standard Methods 5520D (closed reflux colorimetric method), 2540B and 2540E, respectively [25]. Na^+ concentration was analyzed using an ICP Mass Spectrometer (NexION 350D, Perkin Elmer, Waltham, MA, USA).

3. Results and Discussion

Figure 3 presents permeate flux during MF and UF concentration of sewage. MF provided higher levels of flux than UF, most likely as a result of a lower fouling tendency. TMP oscillated on both cases between 0.1 and 0.4 Bar. In the case of FO, observed flux was in the range of range 2.0–2.5 L m^{-2} h^{-1}, which can be considered low, even though it is in the same order of magnitude as others reported when applying CTA membranes to wastewater treatment [21,26]. Direct concentration by membrane processes such as MF, UF and FO will be most likely limited by cake layer formation and fouling. Several authors have suggested the application of pre-treatments to address this potential problem. For example, several authors have pointed out the convenience of applying DMF to pre-settled sludge. Settling is a simple and reliable way to concentrate a large part of the organic matter contained in sewage and can reduce chances of membrane fouling during DMF, as was observed by a few authors [16,27]. Indeed, during this research, pre-settled sewage was used according to the findings of reported experiences. Other authors have proposed the combination of coagulation with membrane filtration, with positive results [28–30]. Several fouling mitigation alternatives have been tested when using membranes for water treatment applications [31]. For example, fouling during the operation of membrane bioreactors has been extensively studied [32,33]. The particularities of membrane fouling during DMF, and the best strategies for its mitigation, definitively require further research.

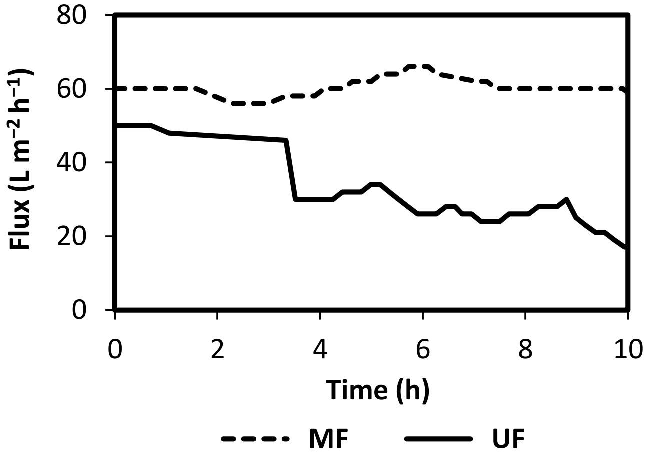

Figure 3. Permeate flux during sewage concentration by MF and UF.

Volume reduction factors during sewage concentration were 8.2, 10.9 and 6.1, for MF, UF and FO, respectively. The time required to achieve such volume reductions was 10 h for MF and UF and 74 h for FO. Assays were originally conceived to produce a volume reduction of eight or higher, which was achieved by MF and UF. However, in the case of FO, the assay was stopped before, considering the time required for concentration as a result of the low flux, to prevent excessive COD decomposition. The high time required for FO-based concentration is the result of the low flux observed for that membrane process. Time of operation could be decreased by simply increasing membrane area or by adjusting operational conditions. For example, increasing cross flow velocity improves hydrodynamic mixing, reducing the external concentration polarization phenomenon [34]. The use of different FO membranes could also be an alternative. For example, membranes such as the Thin Film Composite (TFC) have been reported to provide higher water fluxes [9]. Liang et al. [35] reported the use of vertically oriented porous substrates as supports for minimizing or eliminating the ICP effect of TFC membranes, resulting in water fluxes exceeding 50 L m^{-2} h^{-1}. On the other hand, Wu et al. [36] tested a graphene oxide-modified film nanocomposite (TFN) FO membrane for sewage concentration, reporting an improved filtration performance. Relevant developments in membrane materials are expected to take place during the following years that may provide membranes with enhanced filtration characteristics.

Figure 4 presents COD and VS concentrations of sewage and the concentrates produced by tested membrane processes. Sewage presents low levels of COD, which is the result of the fact that samples were taken after primary settling. The COD content of the concentrated sewage coming from MF and UF presented similar values: 1.46 and 1.52 gCOD L^{-1}, respectively. The COD concentration in the case of the FO process was lower (0.9 gCOD L^{-1}) as a result of a lower volume reduction. Permeate COD concentrations were 0.044 and 0.049 gCOD L^{-1} for MF and UF, respectively. COD concentration in the permeate of FO was in the range of the detection limit of the tested method, most likely because the high rejection of FO membranes can provide for dissolved organic carbon [37,38]. Performance of MF and UF membranes in terms of COD rejection were similar, despite the differences in pore size. Most likely, this is the result of the effect that fouling can have on the rejection of porous membranes since fouling layers can act as a secondary active membrane [39].

Recovered COD, i.e., the proportion of COD present in the sewage that could be found in the concentrate, was 71, 59 and 62% for MF, UF and FO, respectively. These values are in the range of or are slightly below those reported by other researchers when studying DMF with different membrane processes [13,14,21]. Values of recovered COD were influenced by the permanence of a relevant amount of COD in the setup, as fouling layers and contained in the liquid remaining in tubes. This phenomenon is the result of the use of a clean starting system and a batch operation. Continuous operation is expected to minimize COD retention in the system as fouling layers with respect to the fed COD. Moreover, in the case of FO, the long operation time most likely favored biodegradation.

Table 2 presents the Na^+ concentration of sewage and concentrated sewage. As expected, no big changes in Na^+ are observed when the sewage is pre-concentrated with MF and UF processes as a result of the null sodium rejection that these membranes can accomplish. However, Na^+ concentration in concentrated sewage by the FO process increased nine times. Such an increase is 50% higher than that expected, considering full Na^+ rejection. The difference must be then associated with reverse salt flux from draw solution, which could contaminate concentrate sewage, potentially affecting subsequent anaerobic digestion process.

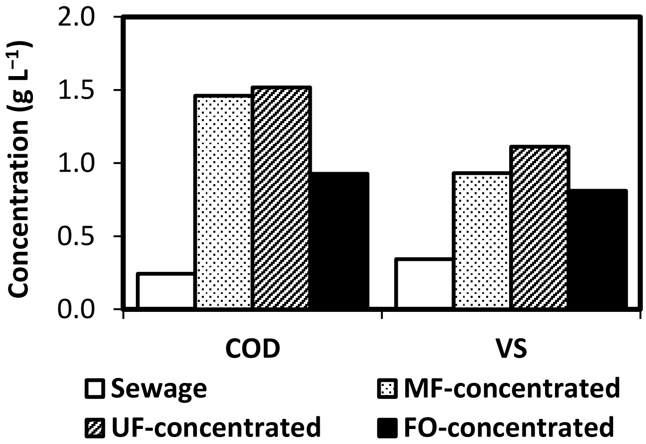

Figure 4. Sewage and concentrated-sewage characterization.

Table 2. Sodium concentration on sewage and concentrated sewage.

Sewage	Na^+ (g L^{-1})
Original Sewage	0.22
UF concentrated	0.20
MF concentrated	0.19
FO concentrated	1.94

Methane production during BMP assays using concentrates from MF, UF and FO are presented in Figure 5a. BMP was very similar for MF and UF, probably as a result of a similar rejection, as already commented. BMP values were lower than the ones reported by Gong et al. [14] and Hafuka et al. [40], who applied DMF using UF and MF membranes, respectively. This could be partially the result of differences in sewage properties. Interestingly, the BMP was higher for FO concentrate; however, the methane production rate was notoriously slower. Such observations are confirmed by the kinetic parameters calculated using the modified Gompertz model, presented in Table 3. P_m for FO was 30% higher than those for MF and UF, which were almost coincident. On the other hand, R_m for FO was only 11% of that evaluated for MF. Even though concentrates produced by MF and UF presented different values for R_m, BMP kinetics were enough in both cases to provide maximum production in about 10–15 days. It is possible that the

COD degradation that occurred during the FO concentration process may have affected the nature of a part of the sewage COD, affecting observed P_m calculated for FO. However, a comparison of observed BMP values may also suggest that solids retained by FO and MF/UF were, at least partially, of a different nature, as a result of the FO rejection of solutes that MF and UF were not able to contain. On the other hand, the lower methane production rate observed for FO (represented by a lower R_m) is most likely the result of the presence of Na^+ or other ions, as a result of the high rejection of FO membrane and the reverse salt flux. Figure 5b presents the effect of Na^+ concentration on the SMA of the anaerobic sludge used for BMP determination. A concentration of 2.5 $gNa^+ L^{-1}$ already produces a decrease in SMP of about 20% during acetate conversion. Sodium concentration during BMP tests for FO concentrate was close to 2 g L^{-1}, so this factor may have played a role in the methane kinetics observed. This result agrees with the report from Gao et al. [41], who also identified the potential inhibitory effect of NaCl during the anaerobic digestion of FO-concentrated sewage.

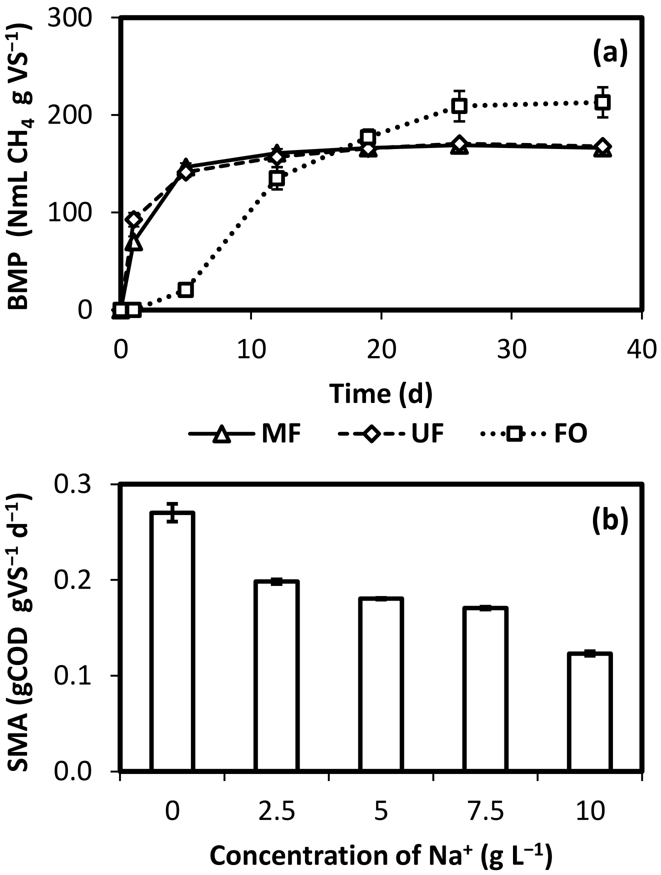

Figure 5. Results of the (**a**) biogenic methane potential (BMP) analyses for membrane concentrated sewage (MF, UF and FO). (**b**) Sludge methane activity (SMA) using acetate as substrate, at different concentrations of NaCl. Error bars indicate standard deviation between replicas.

Table 3. Kinetic parameters obtained from first order exponential model of BMP test for MF, UF and FO process.

	P_m $mLCH_4\ gSV^{-1}$	R_m $mLCH_4\ gSV^{-1}\ d^{-1}$	λ d	R^2
MF	161.9	105.2	0.33	0.988
UF	160.5	146.4	0.36	0.977
FO	210.6	16.4	3.94	0.997

Based on the BMP results, the potential energy contained in the concentrates can be estimated, considering the heating value of generated methane. For these calculations, a lower heating value was considered, 5050 kJ kg^{-1} [42]. The energy potential was 1.6, 1.6 and 2.1 kWh per kg of VS contained in the concentrates obtained by MF, UF and FO, respectively. Based on these values, energetic potential can be calculated per m^3 of the original sewage before concentration. Results are 0.19, 0.17 and 0.28 kWh per m^3 of untreated sewage. These values have been computed considering the levels of organic matter recovery observed during this research, which could certainly be improved. Since membrane concentration during this research was conducted with pre-settled sewage, previous values represent only part of the energetic potential of the wastewater. Then, it is inferred that a combination of settling and membrane concentration may be a suitable alternative for the extraction of the energetic potential of sewage. Even though FO is a separation process that provides a much higher rejection of contaminants, if energy recovery is the main objective driving the concentration process, MF and UF may provide similar levels of potential energy recuperation. However, a post-treatment will be required to remove the remaining contaminants in the permeate from those membrane operations. It is interesting to comment that a combination of FO and anaerobic digestion for sewage treatment has also been addressed in the form of FO-based anaerobic membrane bioreactors (FOAnMBR) [43,44]. However, depending on the way a reactor is operated, performance may be limited by the low organic matter concertation of the feed [18]. Then, the combination of membrane-based preconcentration and anaerobic digestion would still present advantages that should sustain future research oriented to the development of this alternative.

4. Conclusions

- Membrane concentration offers the possibility to convert organic matter contained in sewage into biogas, a source of renewable energy.
- Even though MF and UF can provide the required concentration process, enabling biogas production, rejection is normally limited to solids and colloids, so post-treatment may be required.
- Membrane fouling is a key aspect of membrane performance, and it needs to be further studied to enable the long-term operation of a direct membrane filtration operation.
- FO can provide a better rejection than MF and UF, which may result in higher methane yields. It is inferred then that FO's sewage concentration may be technically feasible. However, some drawbacks need to be overcome, such as low water flux and potentially inhibitory salt concentration, resulting from high solute rejection and reverse salt flux from the draw solution.

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