Universitat de Girona

Facultat de Ciències

Memòria del Treball Final de Grau

Comparison of CTA and TFC FO-membranes for water recovery

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Summary

For centuries, mankind has faced water-related issues for it is a limited good of vital importance for human development. Climate change, pollution and world water scarcity now have given more awareness to these problems. However, it is in human societies nature to consume water and return it to the environment in a form that is no longer profitable for human use. Ages of developing techniques and systems for water distribution has led to nowadays being able to obtain water from our own wastewater. Osmotic membrane bioreactor (OMBR) processes are a very important step in water tertiary treatment and furthermore, forward osmosis OMBR (FO-OMBR), which is at the vanguard of research and innovation. FO-OMBR uses the water gradient that the difference of osmotic pressure creates to separate physiochemically water from wastewater. In this project, first steps at laboratory-scale experiments are carried out to characterize osmotic membranes for further use at pilot-scale. Cellulose triacetate (CTA) has been widely used, and new materials such as thin-film composite (TFC) polyamide membranes have been produced. Therefore, new work is needed to compare their behavior at different concentration of the osmotic agent while also comparing performances at water recovery, lifespan of membrane and costs. In this work, sodium chloride is used as the osmotic agent. Also, the system configuration is considered as a limiting variable at operating, so different experiments were carried in both *suction* and *pulsion* modes. Impact of the pump is contemplated too so different pump speed experiments were held. In this study, it was also considered challenges such as salt loss from the *draw* solution (DS), so *RSD* value is reckoned. However, membrane *fouling* was not contemplated in this study; further work needs to be done. Once the results were obtained, it was regarded that when operated with TFC membrane better performance of the OMBR is done, as *RSD* values were lower and *permeate* volumes higher. Even though no essential differences where seen between suction and pulsion configurations, it was pulsion that showed better performance values.

Keywords: OMBR, cellulose triacetate, thin-film composite, draw solution, permeate, pulsion, suction

Resum

Durant segles, la humanitat s'ha hagut d'enfrontar a problemes relacionats amb l'aigua donat que és un bé limitat de vital importància pel desenvolupament humà. El canvi climàtic, la contaminació i l'escassetat de l'aigua al món han ajudat a crear més consciència per aquests problemes. Forma part de la naturalesa de les societats humanes però, el consumir aigua de tal manera que la que retorna al entorn ja no és aprofitable. Anys d'estudi i desenvolupament de tècniques i sistemes per a la gestió d'aigua han portat a que avui dia siguem capaços d'obtenir aigua neta dels nostres propis residus. Els processos de bioreactors de membranes osmòtiques (OMBR) són una part clau en el tractament terciari de l'aigua i, sobretot, els OMBR d'osmosis directa (FO) estan a la vanguàrdia de la recerca i innovació. La FO-OMBR utilitza el gradient que crea la diferència de pressió osmòtica per separar fisico-químicament l'aigua neta de la residual. En aquest estudi es van dur a terme els primers passos a escala de laboratori per a la caracterització de membranes d'osmosis directa per a la seva posterior aplicació en planta pilot. El tri-acetat de cel·lulosa (CTA) s'ha estat usant àmpliament com a material de membrana per FO-OMBR però darrerament s'ha començat a produir nous materials com ara el compost de poliamida de capa fina (TFC). És per això que es requereix de nous estudis per caracteritzar el comportament d'aquest tipus de membrana a diferents concentracions del agent osmòtic, mentre es compara els rendiments que s'obtenen per a la recuperació d'aigua, la vida mitja de la membrana i els costos. En aquest treball s'utilitzà el clorur de sodi com a agent osmòtic i es considera la configuració del sistema com a factor limitant pel que fa al rendiment de la operació, així que es porten a terme diferents experiments en mode de *pulsió* i *succió*. L'impacte que té la bomba empleada també es contempla i per tant es dissenyen experiments a diferents velocitats. També es tingué en compte la pèrdua de sal en la solució *draw* (DS), i en qüestió es calculà el factor *RSD*. En aquest treball, tot i que es tingué present qüestions com el *fouling*, no es van mesurar; caldrien més estudis. Es va contemplar que quan s'operà amb membranes TFC, s'obtingué millor rendiment ja que els valors de *RSD* foren més baixos i els de volum de *permeate* foren més elevats. Tot i que no s'observaren diferencies gaire significatives entre les configuracions de succió i pulsió, va ser aquesta segona la que donà millors resultats.

Paraules clau: OMBR, triacetat de cel·lulosa, compost de capa fina, solució draw, permeate, pulsió, succió

Resumen

Durante siglos, la humanidad ha tenido que enfrentarse a problemas relacionados con el agua, dado que esta es un bien limitado de vital importancia para su desarrollo. El cambio climático, la contaminación y la escasez de agua en el mundo han ayudado a crear más consciencia para estos problemas. Pero está en la propia naturaleza de las sociedades humana el hecho de consumir el agua y retornarla en el entorno de un modo que ya no es provechosa. Años de desarrollo de técnicas y sistemas para la correcta gestión del agua han llevado a que hoy en día seamos capaces de obtener agua potable de nuestros propios residuos. Los procesos asociados a biorreactores de membranas osmóticas son una parte clave en el tratamiento terciario del agua y, sobre todo los OMBR de ósmosis directa (FO) están en la vanguardia de la investigación e innovación. La FO-OMBR usa el gradiente que se crea por la diferencia de presión osmótica para separar fisicoquímicamente el agua limpia de la residual. En este estudio se llevaron a cuenta los primeros pasos a escala de laboratorio para la caracterización de membranas de ósmosis directa para su posterior aplicación en escala piloto. El tri-acetato de celulosa (CTA) se ha venido usando ampliamente como material de membrana para FO-OMBR, pero últimamente se han empezado a producir nuevos materiales como el compuesto de poliamida de capa fina (TFC). Es por eso que se requiere de nuevos estudios para caracterizar el comportamiento de este tipo de membrana a diferentes concentraciones del agente osmótico, mientras se compara con el rendimiento obtenido en la recuperación de agua, la vida media de la membrana y los posibles costos. En este trabajo se emplea el cloruro de sodio como agente osmótico y se considera la configuración del sistema como factor limitante del rendimiento de la operación, así que se llevan a cabo distintos experimentos en configuración de *succión* y *pulsión*. El impacto de la bomba empleada también es contemplado y por tanto se diseñan experimentos a distintas velocidades de ésta. También se tiene en cuenta la pérdida de sal en la solución salina *draw* (DS) así que se calculó el factor *RSD*. En este trabajo, aunque se tubo presente cuestiones como el *fouling* de la membrana, no fueron mesurados; harían falta nuevos estudios. Una vez operado se contempló que cuando se operó con membrana TFC se obtuvieron mejores rendimientos pues los valores de *RSD* fueron más bajos y el volumen de *permeate* fue mayor. Aunque no se observaron diferencias demasiado significativas entre las configuraciones de succión y pulsión, fue esta segunda la que ofreció mejores resultados.

Palabras clave: OMBR, triacetato de celulosa, compuesto de capa fina, solución draw, permeate, pulsión, succión

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List of acronyms

AL	Active layer
AS	Activated sludge
CTA	Cellulose triacetate
DPR	Direct potable reuse of water
DS	Draw solution
ECP	External concentration polarization
Eq.	Equation
FO	Forward osmosis
FS	Feed solution
HTI	Hydration Technology Innovations (commercial brand)
ICP	Internal concentration polarization
IPR	Indirect potable reuse of water
J_S	Salt flux
J_W	Water flux
MBR	Membrane bioreactor
MF	Micro-filtration
OMBR	Osmotic membrane bioreactor
PRO	Pressure retarded osmosis
RO	Reverse osmosis
RSD	Reverse salt diffusion
SD	Standard deviation
SEM	Scanning electron microscope
SS	Suspended solids
SUL	Support layer
TFC	Thin-film composite
TOrCs	Trace organic compounds
TSS	Total suspended solids
UF	Ultra-filtration

Contents

1 Introduction

1.1 Water resources

It is well known that fresh water is a valuable good of high impact for human development. It is considered a universal human right (United Nations Committee on Economic, Social and Cultural Rights, 2003) [1] and it is also vital for life in Earth's ecosystems. Climate change and population growth, as well as other factors have led to variations in the amount and distribution of fresh water availability. In Figure 1it can be seen that actually, water suitable for human consumption is less than 0.01% of the total amount of water on this planet.

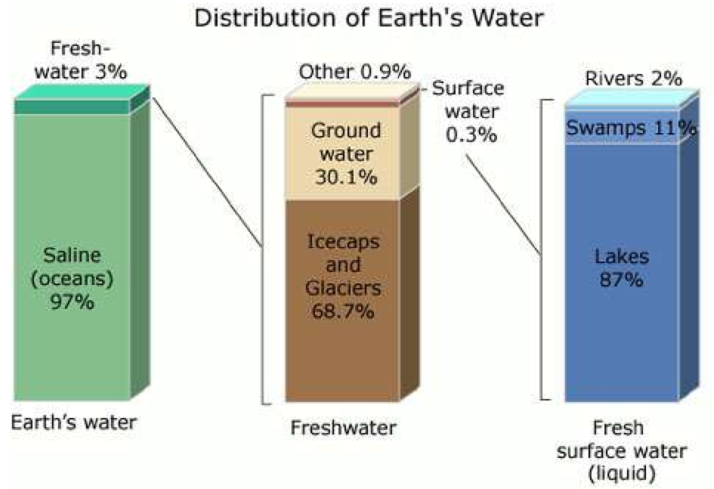

Figure 1. Graphical representation of actual Earth's water resources that are actual suitable for human use and accessible [2].

Humankind has been obtaining water for ages from two different sources; groundwater and surface water. In Figure 2 it can be seen the world's distribution of largest water reservoirs. Notice that the stress on the demand of water is increasing by both population growth and diminishing of these reservoirs.

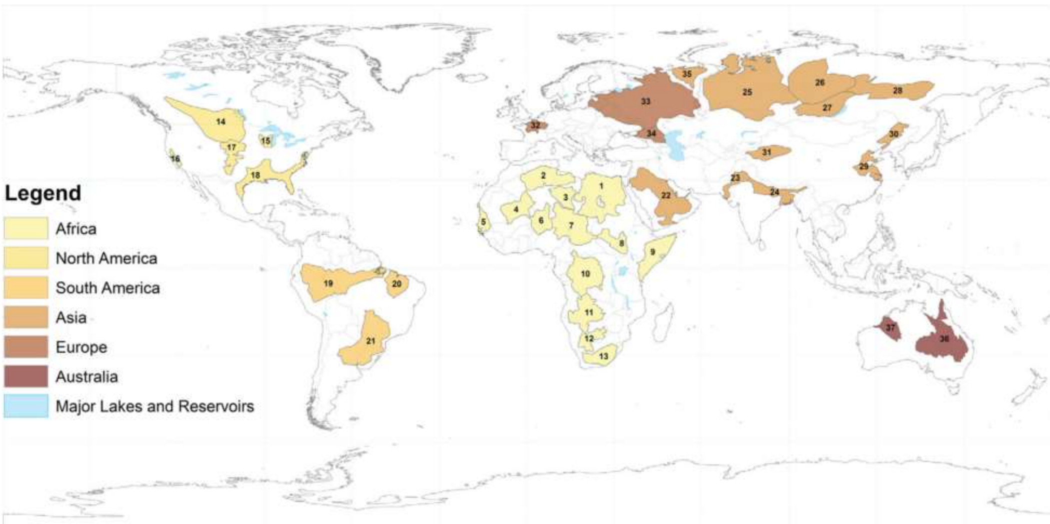

Figure 2. World's groundwater reservoirs and aquifers of most importance, based on WHYMAP delineations [Margat, 2008] [3] and Global Lake and Wetland Database [Lehner and Döll, 2004] [4]. Obtained by Richey et al [5].

Groundwater is largely unregulated [6]. The uncertainty of total water storage leads to a must on investing on renewable water technologies. As the diminishing water is also worrying, GRACE system [5] for example, are able to enlighten and help as to see at what order of magnitude are Earth's reservoirs depleting. For example, Northwest Sahara aquifer system depletion rate is about26960.8 km^3/year.

1.2 Osmosis

Osmosis is a physical phenomenon that is based on the difference of osmotic pressure across a selectively permeable membrane which translates in an unprompted passage of water or other solvents through this membrane (see Figure 3.A and 3.B)[7].This was already known in the ancient times since mankind used this principle to preserve food by dehydration with salt. The selectively permeable membrane permits water flow through while solutes are rejected. Thus, osmosis does not need any hydraulic pressure since its driving force is the difference of osmotic pressure (π) between two solutions, referred as feed solution (FS), with low π, and draw solution (DS) with a high π. π is a function (see Eq. 1) of the number of solute molecules by the solvent volume and temperature

$$\pi = \frac{n}{V_m} iRT \qquad (1)$$

where n is the number of solute molecules, V_m is the volume of solvent, T is the temperature, R is the ideal gas constant and i the dimensionless Van't Hoff factor [8].

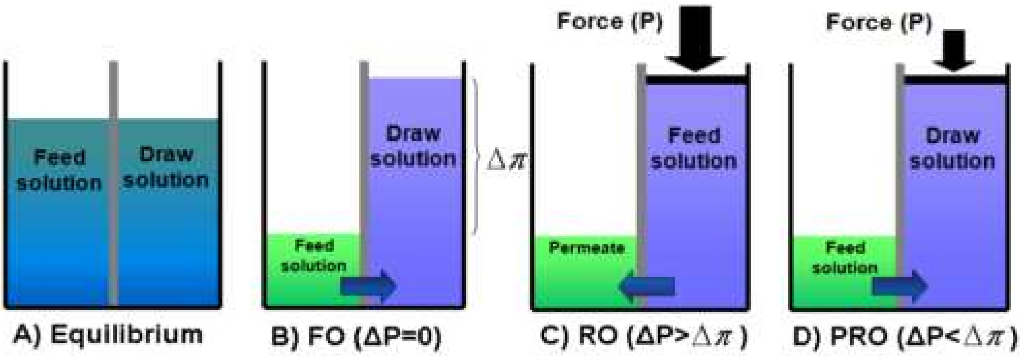

Figure 3. Different system behavior in different conditions. When the system is in osmotic equilibrium, no flux across the membrane is observed. For forward osmosis (FO), no hydraulic force is applied, therefore permeate from feed solution flows to draw solution (notice that ΔP=0). For reverse osmosis (RO), hydraulic pressure is applied to the feed meanwhile that at pressure retarded osmosis (PRO), hydraulic pressure is applied to the draw solution. Blue arrow indicates direction of water flux [8].

The water flux, whose resulting volume is called permeate, is defined as the volume that is moving from the FS to the DS across an area of the membrane by the time it takes to travel within. Therefore, Eq. 2 is defined for J_W, which stands for water flux:

$$J_W = \frac{volumetric\ permeateon\ rate\left(\frac{l}{s}\right)}{membrane\ area\ (m^2)} \qquad (2)$$

In the opposite direction, solute transport takes places from DS to FS. This implicates variations of driving force, as the DS π is being lowered by dilution and solute loss, and thus the performance of the system, which is explained next. This solute flux, named J_S, is defined by solute mass that is moving from DS towards FS across an area section of the semi-permeable membrane by time taken to travel within (see Eq. 3):

$$J_S = \frac{mass\ permeateon\ rate\ (\frac{Kg}{s})}{membrane\ area\ (m^2)} \qquad (3)$$

Reverse salt diffusion (RSD) is defined as the quotient of both fluxes compared (as seen in Eq. 4):

$$\mathrm{RSD} = \frac{J_S}{J_W} \quad (4)$$

The DS solutes loss (J_S) is a phenomenon that diminishes the efficacy as it lowers the driving force [9].

Nowadays, osmosis and even more, reverse osmosis (RO), have gained interest in water recovery fields, as the driven force of osmosis is a very useful tool which allows to obtain water from impaired streams and sewer lines just by osmotic difference principle described before [9].

1.3 Direct potable water reuse

Since water is a scarce good, wastewater needs to be treated in order to meet the potable water standards so it can be reused again. Direct (DPR) and indirect potable water reuse (IPR) have been proposed. IPR technique releases treated wastewater with remaining trace solute to an environmental system for its purification by natural agents. On the other side, DPR obtains purified water from treated water; its process consists in treating wastewater to enhance its quality to drinking water standards and return it to feed line without any environmental intermediate. DPR is the preferred option since its cost saving, less polluting and a more secure manner to verify water quality [9,10].

Water treatments, for DPR, have been studied along with different principles and techniques. Activated sludge (AS) processes have been used since early twentieth century and consist basically in an aeration tank for bio-reactions, a settling tank for separation of solids and wastewater and a recycling of AS to the influent, as shown in Figure 4[11,12]. Air is introduced to a tank with a mixture of primary treatment sewage, which become to biological flocs, also known as AS. This AS fluid lowers its degradable components by biological and chemical effect. By the effluent of the aeration tank, this mixed liquor exits towards settling tanks which will work as a clarifier. The supernatant, which is the treated wastewater will go to further treatment while the settled AS is returned to the aeration tank [11].

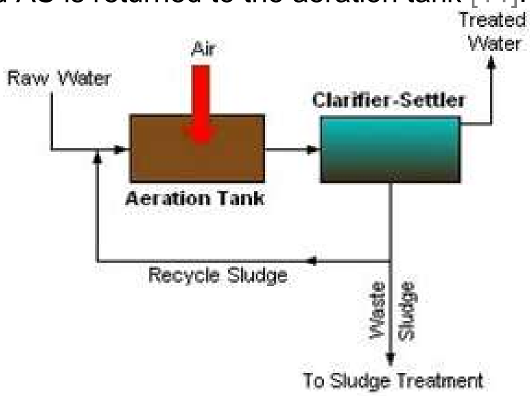

Figure 4. Representative diagram of activated sludge treatment. The treated water, which is the supernatant resulting from the clarifier, will undergo different chemical and biochemical treatments, that will be further discussed [11].

AS processes are used along with conventional treatment processes to improve its performance. Conventional treatment processes are based on a combination of physical, chemical and biological processes to remove solids, solutes and organic matter from water. It consists in preliminary, primary, secondary (with AS processing being part of both primary and secondary treatments) and tertiary treatments and more disinfecting steps can be added in the water treatment process (see Figure 5) [13,14].

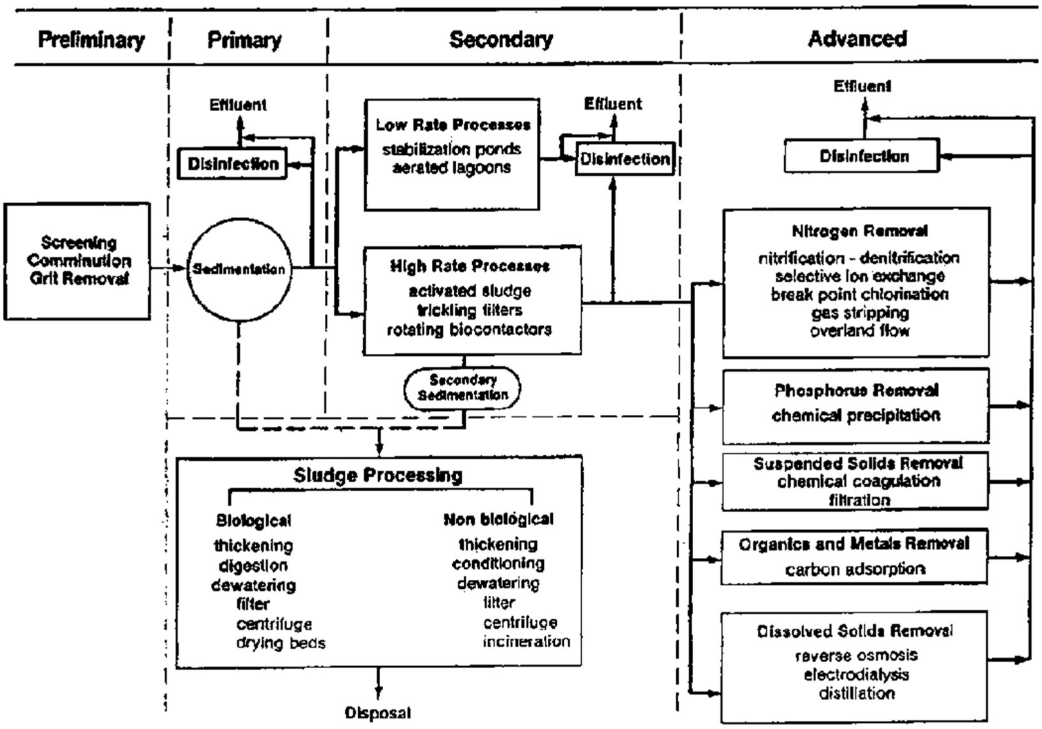

Figure 5. Diagram of all phases that can be found in conventional water treatment. Primary treatment settles solids while some oil-driven extraction can be done. Secondary treatment removes biological components and may require separation processes. Tertiary treatment (here described as advanced treatment) usually is performed prior to treated wastewater discharge to an ecosystem. See that primary and secondary treatment steps are used along with AS processes [13].

1.4 MBR

Apart from conventional treatment processes, there are several different systems for water recovery. Membrane processes such as membrane bioreactor (MBR) processes have been used and studied for many years. Unlike conventional AS and tertiary treatment, MBRs do not need a secondary clarification; membrane processes such as microfiltration (MF) or ultrafiltration (UF) to separate water from activated sludge are used instead [15]. MBRs offer more advantages in comparison with conventional wastewater recovery such as easy operability and automation, low sludge production, high rejection of suspended solids (SS) and pathogenic microorganisms. MBR also offers better quality water purification and besides, it can be coupled with downstream reverse osmosis (RO) [9,15,16,17,18,19,20].

However, a recurrent issue with MBR processes is membrane fouling [9,21,22] caused by deposition of SS on top or inside of the membrane. This not only diminishes the water flux but also the yield of recovery and its quality. Thus, costs of operation are increased and membrane lifespan is reduced [23].

1.5 OMBR

Osmotic membrane bioreactor (OMBR) is a technology useful to lessen the fouling impact on the membrane that occurs in previously commented MBRs. OMBR couples activated sludge processes with a semi-permeable FO membrane for water extraction based on the low osmotic pressure of the activated sludge towards a DS [9,15,31,22]. Given that it is based on osmotic membranes, the driving force is the osmotic pressure difference. The transport in OMBRs occurs when water in the FS, a solution of lower osmotic pressure which in water treatment case is the AS, moves towards a DS, a solution of higher osmotic pressure, through the semi-permeable FO membrane. Conversely, transportation of DS solutes does happen in the opposite direction of water permeation towards FS and itis the salt flux J_S. As J_W occurs, DS is being diluted. Nowadays, there have been described several different methods to maintain a constant DS concentration: whereas it can be released in a water reservoir (if natural, it should meet the standards to do so), it can also be reconcentrated. This may utilize thermal, crystallization or membrane process as separation step [24,25,26,27] and it also can result in a potable reuse of the recovered water [28,29].

If compared, OMBRs show higher rejection of TSS [18,19], ions and TOrCs [30] than MBR, because of its membrane type (FO membranes are semi-permeable whereas MBRs membranes are porous). As previously commented, OMBRs also show lower fouling propensity therefore, the costs of operation are cheapened while also the frequency of cleaning is reduced [9]. OMBRs do not need any additional pressure such as it is needed for RO processes (see Figure3).

Almost every bench-scale study about OMBR water treatment has been conducted by using cellulose based membranes and also without a re-concentration step [9]. Thin-film composite are promising candidates for further large-scale application in FO-OMBR due to good results obtained in RO [21].

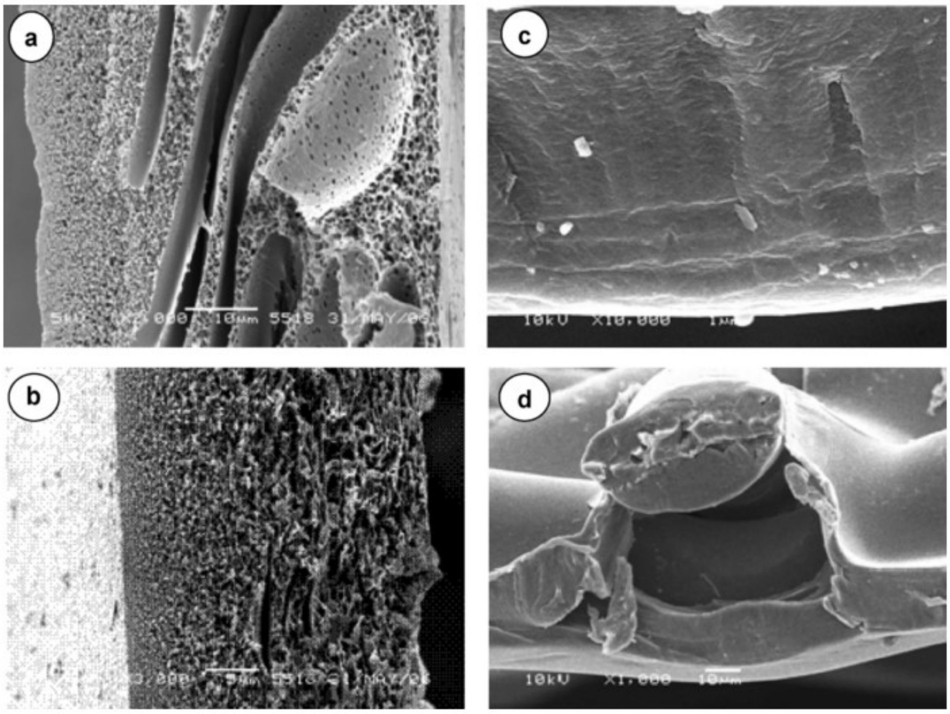

Figure 6. SEM pictures of FO membranes. A, B corresponding to TFC membranes while C, D to CTA [21].

Despite the potential to address key issues surrounding global water and energy demands, osmotically driven membrane processes have yet to progress significantly beyond conceptualization. The major obstacle in advancing this technology is the lack of an adequate membrane. A membrane designed for an OMBR process should reject dissolved solutes, produce high permeate water fluxes, be compatible with the selected DS, and withstand the mechanical stresses generated by the operating conditions [31]. Existing commercial membranes lack one or more of the above-mentioned characteristics, inhibiting their use in osmotically driven membrane processes. For example, commercial FO membranes that are made from cellulose triacetate (CTA), get degraded when exposed to an ammonium bicarbonate DS [32]. Additionally, cellulose acetate membranes have relatively low pure water permeability and salt rejection, which limits their use for desalination. Alternatively, conventional thin-film composite (TFC) reverse osmosis (RO) membranes exhibit high salt rejection and satisfy the chemical stability and mechanical strength requirements. However, TFC membranes yield very poor permeate water fluxes in FO because they are designed for pressure-driven membrane processes, such as in RO [7,21,25]. TFC-RO membranes fail in FO operation because the thick and dense support layers, necessary to withstand large hydraulic pressures, result in internal concentration polarization (ICP).

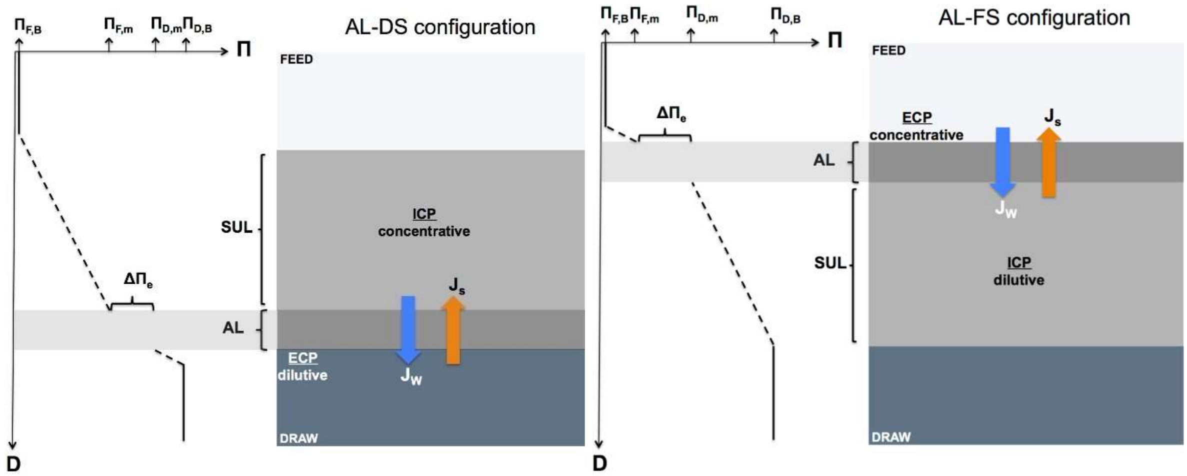

Figure 7. Pictures of ICP and ECP with active layer (AL) facing the FS or the DS. J_S in orange arrows, J_W in blue arrows. The support layer (SUL) acts as concentrative step for salt or solutes when entering to the FS from the AL, whereas when exiting the DS towards the AL, SUL acts as dilutive step [33].

ICP adversely affects the performance of all asymmetric membranes in FO, with the effects being exacerbated for TFC-RO membranes due to their thick and dense porous support. The porous support layer acts as a diffusive boundary layer, which severely reduces the osmotic pressure difference across the active layer [34]. Because this boundary layer is unperturbed by stirring [35], modifying the support layers is essential to minimize the performance limiting effects of ICP that currently hinder TFC membranes [22,35]. Prior studies have demonstrated, through both experiments and modeling, that the additional resistance to mass transfer of this boundary layer is proportional to the support layer thickness and tortuosity, and inversely proportional to the support layer porosity [25,36]. Therefore, the ideal support layers for FO membranes to enhance performance would be very thin, highly porous, and provide a direct path from the DS to the active surface of the membrane. Fabrication of TFC membranes tailored for FO operation. Innovative modifications made to the membrane casting procedure, as well as the resultant effects of these changes on the microstructure of the membranes are described. Salt rejection and water flux of the newly fabricated TFC-FO membranes are compared to commercially available RO and FO membranes. These performance results are linked to the membrane structural properties. This work aims to demonstrate the ability to fabricate membranes with a structure adapted to FO processes, thus providing a basis for further developments of osmotically driven membranes.

2 Objectives

The study was carried in LEQUIA facilities from November 2015 to April 2016. It was started out by planning different tests for polishing the yields of water recovery from a feed solution before scaling.

The goals of this project were:

- To study different osmotic membrane types, cellulose triacetate (CTA) and thin-film composite (TFC), in different conditions such as the process configuration, flowrate of the solutions movement inside the cell and the osmotic force used in the draw solution.
- Characterize the water flux, the salt flux and the reverse salt diffusion of each configuration and conditions.

3 Material and methods

The different experiments that were held during this study are shown in Figure 8, each with its corresponding operation conditions.

TFC				**CTA**			
Conditions				**Conditions**			
N	**System Configuration**	**Pump speed (rpm)**	**DS Concentration (g/l)**	**N**	**System Configuration**	**Pump speed (rpm)**	**DS Concentration (g/l)**
1	Pulsion [1]	50	20	**1**	Pulsion	50	35
2	Suction	50	20	**2**	Suction	50	35
3	Pulsion	50	35	**3**	Pulsion	100	35
4	Suction	50	35	**4**	Pulsion	150	35
5	Pulsion	50	70				
6	Suction	50	70				
7	Pulsion	50	140				
8	Suction	50	140				
9	Pulsion	50	35				
10	Suction	50	35				
11	Pulsion	100	35				
12	Suction	100	35				
13	Pulsion	150	35				
14	Suction	150	35				

Figure 8. Resume table for each experiment that was performed as a combination of four different variables: operating mode (Pulsion, Suction), flow rate (0.13, 0.25, 0.36 l/min), DS salt concentration (20, 35, 70, 140 g/l) and membrane type (TFC, CTA). N stands for number of experiment with each membrane type.

Notice in Figure 8that no study for impact of DS salt concentration was held. Three rates were meant to be calculated from within the experiments that were held: salt flux (*Js*), water flux (*Jw*) and *RSD*. While both *Js* and *RSD* are meant to be obtained as low as possible, *Jw* is what is needed to be as high as possible.

3.1 Membrane type

In this study, two different membrane materials were tested; a thin-film composite (TFC) FO polyamide membrane (Porifera, Inc.) and a cellulose tri-acetate (CTA) FO membrane

[1] Pulsion: as the counterpart of suction, consisting in the operation mode that pushes water away instead of sucking it.

(Hydration Technology Innovations Llc.). Their performances for permeate recovery were tested under different flow, different DS concentration and different experimental set up conditions.

3.2 Feed solution

Deionized water (DI water) was used as FS since it was a laboratory scale for a preparatory study before testing in pilot scale and only optimization of membrane performance under different conditions was considered. FS conductivity was monitored (Crison EC-Meter Basic 30+) during the whole experiment and also before as quality control to prove it was pure and without any trace of salt or osmotic agent.

3.3 Draw solution

As osmotic agent for DS, sodium chloride marine salt (Infosa) was used. The impact of different concentrations (20, 35,70,140 g/l) on the performance of the membranes was tested.

3.4 Membrane cell

Since it was a laboratory scale experiment, a methacrylate filtration cell with filtration surface area of $0.0056 m^2$ was used.

3.5 Operating set up

To test TFC and CTA membranes performance pulsion and suction set ups were used, always operated in countercurrent. For pulsion test, the pump was placed between DS vessel and membrane entrance in order to pump out the DS towards the membrane cell. For suction test, the pump was placed after the membrane cell exit with pipe carrying DS and the permeate.

For mass transfer and permeate calculation, a Kern EWJ-6000 balance was used. Balance measurements were annotated simultaneously with each FS sample collection, to further calculate water flux from FS to DS.

Given that this was a laboratory-scale experiment and pump was needed (Watson and Marlow 300series) to help DS and FS enter and exit the methacrylate cell, its effect on permeate volume and rate obtained had to be considered. Different pump speed experiments were used to elucidate the effect of this parameter. So, in order to test membrane performance pertinent calibration (pump speed to flowrate conversion) was made, as it can be seen in Figure 9.

Pump speed (rpm)	50	100	150
Flowrate (l/min)	0,132	0,247	0,362

Figure 9. Calibration for pump speed to flowrate, with regression (r^2) coefficient of 0.988.

In order to establish a comparison between the different experiments, it was only modified either pump speed or DS at a time. For the measure of the impact on *RSD* with the variation of DS concentration, it was settled to work at a constant flowrate of 0.13 l/min (50 rpm). On the other hand, to study the impact on *RSD* when pump speed is changed, 35 g/l was the DS concentration established to maintain. Therefore, when comparing both operating set ups, focus on 50rpm (in the case of flowrate impact study) and 35g/l of salt (in the case of salt concentration in the DS variation) is needed to elucidate differences between both configurations.

3.6 Sample collection

During each experiment, FS samples were collected to measure its conductivity (Crison EC-Meter Basic 30+) in order to further calculate salt flux from DS to FS. The time between each sample was 10 minutes.

Two samples were collected before and right after turning the pump on, when starting each experiment, to measure conductivity in order to verify no significant differences between both samples nor leakages in the set up. This quality control helps elucidate good regression coefficient when calculating reverse salt diffusion.

4 Results and discussion

The test was held in a combination of different variables to measure impact of different DS salt concentration and at different pump speed, under different system configuration (pulsion or suction) with different membrane types. All values given below are measured averages between all data obtained during each experiment, therefore *RSD* values shall not be regarded as a result of any direct calculation between data given of J_S and J_W. There can be observed meaningful differences in membrane permeability (J_S) and permeate flow (J_W) values when changing different parameters such as membrane type, DS concentration and pump velocity and its configuration. Even though the system always worked with the active layer facing the FS, neither concentrative ICP nor dilutive ECP were considered.

Standard deviation (SD) of all averages calculated are relatively high, thus meaning that the dispersion of data for almost each average calculation is also large. This is mainly because the fluxes tended to increase (not all at the same rate) as time passed.

4.1 Cellulose Triacetate (CTA) membrane

4.1.1 Impact of pump speed

As it a can be regarded in Figure 10, the increase of the pump speed did not have a clear tendency on J_S trend as it decreased in 100 rpm and increased at 150 rpm. J_W increased its flux at a low slope. However, since this J_W increase was not linear, more experiments with lower and higher pump speed should be done to prove if above 150 rpm, it would be prone or not to any saturation point as a result of the pump speed impact on the permeate flux.

J_S showed its minimum value at 100rpm while its maximum at 50rpm. Given that this is not what would be expected, since J_W seemed to behave concurrently with pump speed, this leads to think that some bias was affecting this system. Regarding the sizes of the SD represented also in Figure 10, more experiments should be considered to get an accurate idea of CTA performance for pump speed variation, as great variance of results were obtained.

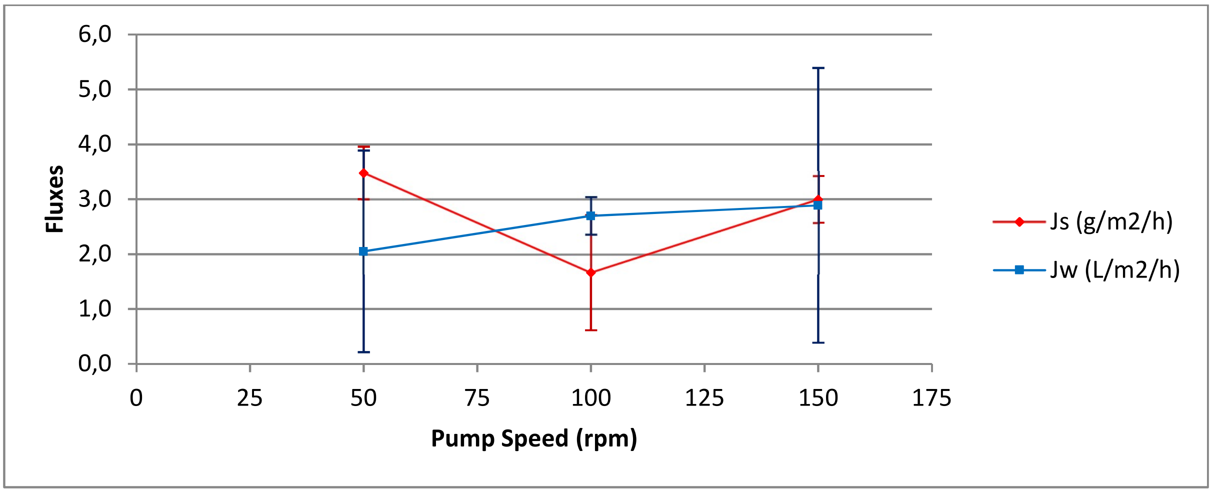

Figure 10. Average results for CTA experiment at different pump speed (50, 100, 150 rpm). Red rhombuses represent salt fluxes -*Js*-, expressed in grams per square meter per hour. Blue squares represent water fluxes -J_W-, expressed in liters per square meter per hour. Green columns represent reverse salt diffusion -*RSD*-, expressed in grams of salt per liter of permeate. For better comprehension. SD of J_S in red, SD of J_W in blue.

Figure 11 shows that, even though *RSD* behavior did not vary considerably, it was slightly affected by J_S results. SD showed great dispersion, thus no clear tendency can be dilucidated;

more experiments should be done. What can be observed is that *RSD* values varied around 2 g/l, which this could indicate that pump speed does have a poor impact on OMBR performance.

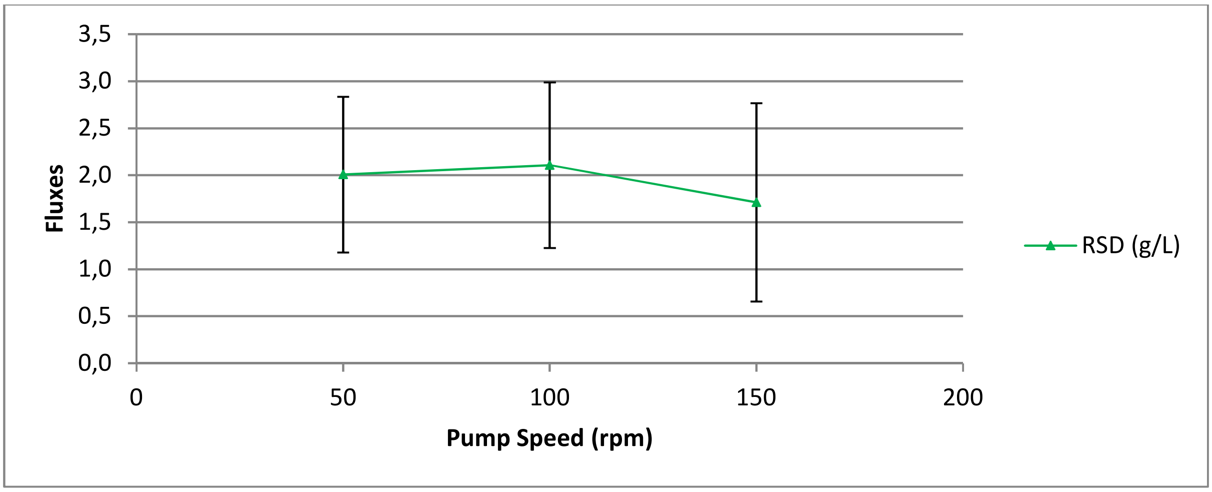

Figure 11. Average results for CTA experiment at different pump speed (50, 100, 150 rpm). Green triangles represent reverse salt diffusion -*RSD*-, expressed in grams of salt per liter of permeate. SD shown in black.

4.1.2 Impact of system configuration

For CTA pulsion and suction mode comparison, a single experiment was performed in suction, as it is described in section 3. In Figure 12 it can be seen the results of suction and pulsion at 50rpm and 35 g/l at the DS. In this pump speed variation experiment, J_S was considerably better obtained -thus, less membrane permeability- by pulsion configuration.

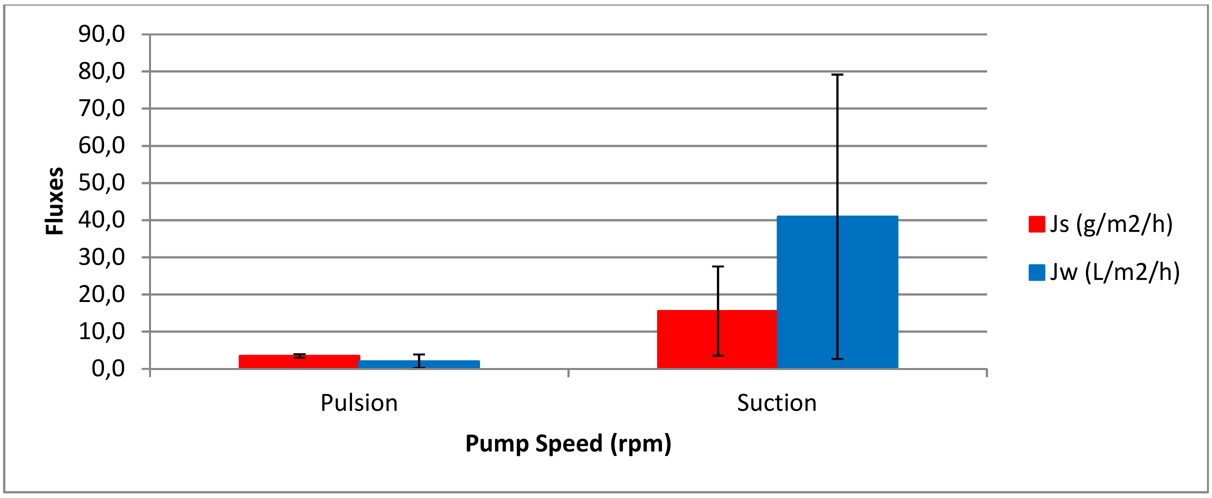

Figure 12. Average results for CTA experiment at 50 rpm for each different system configuration (pulsion and suction). Red columns represent salt fluxes -*Js*-, expressed in grams per square meter per hour. Blue columns represent water fluxes -J_W-, expressed in liters per square meter per hour. SD are shown for both *Js* and J_W in black.

Regarding this suction case, both J_S and J_W showed higher flux values; for J_S case, it did by more than five times of pulsion values while for J_W case, it did by more than twenty times. So, as it can be seen in Figure 12, suction at 50 rpm gave bad membrane permeability while also a very notable permeate recovery performance.

SD for both suction fluxes were larger than when it was operated in pulsion mode. As said in section 4.1.1, the great variance of the data leads to a need for more replicates in order to understand CTA performance when operated in suction mode.

Since it is a new proposal (for previous suction configurations, only submerged MBR plates were experimented), suction configurations may need to be improved [15,32].

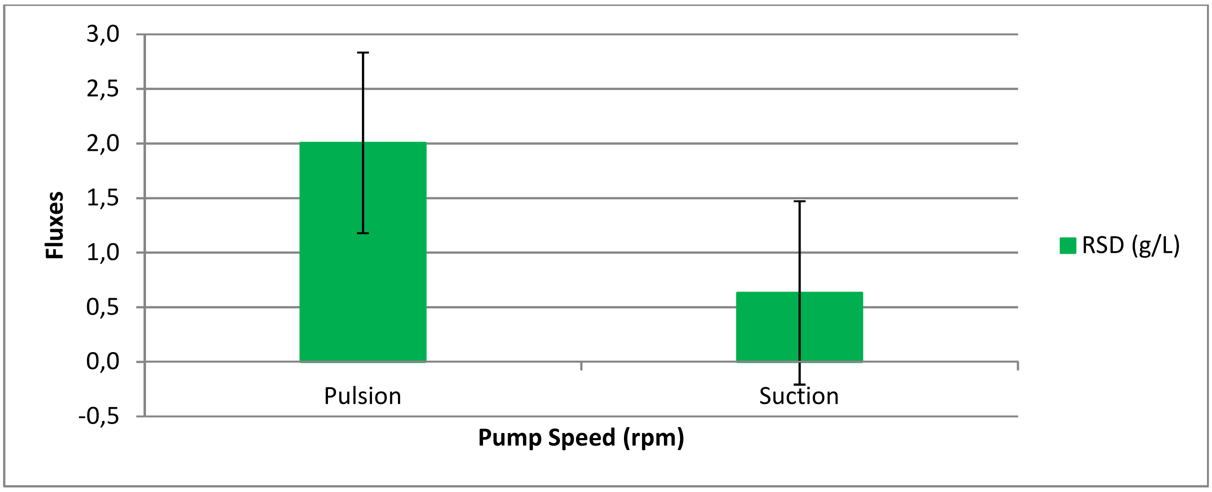

Figure 13. Average results for CTA experiment at 50 rpm for each different system configuration (pulsion and suction). Green columns represent reverse salt diffusion -*RSD*-, expressed in grams of salt per liter of permeate. SD shown in black.

As it can be regarded in Figure 11, the increase of pump speed merely affected the membrane performance, as *RSD* value did not get considerably altered. Moreover, when suction was used instead of pulsion, as seen in Figure 13, performance got worse as *RSD* values were lower, presumably due to the J_S high values.

4.2 Thin-Film Composite (TFC) membrane

4.2.1 Impact of pump speed

Pump speed variation experiment for TFC was also performed. As shown in Figure 14.A, J_W was poorly affected by pump speed increase, with a low increasing slope. Unlike J_W, J_S was affected by pump speed with a maximum value at 50 rpm and a minimum at 100 rpm. SD showed again that there was a great dispersion of data so, even the trend seemed to be quite stable. Further tests should be done to understand the tendency of TFC J_S.

As it can be regarded in Figure 14.B, the increase of pump speed merely affected the membrane performance, as *RSD* hardly increased. Comparing Figure 14.A with Figure 14.B, it can be noticed that the experiment was highly biased, as *RSD* did not give values corresponding with J_S and J_W trends. Thus, this may lead to think that the pump speed variation effect on CTA performance is poor. However, there was a noticeable high deviation at its maximum value -100 rpm-, so tendency is still not clear as SD is too large.

As it was said on section 4.1.1, further studies should be done in order to correlate the effects on the variation of pump speed as a cause of flowrate variance, and which impact it has on the membrane performance.

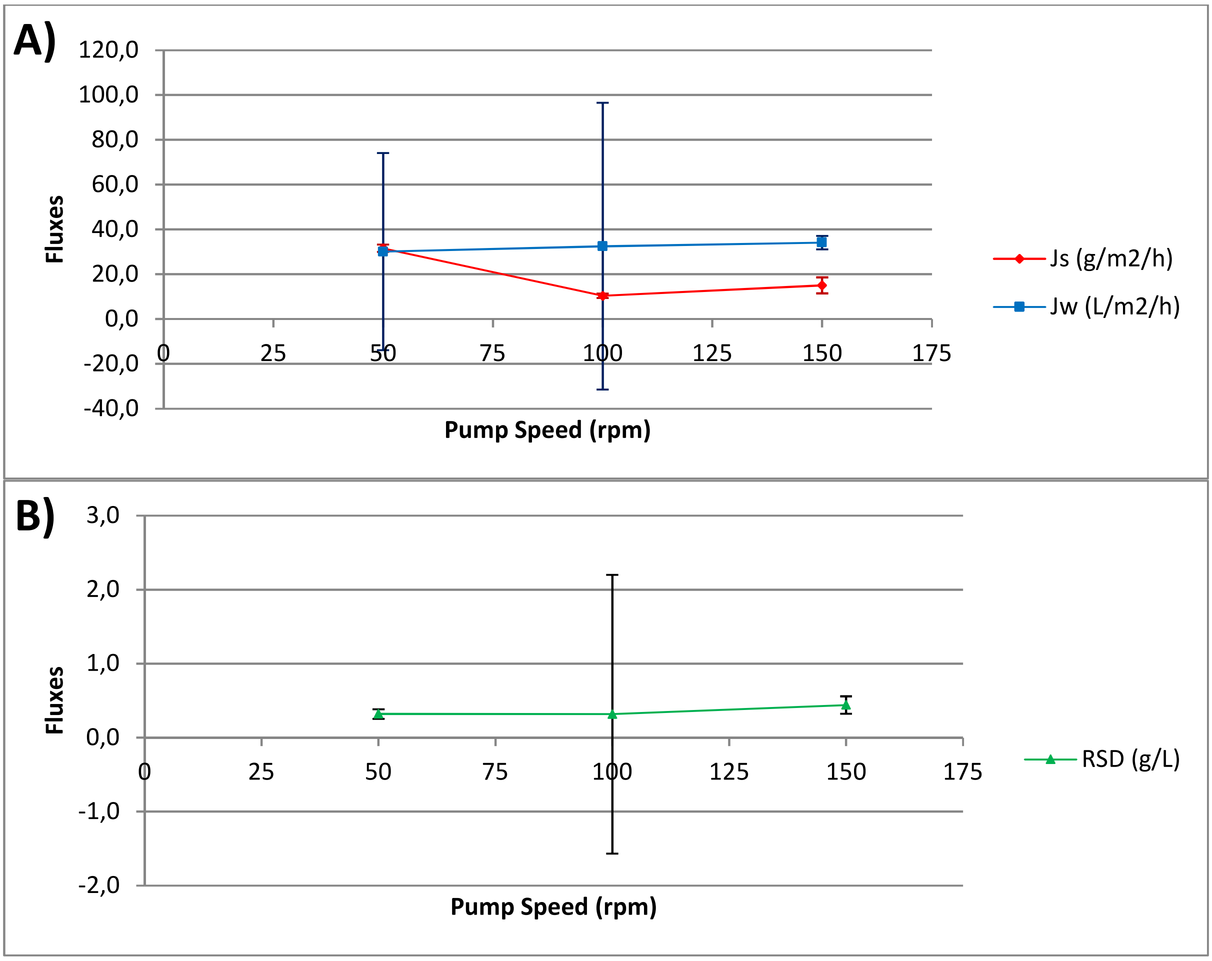

Figure 14. Average results for TFC experiment at different pump speed (50, 100 and 150 rpm) when operated at pulsion mode. A: Red rhombuses represent salt fluxes -*Js*-, expressed in grams per square meter per hour. Blue squares represent water fluxes -*Jw*-, expressed in liters per square meter per hour. SD of J_S in red, SD of J_W in blue. B: Green triangles represent reverse salt diffusion -*RSD*-, expressed in grams of salt per liter of permeate. *RSD* SD is shown in black.

4.2.2 Impact of salt concentration in DS

As it was expected, increasing salt concentration in DS led to an osmotic pressure increase; this entailed water flux increase too. This phenomenon is something that can be found in every study that was considered in this project [37]. In this particular case (Figure 15), water flux increase was clearly visible -accentuated when working at 140 g/l at DS-. Even though from the results shown before it can be dilucidated this tendency, there were isolated cases when this principle did not work as expected. Furthermore, as it can be regarded in Figure 15.A, the increase of salt concentration along with salt flux cannot be seen as a lineal correlation. Comparing with data shown in Figure 15.B, from low salt concentration (20 g/l) to medium salt concentration (35-70 g/l), *RSD* value increased, since salt flux was higher than water flux, in both pulsion and suction mode (see Figure 15.A). Furthermore, J_S did not increase substantially along with DS salt concentration at least until it was worked at 70 g/l, meanwhile showing higher *RSD* values. *RSD* minimum value was reached when working at 140 g/l of DS salt concentration. SD obtained in this experiment were considerably lower than the rest, thus entailing the accuracy of the tendency that it was observed.

As said before, it was settled that the active layer of the membranes would always face the FS, for results [38] in several bibliographic publications have established that is more efficiently worked by doing so. Perhaps, there was some influence of dilutive ICP of DS salt with the permeate inside the membrane, as well as salt concentration on the surface of the membrane layer facing the FS, reducing the osmotic driving force by concentrative ECP. Such as how it was planned in this project, there was not an efficient manner to record the actual impact of this phenomenon on the membrane performance.

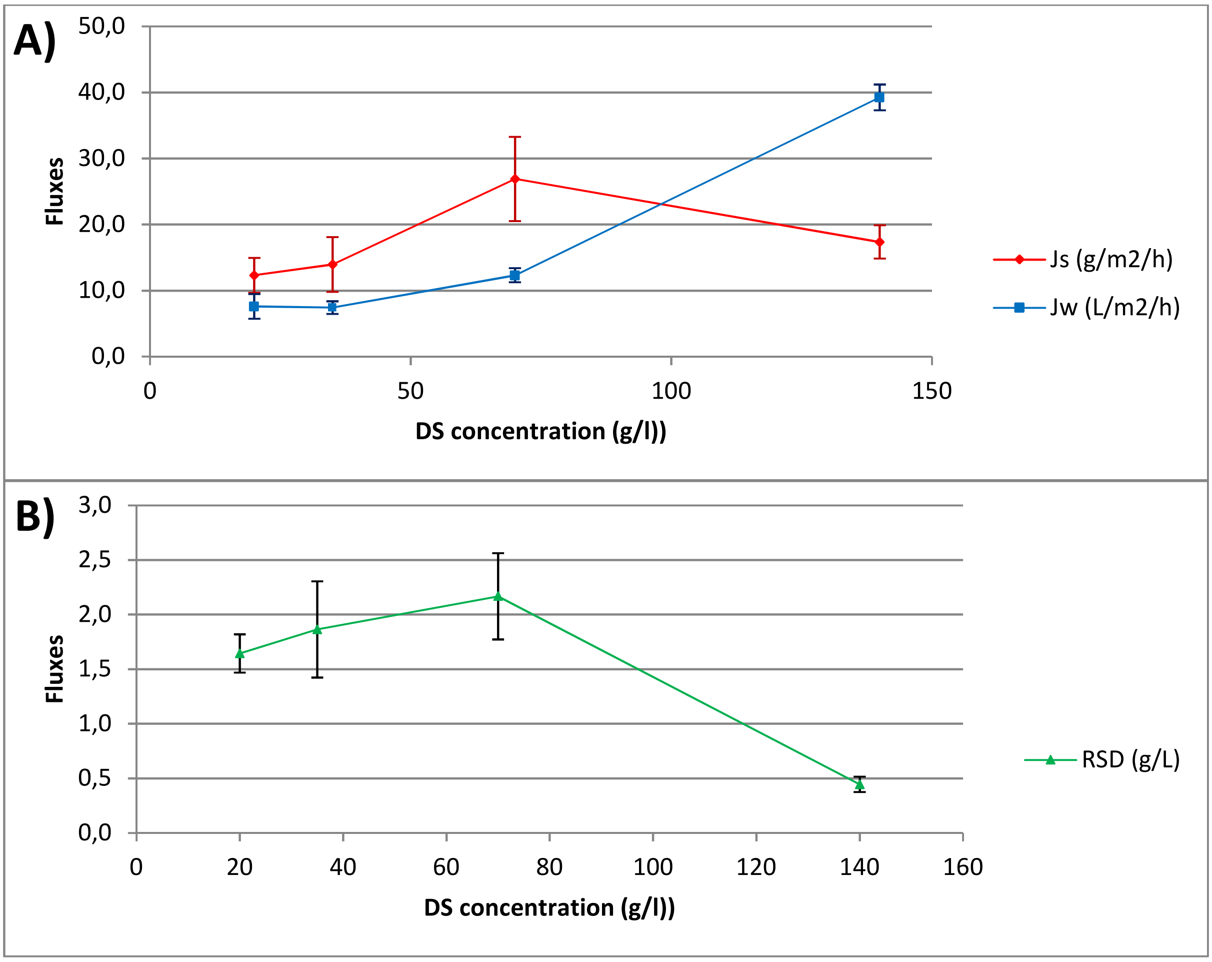

Figure 15. Average results for TFC experiment at different DS salt concentration (20, 35, 70 and 140 g/l) when operated at pulsion mode. A: Red rhombuses represent salt fluxes -*Js*-, expressed in grams per square meter per hour. Blue squares represent water fluxes -J_W-, expressed in liters per square meter per hour. *J* SD of J_S in red, SD of J_W in blue. B: Green columns represent reverse salt diffusion -*RSD*-, expressed in grams of salt per liter of permeate. *RSD* SD is shown in black.

The results obtained in this experiment, shown in Figure 15, suggest that there is some trend for J_W. As it can be seen from the bibliography [37], water flux shall increase along with DS salinity increase; in this current study, it was clearly accomplished when DS salt concentration varied from 70 g/l to 140 g/l. But to trace a solid regression, further studies should be done to strengthen the results that were obtained for this TFC membrane.

4.2.3 Impact of system configuration

J_S and J_W fluxes increased along with an increase of pump speed in suction case (see Figure 16). J_W showed the same behavior for both pulsion and suction case -as it is Figure 14.A and 15.A-, but J_S did not.

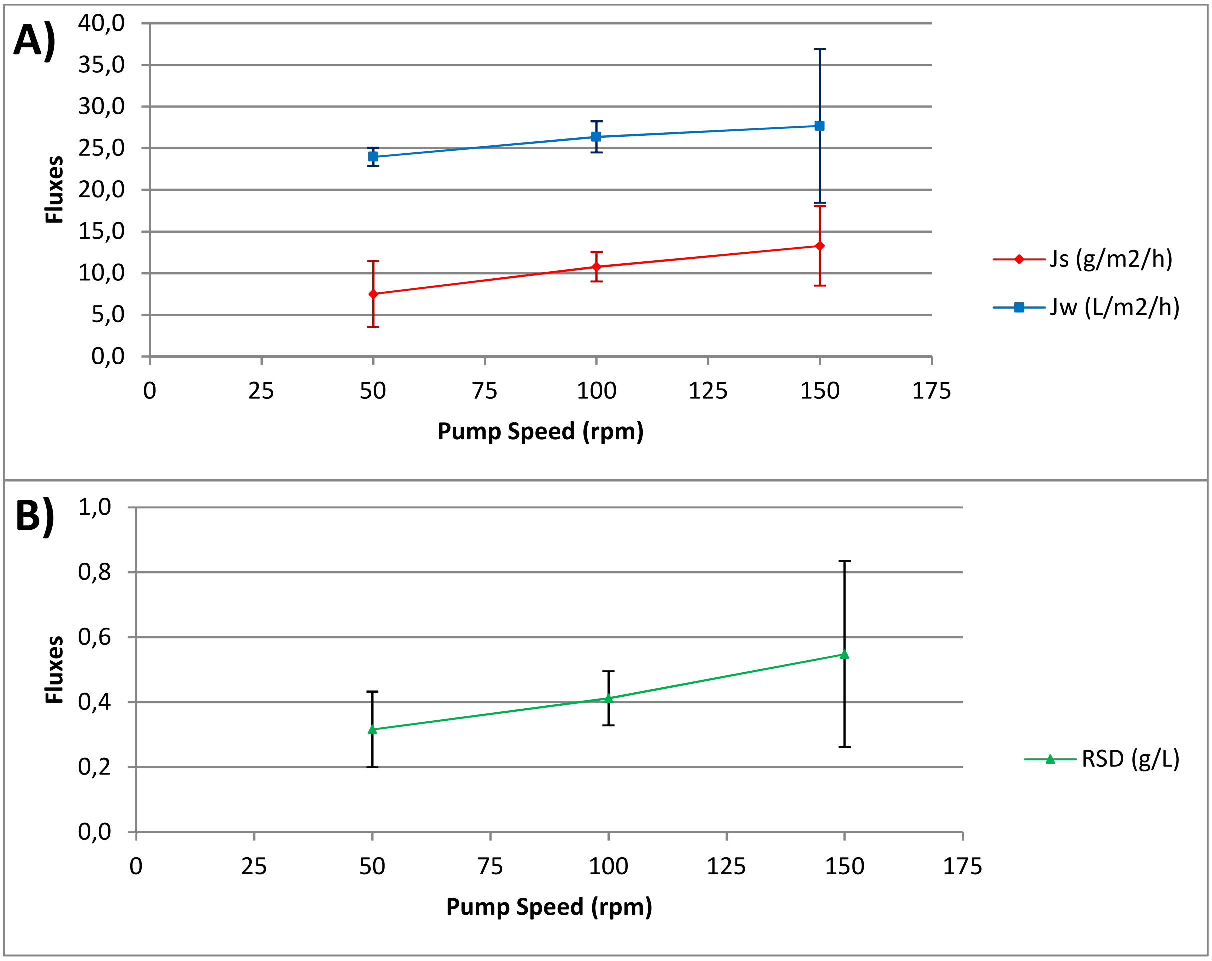

Figure 16. Average results for TFC experiment at different pump speed (50, 100, 150 rpm) when operated at suction mode. A: Red rhombuses represent salt fluxes -*Js*-, expressed in grams per square meter per hour. Blue squares represent water fluxes -*Jw*-, expressed in liters per square meter per hour. SD of J_S in red, SD of J_W in blue. B: Green columns represent reverse salt diffusion -*RSD*-, expressed in grams of salt per liter of permeate. *RSD* SD is shown in black.

Observing results in both pulsion (Figure 14) and suction (Figure 16) experiment cases for different pump speed, since differences between both pulsion and suction experiments cannot be explained by any osmotic principle that is tested here it seems it was due to a failure of design -due to its intrinsic bias- and again, there was no substantial difference between suction and pulsion to offer a reliable explanation on whether it is better to operate in pulsion mode or not. SD were slightly larger in suction experiment, specially in 150 rpm case. This bias at 150 rpm may explain the decay of J_S in pulsion mode at 150 rpm while it did not happen in suction.

Focusing at different DS concentration study case, best water flux for each salt concentration were given by suction configurations (Figure 17.A), meanwhile best *RSD* values were in pulsion modes (Figure 15.B). J_W showed similar trends to pulsion case (Figure 15.A) until 70 g/l, when it was operated in suction (Figure 17.A). Thus, *RSD* decreased because of higher J_W obtained while J_S diminished. It was when working at 140 g/l of DS salt concentration that *RSD* values showed better results better (lower) either in pulsion or in suction configuration and there were obtained substantially high water fluxes. Meanwhile at this high DS salt concentration, salt flux values appeared much lower than when it was operated at lower DS salt concentration.

In Figure 17, DS salt concentration variation test operated in suction mode is shown. When data obtained from the homologous pulsion test (Figure 15) are compared, similar trends can be found, although less SD was observed in suction case.

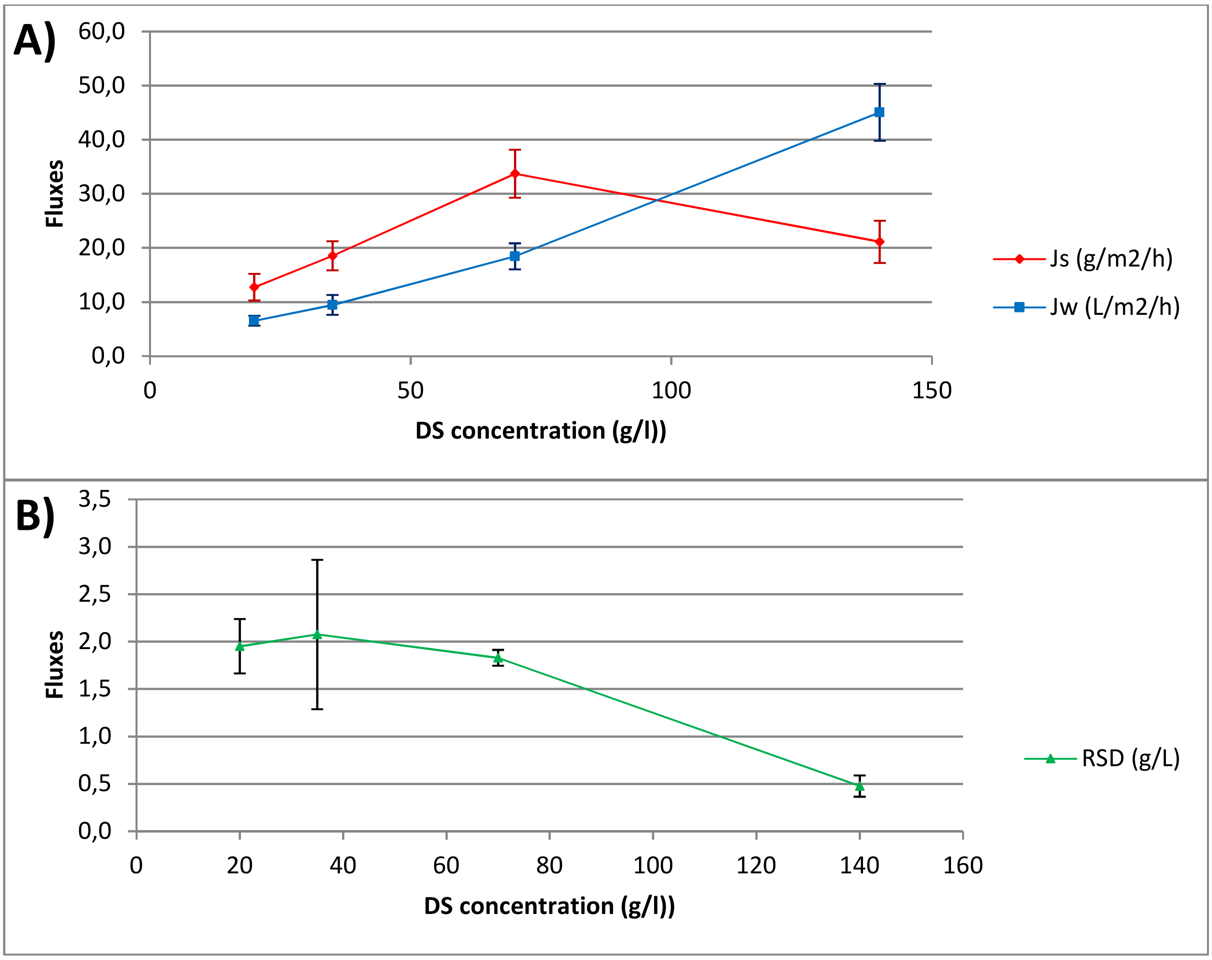

Figure 17. Average results for TFC experiment at different DS salt concentration (20, 35, 70 and 140 g/l) when operated at suction mode. A: Red rhombuses represent salt fluxes -*Js*-, expressed in grams per square meter per hour. Blue squares represent water fluxes -*Jw*-, expressed in liters per square meter per hour. SD of J_S in red, SD of J_W in blue. B: Green columns represent reverse salt diffusion -*RSD*-, expressed in grams of salt per liter of permeate. *RSD* SD is shown in black.

Both pulsion and suction experiment cases for different DS salt concentration showed an increase of J_S and J_W until 70 g/l, with J_S decline at 140 g/l. As a result, *RSD* values from both experiments behave in the same manner. Despite this characteristic increasing pathway, here it was not observed the trend of J_W increasing along with DS salt concentration, as it was observed in all possible bibliography [32,34,37]. The slope relation of both increases (J_S and J_W) in the bibliography [34] does quite reflect the same, with J_W slope being higher than J_S. Thus, by this point, it can be settled that results obtained when DS salt concentration varies, quite matches what was expected.

4.3 Membrane type comparison

Due to the lack of data for CTA performance at DS salt concentration variation, comparison was only allowed for different pump speed and thus, more pulsion tests data are able for comparison.

As seen in Figure 18 it was found that TFC membranes offered a better permeate recovery and salt flux than CTA membrane, even with a higher flux (~ten order of magnitude difference) and less membrane permeability. As well as it is seen in sections 4.1 and 4.2, TFC presented better performance values than CTA for water recovery at same DS salt concentration and pump speed, with higher permeate flow at same pump speed. This entails the arguments that supports the hypothesis that TFC really does better performance at same conditions than

CTA. Actually, *Cornelissen et al*. found[2] that HTI cellulose membrane (CTA) offered much higher water and salt fluxes than TFC membranes [21], which did not happen in the results obtained in this project -while working with NaCL salt-. During *Cornelissen et al.* and as well as *T.Y. Cath et al.* studies [24], the polyester mesh was embedded within the polymer material for mechanical support. This is mainly because HTI CTA FO membrane lacked a thick support layer, thus providing a mechanical support. Since this was not considered during this study, it may explain why CTA results are so poor compared to them.

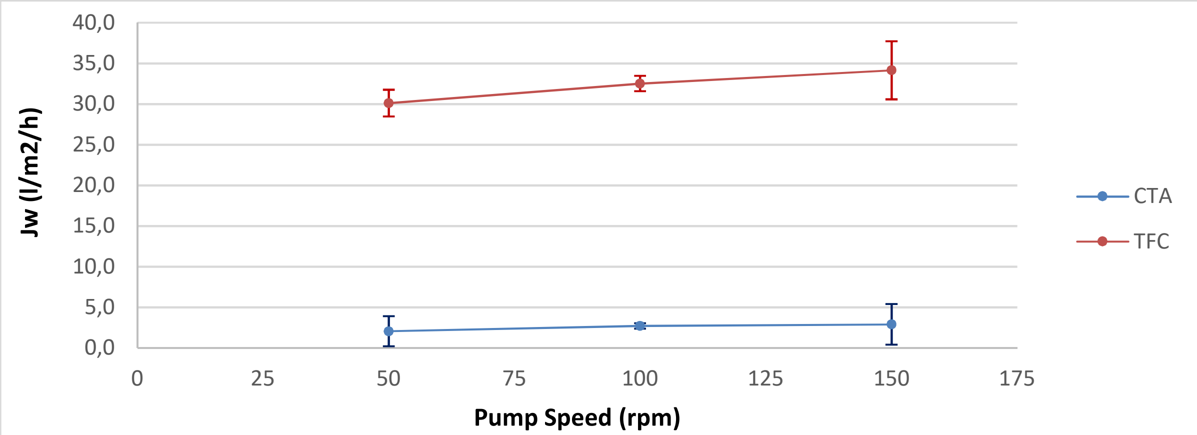

Figure 18. Average permeate fluxes for CTA (blue) and TFC (red) membranes within three different pump speeds, in pulsion configuration. Notice that each dot corresponds to speed pump 50, 100 and 150 rpm, in the same order. TFC SD in red, *CTA* SD in blue.

There is also a notable difference between this membrane studies and the ones that studied in *Cornelissen et al*. study; the CTA membrane used by them was thinner than the TFC membrane (50 to 150 µm) whereas in this current study, TFC membrane used was substantially thinner than the CTA one.

In Figure 19 it is shown the comparison of membrane permeability (J_S) that offered each membrane type within a specific pump speed. In both cases, minimum salt flux was obtained at 100 rpm (equivalent flowrate of 0.25 l/min), while also in both cases their maximum is at 50 rpm (0.13 l/min). Contrarily to the tendency seen in Figure 14, salt flux did not show a continuous increase.

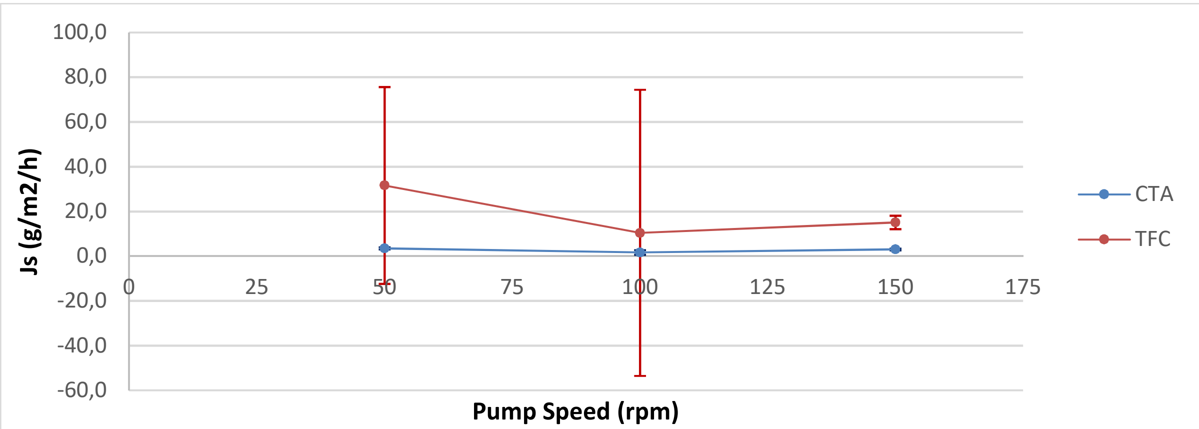

Figure 19. Average salt fluxes for CTA (blue) and TFC (red) membranes within three different pump speeds, in pulsion configuration. Notice that each dot corresponds to speed pump 50, 100 and 150 rpm, in the same order. TFC SD in red, *CTA* SD in blue.

[2] for the operating conditions of DI water as FS, active layer of the membrane facing the FS and 87 g/l of salt (in this case, MgSO4) in the DS

It is clear from Figures 18 and 19 that TFC showed higher salt and water fluxes than CTA at the same operating conditions. However, TFC membrane test in Figure 19 showed very high SD values thus it is not a reliable source of discussion when comparison with CTA is needed. From Figure 19, it can be seen that TFC tended to present higher salt fluxes, whereas at *Cornelissen et al*. found exactly the opposite; higher water and salt fluxes for CTA membranes. But more bibliographic data supports what has been obtained in this project [15,31,37,39,40].

When both membranes behavior at suction operation mode were compared, as it is shown in Figure 20, TFC did not offer better performance than CTA case, as J_W is much higher and J_S much lower. But in J_W, this bias may be explained by its high SD since all the results obtained before led to think the opposite. Thus, due to this high variance, these results were considered as a failure of the system rather than valid values for comparison.

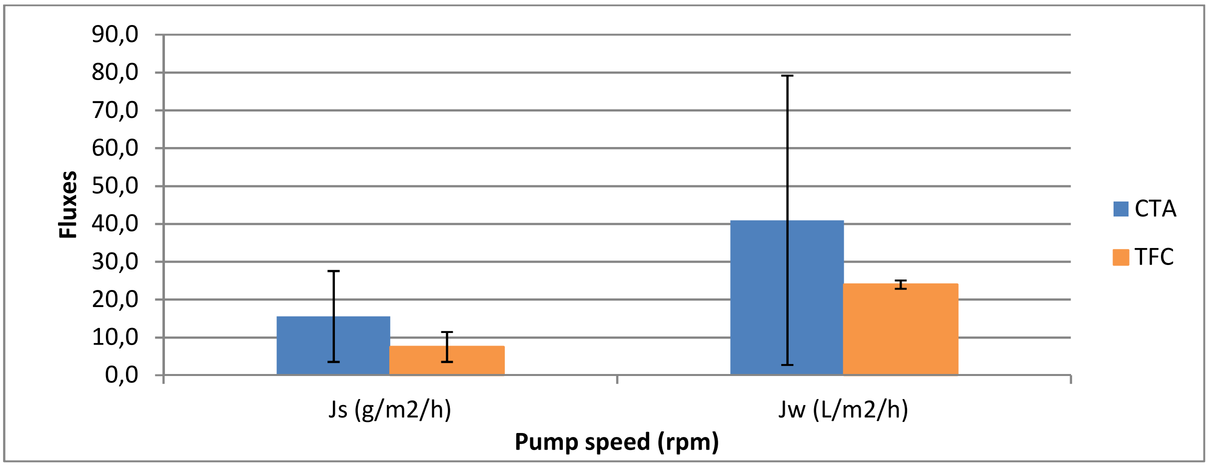

Figure 20. Average salt -J_S- and permeate -J_W- fluxes for CTA (blue columns) and TFC (orange columns) membranes in suction configuration. Operating conditions of 35 g/l salt concentration in DS and pump speed of 50 rpm. SD is shown in black.

4.4 Experimental limitations

There have been some experimental limitations that need to be settled:

- There was not enough time to perform all needed experiments, such as CTA membrane with different DS salt concentration. Thus, comparison with TFC performance was poorer, leading to lack of arguments to support the hypothesis of TFC being better than CTA membranes.
- More replicates need to be done to strengthen the results here obtained. When TFC tests were performed, even though they gave good results no solid tendency was clearly found.
- SD value was likely to appear high due to the lack of control of certain phenomenon, like the increase of the flux rate and its effects on the averages used for calculations.
- There have been some leakage problems during the whole project, because gluing the surrounding rubber needed for the cell to be hermetic, and it did not always work.
- When studying flowrate as a direct variable dependent of pump speed, no specialized bibliography was found about impact of pump speed on OMBR performance, as it was performed as a control test for measuring the impact on the whole study system. Thus, it presumably can be regarded that higher values of pump speed (leading to high values of flowrates) may increase the tendency to membrane permeability, since J_S seems to increase in both CTA and TFC membrane systems. There is no solid tendency about the impact of flowrate, as a consequence of the pump speed, on the behavior of water flux (J_W); further studies should be done.

4.5 Ethical view

Finally, regarding the ethics and sustainability of this project, it is believed that this kind of work enhances the state-of-art of green chemistry. Since water is a limited good, humankind needs to learn to improve its administration.

It is well known that water is a scarce. While hydrosphere has been suffering various natural and artificial changes, altering the water cycle and its balance has led to make a worrying call. New instrumentation and techniques has permitted quantify more accurately the fresh water and groundwater loss over the past decades: sensitive space-based GRACE (Gravity Recovery and Climate Experiment) [5].

Though this work only was performed in a laboratory scale, finding a suitable membrane is highly beneficial for our society, in both domestic and large-scale implementation. For this project is ecofriendly with the environment as its aim is to recover water, there is still more work to do on osmotic membranes [5,6,41].

5 Conclusions

- There is a clear tendency that when salinity in DS is increased so does the water flux generated and the permeate recovery, as it is established in the revised bibliography.
- There is no solid evidence that the pump speed has had large impact on these variations from bibliographic data. When compared, TFC also showed better *RSD* values.
- Since high variance of data was obtained comparison is difficult. More control of the system is required to reduce its intrinsic bias.
- The majority of the bibliography considered here supported the results here obtained; TFC permits higher fluxes for both J_S and J_W. When compared with CTA, TFC also showed better *RSD* values. Therefore, TFC seems to be the preferable option for future studies.
- Suction mode tests do not show clear tendency on whether it is preferable to operate in pulsion or suction. Even though, better results were obtained in pulsion, more experimental tests for comparison of both pulsion and suction modes should be done to elucidate the differences on the actual impact on the performance of the membrane.

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