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Article

Assessment of cellulose acetate membranes separation efficiency in wastewater treatment

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Abstract
Water as a high value resource is constantly used in every activity (from households to industries) and that's why wastewater treatment is essential to ensure the protection of human and environmental health by removing pollutants and pathogens from wastewater before releasing into environment or reused.
Among the emerging technologies, membrane separation processes are efficient, eco-friendly and relatively low-cost solutions that address this problem.
This work explores the benefits of cellulose acetate membranes in wastewater treatment. These natural based membranes offer a sustainable and eco-friendly approach in removing pollutants due to their biodegradability, high permeability and selectivity, showing high efficiency in the separation process. Also, the fouling degree is quite low which ensures multiple separation cycles without anti-fouling treatment which overall translates as cost-effective.
Three types of cellulose acetate membranes (8%, 10%, 12% wt.) were prepared by phase inversion. Each membrane was subject to morphological analysis using SEM which revealed an active layer between 4.468 – 8.324 µm. Secondly the membrane flow characteristics were measured at various pressures, obtaining a maximum average ultrapure water flow of 7868 L/m2h at 3 bar pressure for 8% CA membrane.
Preliminary tests for real wastewater membrane separation showed a maximum efficiency of 93% for CA 8% and 12%, and 81% for CA 10%. The fouling degree was measured after 5 separation cycles showing 15.27% clogging for 12%, 45.12% for 8% and 45.30% for 10% CA. Total suspended solids were reduced from an initial concentration of 110÷134 mg/L to values lower than 5 mg/L.

Keywords: *cellulose acetate membrane, waste water treatment, membrane separation*

INTRODUCTION

Membrane separation processes offer a promising and versatile solution for wastewater treatment, particularly when combined with other "green" technologies. This synergistic approach can enhance the removal of micro pollutants and emerging contaminants, leading to more efficient and sustainable wastewater management.
The European Commission's objective is to achieve climate neutrality by 2050, with a specific target to reduce emissions up to 55% by 2030 [1]. In 2021, the European Commission adopted the Action Plan to reduce pollution of water, air and soil, which works in synergy with the principles of the circular economy to develop sustainable industrial systems, clean technologies, and reduce the consumption of energy and raw materials [1, 2].
Membrane processes are currently one of the most efficient separation processes and are constantly improving. Recent advances in polymer chemistry, materials science and engineering open perspectives for new applications of membrane processes [3]. There are many possibilities for the separation of large streams of heterogeneous or homogeneous mixtures by using membranes and hybrid membrane-based processes which promises to be very efficient and profitable.

The separation principle in membrane processes is realised by:

- *Size exclusion*: membranes have specific pore sizes that allow smaller molecules (such as water) to pass through while blocking larger molecules (such as oil droplets and other contaminants) [4];
- *Selective permeability*: the membrane material is designed to be selectively permeable, which means that it allows certain substances to pass through and retains others, depending on their size, shape and chemical properties [4].

The main factors influencing separation efficiency are: membrane composition, pore size and the speed at which mass transport takes place. Mass transport through the membrane can be diffusive or convective and it is influenced by pressure gradient, concentration, temperature or electrical potential difference. Membrane technologies work without the addition of chemicals and with relatively low energy consumption. They are used to treat groundwater, surface water and wastewater. The membrane separation process can be used as an alternative for flocculation, sedimentation, adsorption, extraction and distillation.

The selectivity and process productivity is given by the efficiency of membrane separation. Selectivity is expressed as retention or separation factor while productivity is expressed as flow [5].

Microfiltration (MF) and ultrafiltration (UF) are used for the removal of larger particle sizes, while reverse osmosis (RO) and nanofiltration (NF) are used for the removal of salts [6]. The correlation between membrane processes and pollutant removal depends on the pore size of the membrane and the molecule size of the pollutant. Membrane processes such as NF and RO can be used with promising results in wastewater treatment containing micropollutants such as endocrine disruptors, pharmaceuticals and personal care substances [7].

The most commonly used materials in membrane fabrication are polymeric materials. The table below shows which types of polymers are mostly used.

Table 1. Types of polymer and the applications of the fabricated membranes

Polymer	Application	Information	Efficiency	Reference
Cellulose acetate (CA)	Dyes, salts, heavy metals removal	Hybrid membrane: graphene oxide + carboxylated titanium dioxide blended with CA Composite membrane: (hydroxyapatite nanoparticles + CA)	94.94% Methyl violet, 91.28% methyl orange, 88.28% methylene blue, ~40.40 % NaCl, ~ 42.97 % Na_2SO_4; 99.7% Pb, 95.46% Fe (III) removal	[8, 9]
Polyethylene (PE)	Heavy metal removal Microplastic removal	Modified PE with Polyamide; Nanocomposite membrane: PET+carbon nitride	94.5% Mg^{2+} removal; 72.8 % organic matter removal from drinking water and 72.3 % from wastewater effluent	[10, 11]
Polysulfone (PSf)	Anion exchange for water electrolysis; Protein separation	Polysulfone membrane cross linked with chloromethylene; Doped catalyst $FeCl_3$, $CuCl_2$ on modified PSf membrane	1.58 mmoL/g ion-exchange capacity; 453.3 L/m^2 permeation flux for PSF/PANI-$CuCl_2$	[12÷14]
Polypropylene (PP)	Microfiltration of wastewater from car washes	Washing agents present in wastewater (alkaline pH) could reduce the fouling effect of the membrane	Reduces tubidity (0.2÷0.8 NTU in permeate), bacterial removal (almost 100% for E. Coli)	[15]
Polyvinylidene fluoride (PVDF)	Dye removal	Cellulose acetate esters extracted from peanut shell used as filler for PVDF membrane to reduce fouling	95.75 ± 0.78 % for rejection of MB (methylene blue), Increased hydrophilicity 23.49 ± 2.40 L m^{-2} h^{-1}	[16]
Polyester (PE)	Drinking water treatment	Interfacial polymerization between erythritol and trimesoyl chloride (TMC) to decrease pore dimension.	77.11 up to 85.00 % DOC removal; high rejection of Na_2SO_4, $MgSO_4$ and NaCl compared to other PE membranes	[17]

Polymer	Application	Information	Efficiency	Reference
Polyethersulfone (PES)	Pharmaceutical wastewater treatment	Blending nanofiltration with electrochemical oxidation to remove antibiotics	99.7% rejection for azithromycin, 98.8% rejection for Na_2SO_4 and 74.9% for $MgCl_2$	[18]
Polyamide (PA)	Oily wastewater treatment	Hybrid techonolgy: PA membrane coupled with activated carbon	Up to 99% oil removal	[19]
Polyacrylonitrile (PAN)	Dyes and organic matter removal	Composite membrane: PAN blended with PEG (polyethylene glycol)	Above 98% separation efficiency for BSA, HA, CR and CB dyes	[20]

Cellulose / cellulose acetate

Cellulose is an abundant organic compound found in the structure of most plants which is used in the paper, plastics and filter membrane industries. It has limited solubility in common organic solvents due to the inter and intramolecular hydrogen bonds in its structure, therefore cellulose derivatives such as cellulose acetate (CA), cellulose triacetate (CTA) and carboxymethyl cellulose (CMC) are preferred for membrane production [9].

If we take a look at the global plastic production, about 10÷24% of plastic is recycled while at least 76% is irresponsible dumped in the environment. This is where the advantages of cellulose such as biodegradability and natural origin come into play, as it is considered a renewable source. This means cellulose based membrane represents a sustainable wastewater treatment solution [21].

Cellulose acetate (CA) is the main ester of cellulose, synthesized by partial or total acetylation of the available hydroxyl groups in the anhydrous glucose units. Depending on its acetyl content, CA can be classified as mono-, di- or tri-substituted. The degree of substitution is an important characteristic as it influences both the solubility and biodegradability of this cellulose derivative. At industrial level, CA is obtained through the esterification process of the cellulose molecule in the presence of sulphuric acid (H_2SO_4) with acetic acid (CH_3COOH) and acetic anhydride ($C_4H_6O_3$). The cellulose derivatives are found mainly in wood, flax and cotton. Various studies have reported the use of environmentally friendly synthesis processes using agricultural residues (corn starch, rice husk, sugar cane) and replacing the use of sulphuric and acetic acids with less polluting substances [22]. Agricultural and industrial residues contain mainly cellulose (30÷60%), hemicellulose (14÷40%) and lignin (7÷20%) [23]. In this regard we can consider that there is a renewable source of raw material available at low cost for the synthesis of cellulose acetate membranes.

Substitution degree

The degree of substitution (DS) is an important parameter of the modified cellulose because it influences the physical and chemical properties such as its solubility, water-holding capacity, and thermal stability. During the acetylation process, the degree of substitution (DS) is a key indicator of how many hydroxyl (OH) groups on each glucose unit have been replaced with acetyl groups. A higher DS value indicates that most of hydroxyl groups of the glucose unit have been replaced by acetyl groups within the cellulose structure. The maximum DS value obtained can be equal to 3, which translates into high solubility in solvents like acetone and tetrahydrofuran (THF), while cellulose with DS = 0 means it is insoluble in these solvents [23].

This substitution significantly impacts properties like hydrophobicity, mechanical strength, and protein binding. Cellulose is mostly hydrophilic, and may have higher protein binding effect, also can be affected by chemicals. On the other hand, cellulose acetate membranes have high hydrophilicity and lower protein adsorption, have good mechanical strength and stability, are non-toxic, quite easy to fabricate (phase inversion technique), and they are also affordable. These traits are essential when developing large-scale applications that are safe to use for water treatment, assures high performance of separation, are easy to produce and cost-effective [4]. Widely used in filtration, especially in water and wastewater treatment, gas separation, and energy generation.

Cellulose acetate membranes were amongst the first types of filtration membrane developed along with cellulose and ethyl cellulose membrane [24].

Applications of CA membranes

Listed below are a few examples found in literature about the wide spread applicability of cellulose acetate membranes showing their great potential in environmental protection: crude oil wastewater separation [25,26]**;** heavy metal removal from wastewater [9,27]**;** removal and degradation of dyes [28,29]; treating water contaminated with natural organic matter (NOM) [30]; separation of macromolecules (proteins) [31]**;** removing organic pollutants from wastewater [32]; separation of micro plastics, nano plastics and bacteria [33,34]; drinking water purification [35,36]; indoor air purification [37,38]**;** virus removal from biological products [39]; air filters for the automotive industry [40]; nature-Based Solution for wastewater treatment [41]; catalytic degradation of organic pollutants form wastewater [32]; removal of pharmaceuticals from wastewater [18,42].

EXPERIMENTAL PART

Membrane preparation

Three types of cellulose acetate membranes (8%, 10% and 12% wt) were prepared by phase inversion. Firstly, the cellulose acetate (50000 g/moL) was dissolved in 1-methyl 2-pyrrolidone (NMP) solvent higher than 99.5% purity, then the additives polyvinylpyrrolidone (PVP), K30 (40000 g/moL) and K25 (24000 g/moL), respectively polyethylene glycol (PEG) 4000 (M = 3500 ÷ 4500 g/moL) were added.

The mixture was continuously stirred for 24 hours. After 24 h, the polymer solution was deposited on the flat glass sheet and stretched with the doctor blade device (300 μm slit), followed by rapid immersion in the ultrapure water coagulation bath (conductivity less than 5.6 x 10^{-5} mS/cm) where precipitation was performed. Finally, glycerol 99.5% and ethanol 96% were used for post-treatment and membrane conditioning, followed by drying at room temperature for 24 hours.

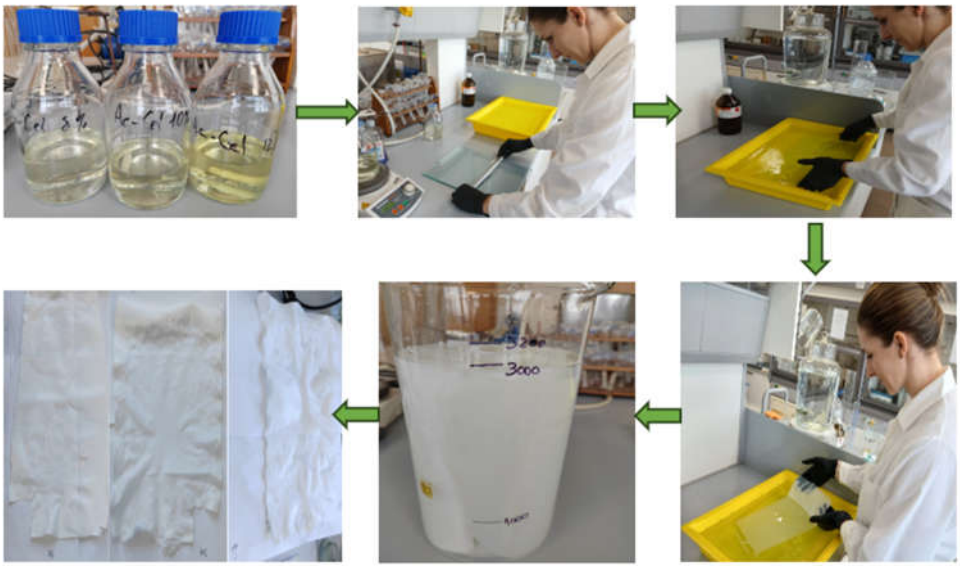


Fig. 1. The steps in membrane preparation

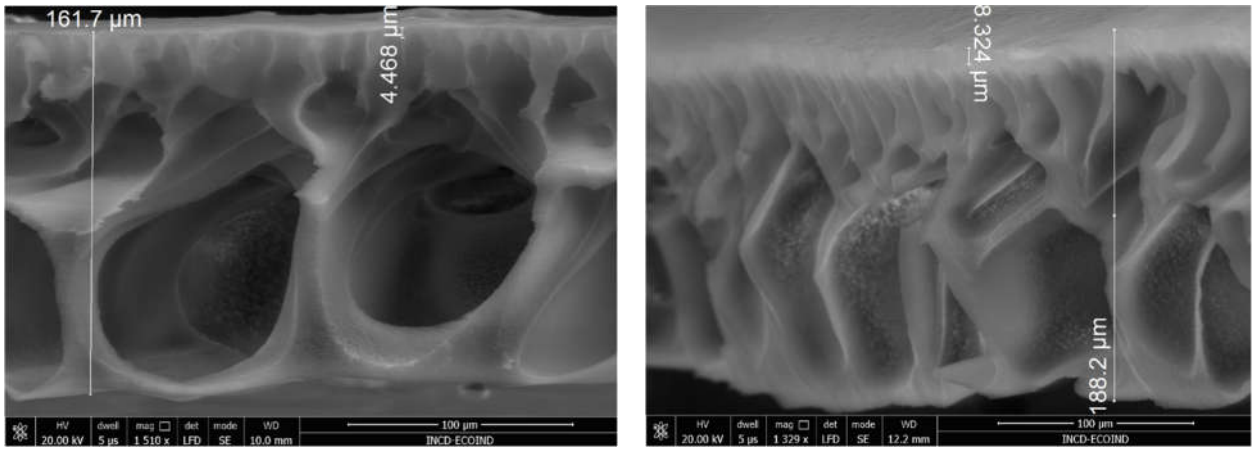
SEM analysis

Scanning Electron Microscopy (SEM) is a technique for analysing the morphology of a sample by scanning it with a focused electron beam. High resolution images (down to 1.4 nm) are taken and the composition is analysed by energy dispersive X-ray microanalysis [43].

The membranes were morphologically characterised by scanning electron microscopy using a FEI Quanta FEG 250 (Thermo Fischer).

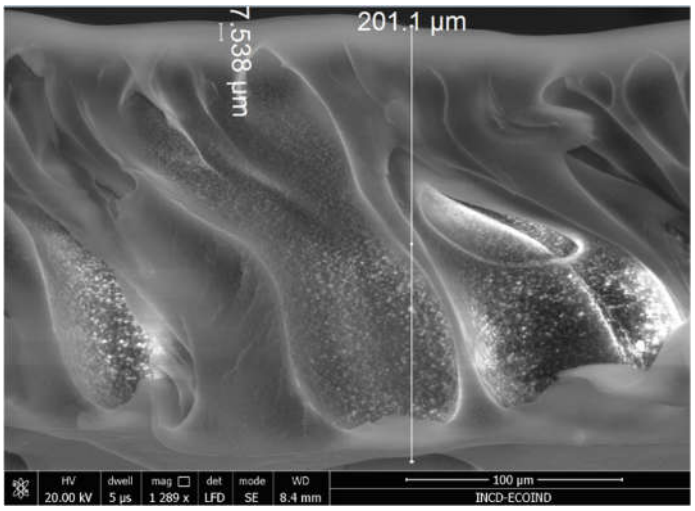
SEM images of the CA membranes showed mostly uniform distributed pores on the active surface, with pore size that varies in order CA 8% > CA 10% > CA 12%. It is observed that the increase in CA concentration decreases the pore size.

Total membrane thickness showed values between 161.7÷201.1 µm. The ***thickness of the active layer*** showed values between 4.468÷8.324 µm revealing a direct correlation with the increase of the initial CA solution concentration used in the membrane synthesis.



2a. CA 8% section

2b. CA 10% section



2c. CA 12% section

Fig. 2. SEM section images of CA 8%, CA 10% and CA 12%

Determination of membrane flow characteristics

In order to determine the flow characteristics of CA membranes, it was used a KMS Laboratory Cell CF-2 instrument (figure 3). The average ultrapure water flow was determined at different working pressures between 1 and 3 bar.

The average ultrapure water flow was determined by monitoring the permeate volume and time after 4 feed cycles (tests), using the formula [44]:

$$Jw = V/(S \times t) \quad (1)$$

where:

J_w = ultrapure water flow passing through the membrane;

V = volume of ultrapure water passing through the membrane;

t = the time in which the volume of water V was collected;

S = specific surface area of the membrane (28 cm^2).

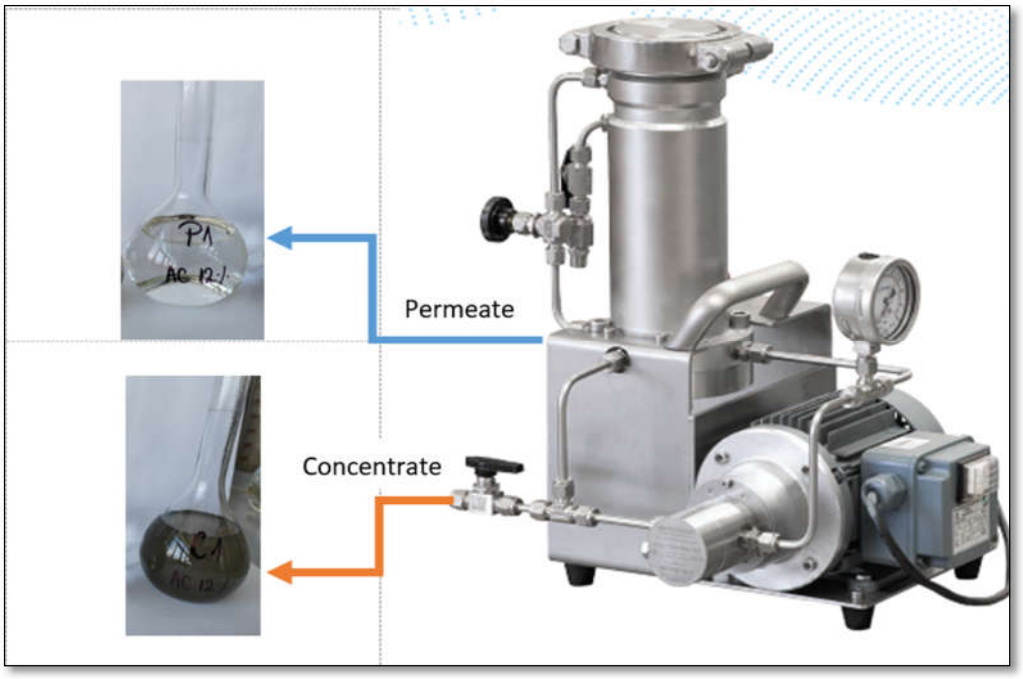


Fig. 3. KMS Laboratory Cell CF-2 equipment used in the separation process

RESULTS AND DISCUSSION

Ultrapure water flow determination

The average ultrapure water flows varied in direct correlation with the concentrations of the polymeric solutions from which the membranes were obtained and their morphological characteristics: 8% CA > 10% CA > 12% CA.

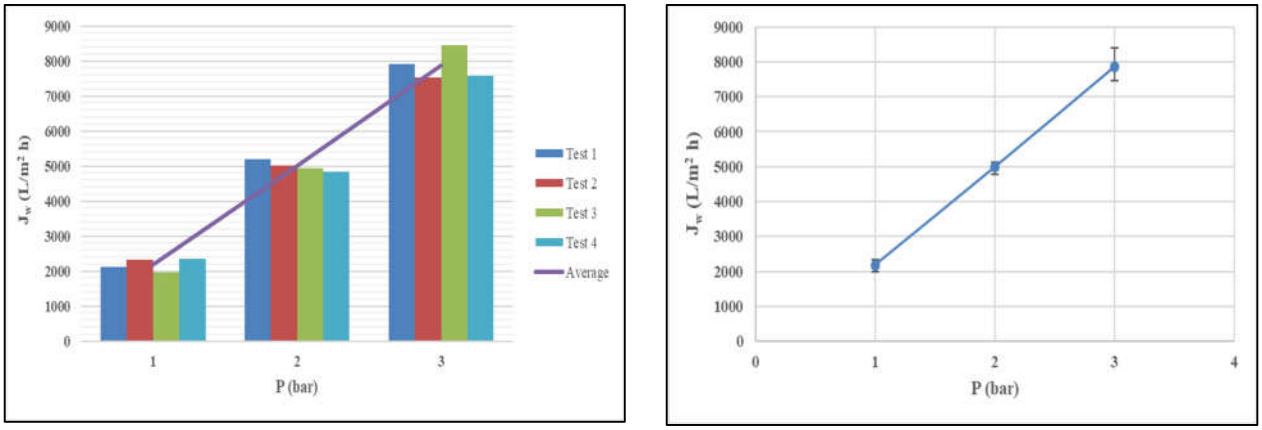


Fig. 4. Ultrapure water average flow for CA 8%

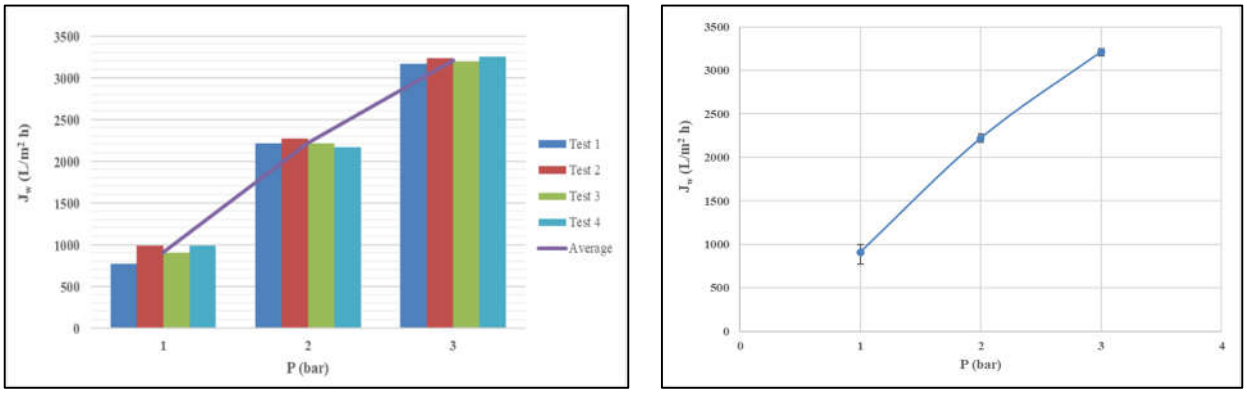


Fig. 5. Ultrapure water average flow for CA 10%

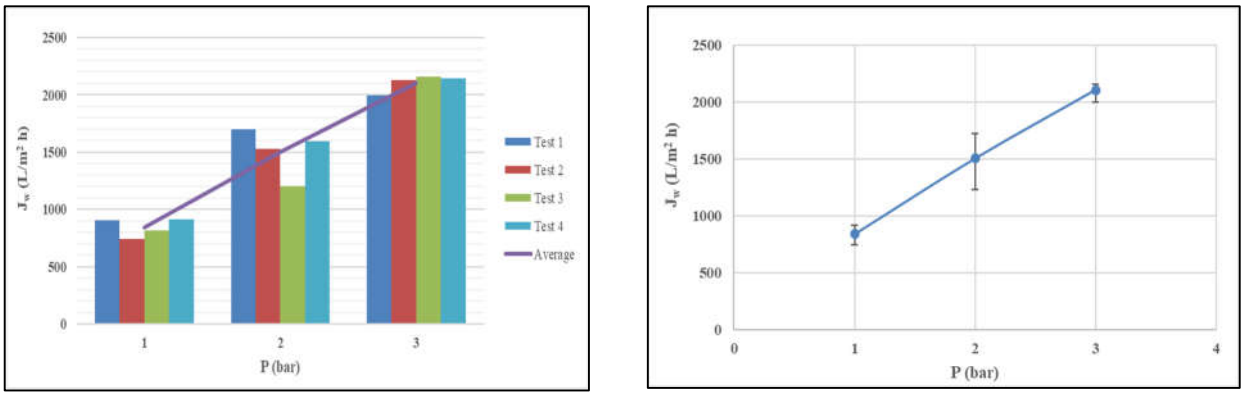


Fig. 6. Ultrapure water average flow for CA 12%

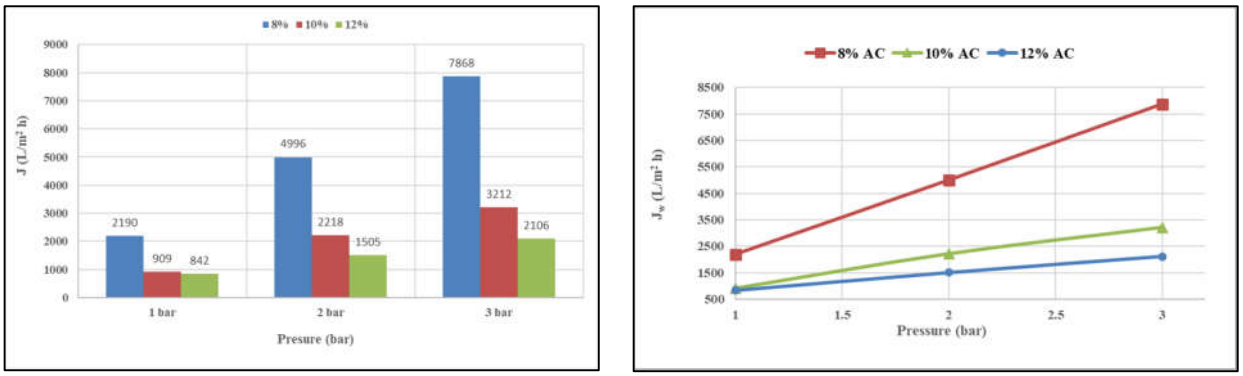


Fig. 7. Comparison of the pure water average flow obtained for each of the three CA membrane at different pressure

For the separation experiments was established a working pressure of 2 bar due to the better results of ultrapure water flow obtained in the initial tests also because the errors resulting from the three measurements were lower.

Membrane separation performance tested on real wastewater

Separation tests were performed using the three types of membranes prepared (CA 8%, CA 10% and CA 12%) which were subjected to a series of 5 tests for each type of membrane, at a working pressure of 2 bar. A volume of 400 mL raw wastewater was introduced in the feed tank and started the separation process at 2 bar pressure while measuring the time in which 200 mL permeate was collected after separation. After each test the installation was washed with 400 mL ultrapure, measured the time required for extracting 200 mL of permeate in order to determine the fouling effect of the membranes.

After each separation were recorded: average separation flow; average ultrapure water flow to determine the fouling effect; COD indicator was monitored to determine the separation efficiency; Total suspended matter (TSM) was monitored in order to establish the separation efficiency.

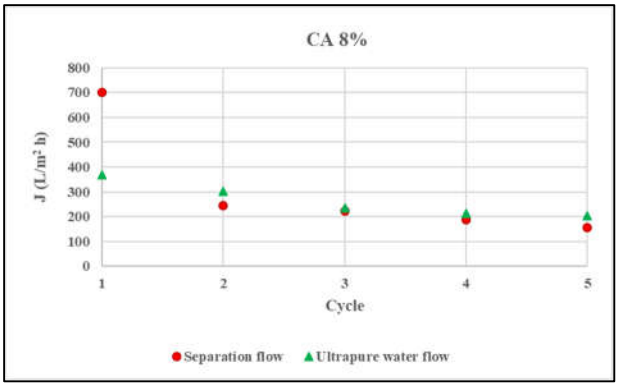


Fig. 8. Separation flow and ultrapure water flow vs. separation cycles using CA 8%

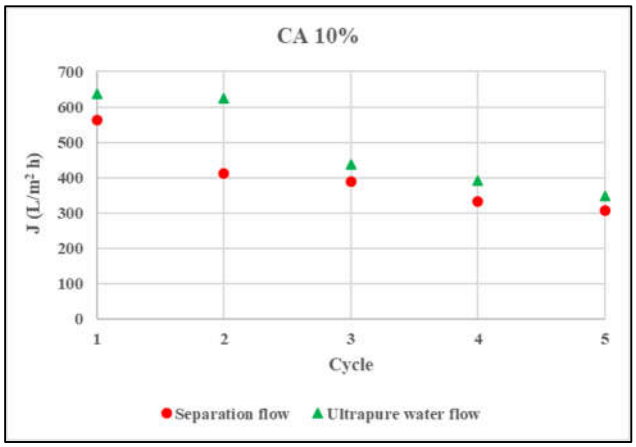


Fig. 9. Separation flow and ultrapure water flow vs. separation cycles using CA 10%

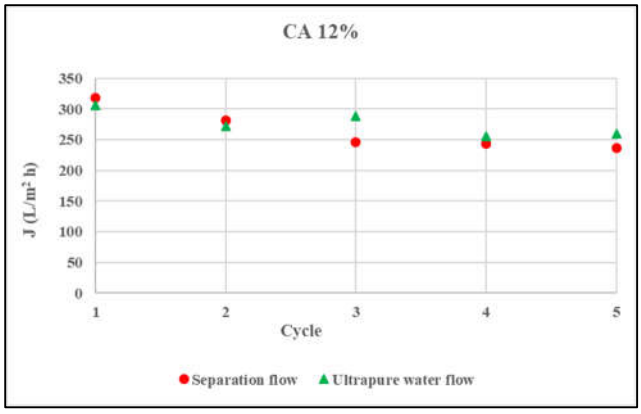


Fig. 10. Separation flow and ultrapure water flow vs. separation cycles using CA 12%

Separation efficiency

After each separation cycles the permeate and concentrate were collected, and determined the organic content by measuring the COD indicator in order to establish the removal efficiency. The results showed efficiencies between 78÷93% for CA 8%, 36÷93% for CA 12% and 56÷81% for CA 10%.

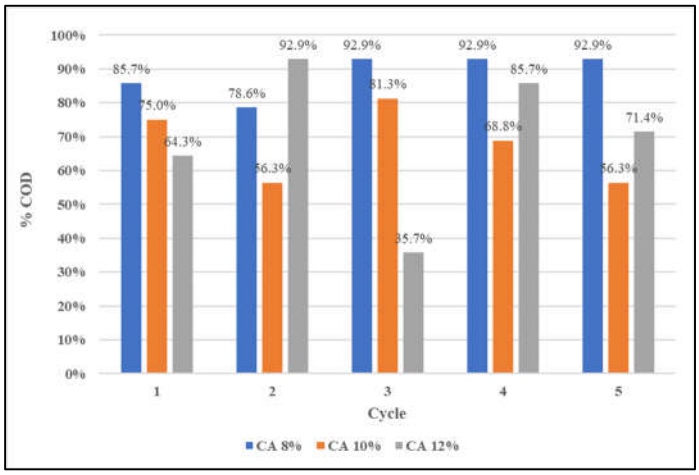


Fig. 11. Separation efficiency expressed in COD after each cycle for the 3 membranes

The fouling effect was determined by measuring the average ultrapure water flow after 5 separation cycles and the results showed:

- 202 L/m^2 h for CA 8% (45.12% degree of clogging);
- 348 L/m^2 h for CA 10% (45.30% degree of clogging);
- 260 L/m^2 h for CA 12% (15.27% degree of clogging).

The calculated values after 5 separation cycles showed that the membranes can be further used without applying an antifouling treatment.

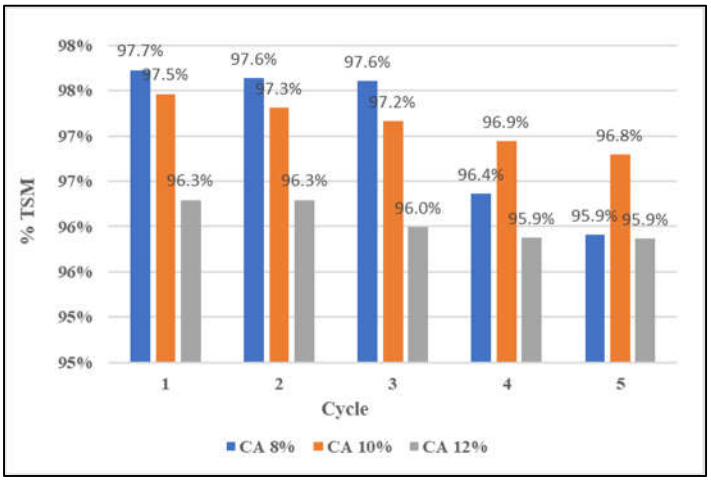


Fig. 12. Separation efficiency expressed in TSM after each cycle for the 3 membranes

TSM loading was reduced from initial values between 110÷134 mg/L to values less than 5 mg/L. After 5 separation cycles conducted for each membrane, they were again subjected to SEM analysis in order to observe the fouling degree, as presented in the figures 13a, 13b and 13c below. SEM images revealed the presence of unicellular organisms and microplastics on the membrane surface because of the pollutant load present in the wastewater.

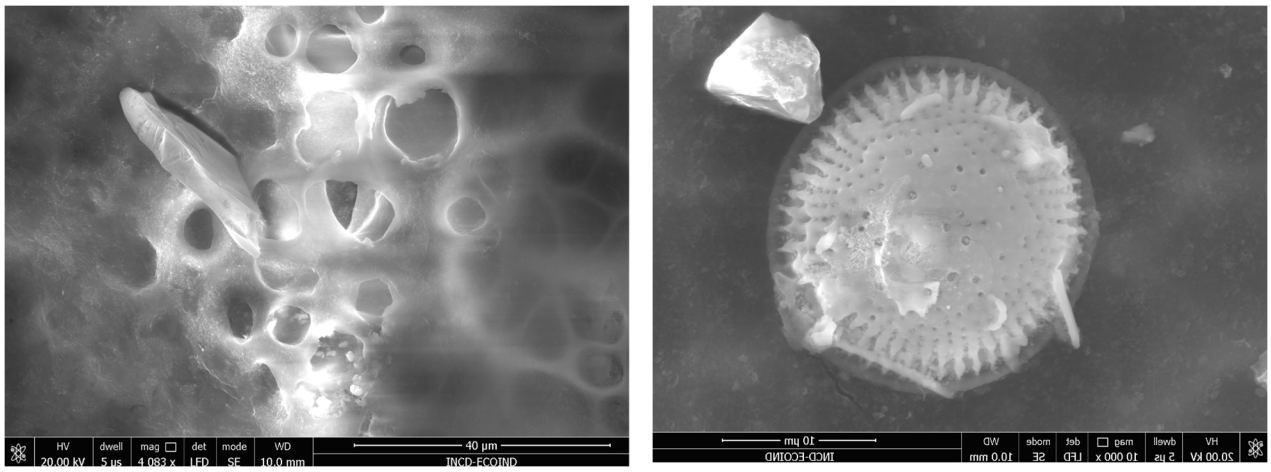


Fig. 13a. SEM images of CA 8% after 5 separation cycle

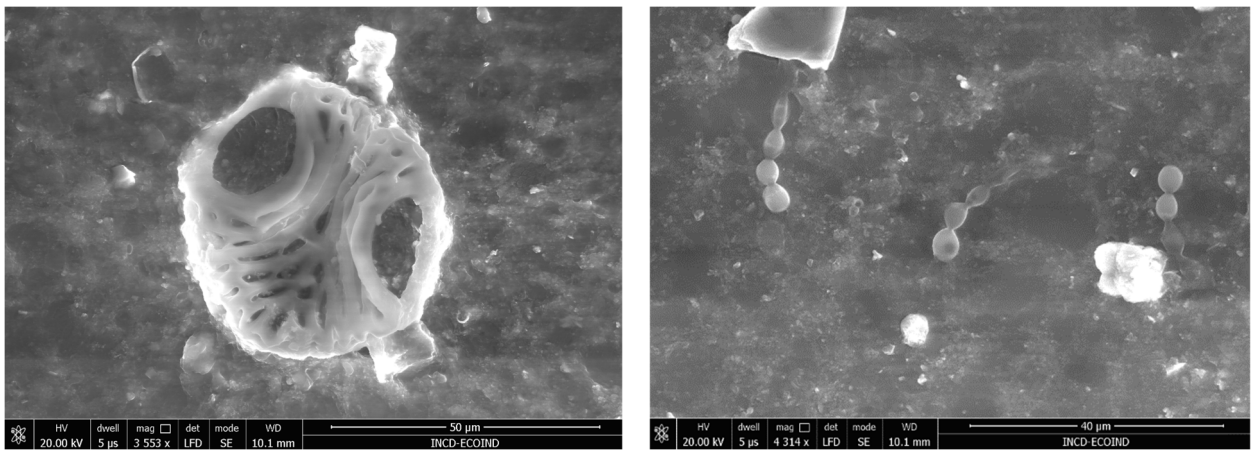


Fig. 13b. SEM images of CA 10% after 5 separation cycle

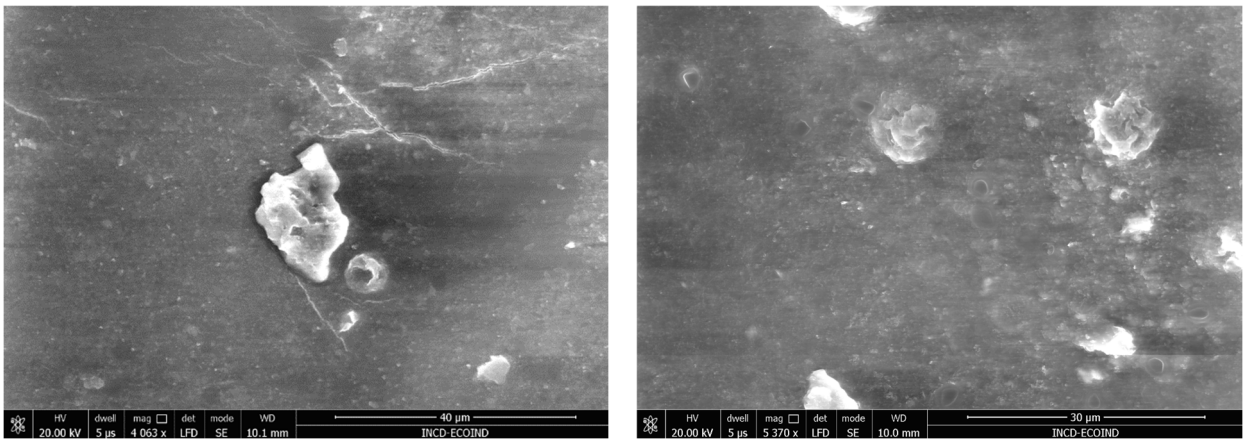


Fig.13c. SEM images of CA 12% after 5 separation cycle

CONCLUSIONS

Three types of CA membranes were synthesized using polymeric solutions of 8%, 10%, and 12% wt CA mass percentage.

SEM images of the CA membranes revealed evenly distributed pores on the active surface, with pore sizes decreasing as the CA concentration increases (CA 8% > CA 10% > CA 12%). In other words, higher CA concentrations in the membrane material generate smaller pores.

The ultrapure water flow passed through the membranes decreased as the concentration of the polymeric solutions increased. Specifically, CA 8% membrane had the highest flow rates, followed by CA 10%, and then CA 12%, due to variations in their morphological characteristics.

For the preliminary tests it was used a real wastewater sample with an organic load of 120÷140 mg O_2/L COD and TSM of 110÷134 mg/L. Each of the 3 prepared membranes were tested for 5 separation cycles alternating with ultrapure water after every separation cycle in order to assess the fouling degree.

The fouling degree calculated for each membrane ranged between 15.27% for 12% CA, 45.12% for 8% CA, and 45.30% for 10% CA. After 5 separation cycles the average ultrapure water flux maintained above 200 L/m^2 h, which shows that the membranes can still be used.

The test results indicated that the membrane prepared with CA 8% achieved higher performance, about 88.6% efficiency in COD removal and TSM removal efficiency of 97% at 2 bar working pressure.

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