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# A study on cross flow membrane module with laboratory synthesized nano-filtration membrane

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## ABSTRACT

The evolution of new polymer membrane material is one of the important factors in the quest for advances and improvements in membrane technology. The various membrane processes and range of particles diffusing through or retained are based along the membrane pore sizes. Membrane process like Micro filtration, Ultra filtration, Reverse osmosis, Electro dialysis, and so forth are used individually or integrated appropriately with other traditional techniques, in many industries for the recycling of rare materials, toxic chemicals, polymer binders, etc. The primary aim of this paper is to make a blend membrane with CA/PSf compounds by using the phase inversion technique. Then form Nano-filtration membrane by the extra methods. This composite membrane installed in the cross flow module, and ticking off the performance of the Nano-filtration membrane using drain water.


## KEYWORDS

Cellulose acetate/ polysulfone composite membrane;
Membrane preparation;
Phase inversion technique;
Cross flow membrane module;
Drain water treatment.

## INTRODUCTION

The demand of, not always sufficiently available, ground and/or surface water, is expected to increase in the future. Therefore alternative sources of water among of which wastewater will gain more importance compared to the more traditional water sources. Waste water reuse after purification does not only help to overcome water shortages, but it also decreases the volume of wastewater to be discharged. The decrease, by pretreatment, of the volume or harmful loading of waste streams is of high importance in view of new legislations for waste discharge. Application examples are already published in literature.

A complex and sometimes more controversial force driving the introduction of membranes in environmental engineering applications is the change in the market, the size and features of which are intimately linked to changes in the available technologies. Municipal water treatment is not generally seen as an open grocery store. The immense majority of water treatment plants are supported by municipalities that do not behave according to common market incentives or business conventions. Cross flow membrane filtration became a popular issue in the late 1960s and early 1970s as methods of fabricating micro porous and infiltration membranes were com-

mercialized and new methods for harvesting fermentation broth were sought. Early references in this field from Michaels, Blatt et al., Porter and Michaels, Henry and Allred, and many others sought to apply and characterize this filtration technique, and to develop prognostic models of its execution.

Cross flow filtration is the most universal condition to identify the various techniques by which slurries of particles or solutions of macromolecules flow across the airfoil of a filtration medium with just a fraction of the liquid volume permeating the membrane purpose. Through several mechanisms, depending on the size of the molecule or particle, cross flow reduces the accumulations of materials on the membrane surface, allowing filtration to continue beyond the stage, which would be possible using traditional dead –end filtration. The flux will always refer to the permeate flow rate per unit area of membrane.

While reverse osmosis and ultra filtration were being built in several applications, there was a deficiency of available membranes with cutoffs between 400 and 4,000 g/mol. An increasing interest in NF membranes developed in the final ten. One of the main applications is water softening. The following membranes are recommended for use in water softening systems: UOP Fluid system modules 8231- LP (cellulose acetate blend) and 8921- UP (TFCS polyamide,), FILMTEC NF 70 and NF 40, Toray modules SCL – 100 (modified cellulose acetate) and SU 600 (composite polyamide), Nitto Denko NTR – 729 HF, Desalination Systems Desal – 5 and and Dupont SM 15[33, 36].

## MATERIALS AND METHODS

### Materials

Cellulose Acetate (CA) was provided by Central House (P) Ltd., New Delhi. It is a white amorphous powder, insoluble in water and soluble in acetone. It has a viscosity of 6% solution in 95% acetone and water mixture 25°C. It causes the acidity as (acetic acid.).

Poly(sodium4-styrenesulfonate),$[-CH_2CH(C_6H_4SO_3Na^-]_{n,}$ (PSF) was obtained from Aldrich Chemical Company, Inc. (USA).

N, N – Dimethylformamide, $C_3H_7NO$, (DMF) M =73.10g/mol, (1l=0.95kg) was procured from Merck, Merck Limited, Worli, Mumbai. It has specified: Assay (GC) ≥ 99.5%, Density (d 20 °/4°) 0.948 – 0.949, Water ≤ 0.1%.

Polyvinylpyrrolidone (K -30) pure, $(C_6H_9NO)$

**TABLE 1 : Commercially available membranes for ultra-filtration**

| Trade Name | Membrane Material | Cut off (g/mol) | pH Range | Maximum Operating Temperature (°C) | Protein Adsorption (mg/cm²) |
|---|---|---|---|---|---|
| | | **Millipore** | | | |
| PL Ultra filter | Cellulose | 1,000-300,000 | 2-13 | 35-50 | 1 |
| PT Ultra filter | Polysulfone | 10,000-300,000 | 1-14 | 50 | <100 |
| PALL Gelman | PALL Gelman | PALL Gelman | PALL Gelman | PALL Gelman | PALL Gelman |
| Nova | Nova | Nova | Nova | Nova | Nova |
| Omega (TM) | Omega (TM) | Omega (TM) | Omega (TM) | Omega (TM) | Omega (TM) |
| Desal | Desal | Desal | Desal | Desal | Desal |
| P-series | P-series | P-series | P-series | P-series | P-series |
| G-Series | G-Series | G-Series | G-Series | G-Series | G-Series |

**TABLE 2 : Characteristics of NF membranes in comparable condition**

| | Permeability ($m^3/m^2$day Mpa) | Cut off Retention (%) | | | | | | |
|---|---|---|---|---|---|---|---|---|
| | | (g/mol) | $Fe^{3+}$ | $Cr^{3+}$ | $Ni^{2+}$ | $SO_4^{2-}$ | $NO^{3-}$ | F |
| Desal -5 | 1.3 | 150-300 | 89.8 | 90.1 | 90.4 | 64.8 | 36.8 | 61.3 |
| PVD -1 | 0.8 | 180 | 96.0 | 96.4 | 96.4 | 71.4 | 24.8 | 69.6 |
| NF-45 | 1.2 | | 99.0 | 99.6 | 99.7 | 56.9 | 25.0 | 71.0 |

x, M. W. ~ 40000 (PVP) was purchased from Sisco research laboratories pvt. Ltd., Mumbai. It has analysis: pH (5% aq. Soln.) 3.0-7.0, Maximum limits of impurity water (5%).

Polyethyleneglycol 400 HO $(C_2H_4O)_{nH}$, 1l = 1.13 kg (PEG) was supplied by Merck, Merck Limited, Worli, Mumbai. It has specification: Mean M.W. 380420, Hydroxyl Number 267 295, Density (d20°/ 4°) 1.124 – 1.126.

Sodium Azide ($NaN_3$), M=65.01 g/mol procured from Merck, Merck Limited, Worli, Mumbai.

Distilled Water was prepared in the lab with the help of a distillation unit. Ultra-pure Water was prepared from Distilled Water by water purification system (Millipore Synergy 185) in the laboratory.

**Preparation of membrane**

The first solution was prepared by dissolving 14 gms of CA in 50 gms of DMF solvent. Then the solution was under constant mechanical stirring by an agitation for 0.5 hr. The second solution was prepared by dissolving 6 gms of PSF in 50 gms of DMF solvent. The second solution was stirred for 0.5 hours.

Then the two solutions were mixed. Afterward that the additives like PVP (5gms.) and PEG (5gms) was added to the blend solution. The blend solution based on CA and PSF in the presence of additives PVP and PEG, insolvent DMF was under constant mechanical stirring by an agitator for 0.5 hours to produce the polymers swelled. The solution obtained was set apart to stand for at least 0.5 hours to get rid of air bubbles. The temperature and viscosity of the solution were noted. The temperature of the solution was 33°C. CA/PSF blend solution was cast on a glass plate by phase inversion technique. The thickness of the spread casting solution was controlled by manually setting the top of the casting blade.

The thickness of the casting membrane was measured by micrometer. The thickness of the membrane is 250 μm. The roll film was kept in ambient air (35°C) for 2 minutes for the evaporation. The gelatin bath was prepared with 1 liter of ultra-pure water, 2 to 3 grams of DMF and 1gm of surfactant (SPAN 80). Then cast film along with glass plate was immersed in a 10°C gelatin bath and stuck in the gelatin bath for at least 1 hour for complete precipitation and the establishment of the membrane.

Then the membrane was removed out of the glass plate and rinsed it with distilled water or ultra-pure water. The tissue layer was again stored in ultra-pure water containing 0.5 to 1% Sodium Acid to prevent microbial attack on the membrane. The membrane was kept in this condition still it was engaged. The first solution was prepared by dissolving 14 mg of CA in 50 grams of DMF solvent. Then the solution was under constant mechanical stirring by an agitation for 0.5 hours. The second solution was stirred for 0.5 hours.

**Preparation of nano-filtration membrane**

The CA/PSF Membrane synthesized in the labo-

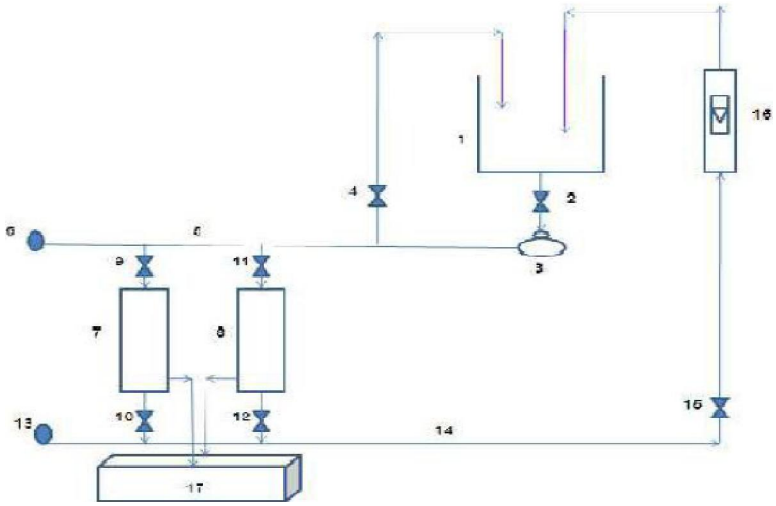


**Figure 1 : Schematic diagram of laboratory scale cross flow nano- filtration system**

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**Figure 2 : Diagrams of experimental setup shows the cross flow membrane module installed in setup**

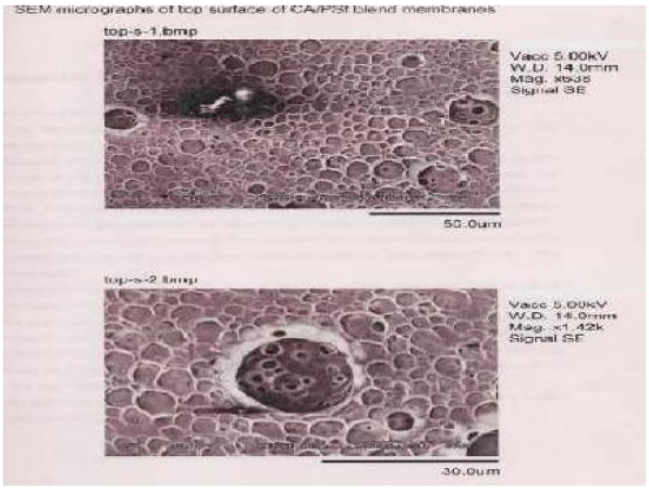


**Figure 3 : SEM micrographs of top surface of CA/PSF blend membranes**

**TABLE 3 : Results obtained from CA/PSf Nano filtration membrane using cross flow module**

| Initial time(t1) in min | Final time in (t2) in min | Sampling time (Δt) in min | Trans membrane pressure(Δp) in bar | Permeate amount of pure water(Vp) in ml |
|---|---|---|---|---|
| 0 | 5 | 5 | 1.5 | 250 |
| 5 | 13 | 8 | 1.75 | 370 |
| 13 | 25 | 12 | 2.1 | 450 |
| 25 | 40 | 15 | 2.4 | 480 |
| 40 | 60 | 20 | 2.7 | 550 |
| 60 | 85 | 25 | 3 | 550 |

| Purewater flux(lit/min.$m^2$) | Membrane resistance (bar.$m^2$.min/Lit) | Recycle liquid amount(ml) | Percentage of rejection |
|---|---|---|---|
| 5 | 0.3 | 3700 | 93.24 |
| 4.625 | 0.38 | 5500 | 93.27 |
| 3.75 | 0.56 | 6000 | 92.5 |
| 3.2 | 0.75 | 7200 | 93.33 |
| 2.75 | 0.98 | 7500 | 92.67 |
| 2.2 | 1.36 | 7500 | 92.67 |

ratory was used for preparation of composite NF membrane. The membrane was soaked in 10 (weight) $Zncl_2$ solution for 3 days. After 3 days it was dried at 40°C for 6 hours, and cooled subsequently to 30°C by ambient air. The membrane sheet was then soaked in dilute HCl solution ($P^H$ = 4). This resulted in formation of thin membrane sheet on removal of $Zncl_2$Using 1M NaoH solution at 30°C, the membrane was hydrolyzed. After 3 hours the membrane was immersed in 1M HCl at room temperature for 6 hours. The membrane was immersed in NaOH solution ($P^H$=8. 5) for 3 hours. This resulted in the formation of NF membrane. The membrane was then studied for flux, selectivity and morphology.

## Experimental set – up

## Morphological studies

A scanning electron microscope (SEM) (-3000 N HITACHI) was used to examine the top‘ surface and cross sectional view of the membrane. The membrane was cut into pieces of various sizes and mopped with filter paper. These dry samples were gold sputtered for producing electric conductivity and photomicrographs were taken in very high vacuum conditions. Several SEM images were taken at various magnifications for the top surface and cross sectional perspective of the polymer membrane.

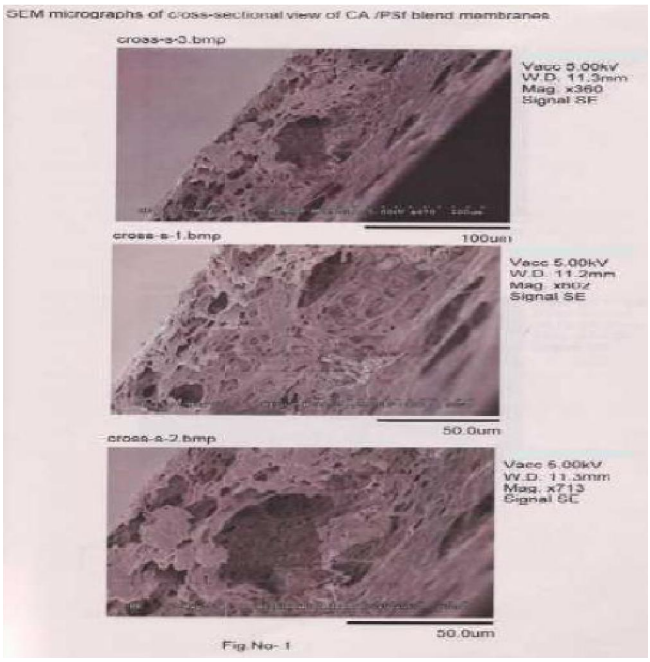


**Figure 5 : Time *vs* pure water flux**

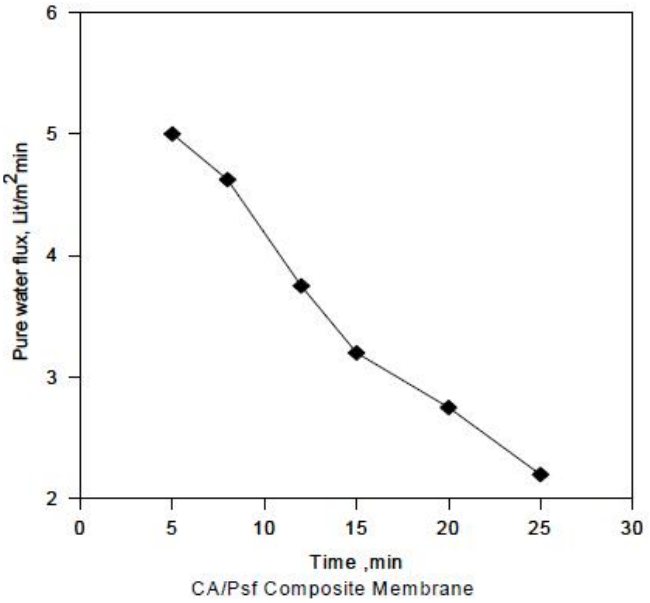


**Figure 6 : Time *vs* permeate amount of water**

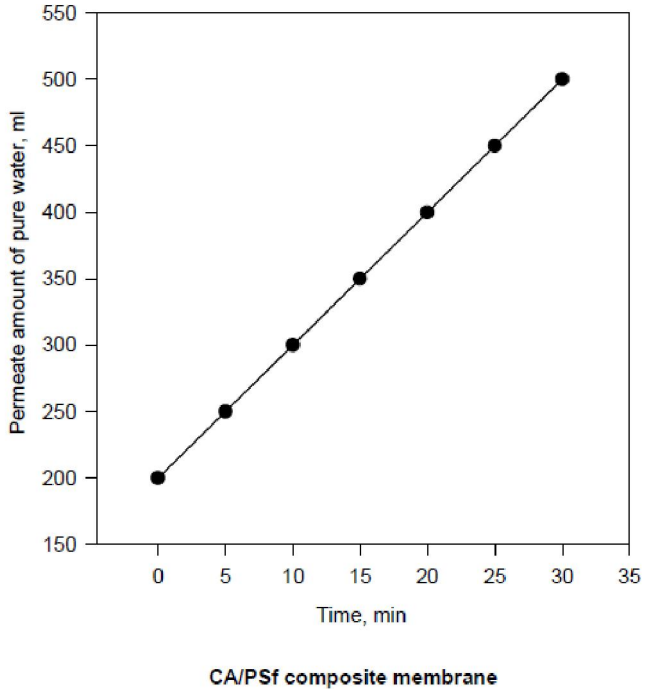


**Figure 7 : Pure water flux *vs* transmembrane pressure**

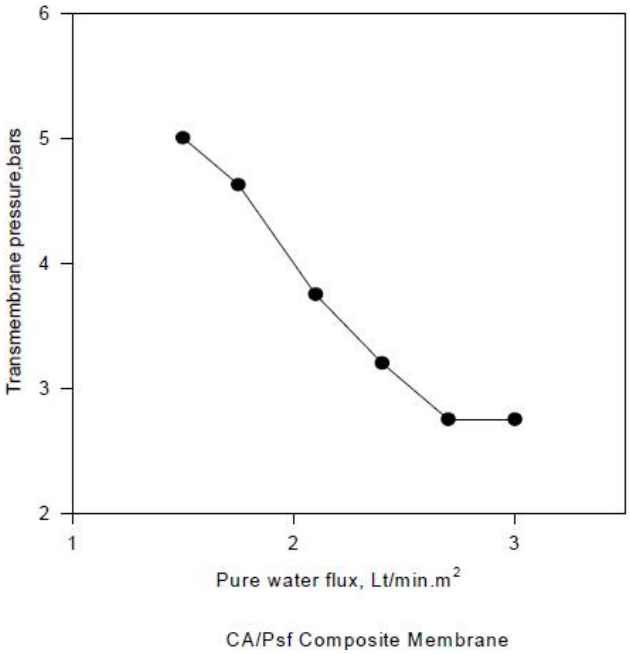


**Figure 4 : SEM micrographs of cross – sectional view of CA/PSF blend membranes**

## RESULTS AND DISCUSSION

The Nano filtration membrane was washed by ultra-pure water. Then washed NF membrane was fitted in cross flow membrane module. We take 10 lit of drain water filled into the tank and pop out the experiment to calculate water flux and membrane resistance.

Thickness of the membrane ($Z_m$) = 2.5×10-3 m;

cross sectional of the membrane (A) = breadth × length

Breadth of the membrane = 0.05 m; Length of the membrane = 0.2 m; A = 0.01 $m^2$;

Flow rate indicated by Rota meter (Q) = 8.8 lt/ min;

Cross sectional area of the stainless steel pipe ($A_c$) = $\pi d^2/4$;

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Diameter of the pipe (d) =0.00635 m; $A_c$ = 0.316×10-4 $m^2$; Cross flow velocity (v) =Q / $A_c$; V=278.48 m/min; Pure water flux $J_w = \frac{V_p}{\Delta t \, x \, A}$ = permeate amount of pure water / (sampling time × membrane area),lt/min $m^2$; Percentage of water content of membrane ($W_w$) =(1-(wt of dry membrane/ wt of wet membrane))×100;Weight of dry membrane = 0.6 gm; Weight of wet membrane = 1.1 gm;$W_w$=(1-(0.6/1.1))×100= 43.45%; Membrane resistance ($R_m$)=Transmembrane Pressure / Pure Water Flux = $\frac{\Delta p}{J_w}$; Rejection ratio (Rr) =(1-(permeate amount of pure water/amount of recycle water)) *100.

## CONCLUSIONS

The characterization of CA / PSf blend membranes in terms of pure water flux, membrane resistance, percentage of rejection and morphological studies was examined in the presence of Pore former, PVP and PEG.

Ultra filtration carried out at different transmembrane pressures up to 2 bars and nanofiltration operated in all the ranges of pressures present a respectable operation in terms of permeation in terms of permeation fluxes indicating a low level of membrane fouling for both membranes.

Cross flow membrane technology, in form of NF and RO, began to be recognized as an efficient, economical and reliable separation process, purification systems utilizing cross flow membrane

Filtration such as Nanofiltration (NF) or ultrafiltration (UF) can be a safe option to a conventional arrangement.

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