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Cellulose acetate membrane preparation by phase inversion to estimate optimized parameters and its performance study

Heena N Katariya*[1] and Tejal M Patel[2]

[1]*Department of Chemical Engineering, S. S Agrawal Institute of Engineering & Technology, Navasri- 396445 Gujarat, India*
[2]*Department of Chemical Engineering, G H Patel College of Engineering & Technology, Vallabh Vidyanagar-388 120, Gujarat, India*

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Abstract. Development in advanced separation processes leads to the significant advancement in polymeric membrane preparation methodology. Therefore, present research investigated the preparation and characterization of cellulose acetate membrane by phase inversion separation method to determine optimized operating parameters. Prepared CA membrane's performance was been analyzed in terms of % rejection and flux. Investigation was conducted to study effect of different parameters such as polymer concentration, evaporation rate, thickness of film, coagulation bath properties, temperature of polymer solution and of the coagulation bath etc. CA membrane was fabricated by taking polymer concentration 10wt% and 11wt% with zero second evaporation time and varying film thickness over non-woven polyester fabric. Effect of coagulation bath temperature (CBT) and casting solution temperature were also been studied. The experimental results from SEM showed that the surface morphology had been changed with polymer r concentration, coagulation bath and casting solution temperature, etc. Lower polymer concentration leads to lower precipitation time giving porous membrane. The prepared membrane was tested for advanced waste water treatment of relevant effluent stream in pilot plant to study flux and rejection behavior of the membrane.

Keywords: cellulose acetate; flux; nanofiltration; membrane; polymers; rejection; thin film composite; transport

1. Introduction

Entire world is suffering from the most severe problem of water shortage termed as "water scarcity". It is quite obvious that no one in this world can survive without water as it is essential component for every life exists on this planet. Main causes behind water scarcity are unnecessary waste of water, overpopulation, water pollution, global warming, depletion of ground water, degradation of forest lands, etc. One third of the developing world will face serious water shortage by 2025 (Keller *et al.* 2000). One of the possible ways to control water shortage includes the recycling and reusing of available waste water from its sources by the effective water treatment technologies. Waste water treatment involves breakdown of complex organic compounds of waste water into the stable and nuisance free compounds, either physico-chemically and/or by using biological treatment. Conventional water treatment techniques are carried at different level as preliminary, primary, secondary followed by advanced or tertiary treatment. Preliminary treatment involves removal of large substance such as sticks, grit etc. whereas primary treatment removes floating and settable materials usually by sedimentation. Secondary treatment is used to treat further the water from the primary treatment unit by biological and chemical processes. Finally the advanced treatment which involves the removal of dissolved and suspended impurities that the conventional secondary treatment process cannot be removed. With the conventional water treatment process, it has high maintenance and labor cost as well as the sludge handling is the most critical problem and it is quite costlier, too. Water rejection from such treatment process cannot be reutilized as it requires further treatment to make it suitable for the reuse. Conventional water treatment can reduce the water pollution, one of the causes of the water scarcity but by combining conventional treatment with the membrane process such as reverse osmosis, nanofiltration, and gives advantages of reutilization of effluent as a one of the solution to problem of water shortage. Large variety of materials especially polymers are extensively used in the preparation of membrane because of their chemical and thermal stability, high performance in terms of permeability flux and solute rejection, great flexibility as well as mechanical strength. Methods available for the membrane preparation are sintering, stretching, track-etching, template leaching and phase inversion.

Among all these methods, phase inversion is most extensively used as it provides large variation in surface morphology (Tan and Rodrigue 2019). Loeb and Sourirajan have invented first viable membrane by phase inversion method in 1960 (Kusumawati *et al.* 2018). In this method, a homogenous liquid polymer solution was converted into solid membrane film in a controlled manner and the mechanism was termed as liquid-liquid demixing (Sifat *et al.* 2017). Membrane morphology can controlled by controlling initial stage of phase transition, i.e., it decides whether the porous or nonporous membrane will be formed. This phase transition can be done by variety of techniques

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*Corresponding author, Assistant Professor
E-mail: hnk.ssaiet@gmail.com

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comprises of thermal precipitation, solvent evaporation, precipitation by controlled evaporation, immersion precipitation, precipitation from the vapor phase (Mulder 1996) and (Alihemati *et al.* 2020). In the present research work, membrane was prepared by immersion precipitation which sometimes called as Nonsolvent induced phase separation (NIPS) technique. As this technique allows the many membrane morphologies, it is most general and commercially explored method. For this study, Cellulose acetate was selected as a membrane material, it is a natural polymer and after it's fouling it can be degraded whereas with other polymeric materials, it is almost impossible to decompose it off. Choice of such a natural polymer promotes sustainable development and can be contributed to overcome global environmental issues. Moreover large varieties of solvents are made available for cellulose acetate. Characterization of prepared membrane was tested in terms of flux, porosity, solute rejection and water content (Haan *et al.* 2018). In this research work, cellulose acetate membrane was prepared by setting different parameters (choice of solvent-non solvent pair, polymer concentration, nonsolvent bath and casting solution temperature, evaporation time, etc.) as optimized conditions based on literature review. SEM results described morphological behavior (porous or nonporous) of 10% CA/Acetone and 11% CA/Acetone membrane. Both the membranes were prepared via phase inversion technique at optimized conditions and tested with water in nanofiltration pilot plant. This research will help to decide/fix optimized parameters at which membrane is capable of giving best performance in terms of solute rejection and flux.

2. Materials and methods

2.1 Chemicals

Cellulose acetate (CA) was selected as a membrane material was procured from Chemdyes Corporation Rajkot, Gujarat. Extrapure acetic acid was used as a solvent and distilled water as a nonsolvent.

2.2 Preparation method

Membrane preparation by nonsolvent induced phase inversion consists of three steps first : preparation of casting/ polymeric solutioncontaining at least one polymer and one or more solvent having high solubility for polymer. This casting solution may contain additives. Second: casting of homogenous liquid polymeric solution over suitable support with the help of film applicator. Third: polymeric film casted over suitable support then immerse in nonsolvent coagulation bath where solidification occurs because of diffusion between nonsolvent and solvent resulting in either porous or nonporous (Guillen *et al.* 2011). Preparation of homogenous polymeric casting solution involves mixing of cellulose acetate (concentration ranging between 10-15 weight %) in acetic acid under continuous stirring by using magnetic stirrer for at least 4-5 hours until solution becomes homogenous showing complete dissolution. After complete dissolution polymer solution is left without stirring until no bubbles appeared. After complete dissolution, polymer solution is left without stirring until no bubbles appeared. Glass plate/non woven polyeser fabric was selected as a suitable support on which this polymeric solution was casted by means of automatic film applicator or casting knife with predefined film thickness (Mulder 1996). Polymeric film over glass plate/ non woven polyester is then immersed in a coagulation bath containing distilled water where solidification takes place resulting into formation of porous/nonporous membrane. This prepared membrane CA/Acetic acid/ Water is washed and dried and stored in distilled water for at least 24 hr before testing (Phale and Chendake 2013). Properties of prepared membrane is influenced by different parameters such as polymer and solvent types and their concentration/ composition, casting solution temperature, non-solvent coagulation bath temperature and composition, evaporation time etc (Tiron *et al.* 2017). During the preparation of membrane via phase inversion, there are three possible membrane structure depending upon the choice of ternary system (Polymer/Solvent/Nonsolvent): porous film, nonporous film, dense top layer on porous supporting layer (El-Gendi *et al.* 2012).

(a) 10% CA/acetic acid over nonwoven polyester fabric

(b) 11% CA/acetic acid over nonwoven polyester fabric

Fig. 1 CA membrane prepared by phase inversion method

2.3 Characterization of membrane

2.3.1 Water content and porosity

Water content of prepared membrane can be determined by the weight difference between wet and dry membranes. To do so a piece of membrane is placed in water for 24 hours and weighed. This wet membrane is then quickly placed between two sheets of filter paper for removal of additional water and weighed. After that it is dried in an oven at a fix temperature for 24 hours and again weighed.

Table 1 Membranes casted at different casting conditions

Membrane Code	Polymer concentration (wt%)	Casting solution temperature (°C)	CBT (°C)	Precipitation time (sec)	Formation Mechanism	Surface Morphology
CA10a	10	24	24	93	Delayed Demixing	Even Surface
CA10b	10	42	50	45	Instantaneous Demixing	Uneven Surface (Due to high CBT)
CA11c	11	24	24	97	Delayed Demixing	Even Surface
CA11d	11	43	50	80	Instantaneous Demixing	Uneven Surface (Due to high CBT)

By using following formula water content can easily be calculated.

$$\% \, water \ content = \frac{w_w - w_d}{w_w} \times 100\% \qquad (1)$$

where, w_w = weight of wet membrane and w_d= weight of dried membrane

$$\text{Porosity} = \frac{w_w - w_d}{\rho_f v_m} \times 100 \qquad (2)$$

where, ρ_f is density of water (g/cm³) and v_m is membrane small size volume.

Membrane mean pore radius size (r_m) is determined by Guerout-Elford-Ferry equation on the basis of pure water flux and porosity (Bagheripour *et al.* 2016).

$$r_m = \sqrt{\frac{(2.9 - 1.75 \, porosity) 8 \mu Q}{porosity * A * \Delta p}} \qquad (3)$$

where μ = Viscosity of water

Q = Volumetric flowrate of the permeated water (m^3/s)

Δp = Operating pressure.

2.3.2 Flux and rejection

Prepared membrane was tested to determine its filtration performance in terms of % solute rejection and permeation flux. Cell size membrane was placed into cell with the upper surface contacting feed solution. Pure water permeability flux (PWP) is found at different pressure by passing distilled water through the membrane to be tested. Then aqueous salt solution is used as a feed for testing to calculate %rejection Zinadini and Gholami (2016).

$$\text{Porosity} = \frac{w_w - w_d}{\rho_f v_m} \times 100 \qquad (2)$$

where, J_w= Permeation flux in L/m² h, Q = Volume of permeated water in L, Δt = Sampling time in h, A = Membrane area in m².

$$\% \, rejection = 1 - \left(\frac{c_p}{c_f}\right) \qquad (5)$$

where c_p is the concentration of salt in permeate and c_f is the concentration of feed respectively.

3. Results and discussion

Present research work has been greatly emphasis on the preparation of micro porous membrane at identified optimized parameter (composition of casting solution and coagulation bath/choice of solvent-nonsolvent system/ evaporation time/formation and coagulation bath temperature) and its characterization of membrane in terms of flux and rejection. With trial and error, efforts were made to identify optimum condition for CA/acetic acid membrane preparation.

3.1 Polymer/solvent/nonsolvent system selection

Selection of polymer/solvent/nonsolvent system is a critical factor as it can limit membrane preparation, with the aspects of sustainability CA (organic polymer) chosen for membrane preparation in comparison of other polymeric materials like PVDF, PSF, etc. (Hazrati *et al* 2020). Low solubility parameter difference should be considered while choosing solvent for previously selected low molecular weight polymer (Guillen *et al.* 2011) and (Buonomenna *et al.* 2007). Cellulose acetate has an excellent compatibility with dioxane, but hands on practice with acetic acid/acetone and its easy availability, acetone/acetic acid was chosen as a solvent for cellulose acetate and distilled water as a nonsolvent.

3.2 Effect of Polymer concentration on membrane formation mechanism

From Table 1, it was been observed that the lower polymer concentration results in the lower precipitation time promoting instantaneous liquid-liquid demixing thereby yielding porous membrane matches with findings of (Alihemati *et al*. 2020). Such results matched with (Hwang *et al.* 1996) confirmed that at high polymer concentration, diffusional rate between solvent and nonsolvent was slowed down resulting in more precipitation time for phase separation. As an optimized polymer concentration was been selected as 10wt%.

3.3 Support material selection

Initially membranes were casted over glass plate with the thickness ranging between 50-150 µm. Problem encountered was a poor mechanical strength of prepared membranes. Thickness increment was a prime solution to improve mechanical strength. After casting membrane even at increased thickness ranging between 200-350 µm similar problem was again observed. Another key is to cast a membrane over another substrate rather than over a glass plate. Membrane casted over filter cloth was unable to create pressure drop as entire feed water passes through permeate line without generating retentate. Finally non-

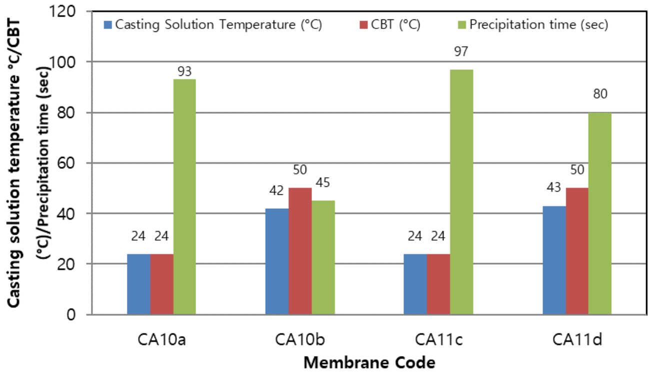


Fig. 2 Comparative analysis of different membrane at different casting condition

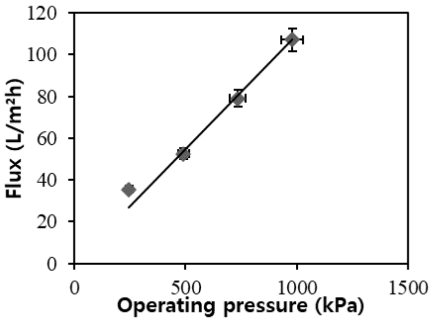
woven polyester support was used as a support material for casting and successfully pressure drop was generated while testing a membrane.

3.4 Effect of coagulation bath temperature/casting solution temperature and evaporation time on membrane morphology

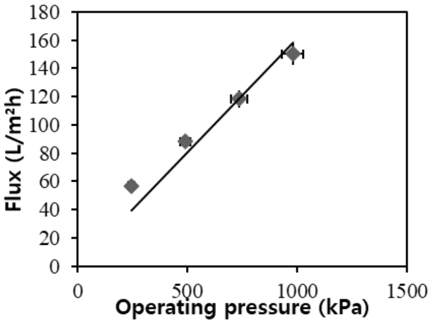
Four experiments were conducted for this parameter evaluation, illustrated in Table 1 with the membrane code CA10a, CA10b, CA11c, CA11d. In comparison of CA10a and CA11c membrane, precipitation time was quite higher than for the membrane CA10b and CA11d. This was because of increased casting temperature and CBT for CA10b and CA11d which exactly matches with findings of (Wang *et al*. 2019). Lesser precipitation time higher will be the mutual affinity between solvent and nonsolvent (Amirilargani *et al*. 2010). But at higher CBT uneven structured membrane was resulted. Viscosity of the casting solution was decreased when it is heated to high temperature therefore this lesser precipitation time is due to decreased viscosity, too. Zero evaporation time is favourable in this research work. As an optimum condition it is convenient to cast a membrane with varying casting solution temperature as per surface morphology at ambient CBT and zero evaporation time for casting micro porous membrane. Because during evaporation of solvent from the solution/ air interface, solvent molecule was exchanged places with air molecules and air molecules will occupy the empty space left by solvent. Closure of space by air molecule getting larger with longer evaporation time and as a result flux decreased due to formation of membrane with smaller pore size (Kusworo *et al*. 2017). Fig 2 illustrates this comparative study based on Table 1.

3.5 % water content

Hydrophilicity/water content promotes membrane antifouling properties. Since CA10a/CA10b and CA11c/ CA11d had same polymer concentration, % water content was different (for CA10a, CA10b, CA11c, CA11d % water content is 66.67%, 98.90%, 59.40% and 94.08% respectively) may be due to varying casting condition. But this was not true in every situation as this result contradicts with (Bagheripour *et al*. 2016). As per his research he concluded that water content can be improved by surface modification. One cannot conclude that increased CBT and casting solution temperature solely caused in improved hydrophilicity.



(a) 10% CA/acetic acid over nonwoven polyester fabric



(b) 11% CA/acetic acid over nonwoven polyester fabric

Fig. 3 Plot of flux vs. operating Pressure

3.6 Flux and rejection

Membrane casted over non-woven polyester fabric) was analyzed to check it performance at various operating pressure. Plot of flux vs. operating pressure shown in Fig. 3 was a straight line with the slope providing permeability.

To find rejection by membrane, aqueous solution of salt is passed through it. Initial concentration and permeate salt solution concentration was measured by digital TDS meter. Then by using equation (5) %rejection can be found . % rejection for CA membrane having 10% and 11 % polymer concentration were 76% (permeability 0.1097 L/$m^2 h\,pa$) and 70% (permeability 0.1617 L/$m^2 h\,pa$) respectively while used for pasing salt solution through it. Conclusion was obtained from this performance analysis is that the permeability increases with increase in polymer concentration.

Performance of the prepared membrane was tested by pasing a dye soltution of reactive red dyre through it at different opetaing pressure. first the feed solution is prepared by mixing 12 gram of reactive red dye into 30 liter of water. This feed solution is allowed to pass through flat sheet membrane at different pressure ranging from 245 Kpa to 980 Kpa. Concentration of collected permeate was measured by spectrophotometry. It involves interpretation of Absorbance value into the concentration of permeate at given wavelength. It requires the calibration plot to read this unknown concentration value of a given permeate. Usually

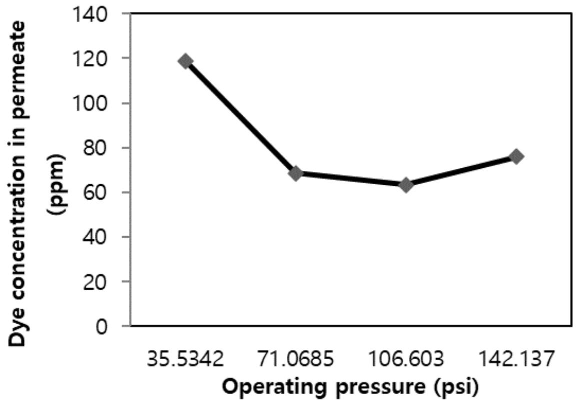
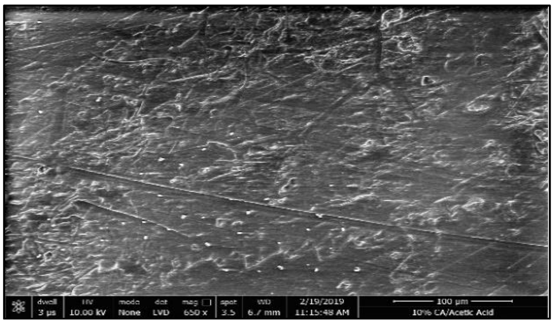
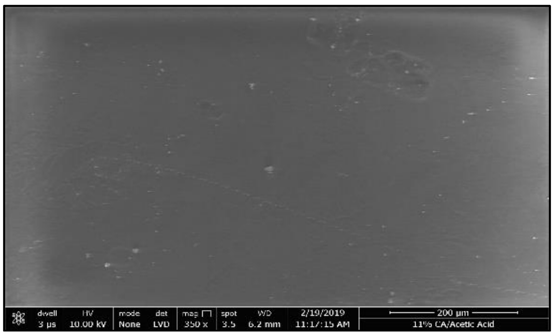


Fig. 4 Dye concentration in permeate Vs. operating pressure for membrane prepared at optimized condition (10 wr% CA, Ambient casting solution and CBT, Zero second evaporation time)



(a) SEM of CA10a (10wt% CA concentration)



(b) CA11c (11wt% CA concentration)

Fig. 5 Scanning electron microscopic analysis photographs

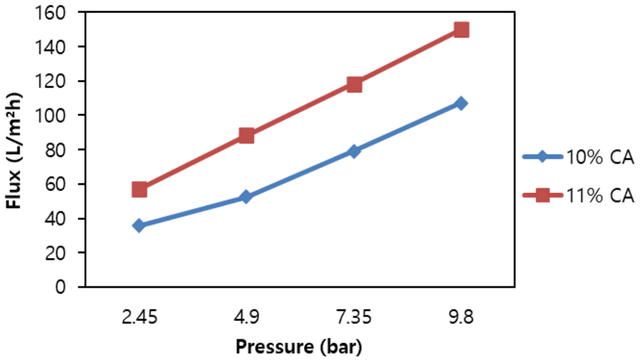


Fig. 6 Flux Vs. pressure for membrane of 10% CA & 11% CA (10 wr% CA & 11 wt% CA, Ambient casting solution and CBT, Zero second evaporation time)

the calibration chart is prepared by measuring absorbance value of known concentration and by plotting the graph of absorbance vs. concentration. As per beer's law this relationship between the absorbance and concentration is linear and one can easily find the unknown value of concentration with known absorbance from this chart. Below plot of Fig. 4 describes the change in dye concentration with operating pressure during performance analysis for reactive red dye feed solution.

3.7 SEM (scanning electron microscopic analysis)

Scanning electron microscope is use to observe morphology of prepared membrane. A narrow beam of highly energetic beam hits the membrane sample by means of electric gun. Pair of condenser lenses are used to focus the electron beam which are capable of bending electrons path. The incident electrons are known as high energy or primary electrons and electrons that get reflected are known to be low energy (secondary) electrons. Secondary electrons are not reflected but liberated from the atoms in the surface, determining image that can be seen on the screen. Sample is coated with the thin gold conducting layer to prevent burning and/or damage of sample membrane due to high intensity of energy. Upper surface of cross sectional morphology signifies the membrane skin layer and bottom layer cress section signifies the porous membrane containing microspores and macrovoids.

However aim is to prepare micro porous membrane at optimized condition, SEM photographs were taken to analyze surface morphology. Parameters (polymer concentration, casting solution temperature, CBT, evaporation time) affecting the membrane morphology were set first as shown in table 1 then CA10a and CA11c were examined. Fig. 4(a) represents surface morphology for membrane with 10wt% polymer concentration while Fig. 4(b) represents same for the 11wt% polymer concentration. For low CA concentration membrane was formed with uneven pores. In comparison with SEM photograph of CA10a, CA11c (11 wt% CA) shows dense homogenous surface layer.

4. Conclusions

Present research work is focused on the estimation of optimized parameter for casting membrane by most favorable method that can be able to give best performance in terms of flux and rejection for utilization in advanced waste water treatment. From this research work following conclusion has been made:

1. For casting polymeric membrane choice of the polymer depending upon its application is most important. Choose a low molecular weight polymer having wide range of solvent-nonsolvent system. Moreover, choice of solvent-nonsolvent pair is the second important criteria to be considered. Choose compatible solvent for previously selected polymer based on solubility parameter (low difference in solubility parameter). Study was carried out by choosing Cellulose acetate/acetic acid / water system. Phase inversion method by immersion precipitation, most largely

used commercialized technique was used as it provides wide variation in the morphology. It is concluded that mechanical strength to the membrane is improvised by selecting suitable support. Experiment conducted using glass plate and filter cloth wasn't able to bare operating pressure. Alternative is to use nonwoven polyester support.

2. After selection of materials, casting method and support material, different parameters (polymer concentration, casting solution temperature, coagulation bath temperature, evaporation time) were fixed as follows:

- Select the low polymer concentration ranging between 10-15 weight %.
- Additive should be selected based on required membrane morphology and selected polymer.
- No needs to be add any solvent in the coagulation that may increase complexity.
- Evaporation time is selected as zero second resulting in lesser precipitation time.
- Casting solution temperature should be kept normal to high and pick temperature that promotes instantaneous demixing.

3. Porous membrane has been obtained due to low polymer concentration, ambient CBT and casting solution temperature as it decreases the precipitation time resulting in the instantaneous demixing mechanism. Prepared cellulose acetate membrane is suitable for the desalination. It can be termed as a reclamation membrane for desalination can be able to reduce TDS level at desired value for reutilization to fulfill domestic demands. Cellulose acetate membrane has great antifouling properties and can be able to give the best performance while casting over nonwoven polyester rather than over filter cloth or filter cloth.

4. Flux and rejection are the main parameters to judge membrane performance. Cellulose acetate membrane with 10% (weight) concentration over polyester can give the similar type of performance with high flux values than the membranes were prepared and tested by Pinem, (2016) with polymer concentration 17%, 18%,19% (weight % polymer concentration).

5. CA membrane was also been tested/ checked by conducting pilot plant study with reactive red dye feed solution. Results obtained somewhat are matched with (Sutedja *et al*. 2017). As per (Sutedja *et al*. 2017), solute rejection for polysulfone membrane with surface modification is 88% with reactive red dye feed solution whereas Cellulose acetate membrane can be able to give solution rejection of 84.17% for the same feed system. With the surface modification this % solute rejection may be increased.

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Nomenclature

CA	Cellulose Acetate
CBT	Coagulation Bath Temperature
SEM	Scanning Electron Microscope
NIPS	Nonsolvent Induced Phase Separation
TDS	Total Dissolved Solids
PVDF	Polyvinylidene fluoride
PSF	Polysulfone