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Fabrication, characterization and permeation study of ultrafiltration dialysis membranes

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ABSTRACT

In this work, cellulose acetate-based flat sheet membranes were fabricated using PEG 400 and glycerol as an additive in various composition of doped solutions. The fabricated membranes were characterized using SEM, AFM, and FTIR analysis techniques. According to SEM, it was observed that the addition of PEG 400 wt% resulted in the formation of membranes with micro and nanopores and the distribution of pores was not uniform. To avoid heterogeneity in pore sizes, glycerol was added in the dope solution containing CA and PEG 400 (6.2 wt%). The addition of 10.1 wt% glycerol resulted in the formation of membrane with uniform distribution of pores. The permeation study of synthesized membranes was carried out using pure water, urea, and glucose solution which revealed that CA PEG-10.1 wt% glycerol-blended membrane (M8) is most sugar selective and selectivity up to 15.21 is documented.

Keywords: Cellulose acetate; Ultrafiltration membranes; Glucose; Water; Urea; Dialysis

1. Introduction

With the advancement in membrane technology, separation, concentration, and purification have become industrially workable unit operations due to its high efficiency of separation. Further, low energy of operation, spatial requirements, simplicity of operation using modern compact modules as well as recycling and reuse of chemicals and water promote membrane processes as a promising technique in separation processes [1]. Membrane being the important part of the process plays a key role in uttering the applicability and effectiveness of the process. Membranes are being increasingly used in laboratory as well as industries since five decades.

Dialysis had been familiarized since 1861 and frequently utilized as a laboratory technique for the purification of small quantities of solutes [2]. For the last 20 years, dialysis has been considered as replacement of kidney failure. Basically, it is lifesaving technique, as from the human body, the removal of excess water and waste is essential. Kidney is the main organ, which removes toxins such as urea, creatinine, uric acid, and other components from blood, in the form of urine. The same function is performed by hemodialysis membrane in the absence of natural kidney.

Dialysis membranes can be distinguished by their material of fabrication such as cellulose acetate (CA), polymethyl methacrylate (PMMA), polyacrylonitrile

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(PAN), polysulfone (PS), ethylene vinyl alcohol copolymer, and polyamide [3]. Previous research revealed that different dialysis membrane materials have different ability in terms of biocompatibility and performances efficiency. CA is the most commonly used material for making dialysis membranes. This is due to its excellent properties such as biocompatibility, good desalting, high flux, and relatively low cost [4]. The first formed CA membranes produced low flux and are much liable to bacteriological and chemical agents [5]. The performance of CA may be upgraded by blending it with suitable additives to fulfill new requirements and supplementary membrane properties. Additives, namely polyvinylpyrrolidone (PVP) [6,7], polyethylene glycol (PEG) [8], lithium chloride (LiCl) [9,10], ethanol [11], polystyrene sulfonic acid [12], water [13], ethylene glycol [14,15], glycerol, and phosphoric acid [16] have been commonly used. Polyethylene glycol (PEG) and polyvinylpyrrolidone (PVP) as an alternative to chemical modification has produced membranes with intensified hydrophilic property [17,18] and also the diffusive transport properties of solute through the ultrafiltration membrane. Latest research has shown that PEG additives in CA ultrafiltration membrane influenced the membrane characteristics in terms of pore size, permeate flux, and the protein rejection rates [19].

In this context, CA membranes were fabricated for ultrafiltration purpose. PEG (MW 400) and glycerol were incorporated as a plasticizer as well as pore-forming agents. This work has opened new ways for the facile structural modification in CA polymeric matrix using plasticizer and pore-forming agents. Phase inversion technique was followed to synthesize membranes. The fabricated membranes were characterized by SEM, FTIR, and AFM analysis techniques. The permeation study of water, urea, and sugar via these membranes resulted in interesting results, which are discussed here.

2. Experimental

2.1. Material

CA with the average molecular weight of 30,000 Da (Sigma–Aldrich) was used as the membrane-forming polymer. The solvent used was acetic acid (sigma aldrich) with analytical purity >98% and distilled water was used as non-solvent agent. Polyethylene glycol (PEG) 400 (Panreac) and Glycerol (Fisher Chemicals) were used as additives. Experiments were performed using urea with molecular weight of 60.02 obtained from Sigma–Aldrich and table sugar was used for testing synthesized membranes.

2.2. Fabrication of membranes

CA of 10% weight concentration was dissolved in mixtures of acetic acid and PEG of various ratios as shown in Table 1. The formulation compositions were used for the different PEG weight percentages. Other components of recipe were kept constant. Changing numerical values of components other than PEG shows adjustment for net 100% of doped solution. Membrane without polyethylene glycol as additives containing 10% CA was also casted so as to observe the effect of absence of PEG on the dialysis membrane performance and this membrane is named Mo. The solution temperature is maintained at 70˚C with continuous stirring for 6 h to ensure that the polymers are homogenized properly. When the entire polymer is completely dissolved, as indicated by the clear solution obtained, it was cooled down and poured into a storage bottle. The solution was subsequently degassed in an ultrasonic bath for about two hours to remove any air bubbles present and kept away from direct sunlight so as to slow down its aging process.

Another set of experiment was also run by keeping PEG constant and varying the weight percentage of Glycerol Table 2. This was done to study the effect of glycerol presence on PEG-modified CA membranes.

2.3. Membrane casting

The CA polymer solution was casted on a glass plate using a casting knife. The casted membrane was then immersed in water bath to complete the phase separation, where exchange between the solvent and water is induced. The Loeb and Souriajian or Phase separation (inversion) process is one of the most widespread methods used to produce porous polymeric membranes [20]. During the process a homogeneous polymer solution is casted as a thin film. The casted film is then immersed into a non-solvent coagulant bath. The diffusional exchange of solvent and non-solvent will occur through the interface between cast-

Table 1
Formulations of four different dope solutions

	Weight percentage %			
Element	M1	M2	M3	M4
Cellulose acetate	10.2	10.2	10.2	10.2
Acetic acid	76.1	74.4	72.9	71.5
Polyethylene glycol	*6.2*	*7.9*	*9.9*	*11.5*
Distill water	7.5	7.5	6.9	6.8
Net total	100	100	100	100

Note: The italic values shows the best composition with PEG that was selected and then used later to modify with PEG.

Table 2
Formulations of four different dope solutions keeping PEG constant

Element	Weight percentage %			
	M5	M6	M7	M8
Cellulose acetate	10.4	10.2	10.2	10.2
Acetic acid	72.5	71	70	69
Polyethylene glycol	6.2	6.2	6.2	6.2
Glycerol	*4.3*	*6.1*	*8.1*	*10.1*
Distill water	6.8	6.4	5.5	4.5
Net total	100	100	100	100

Note: The italic values represent the best membrane formed after modification with Glycerol.

ing solution and non-solvent, resulting in phase separation of casting solution to form a membrane with a symmetric or asymmetric structure [21,22].

After that the casted membrane was transferred to another container containing glycerol for post treatment to remove the excess acetic acid from the membrane [23]. In the final step, the membrane was placed in distilled water before permeation study.

2.4. Membrane testing cell

The permeation study of CA membranes, doped with PEG and glycerol was done using membrane testing rig as shown in Fig. 1. The permeability tests were performed using pure water, 10% urea, and 10% glucose. The membranes were cut in the circular diamerter of 3.78 cm (effective area of the membrane is 11.25 cm^2) and placed in the membrane cell. The membrane cell was having a circular porous membrane plate at which the membrane was placed. Vaccum was created using vaccum pump in the collection cell of apparatus to facilitate the flow. The known quantity of sample solutions (water, urea, and sugar solutions) were poured and the time was calculated using stopwatch. Using the quantity of sample solution and time taken, the flow rate of solutions was calculated accordingly.

3. Membrane characterization techniques

3.1. FTIR spectroscopy

FTIR spectroscopy (FTIR Spectrum 100 PerkinElmer, MID IR) was used to obtain the qualitative structural analysis of the pure and blended CA membranes (membranes having PEG and Glycerin) with 1 cm^{-1} resolution in transmission mode with wave numbers from 450 to 4,000 cm^{-1}. The small portion of the pure and blended membranes were cut in circular shapes and placed in a pallet holder. The holder was then mounted in an FTIR instrument (PerkinElmer). All of the spectra (subtracted from the background spectra) were recorded at room temperature.

3.2. SEM

SEM (JSM 6409A, JEOL, Japan) was used to get cross-sectional images of synthesized membranes after sputter coating with a thin gold film. The samples were attached on brass plates with double-sided tape.

3.3. AFM

The root mean square values of the surface roughness of the membranes were measured by AFM. The pure and blended membranes were heated to remove

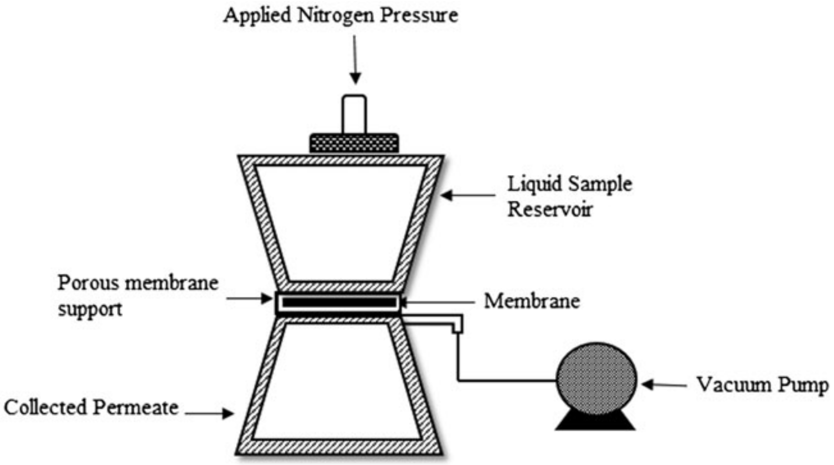


Fig. 1. Schematic diagram of membrane testing cell.

moisture and then mounted on slab with double-sided tape. The slab containing membranes were placed in a JSPM-5200 (Japan) to get the surface images.

4. Membrane permeability

The performance of the dialysis membrane is measured in terms of pure water flux and selectivity of synthesized membrane for 10% urea solution and 10% sugar solution in water. 10% Urea testing solution was prepared by adding 10 g of urea average molecular weight 60.02 obtained by Sigma–Aldrich) in 100 ml water. Similarly, 10% sugar solution was prepared by adding 10 gm table sugar in 100 ml distilled water. Flow rates of each sample solution was calculated using known volume of the solution and time taken by solution to get filtered by membrane. For every run, 20 ml of sample solution was used and the flow time was calculated. All the testing was done at room temperature and the pressure difference was maintained at 81.325 kPa.

Using these information permeate flux, J, can be calculated from its volume, V, collected during time, t.

$$J = V/A\,.\,t \tag{1}$$

where A represents the effective membrane area. All experiments have been repeated three times to ensure reproducibility of the results. Results tabulated were the average values with an error of ±3.5 to ±4.2%.

5. Results and discussion

5.1. FTIR analysis

Fourier transform infrared spectroscopy is one of the important techniques for qualitative analysis of polymeric membranes. The FTIR spectra of selected membranes are collectively shown in Fig. 2. The FTIR spectrum of Mo (pure CA membrane) [24] is compared with membranes with different wt% of PEG in casting solution (M1 and M3) and membranes with constant PEG and varying glycerin wt% in casting solution.

In spectrum of pure CA membrane Mo, the peak at 3,417 cm^{-1} is attributed to the stretching vibrations of carboxylic acid group (−COOH) [25]. At 1,790 cm^{-1}, the prominent peak represents carbonyl group (C=O). Another peak shows presence of alkyl group ($C–CH_3$) and at 1,235 cm^{-1} the peak is because of asymmetric stretching of ether group (C–O–C). In case of polyethylene glycol the characteristic peaks are found at 3,410, 2,869, and 1,100 cm^{-1} represent the –OH, $–CH_2$, and C–O–C, respectively. FTIR spectrum of membrane M1 and M3 the broadening of peaks in –OH region displays confirmation of interaction between CA and PEG.

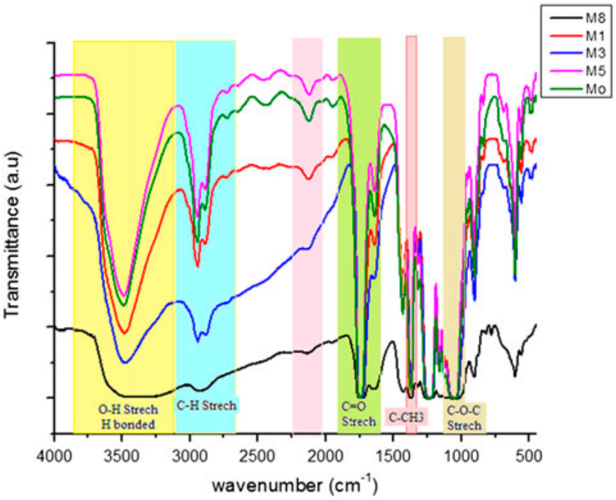
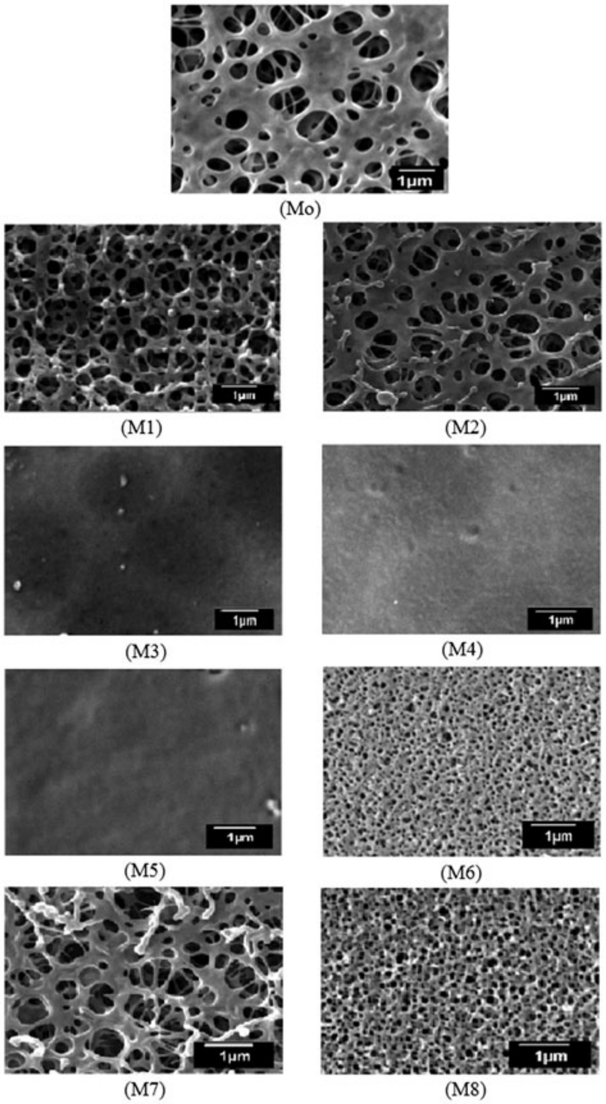


Fig. 2. FTIR spectra of selective synthesized membranes.

In case of glycerin added membranes M5 and M8, the presence of peak at 3,339 cm^{-1} shows –OH bond stretching and C–H bond vibration is shown at 2,935 cm^{-1}. The small peak at 2,879 cm^{-1} is because of alcoholic stretching in glycerol. The broadening of –OH peak and C–H stretching peak shows the perfect blending of PEG and glycerol with CA.

5.2. SEM analysis

SEM images were taken to explain the influence of amount of PEG and Glycerol in ultrafiltration dialysis membrane. Fig. 3 represents the SEM surface images of all fabricated membranes. In case of Mo (membrane produced without additives) a dense spongy structure with thick asymmetric skin is observed. The macro void formation is enhanced by the addition of hydrophilic additive PEG in dope solution as shown by the SEM images of M1, M2, M3, and M4. The membrane morphology changes from thick dense skin to porous asymmetric structure. Presence of small quantity of hydrophilic additive promotes instantaneous demixing which enhance pore formation and sugar flux of produced membranes. The hydrophilic additive PEG, also acting as non-solvent boost phase inversion mechanism from delayed demixing to instantaneous demixing resulting in the formation of pores in membranes structure. It is assumed here that PEG played an important role in changing the characteristics of CA in improving the permeability of dialysis membranes.



(Mo)

(M1) (M2)

(M3) (M4)

(M5) (M6)

(M7) (M8)

Fig. 3. SEM surface images showing the influence of PEG on M1, M2, M3, M4, and influence of combination of PEG and glycerin on the morphology of membranes M4, M6, M7, M8.

It is possible that the presence of low amount of PEG most likely caused speedy formation of nuclei having additive rich phase compared to diffusion of non-solvent into polymer solution so that nuclei with high solvent concentration are present and thus promotes pore or macrovoids formation [26]. Additive also serves to increase the membrane content of the synthesized membranes which results in enhanced degree of swelling. Former studies in ultrafiltration [27,28] showed that appropriate amount of non-solvent additives improves the formation of macrovoids and pores, while high volume of non-solvent suppressed their formation due to the delayed demixing in the growth stage. For the case of the dialysis membranes produced in this study, it is apparent that there is similarity such that pores were essential in supporting the solute clearance in dialysis process.

In addition, increasing the amount of additive increases the viscosity of dope solution and results in difficulty in casting the membrane as well as drastic decrease in flux of produced membrane. Lowering the quantity of hydrophilic additive causes the surface layer's polymer particles to swell in vertical direction which produces space between particles thus generating pores and in case of high amounts of additive the particles will be overlapped and will cause swelling in horizontal direction resulting in the production of dense membranes with low flux. Hence we can say that up to certain limit, the CA membranes with PEG will give better flux for water and sugar clearance.

In case of membranes M5, M6, M7, and M8 with the addition of glycerol in dope solutions results in uniform distribution of nano-sized pores and also resulted in better appearance. The uniform pore distribution is an essential parameter for constant flux. Although rest of the membranes have nano-sized pores but in case of M8 the pores were uniformly distributed that bring about high sugar clearance rate and reasonable flux measurement.

5.3. Atomic force microscopy

AFM is a well-recognized technique to analyze surface topography of membranes. Therefore, the pure and modified CA membranes were examined under AFM in tapping mode. Three-dimensional AFM images of the top surfaces of all membranes with scanning area of (10 × 10 µm) are presented in Fig. 4. The light regions in AFM images correspond to height, whereas dark regions denote depression [29,30].

From results, it can be inferred that pure CA membrane has smooth surface. However incorporation of PEG at varying concentration somehow increases surface roughness with respect to pure CA membrane [24,25]. The obvious interpretation for this behavior is the formation of micro and nanopores in membranes, which generates somewhat heighted features. In the membranes M5–M8, the glycerol was included to control the pore size in membranes and the roughness of membranes was more in M5. However, a decreasing trend in surface roughness was observed in M6, M7, and M8 by increasing the concentration of glycerol. This was probably due to a reason that at (max) 10.1 wt% glycerol, the uniformity in nanopore size was observed in membrane (M8).

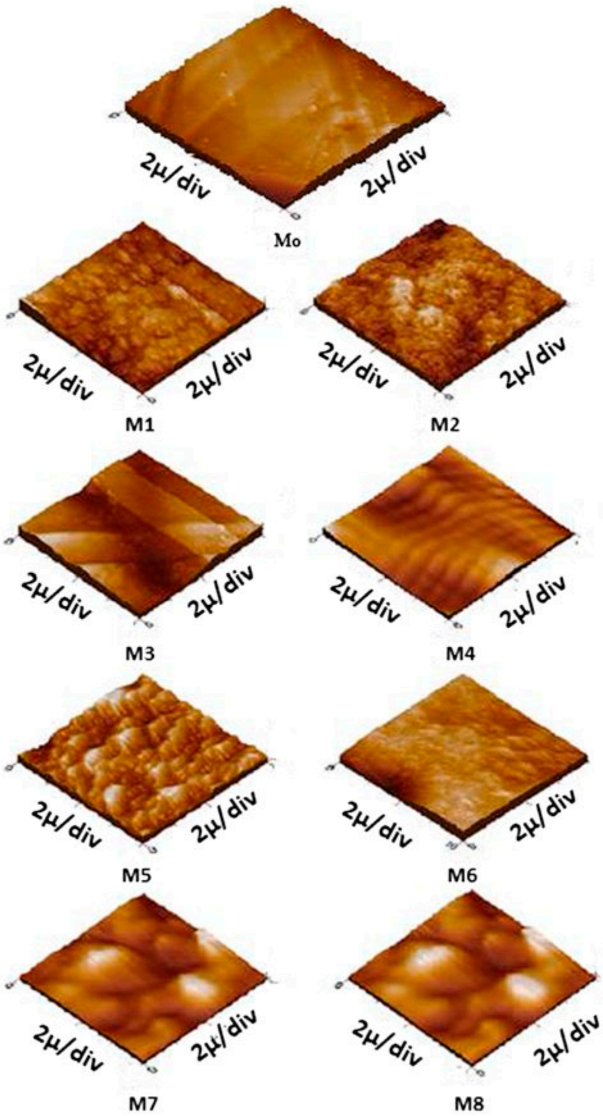


Fig. 4. AFM scans of fabricated membranes.

5.4. Membrane performance

The CA blended with PEG and glycerol was used to study the permeability of pure water, 10% urea, and 10% glucose. The membranes were cut in the circular diameter of 3.58 cm and placed in the membrane cell. The membrane cell was having a circular porous membrane plate at which the membrane was placed. Vaccum was created using vaccum pump in the collection cell of apparatus to facilitate the flow. The known quantity of sample solutions (water, urea, and sugar solution) was poured and the time was calculated using stopwatch. Using the quantity of sample solution and time taken, we calculated the flow rate of solutions accordingly. The results for the flux and selectivity of all eight membranes are tabulated below.

In Table 3, it is illustrated that the membranes were synthesized using CA polymer having hydrophilic additives like PEG and glycerol. Addition of hydrophilic additive like PEG effected the properties of fabricated membrane and hence the characteristics changed accordingly [26–28,31]. The results showed that M1 having 6.2% PEG is showing good sugar selectivity i.e. 11.5%, however the addition of glycerol in the fabrication recipe increased the sugar selectivity up to 15%.

In case of first set of membranes i.e. membranes having PEG hydrophilic additive in the dope solution resulted in membranes with small pores ranging in micro and nanosize range. According to Fig. 5 the fluxes of water, urea, and sugar via M1, M2, M3, and M4 showed same trend. The fluxes of water and urea are high compared to sugar solution. However, the membrane M1 has revealed interesting results. In this membrane water flux was much higher as compared to sugar and urea fluxes. The non-uniform pore distribution of pores resulted in the blocking of nano-sized pores and the micro pores cannot stop the flow of sugar molecules across the membrane and hence the membrane offered less hindrance or resistance to the flow and resulted in low permeability of sugar. Also the amount of hydrophilic additive effected the formation and distribution of pore. Increasing the weight percentage of additive resulted in larger pore diameter and hence high permeability which is against the requirement. M1 showed good pore diameter and distribution hence resulted in better sugar permeability as compared to other three membranes.

The performance and characteristics of M1 was further enhanced by adding glycerol in the dope solution. The addition was made gradually and at 10.1 wt% addition of glycerol, the membranes were formed with uniform distribution of nano-sized pores. Fig. 6 presents similar trend of water, urea, and sugar solution as represented by set 1. However, M8 shows lowest sugar flux in comparison to water and urea solution fluxes, which means that M8 resists the flow of sugar molecules through CA-modified membrane and thus reduces the concentration of sugar molecules in the filtrate.

Fig. 7 shows that M8 membrane is with highest sugar selectivity 15.21 in correlation to that of water. The addition of glycerol to the PEG/CA dope solution resulted in the formation of non-porous membrane which hinders the passage of sugar molecules. The molecular weight cut-off for nanofiltration is 200–1,000 Da, whereas the molecular weight of sugar molecule is 342.2965 Da. The separation in case of nanoporous membranes is because of size difference therefore the sugar molecules cannot pass through the non-porous membranes.

Table 3
Flux and selectivity results of synthesized membranes

Fabricated membranes	Pure water flux (L/h m^2)	10% urea flux (L/h m^2)	10% sugar flux (L/h m^2)	Sugar selectivity
M1	639.91 ± 3.9	278.22 ± 3.7	55.46 ± 4.6	11.5
M2	254.93 ± 4.7	246.13 ± 3.4	135.29 ± 4.8	1.19
M3	941.6 ± 4.2	901.5 ± 4.6	771.38 ± 3.9	0.12
M4	367.82 ± 4.3	329.86 ± 4.7	227.73 ± 4.0	1.62
M5	743.82 ± 4.3	522.6 ± 4.0	132.3 ± 4.7	5.62
M6	1,280.8 ± 4.1	1,142.2 ± 4.4	666.67 ± 4.3	1.92
M7	772.89 ± 4.5	694.4 ± 3.9	260.6 ± 4.2	2.96
M8	693.86 ± 3.7	584.8 ± 3.8	45.6 ± 4.5	15.2

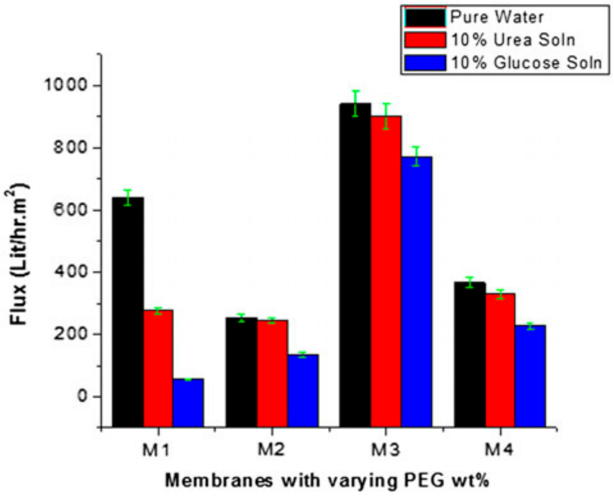


Fig. 5. Graph presenting fluxes of pure water, urea, and glucose solutions by first set of membranes.

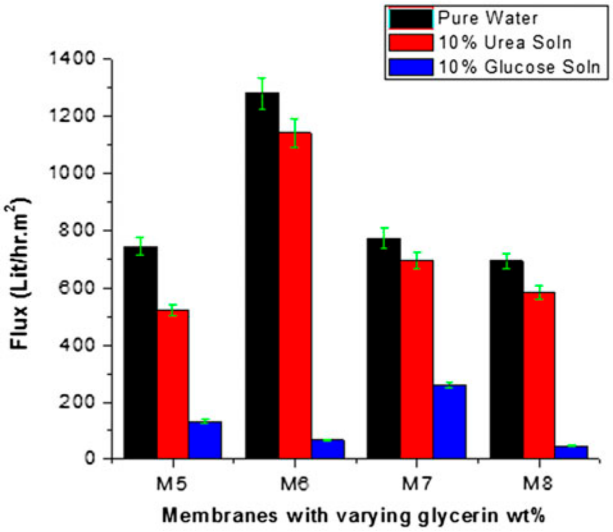


Fig. 6. Graph presenting fluxes of pure water, urea, and glucose solutions by second set of membranes.

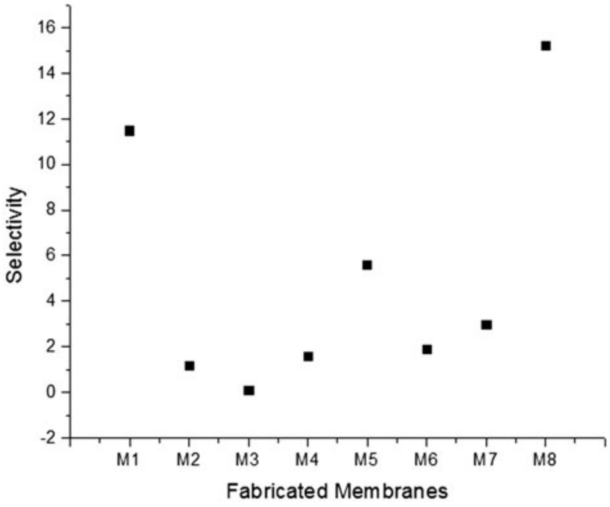


Fig. 7. Graph showing selectivity of fabricated membranes.

6. Conclusion

The present study elaborates the effect of PEG and glycerol on membrane morphology and permeation results of CA ultrafiltration membrane. The membrane performance in terms of glucose clearance was significantly influenced by the amount of glycerin in dope solution having PEG. Higher amount of glycerin up to 10.1 wt% in PEG dope solution strongly enhanced the urea clearance, while increasing the quantity of glycerin more than 10.1 wt% reduced the membrane performance. Combination of glycerin and PEG produced hemodialysis ultrafiltration membranes that can separate glucose better due to uniform nano-sized pore distribution on membrane surface.

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