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# Polysulfone-mesoporous metal oxide based tubular nanocomposite ultrafiltration membranes for turbidity removal of seawater

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**ABSTRACT**

In this study, ultrafiltration membranes are prepared in outside skin tubular configurations using polysulfone polymer with and without mesoporous silica and aluminium oxide powder (~3.8–4.0 nm pore size) independently. Prepared membranes are characterized in terms of pure water flux, separation of single uncharged solutes like polyethylene oxide (PEO), water contact angle and average surface roughness. After filtration of turbid seawater (50–60 NTU) to remove the turbidity, pure water flux recoveries of all the membranes were evaluated after cleaning by backwashing with deionized (DI) water. It was found that flux recovery is better in nanocomposite membranes than in purely polysulfone coated membranes. The extent of turbidity removal was studied as a function of turbidity load and temperature of seawater. Incorporation of the porous nanoparticles in pure polymer matrixes not only enhanced the water flux without sacrificing selectivity but it also increased the fouling resistance of the UF membranes. After 2 month operation, no performance deterioration was observed for all the tubular membranes.

*Keywords:* Nanocomposite; Tubular membrane; Ultrafiltration; Selectivity; Turbidity removal

## 1. Introduction

Ultrafiltration (UF) membranes are used for applications in separation of various components in food, dairy, paper, textile and chemical industries including pretreatment of seawater reverse osmosis in place of conventional pretreatments [1,2]. However, it is well known that UF membranes are prone to fouling with time and the extent of fouling depends not only on the chemical nature and surface morphology of the membrane surface but the membrane assembling configuration also [3]. Generally, regular backwashing and/or flow reversal water flushing are used for restoration of UF membrane performance after fouling [4]. Among assembling configurations, UF membranes are used more in capillary/hollow fiber and tubular configurations than the commonly used spirally wound modules. The tubular module is best suited for applications with a higher loading of foulants inspite being the less compact configuration. We have chosen tubular with outside skin configuration in our study as the membrane surface is most exposed for cleaning after fouling in turbid seawater. Development of fouling resistant UF membrane is also an important research area for minimization of frequent backwashing to restore the same flux. Several approaches like plasma and redox initiated graft-polymerization [5], surface coupling of polymers and biomolecules [6], additive segregation to form a polymer brush layer [7] etc. have been used for membrane surface modification to overcome membrane fouling. Nowadays incorporation of different types of nanoparticles like zeolite [8–12], silica [13–16], titanium oxide [17–19], silver [20–22], carbon nanotube [22–24] etc. into polymeric membranes has been adopted for better water permeability, anti-fouling property, thermal and mechanical stability.

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Nanocomposite UF membranes were developed mostly using metal and metal oxides nanoparticles as filler material in commonly used polymers like polysulfone, polyethersulfone, polyvinylidene fluoride etc. matrix and the latest status of the metal/ metal-oxide nanoparticle based nanocomposite membranes are reviewed by L.Y. Ng et al. [25] and M. Homayoonfal et al. [26]. The intrinsic membrane properties of nanocomposite membranes were found to vary with the type of nanoparticles and their size, chemical nature of the polymers, interaction of the nanoparticles with the components of the casting mixture etc.

This work presents effects of mesoporous silica and alumina nanoparticles on the overall membrane structure and performances of tubular UF membranes. The nanoparticles were incorporated in the PSf matrix in outside coating of porous high density polyethylene (HDPE) tubes. The membranes were characterized in terms of pure water flux, separation of PEO, water contact angle and average surface roughness. Then the membranes were tested for turbidity removal from seawater and thereafter the pure water flux recovery was evaluated after cleaning by forward flush as well as by backwashing independently with DI water.

## 2. Experimental

### *2.1. Materials*

Polysulfone (PSf) polymer is obtained from M/s. Solvay Specialities India Pvt. Ltd. N-methyl pyrrolidone (NMP) is received from M/s. Sisco Research Laboratory, India and used as such without further purification. Polyethylene oxide with molecular weight of 100,000 (PEO-100K), mesoporous-silica nanopoparticles (200 nm particle size, pore size ~4.0 nm) and aluminium oxide powder mesoporous (5.65 μm particle size, pore size ~3.8 nm) were procured from M/s. Sigma-Aldrich, India. Starting point for the 3 different hand-crafted membranes were HDPE tubes of 500 mm length with 20 mm inside and 30 mm outside diameter, supplied by M/s. Sonadka, Mumbai, India.

### *2.2. Preparation of tubular membranes*

In an airtight glass bottle, 150 g of polymer (PSf) was taken with 650 mL of NMP solvent and kept agitated for several hours for complete dissolution to form a casting solution. For mixed matrix nanocomposite membranes, 15 g (1% w of the polymer weight) of inorganic filler was dispersed in NMP first and then PSf was added. Then the central holes of HDPE tubes were blocked from both sides with rubber corks in order to avoid inside coating. Subsequently the tubes were dipped in the casting solution for 45 s. The outside polymer coated tubes were then taken out from the casting solution and positioned vertically to drain the excess solution until the dipping stopped. Then the tubes were immersed in demineralized water to induce phase inversion at room temperature. After gelling, the tubular membrane was washed thoroughly with demineralized water several times and finally stored in a laboratory refrigerator at 7°C. A pictorial scheme for preparation of the outside skin tubular UF membranes is given in Fig. 1.

Later the polymer coated tubes were put in 10.0% glycerol solution for 5 h and then they were air dried. As it is an outside-in configuration (membrane skin is outside), the membranes were tested for being leak-proof in a pressure vessel. For this purpose, end connectors were glued to both ends of the dried tubes.

### *2.3. Membrane characterization, performance evaluation and fouling studies*

Water contact angle on membrane surface indicates its hydrophilicity and was determined by measuring pure water contact angles on small pieces of membrane peeled from the tubes using the sessile drop method on a standard drop shape analysis system (DSA100, KRuSS GmbH, Germany). The average roughness of the membranes was measured using an atomic force microscope (AFM) (NT-MDT-Multimode 3, Ireland). Air-dried membrane samples peeled from the tubular membrane were fixed on a specimen holder and 10 μm × 10 μm areas are scanned by tapping mode in air.

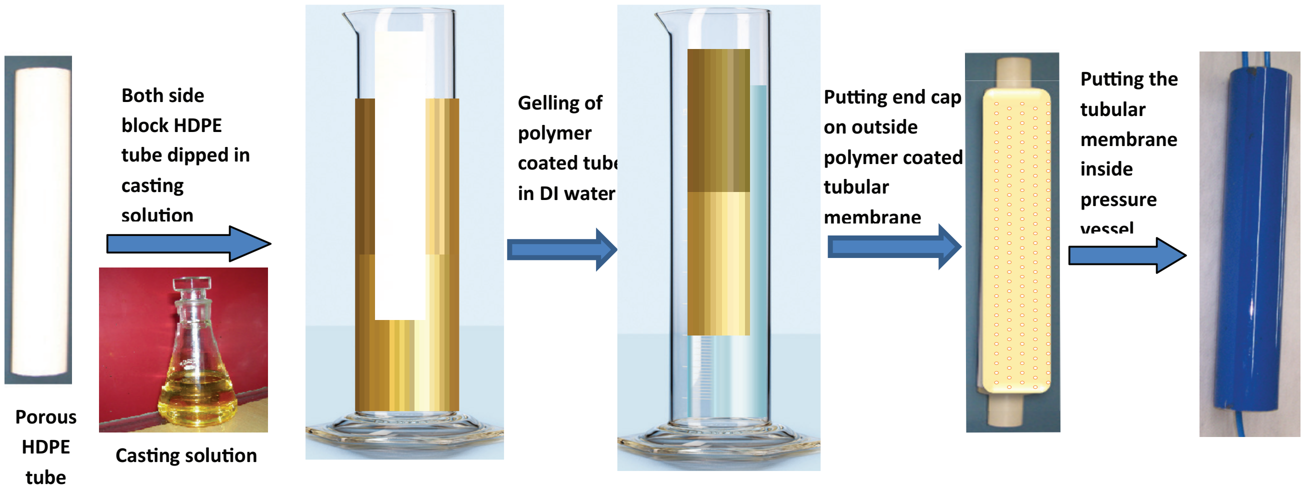

Fig. 1. Scheme for preparation of outside skin tubular UF membrane.

The performance of the outside-in tubular UF membranes was evaluated in a cross-flow filtration system (flow velocity ~2.0 m/s) after assembly in a pressure vessel. These membranes are initially pressurized with demineralized water at 450 kPa until a constant flux was reached. Subsequently, these pre-pressurized membranes are used for ultrafiltration experiments at 300 kPa. After taking pure water flux data, the feed was changed to 1 g/L PEO-100KDa aqueous solution to evaluate the rejection of single uncharged solute. The separation of PEO-100KDa is determined by measuring total organic carbon (TOC) of feed and permeates samples for all the membranes. Then the feed was changed to seawater and removal of naturally occurring turbidity by the membranes was evaluated. Turbidity removal rates were obtained by measuring the turbidity of feed and filtrate samples using standard turbiditimeter.

For fouling experiments, deionized (DI) water was first passed through the membrane at 300 kPa pressure until the flux remained constant over at least 30 min. The end of the stabilization period was taken as the zero time point in the filtration plots. Then the raw seawater was passed at constant flux using all the membranes. A sample of filtrate was collected after every 1 h of filtration. After 8–10 h of operation when the flux remained constant, DI water was used as a feed to determine the reversibility of fouling. A scheme for the performance evaluation and the cleaning processes (forward flush and backwashing) for outside skin tubular UF membrane is given in Fig. 2. Fouled membranes were cleaned by forward flushing for 1 h with cross flow velocity at 2.0 m/s i.e. exactly with the same velocity as used during regular product production. Similarly, the fouled membranes were cleaned by backwashing for 1 h using 100 kPa applied pressure i.e. one third of the pressure as used during regular product production. Fouling studies were carried out at constant initial flux (40.0 $L\cdot m^{2}\cdot h^{-1}$) for proper comparison of the fouling tendency of pure polymer membrane vs. generic nanocomposite membranes.

## 3. Results and discussions

### 3.1. Physico-chemical characterizations of tubular membranes

The pure water flux of the tubular membranes was evaluated at 300 kPa pressure on pre-pressurized membrane at 450 kPa for 3 h. Subsequently, pure water was changed to solutions of standard uncharged solutes PEO-100KDa and the separation data was collected when the product flux remained constant over 45 min. The polymer casting solution composition, the pure water flux, the PEO rejection data for both polysulfone and nanocomposite membranes, the membrane/water contact angle and the average surface roughness of the membranes are given in Table 1. The pure water flux of the polsulfone-silica and polsulfone-alumina based nanocomposite membrane was respectively 33.9% and 18.5% higher with almost the same rejection of PEO as the membrane with only PSf coating. From similar PEO rejection of all three membranes, it can be assumed that the pore sizes on the membrane surfaces for all the membranes are almost the same. The higher water flux in nanocomposite membranes could be due to the combined effects of increase in porosity (as nanoparticles are porous) and more hydrophilic membrane surface than only PSf coated membrane (as evident from the lower water contact angles). As expected, the nanocomposite membranes have rougher surfaces than the only PSf coated membranes and the roughness of nanocomposite membranes are more than double than that of the only PSf coated membrane.

### 3.2. Turbidity removal from seawater as a function of turbidity loading and temperature of seawater

Experiments were carried out for removal of turbidity from seawater using feed seawater of turbidity 60 nephelometric turbidity units (NTU) and the membrane performance is given in Table 2. It was found that all the membranes remove more than 99% turbidity from seawater with high turbidity but the flux decreased substantially in comparison

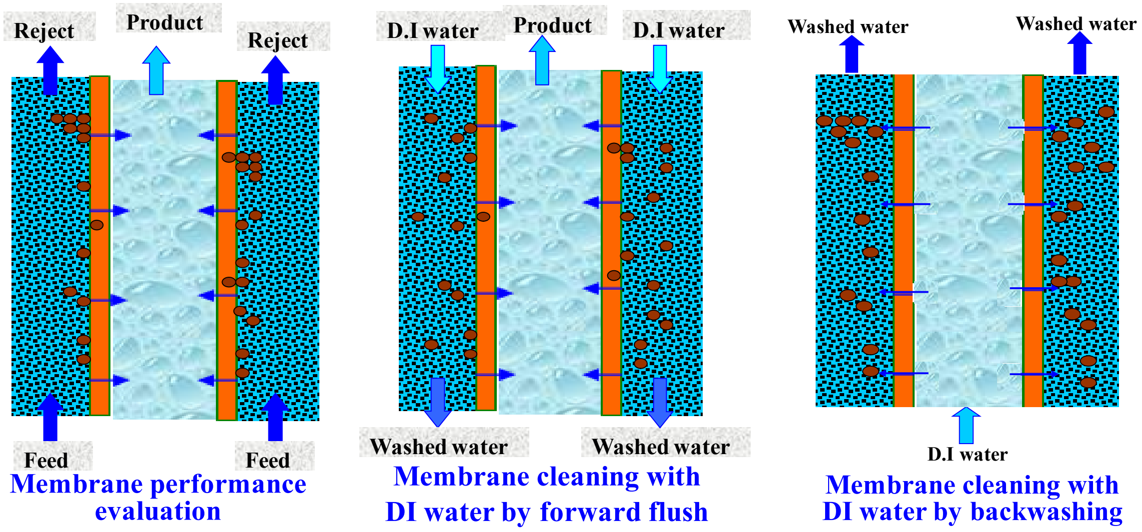

Fig. 2. Schemes for performance evaluation and cleaning process for outside skin tubular UF membrane.

Table 1
Casting solution composition, separation characteristics with water contact angle and roughness value of polysulfone and nanocomposite tubular UF membranes

| Membrane | Composition of casting solution | | | Pure water flux | PEO-100K rejection | Water contact angle | Average roughness |
|---|---|---|---|---|---|---|---|
| | Polymer (gm) | Solvent (ml) | Fillers (gm) | (L.m$^{-2}$·h$^{-1}$) | (%) | (°) | (nm) |
| PSf | 150 | 650 | 0 | 66.0 ± 6.3 | 91.8 ± 1.0 | 73.0 ± 1.0 | 10.7 ±1.6 |
| PSf-Silica | 150 | 650 | 15 | 88.4 ± 9.5 | 92.0 ± 1.3 | 67.5 ± 1.8 | 24.3 ± 4.0 |
| PSf-Alumina | 150 | 650 | 15 | 78.2 ± 8.2 | 91.5 ± 1.4 | 66.4 ± 2.1 | 29.6 ± 4.7 |

Table 2
Membrane performances on turbidity removal from seawater

| Membrane | Product flux (L.m$^{-2}$·h$^{-1}$) | Turbidity of the permeate (N.T.U) | % Turbidity removal |
|---|---|---|---|
| PSf | 28.2 ± 4.3 | 0.32 ± 0.03 | 99.47 ± 0.05 |
| PSf-Silica | 40.6 ± 5.6 | 0.29 ± 0.08 | 99.52 ± 0.13 |
| PSf-Alumina | 36.1 ± 5.1 | 0.33 ± 0.07 | 99.45 ± 0.12 |

Turbidity of the diluted seawater : 60 N.T.U., Temperature: 27°C

to pure water flux. This is due to the deposition of colloidal and particulate matters present in the turbid seawater on the membrane surface. Due to seasonal variations, temperature and turbidity loads in the raw seawater are changing. Figs. 3 and 4 show the turbidity removal efficiency of the differently coated membranes as a function of feed turbidity and temperature of the seawater. It was found that with increase in feed turbidity from 30 to 120 NTU, the turbidity removal efficiency decreased slightly for all the membrane but was still above 98% removal for 120 NTU (highest feed turbidity studied). The lowering of the turbidity removal could be due to increased solubility of the organic substances present in seawater with increase in temperature. Effects of temperature seasonal change on sludge characteristics and membrane fouling in a pilot-scale submerged membrane bioreactor (MBR) were studied by Z.Wang et al. [27] and the results showed that bound extracellular polymeric substances (EPS), polysaccharides, and proteins in the supernatants all increased with lowering of temperature. Similarly, in another study [28], it was shown that solubility was found to increase exponentially with temperature (°C) for a literature data set of 50 organic molecules in water. The nanocomposite membranes appear to be less susceptible to a performance decline in turbidity removal than the membrane with only PSf coating. Similarly, with increase in feed seawater temperature from 24–33°C, all the membranes show slightly lower removal of turbidity but still give a turbidity removal of 97.8% or more at highest temperature studied (33°C). In this case also, nanocomposite membranes show less performance decline in turbidity removal [0.75–0.85%] than the membrane with only PSf coating [1.5%].

### 3.3. Flux recovery of fouled membranes on water flushing

The seawater fouling studies are carried out at initial flux of 40 L·m$^2$·h$^{-1}$ for all the membranes. The plot of normalized flux (defined as ratio of instant seawater flux to water flux at the end of the compaction i.e. 40 L·m$^2$·h$^{-1}$) as a function of time of operation is shown in Fig. 5. The reversibility of fouling was determined by flushing the fouled membranes in reverse flow direction (reject to

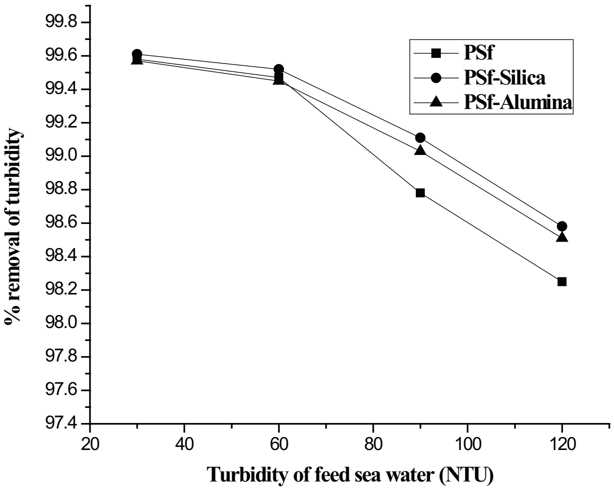

Fig. 3. Turbidity removal by ultrafiltration membranes as a function of feed turbidity of seawater.

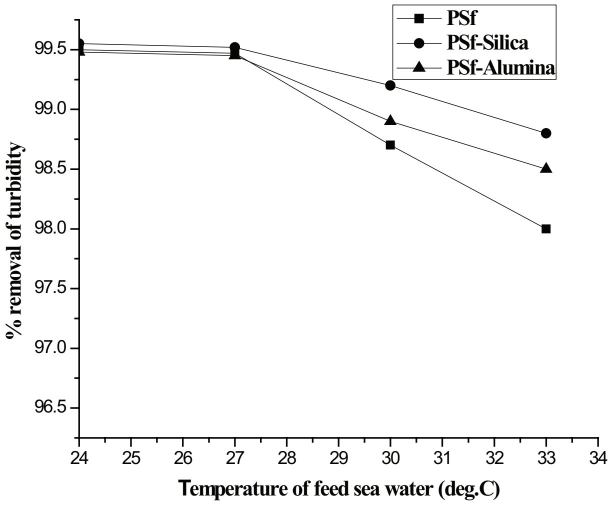

Fig. 4. Turbidity removal by ultrafiltration membranes as a function of temperature of seawater.

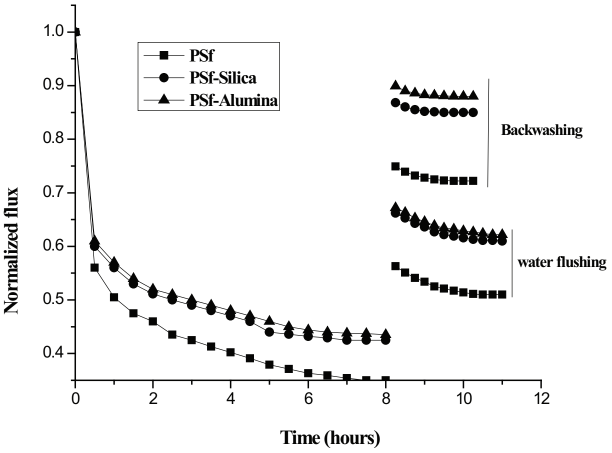

Fig. 5. Flux decline and recovery for only PSf coated and PSf based nanocomposite membranes for turbidity removal from seawater.

Table 3
Comparison of flux of the membranes after 1hr. of seawater filtration with flux after 15 min (0.25 h) of backwashing and water flushing of the fouled membranes

| Membrane | Product flux after 1 h operation in seawater (L·m$^2$·h$^{-1}$) | Water flux after 1 h of DI water flushing (flux after 9 h operation) (L·m$^2$·h$^{-1}$) | Water flux after 1 h of backwashing with DI water (flux after 9 h operation) (L·m$^2$·h$^{-1}$) |
|---|---|---|---|
| PSf | 20.2 | 21.36 | 29.12 |
| PSf-Silica | 22.4 | 25.44 | 34.08 |
| PSf-Alumina | 22.8 | 25.84 | 35.32 |

Starting flux = 40.0 L·m$^2$·h$^{-1}$

feed side) as well as by backwashing using DI water as a feed. The more hydrophobic PSf membrane shows more flux decline (end constant flux on seawater filtration) than the relatively hydrophilic nanocomposite membranes. PSf-silica and PSf-alumina based nanocomposite membranes recovered 61.0% and 62.2% of its initial flux respectively but PSf membranes recovered only 51.0% of initial pure water flux after flushing with DI water. But after backwashing, the PSf-silica and PSf-alumina based nanocomposite membranes recovered 85.0% and 88.0% of its initial flux respectively but PSf membranes recovered 72.2% of initial pure water flux. The results demonstrate that the hydrophobic PSf membrane is more prone to fouling than the nanocomposite membranes. A flux comparison for the membranes after 1 h of seawater filtration, after 15 min of backwashing and after 15 min of forward flush of the fouled membranes (reference to Fig. 5) is given in Table 3. Flux decline with time during seawater filtration of fresh membrane is much more than the flux decline during DI water filtration on washed/flushed membranes.

### *3.4. Studies on change in performances of tubular membranes with time*

In any nanocomposite membrane, the leaching of nanoparticles from polymer matrix may be an issue and hence performance testing of PSf coated and PSf based tubular nanocomposite membranes (PSf-silica and PSf-alumina) were carried out with time for turbidity removal from seawater. Experimental run was going on for more than 2 months (in 8 h/d and 5 working days in a week) with regular backwashing. Comparison of average performances in terms of flux and % turbidity removal for both PSf coated and PSf based tubular nanocomposite membranes are given in Fig. 6 with week-wise brake up for 8 weeks (2 months). For all the membranes, some extent of flux decline was found but no deterioration in rejection properties was observed. For PSf coated tubular membrane, the flux decreases to 23 L·m$^2$·h$^{-1}$ in 8th week from 30 L·m$^2$·h$^{-1}$ in 1st week with almost no change in % turbidity removal from seawater (99.4 ± 1%). For PSf-silica based nanocomposite tubular membranes, the flux was changes from 44 L·m$^2$·h$^{-1}$ in 1st week to 38 L·m$^2$·h$^{-1}$ in 8th week with 99.4 ± 1% turbidity removal from seawater. Similarly, for PSf-alumina based nanocomposite tubular membranes, the flux was changes from 36 L·m$^2$·h$^{-1}$ in 1st week to 31 L·m$^2$·h$^{-1}$ in 8th week with 99.3 ± 0.8% turbidity removal from seawater. It indirectly gives the indication on the stability of the membrane and no nanoparticle leaching takes place from nanocomposite membranes at the end of 2 months.

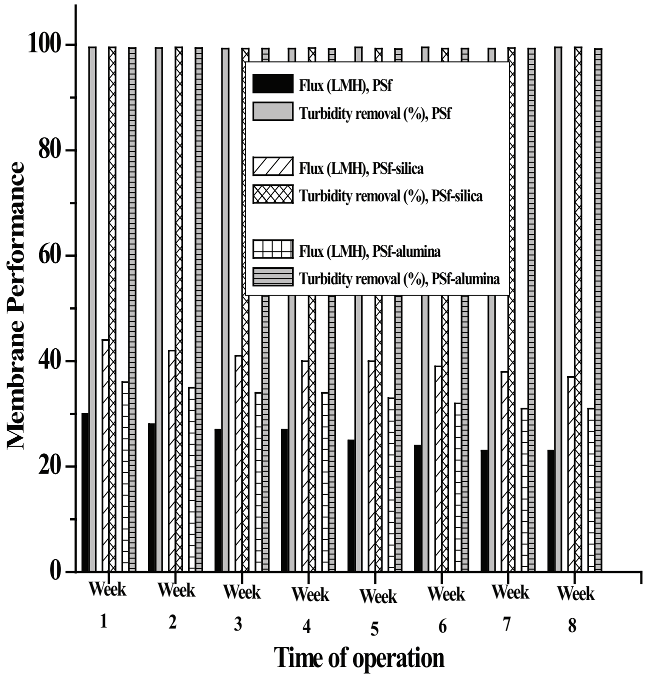

Fig. 6. Performance of PSf coated and PSf based tubular nanocomposite membranes with time for turbidity removal from seawater.

## 4. Conclusions

Silica and alumina based polysulfone nanocomposite ultrafiltration membranes are prepared successfully in tubular configurations for removal of turbidity from seawater. Nanocomposite membranes with porous silica and alumina nanoparticles give more water flux than the membranes with polysulfone coating. This study on turbidity removal efficiency as a function of feed turbidity and temperature of seawater shows that higher feed turbidity and temperature leads to slightly higher filtrate turbidity for all tested membranes. The extent of fouling after seawater filtration in relatively hydrophilic nanocomposite membranes is less than the more hydrophobic membranes with only polysulfone coating. The pure water flux recovery after fouling by both forward flushing with DI water and backwashing is better for nanocomposite membranes than for more hydrophobic membranes with only polysulfone coating. No performance deterioration was observed in 2 months of operation which indirectly gives the indication that no nanoparticles are leaching out with time.

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