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Fabrication, characterization, and performance study of acriflavine TFC nanofiltration membrane for brine concentration

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ABSTRACT

A novel thin-film composite (TFC) membrane is prepared by coating titanium dioxide (TiO_2) nanoparticle-incorporated acriflavine and trimesoyl chloride (TMC) on polysulfone (PSf) substrate with the aim of enhancing the permeation and salt rejection properties of PSf membrane. The membrane characterization was performed using Fourier transform infrared (FT-IR), atomic force microscope, field emission scanning electron microscope, and contact angle (CA) goniometer. The FT-IR spectral studies confirmed the interfacial polymerization of acriflavine and TMC. The study also confirmed the effective incorporation of TiO_2 nanoparticles, and the addition of TiO_2 nanoparticle in acriflavine TFC active layer improved the morphology of the membrane and CA. In terms of water flux, the TFC membrane with 0.1 wt.% TiO_2 nanoparticle content achieved water flux of 67.1 L $m^{-2}h^{-1}$ against deionized water and 53 and 44.5 L $m^{-2}h^{-1}$ against Arabian Gulf seawater and reverse osmosis brine, respectively, when tested using dead-end filtration. The acriflavine TFC membranes showed excellent antifouling characteristics when the loading of TiO_2 was 0.1 wt.% with >99% rejection for magnesium (Mg^{2+}), calcium (Ca^{2+}), and sulfate (SO_4^{2-}) ions. Overall, this study demonstrated the potential of using acriflavine as an amine monomer for the fabrication of polyamide layer with increased membrane performance toward seawater pretreatment and brine concentration applications.

Keywords: Thin-film nanocomposite; Acriflavine; TiO_2 nanoparticle; Interfacial polymerization; Seawater pretreatment

1. Introduction

Freshwater scarcity is a major challenge all over the world, and demand for fresh water is increasing continuously in proportion to the increasing population [1–3]. The membrane-based desalination technologies gained great interest among researchers and commercial desalination developers to produce ultrapure water with low energy and environmental-friendly conditions [4–6]. The membrane technology has a wide range of applications like seawater desalination, wastewater treatment, concentrating fruit juice, wine industries, organic isomer separation, degasification, separation, absorption of gas from atmosphere, etc. [7,8]. In the recent years, thin-film composite (TFC) membranes showed a promising way of producing fresh water at low energy consumption and high purity. The selectivity, low resistance, and high flux attracted researchers to search new effective thin-film coating for the TFC membrane fabrication process [9–11]. The microfiltration and ultrafiltration membranes are used as base polymer membranes for producing TFC membranes. The selectivity of ions in desalination process depends on the pore size, charge on the surface of membrane, and hydrophilicity [12–14]. The hydrophilicity of the membrane can be altered by incorporating nanoparticle and modification of selective layer in the thin film. Among the TFC membranes, the polyamide (PA)-type cross-linking

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coating has attracted due to its excellent pH stability, ion selectivity, and thin-film-forming capability [11,15,16]. The disadvantage of PA-based TFC membrane is its low chlorine tolerance, less chemical stability, and low antifouling behavior. The aforementioned drawbacks can be overcome by developing new PA cross-link polymer with chemically stable functional groups. Further, the modification of TFC membrane by incorporating nanoparticle showed a promising approach to overcome the chemical stability, chlorine tolerance, and antifouling characteristics [11,17,18].

Acriflavine is one of the 3,6-diamine with a quaternary amine at tenth position of the anthracene ring. The diamine at the end of the anthracene ring is more basic and highly reactive. The planar structure of the acriflavine is one of the advantages for cross-link polymerization and to develop a continuous defect-free thin film [19–21]. In the pharmaceutical field, acriflavine is generally used as an antiseptic and antibacterial agent in the drug development. In the aquarium hobby, it is used as an antifungal agent to treat various fungal infections in fish and preventing fish egg loss to fungus. The acriflavine family drugs induce cellular strain to fungus and reduce the further growth, and on the other hand, acriflavine derivatives are good antibacterial and antimalarial agents [22].

In the present study, a novel approach for the fabrication of PA layer is demonstrated using acriflavine as amine monomer and trimesoyl chloride (TMC) as acid monomer via interfacial polymerization (IP) technique. The acriflavine was selected to fabricate TFC with TMC monomer due to its antimicrobial property and the presence of quaternary amine in it. The TFC made by the acriflavine monomer retains the quaternary amine in the TFC polymer backbone which will increase the surface charge on the membrane. Due to this increased charge on the membrane surface, acriflavine TFC membrane is tested for desalination application in this study. Further, titanium dioxide (TiO_2) nanoparticle was incorporated in TFC layer to study the effect of nanoparticle on permeation and selectivity. The membranes were characterized in detail using instrumentation techniques such as Fourier transform infrared (FT-IR), atomic force microscope (AFM), field emission scanning electron microscope (FESEM), and contact angle (CA) goniometer. The performance of this novel TFC membrane was evaluated by performing laboratory-scale rejection and permeation studies using Arabian Gulf seawater (AGS) and brine discharge obtained from the reverse osmosis (RO) desalination plant.

2. Experimental

2.1. Materials

TiO_2 nanoparticles and polysulfone (PSf) with a molecular weight of 35,000 Da were purchased from Sigma-Aldrich (India). *N*-methyl-2-pyrrolidone was purchased from Merck Co., (Germany). The active monomers acriflavine and 1,3,5-benzenetricarbonyl trichloride or TMC were purchased from Alfa-Aesar (United Kingdom). The beach-well seawater and RO brine samples were collected from the Desalination Research Plant of Kuwait Institute for Scientific Research. All other laboratory grade reagents were purchased from Merck (Germany) and were distilled before use.

2.2. Preparation of acriflavine TFC membranes

Acriflavine TFC membranes were fabricated on the surface of precast PSf substrate by IP method. The 0.01, 0.05, and 0.1 weight percent (wt.%) of TiO_2 nanoparticles were dispersed in 2 wt.% of acriflavine aqueous solution with 1.1 equivalent of triethylamine (TEA). Clearly dispersed nanoparticle acriflavine solution was filtered to remove undissolved and agglomerated nanoparticles from the solution. The substrate membrane was clamped horizontally on the glass plate, and aqueous solution of nanoparticle-dispersed acriflavine solution was poured and kept for 2 min in order to penetrate the solution into pores of the substrate. The excess solution was drained off, and residual drops were removed by soft rubber roller. After 1 min, 0.1% (w/v) TMC solution in hexane was poured and kept for 1 min for IP to occur, and TMC solution was drained off. The unreacted TMC was removed by hexane washing and kept inside the oven at 60°C for 10 min to complete polymerization. The newly fabricated TFC membrane was washed with deionized (DI) water and stored in DI water [23]. Based on the concentration of TiO_2 used for fabricating TFC membranes, the prepared TFC membranes are labelled as TFC 1 (control: 0% TiO_2), TFC 2 (0.01% TiO_2), TFC 3 (0.05% TiO_2), and TFC 4 (0.1% TiO_2). The compositions of newly fabricated TFC membranes and CA are tabulated in Table 1. The chemical reaction involved in the formation of PA layer is shown in Fig. 1. The reaction mechanism involved in the formation of IP of acriflavine and TMC monomer in the presence of TEA base is shown in Fig. 2.

2.3. Characterization of TFC membranes

ALPHA-FT-IR spectrophotometer (Bruker Company, Germany) was used to characterize the chemical structure and confirm the incorporation of TiO_2 nanoparticle in the acriflavine TFC membranes. The CAs were measured using CA goniometer from KINO USA (model-SL200KB). Keysight 8500 FESEM (USA) was used to study the surface and cross-sectional morphology of the membranes. The membrane surface topological features were analyzed using Concept Scientific Instrument (Nano-Observer), France, by scanning the membrane surface over 10 μm × 10 μm dimensions.

2.4. Evaluation of membrane performance

The performance of TFC membranes was studied on dead-end membrane filtration cell (Sterlitech HP4750 STIRRED CELL, USA) having effective membrane diameter

Table 1
Composition of TFC solution and contact angle

SL No	Code	% composition			Contact angle (°)
		Acriflavine wt.%	Trimesoyl chloride wt.%	TiO_2 wt.%	
1	TFC 1	2	0.1	0	66.89
2	TFC 2	2	0.1	0.01	53.97
3	TFC 3	2	0.1	0.05	51.89
4	TFC 4	2	0.1	0.1	51.12

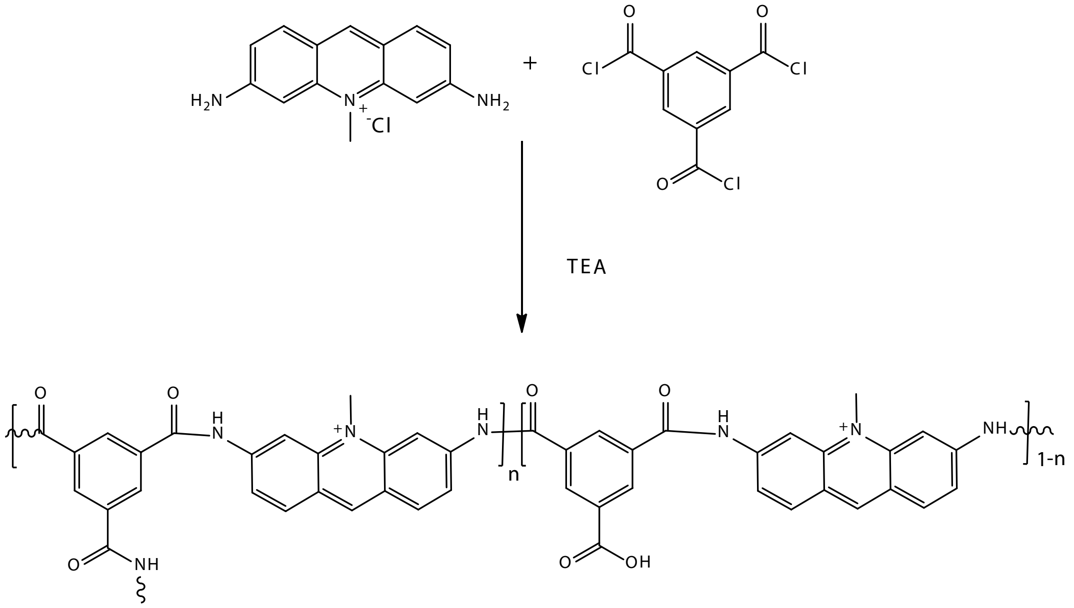

Fig. 1. The chemical reaction of acriflavine and trimesoyl chloride (TEA: triethylamine).

of 5 cm at 9 bar. The feed used for the experiments are DI water, AGS, and RO brine. The water flux (J_w) and salt rejection (R) of the TFC membranes were calculated using Eqs. (1) and (2), respectively.

$$J_w = \frac{Q}{A \times \Delta t} \tag{1}$$

where J_w (L m^{-2}h^{-1}) is the pure water flux (PWF) and Q is the volume of water (L) permeated through the membrane of effective area A (m^2) over a time Δt (h).

$$\%\text{Rejection} = \left(1 - \frac{C_p}{C_f}\right) \times 100 \tag{2}$$

where C_p and C_f are the concentrations of the feed and permeate. Each membrane was tested three times, and average values were reported.

3. Results and discussion

3.1. FT-IR analysis

The FT-IR spectra of acriflavine, PSf, TFC, and TiO_2 TFC are presented in Fig. 3. The sharp peaks at 1,457 and 1,601 cm^{-1} correspond to C=N and C=C of the acriflavine aromatic ring, respectively. Aromatic C–H and *N*-methyl ($N–CH_3$) stretching peaks have appeared at 2,913, 2,919, 2,954, and 3,030 cm^{-1} and free amine (NH_2) as a doublet at 3,315 and 3,431 cm^{-1}. In PSf spectrum, two peaks at 1,294–1,324 cm^{-1} correspond to O=S=O of the PSf and high intense peak at 1,586 cm^{-1} is due to C=C of the aromatic rings present in the PSf polymer. The aromatic C–H and CH_3 stretching peaks have appeared at 2,873, 2,969, and 3,038 cm^{-1} [24]. In neat TFC PA, the peak 1,445 cm^{-1} corresponds to C=O attached to amide link (C–N) and 1,602 cm^{-1} corresponds to the C=O of the carboxylic acid which is formed due to end of the polymer chain or end of polymer branching. The aromatic C–H and *N*-methyl ($N–CH_3$) stretching peaks have appeared at 2,987, 3,008, and 3,032 cm^{-1}. The N–H of the amide appeared as a singlet at 3,343 cm$^{-1,}$ and disappearance of NH_2 peaks at 3,315 and 3,431 cm^{-1} and absence of 1,770 cm^{-1} C–Cl peaks conform the formation of PA layer [25]. The spectrum TiO_2 TFC shows all the corresponding peaks of PSf, PA, and a broad peak of NH, and –OH (TiO_2) at 3,389 cm^{-1} confirms theTiO_2-incorporated acriflavine TFC.

3.2. Membrane morphology

The FESEM is the prominent technique to study the morphological variation of newly developed acriflavine TFC membrane [26,27]. Fig. 4 shows the surface and cross-sectional images of TFC 4 membrane. All the membranes exhibited rigid-valley structure, which is due to the IP of acriflavine amine and TMC [28]. The cross-sectional image clearly shows the formation of thin (~100–300 nm) PA layer on the PSf substrate membrane. In the cross section of the substrate membrane, the finger-type interconnected structure provides little resistance to permeate transportation and mechanical strength. On the top of the finger-type projection, a very thin distinctive sublayer of PA confirms the formation of active TFC layer which is responsible for the selectivity of ions as well as flux [29,30]. The increase of TiO_2 nanoparticle concentration in the TFC layer increased the porosity and decreased the pore size. The decrease of pore size with increase of TiO_2 nanoparticle concentration may be due to participation/interaction of TiO_2 in the IP resulting from the faster reaction between acriflavine and TMC monomers [31].

The surface analysis was carried out to study the topological feature and surface roughness of acriflavine TFC

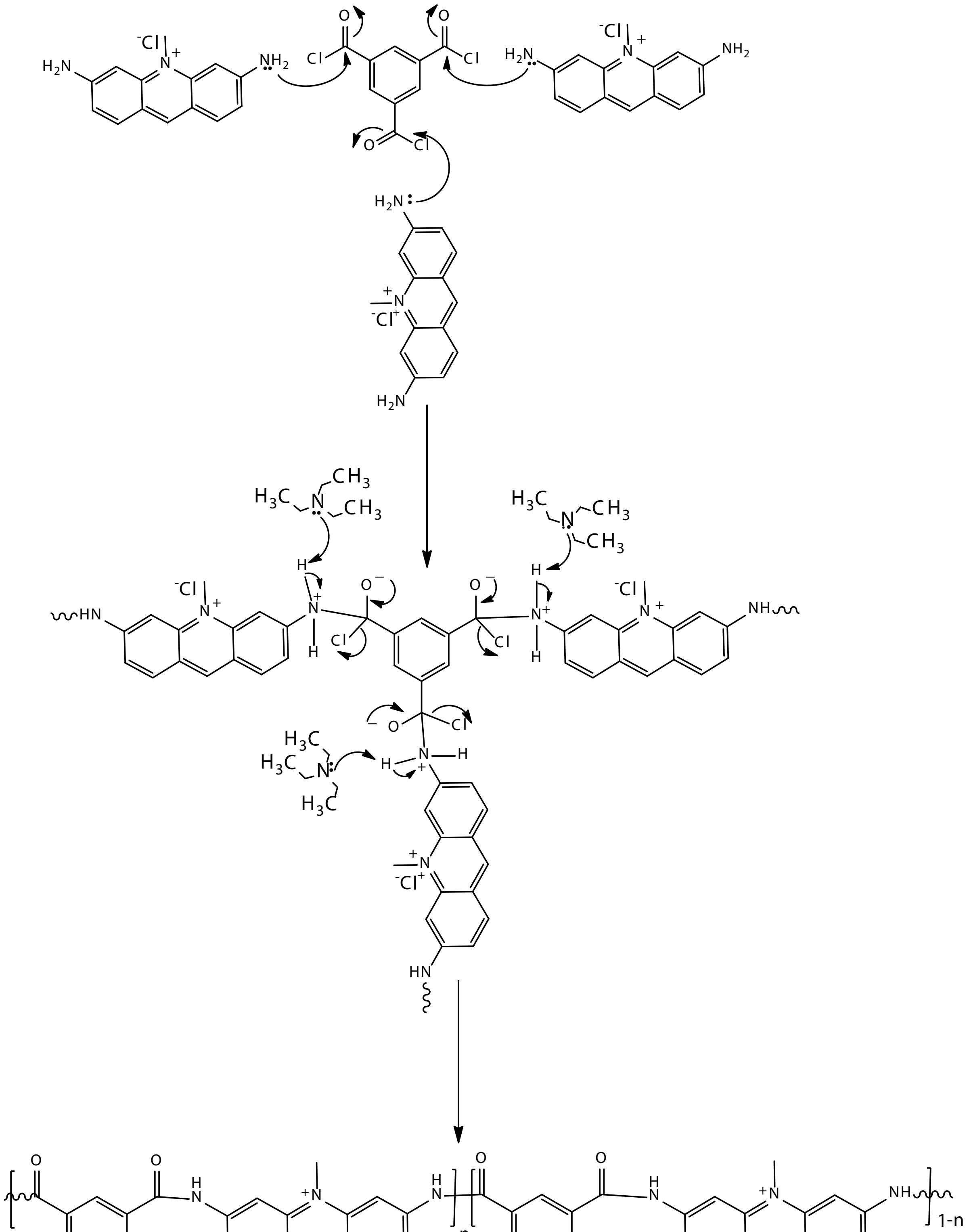

Fig. 2. The reaction mechanism of interfacial polymerization of acriflavine and trimesoyl chloride monomer.

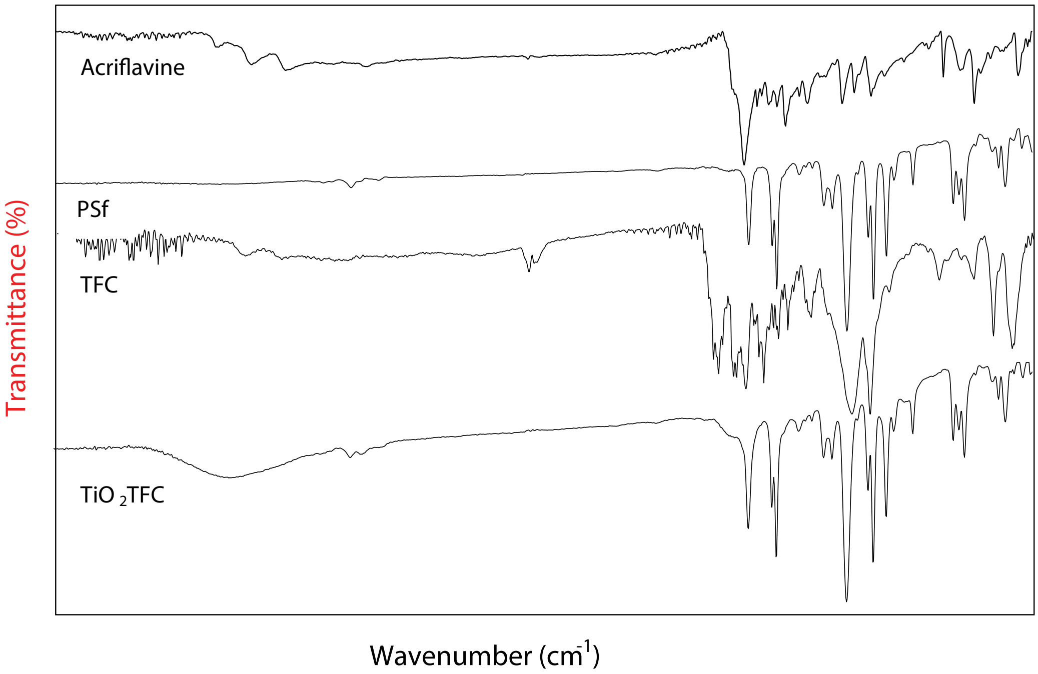

Fig. 3. The FT-IR spectra of acriflavine, PSf, TFC, and TiO_2 TFC.

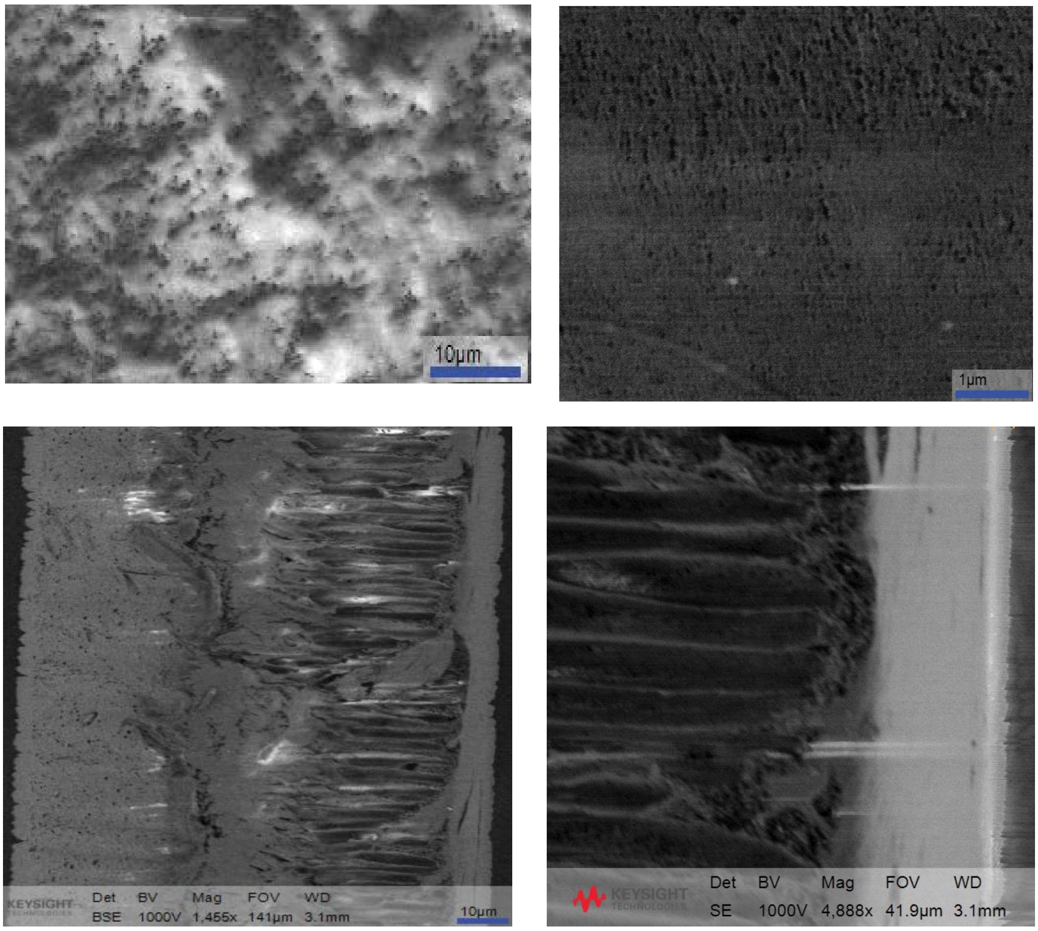

Fig. 4. The magnified surface, cross-sectional FESEM images of TFC 4.

membranes using Nano-Observer AFM instrument by scanning the membrane surface over 10 µm × 10 µm dimensions. The three-dimensional AFM images are shown in Fig. 5, and surface roughness parameters are presented in Table 2 as maximum mean roughness (R_a), route mean square roughness (R_q), and maximum feature height ($R_{\max}$). From the results, increasing trend of roughness values by increasing the TiO_2 concentration in the acriflavine TFC membrane was observed. The lowest maximum mean roughness (R_a) observed was 19.2 nm for acriflavine TFC membrane without any nanoparticle addition, and the highest was observed for TFC 4 (71.7 nm). It is interesting to note that the roughness of the membrane increases as the concentration of TiO_2 increases in acriflavine TFC. This may be due to participation/interaction of TiO_2 nanoparticles in the process of the IP reaction and also fast reaction rate between acriflavine and TMC in the presence of TiO_2 nanoparticles [31].

3.3. CA study

The membrane hydrophilicity is a very important parameter for high water flux and antifouling property. The change in hydrophilicity with the addition of TiO_2 nanoparticle was investigated by measuring CA. In general, lower CA of the membrane indicates more hydrophilic nature [32]. From Table 1, highest CA (66.89°) was observed for acriflavine neat TFC membrane compared with TiO_2 nanoparticle-incorporated membranes. The TFC 1 (neat TFC) having N–CH_3 group in the acriflavine aromatic ring may be the reason for the highest CA. The CA of the acriflavine TFC membrane was decreased with the loading of 0.01%–0.05% and 0.1% of TiO_2; this is due to the additional porous structure on PA and both chemical and structural properties of the membrane surface [33].

Table 2
The surface roughness parameters of membranes

Membrane	Roughness		
	R_a (nm)	R_q (nm)	$R_{\max}$ (nm)
TFC 1	19.2	24.5	163.3
TFC 2	37.1	47.4	170.1
TFC 3	69.9	86.2	324.5
TFC 4	71.7	104.9	593.6

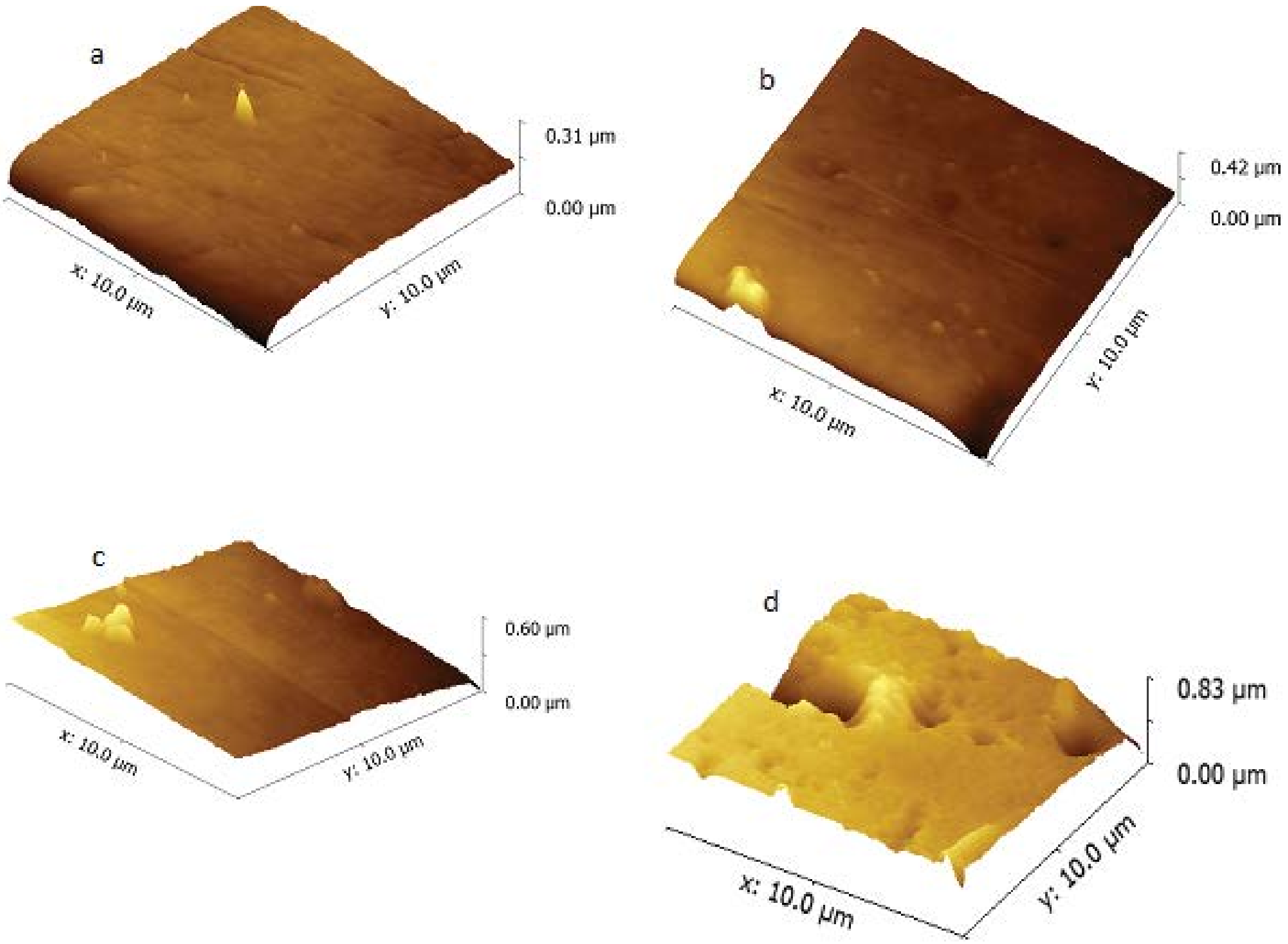

Fig. 5. The three-dimensional AFM images of (a) TFC 1, (b) TFC 2, (c) TFC 3, and (d) TFC 4.

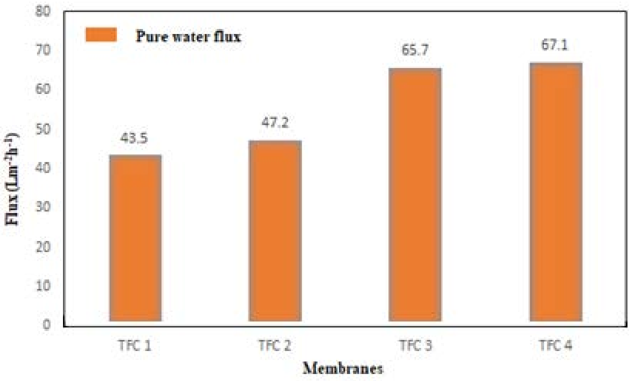

Fig. 6. The pure water flux of the membranes at 0.9 MPa pressure.

Table 3
Physiochemical analysis of AGS and RO brine

Parameters/unit	AGS	RO brine
Total dissolved solids, mg L^{-1}	45,377	54,900
Conductivity, mS cm^{-1}	58.3	69.4
Magnesium, mg L^{-1}	1,325	1,673
Calcium, mg L^{-1}	730	1,090
Boron, mg L^{-1}	3.7	9.8
Lithium, mg L^{-1}	1.21	1.7
Strontium, mg L^{-1}	14.6	121
Sodium, mg L^{-1}	14,488	17,905
Potassium, mg L^{-1}	316.4	997
Chloride, mg L^{-1}	24,876	68,593
Sulfate, mg L^{-1}	3,430.5	4,159

3.4. Membrane performance study

The desalination performance of the TFC membrane mainly depends on the structural properties of thin film and its chemical nature. However, nature of feed solution also plays a major role. Preliminary permeation test was conducted for all the membranes using DI water as feed. Fig. 6 shows that the acriflavine TFC membrane with 0.1% TiO_2 showed high PWF compared with other membranes.

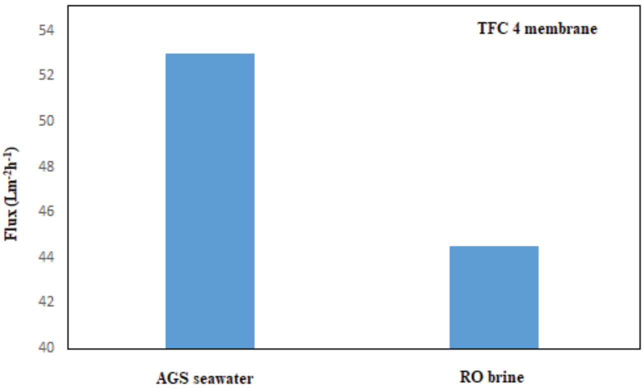

Fig. 7. The water flux of the TFC 4 membrane at 0.9 MPa pressure for AGS and RO brine feeds.

This increasing trend of PWF with increasing concentration of TiO_2 in acriflavine TFC is due to the additional porous structure of PA and both chemical and structural properties of the membrane surface. On the other hand, the charge on the PA layer due to the quaternary amine (positive charge in the acriflavine aromatic ring: $^{+}N–CH_3$) will attract the water molecules on the surface of the membrane and leads to enhanced permeation.

The actual seawater desalination study was conducted for TFC 4 membrane using AGS and RO brine as feeds. Table 3 shows the physicochemical analysis of AGS and RO brine. The desalination performance of the TFC 4 membrane for AGS and RO brine is shown in Fig. 7, and flux obtained were 53 and 44.5 L m^{-2}h^{-1}, respectively. The physicochemical parameters of permeate water and reject from the desalination of AGS and RO brine are shown in Table 4, and the percentage of salt rejection is presented in Fig. 8. The permeate flux for RO brine feed is less compared with AGS, and this may be due to the high salinity of RO brine. TFC 4 membrane showed significant high rejections for divalent ions compared with monovalent ions for AGS and RO brine feeds. The reason for highest rejection for divalent ions is due to the larger size and higher charge of ions [34]. The rejection percentages of ions are 88.2 (Mg^{2+}), 62.6 (Ca^{2+}), 99.8 (SO_4^{2-}), 39.8 (Cl^-), 31.8 (Na^+), and 30.9 (K^+) for AGS feed, and 99.5 (Mg^{2+}), 99.1 (Ca^{2+}), 99.8 (SO_4^{2-}), 68.3 (K^+), 18.6 (Cl^-), and 16.8 (Na^+) for RO brine

Table 4
Physicochemical parameters of permeate water and reject from the desalination of AGS and RO brine

Parameters/unit	AGS		RO brine	
	Acriflavine TFC membrane permeate	Acriflavine TFC membrane reject	Acriflavine TFC membrane permeate	Acriflavine TFC membrane reject
Conductivity, mS cm^{-1}	0.027	62.856	0.071	85.11
Magnesium, mg L^{-1}	5.8	2,039	197	3,013
Calcium, mg L^{-1}	6.0	1,006	407.2	1,507
Sodium, mg L^{-1}	12,051	13,316	12,051	30,520
Potassium, mg L^{-1}	100	340	688	1,223
Chloride, mg L^{-1}	20,230	26,086	41,176	68,603
Sulfate, mg L^{-1}	3.9	6,433	6.84	21,704

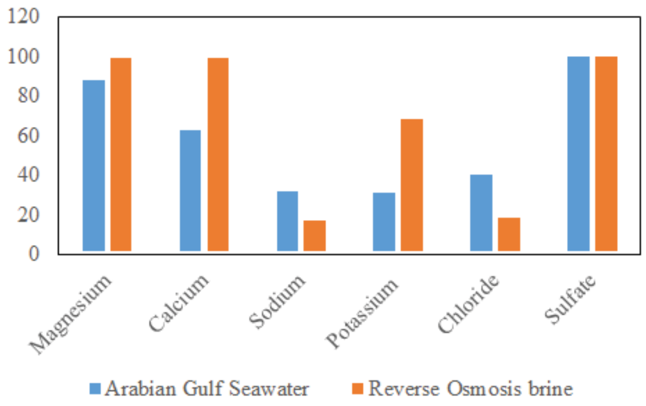

Fig. 8. Percentage salt rejection.

feed. TFC 4 membrane rejected 99% of Mg^{2+}, Ca^{2+}, and SO_4^{2-}. The salt rejection percentage results prove that acriflavine TFC membrane is highly suitable for seawater desalination pretreatment application and RO brine concentration. The long-term experimental (24 h test) test shows less fouling for TFC 4 membrane compared with TFC 1 membrane for RO brine as feed. The antifouling character of TFC 4 membrane may be due to neutralized charge distribution on the acriflavine TFC with TiO_2 nanoparticle on the surface of membrane and change in surface morphology of membrane. The TiO_2 nanoparticle played a major role in decreasing the pore size and increasing the hydrophilicity of acriflavine TFC membrane. The increased hydrophilicity of membrane enhanced the water permeation, and decreased pore size controlled the rejection of divalent ions.

4. Conclusion

The TiO_2 nanoparticle-incorporated acriflavine TFC membranes were fabricated on PSf support by IP method. The inclusion of TiO_2 nanoparticle into acriflavine TFC membrane aided as performance modifier which increased water flux and high salt rejection. The TFC 4 membrane showed CA of 51.12° and PWF of 67.1 L $m^{-2}h^{-1}$. The salt rejection experiments showed excellent rejection for divalent ions for AGS and RO brine feeds. The study concluded that TiO_2 nanoparticle-incorporated acriflavine TFC membranes have high capability of rejecting divalent ions and suitable for desalination pretreatment and RO brine concentration applications.

Acknowledgments

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