

# A NOVEL THIN FILM COMPOSITE REVERSE OSMOSIS MEMBRANE MODIFIED BY IONIC LIQUID

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**Abstract** - Thin Film Composite Reverse Osmosis (TFC RO) membranes have undergone significant changes since inception; particularly the top polyamide layer has been tuned for optimal performance. The present paper demonstrates the novel approach to alter the polyamide membrane performance by subjecting it to ionic liquids. Ionic liquids 1-Butyl-3-Methylimidazolium Chloride [BMIM][Cl], 1-Methyl-3-Octylimidazolium Chloride [C<sub>8</sub>MIM][Cl] and 1-Butyl-3-Methylimidazolium Bromide [BMIM][Br] were used to alter the membrane performance. About a 6.5% increase in MgSO<sub>4</sub> rejection and about an 87% increase in water-flux were noted when the membrane was subjected to 3000 mg/L [BMIM][Cl] after 2000 mg/L sodium hypochlorite each for 2 hours. Also, the decline in contact angle from 52.86° to 43.12° by this treatment demonstrated higher hydrophilicity. Atomic force microscope images showed a decline in surface roughness with the treatment. Scanning electron micrographs were taken to understand the changes in morphology of thin film composite reverse osmosis membranes with ionic liquid treatment. Attenuated total reflectance, infrared spectroscopy and nuclear magnetic resonance analysis were done to evaluate the changes in chemical structure and it was found that the treatment resulted in chemical structural modification of thin film composite reverse osmosis membranes with ionic liquid treatment.

**Keywords:** Thin Film Composite Reverse Osmosis membrane; Ionic liquid; Surface modification; [BMIM][Cl]; [C<sub>8</sub>MIM][Cl].

## INTRODUCTION

The process of reverse osmosis, which was first commercially developed in the mid-1960s, has undergone significant development since then (Glater, 1998). Reverse osmosis, as it is commonly known, is now used extensively in various separation processes, including brackish and sea water desalination. Concentration polarization is one of the major hindrances, which causes a reduction in salt rejection and flux in reverse osmosis thin film composite membranes (Tang et al., 2010). Concentration polarization refers to the gradual increase in concentration of non-permeating components near

the surface of the membrane, effectively reducing its capacity for salt rejection and flux (Morao et al., 2008). The top layer of a reverse osmosis membrane is dense. The thickness of this layer is an important consideration in concentration polarization. Thinning the top layer of the membrane can weaken the effects of concentration polarization and thereby theoretically increase the water flux (Wang et al., 2009). Thin film composite reverse osmosis membranes are either cellulose acetate or polyamide membrane. However, the latter is widely used because of its higher operating pH range, wider temperature range, higher stability to biological attack and ease of surface modification (Petersen et al., 1993; El-Saied et al., 2003; Younos et

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al., 2005; Wethern et al., 1995; Asano, 1998; Kulkarni et al., 1996).

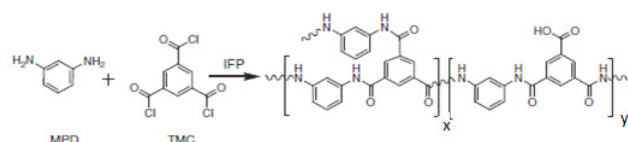
Various experiments have been performed for surface modification of reverse osmosis membranes using different modifying agents. These include the use of sodium hypochlorite and polysaccharides like chitosan (Raval et al., 2016; Raval et al., 2017; Raval et al., 2015). Some toxic, corrosive, flammable or environmentally harmful agents such as concentrated sulfuric acid and hydrofluoric acid (Gilron et al., 2001; Belfer et al., 1998). Room Temperature Ionic Liquids [RTIL] are salts in the liquid state in which ions are poorly coordinated and they exist in the liquid state below 100°C. Ionic liquids have recently gained popularity in use in various chemical applications owing to their unique properties. Ionic liquids are powerful solvents and find applications in decomposing polymers and as additives in nano-filtration and other membranes (Swatloski et al., 2002; Lua et al., 2009; Yung et al., 2010; Mohammad et al., 2015). However, the application of ionic liquid for surface modification of thin film composite reverse osmosis membranes is largely unknown (Schmidt et al., 2008).

This paper aims at using ionic liquids as surface modifiers for commercial thin film composite reverse osmosis membranes and explores their effect on membrane performance. From various different ionic liquids 1-Butyl-3-Methylimidazolium Chloride [BMIM][Cl] was found to give optimum results. The surface modification of the polyamide layer by the ionic liquid [BMIM][Cl] has been characterized to explain the change in performance.

## EXPERIMENTAL

### Materials

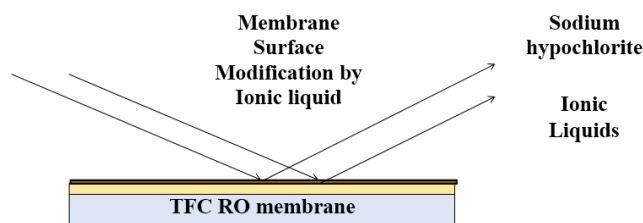
Commercial thin film composite reverse osmosis membrane was purchased from Hydraunatics, USA (CPA2). 1-Butyl-3-Methylimidazolium Chloride [BMIM][Cl] ionic liquid (Molecular Weight = 174.67 g/mol), 1-Butyl-3-Methylimidazolium Bromide [BMIM][Br] (Molecular Weight = 219.12 g/mol), 1-Methyl-3-Octylimidazolium Chloride [C<sub>8</sub>MIM][Cl] (Molecular Weight = 230.78 g/mol) were purchased from Sigma – Aldrich Co. LLC. Sodium hypochlorite (Molecular Weight = 74.44 g/mol) was purchased as laboratory reagent with available chlorine of 4% - 6% w/v from RFCL limited, New Delhi (India). Magnesium sulphate (divalent salt) was purchased from Tokyo Chemical Industry Co., LTD, Tokyo, Japan. The chemical structure of the polyamide layer of thin film composite reverse osmosis membranes is shown in Figure 1 (Rangarajan et al., 2011).



**Figure 1.** Interfacial polymerization reaction to form aromatic polyamide.

### Treatment of membrane with sodium hypochlorite and ionic liquid

Polyamide thin film composite reverse osmosis membranes were cut into a strip of 10 cm x 10 cm, cleaned with deionized water. We stuck the membrane strip on a glass plate to ensure that only the top layer was subjected to treatment and the other components remained air tight and free of any chemical contact. We dipped the membrane into a 2000 mg/L solution of sodium hypochlorite for a period of 2 hours. Membrane was then taken out and once again thoroughly washed with deionized water and then cut into two strips each of 5 cm x 10 cm. One strip was immediately dipped into a solution of 1-Butyl-3-Methylimidazolium Chloride [BMIM][Cl] ionic liquid for 2 hours and the other strip was dipped into deionized water for the same period. A series of experiments were carried out in a similar fashion with varying concentration of 1-Butyl-3-Methylimidazolium Chloride [BMIM][Cl] and other ionic liquids i.e. 1-Butyl-3-Methylimidazolium Bromide [BMIM][Br] and 1-Methyl-3-Octylimidazolium Chloride [C<sub>8</sub>MIM][Cl] as shown in Figure 2.



**Figure 2.** Schematic of membrane surface modification by ionic liquids.

### Membrane performance

Experiments were carried out in a standard reverse osmosis experimentation kit. The modified membranes were subjected to magnesium sulfate solution of 4000 μS in deionized water at 250 psig pressure. The membranes were stabilized for 2 hours and then permeate was collected for 20 minute at 250 psig pressure to measure water-flux and conductivity as per the standard procedure.

### Membrane characterization

We characterized the membranes by Scanning electron micrographs (SEM) to study the surface

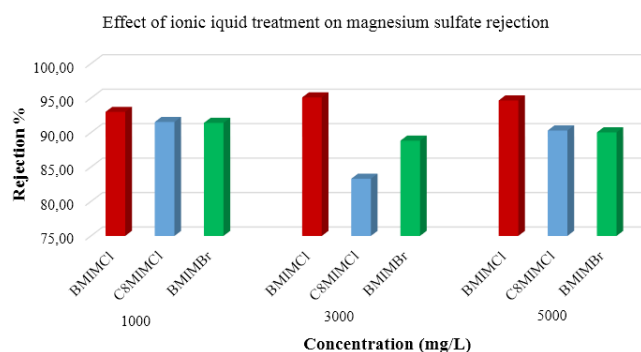
morphology, nuclear magnetic resonance (NMR) and attenuated total reflectance infrared spectroscopy (ATR-FTIR) and to understand the chemical structural modification, atomic force micrographs (AFM) to study the top surface roughness features and contact angle analysis for measuring surface hydrophilicity.

## RESULTS AND DISCUSSION

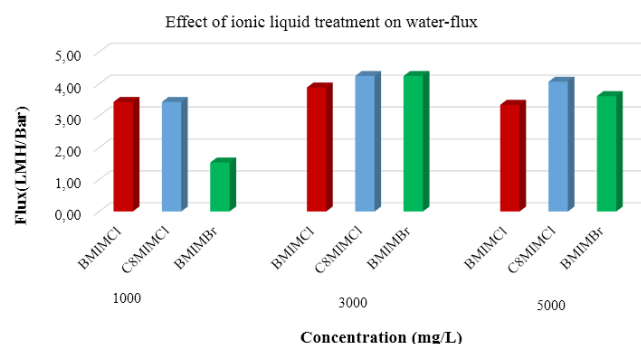
### Divalent salt rejection

Results as shown in Figure 3 and Figure 4 indicate that of the various different ionic liquids 1-Butyl-3-Methylimidazolium Chloride [BMIM][Cl] gives the optimum results in terms of flux as well as divalent salt rejection. Also it can be inferred from Figure 4 and Table 1 that 3000 mg/L concentration of 1-Butyl-3-Methylimidazolium Chloride [BMIM][Cl] gives 6.5% increase in  $MgSO_4$  rejection and about 87% increase in water-flux as compared to virgin thin film composite reverse osmosis membranes and other concentrations of 1-Butyl-3-Methylimidazolium Chloride [BMIM][Cl].

It can be inferred from Table 1 that divalent salt rejection increases until 3000 mg/L 1-Butyl-3-Methylimidazolium Chloride [BMIM][Cl] concentration and then decreases. Water flux also follows the similar trend. It can be deduced from the data in Table 1 that both water flux and magnesium



**Figure 3.** Divalent ion rejection comparison of various ionic liquids with varying concentrations.



**Figure 4.** Water-flux comparison of different ionic liquids with varying concentrations.

**Table 1.** Membrane salt rejection and divalent rejection with different dosage of [BMIM][Cl] ionic liquid.

Membrane	Magnesium sulfate Rejection (%)	Flux (LMH/bar)
Thin Film Composite (TFC)	89.30%	2.08
500 mg/L Ionic Liquid	84.98%	3.16
1000 mg/L Ionic Liquid	93.00%	3.44
3000 mg/L Ionic Liquid	95.10%	3.89
4000 mg/L Ionic Liquid	94.91%	3.62
5000 mg/L Ionic Liquid	94.66%	3.34

sulfate rejection increased for the ionic liquid 1-Butyl-3-Methylimidazolium Chloride [BMIM][Cl] treated membrane with 1000 mg/L to 5000 mg/L concentrations. For 1-Methyl-3-Octylimidazolium Chloride [C<sub>8</sub>MIM][Cl] treated membranes, the water-flux is identical or more as compared to [BMIM][Cl] treated membrane but magnesium sulfate rejection declined for all concentrations, as shown in Figures 3 and 4. For 1-Butyl-3-Methylimidazolium Bromide [BMIM][Br] treated membranes, magnesium sulfate rejection was lower as compared to 1-Butyl-3-Methylimidazolium Chloride [BMIM][Cl] treated membrane.

### Contact angle of membranes

Contact angles of membranes were measured using the drop shape analyser (DSA 100) provided by Krüss Optronic – Germany. The contact angle is a measure to study the hydrophilicity of the membrane, i.e., the contact angle of a water drop will decline with increased hydrophilicity. Contact angles for various membranes are mentioned in Table 2. It can be seen that the contact angle declines from 52.86° to 43.12° for 3000 mg/L 1-Butyl-3-Methylimidazolium Chloride [BMIM][Cl] as compared to virgin thin film composite reverse osmosis membrane. However, it increases further with higher concentration treatment, i.e., 4000 mg/L and 5000 mg/L of 1-Butyl-3-Methylimidazolium Chloride [BMIM][Cl].

**Table 2.** Membrane contact angle with different dosage of [BMIM][Cl] Ionic Liquid.

Membrane	Contact Angle (°)
Thin Film Composite (TFC)	52.86
500 mg/L [BMIM][Cl]	57.00
1000 mg/L [BMIM][Cl]	54.98
3000 mg/L [BMIM][Cl]	43.12
4000 mg/L [BMIM][Cl]	47.42
5000 mg/L [BMIM][Cl]	48.60

### Scanning Electron Micrographs (SEM)

The morphology of the membrane modified by 3000 mg/L 1-Butyl-3-Methylimidazolium Chloride [BMIM][Cl] was studied using field emission scanning



electron microscope (JSM-7100F Japan). Figure 5 shows the SEM images.

Presence of ionic liquid can be observed in Figure 5 (b) & (c). White spots can be clearly identified in Figure 5 (b) that shows the structural modification with ionic liquid treatment. The cross-section in Figure 5 (c) and (d) shows the thinning of the polyamide layer with the ionic liquid treatment. Top polyamide layer thickness was reduced to 60 nm as compared to average polyamide layer thickness, i.e., about 200 nm as reported in case of interfacial polymerization with hexane solvent (Ghosh et al., 2008). The decline in thickness of the membrane is because of interaction between polyamide and ionic liquid. Decline in thickness of the polyamide layer may be attributed to  $\pi$ - $\pi$  interactions of ionic liquid with polyamide.

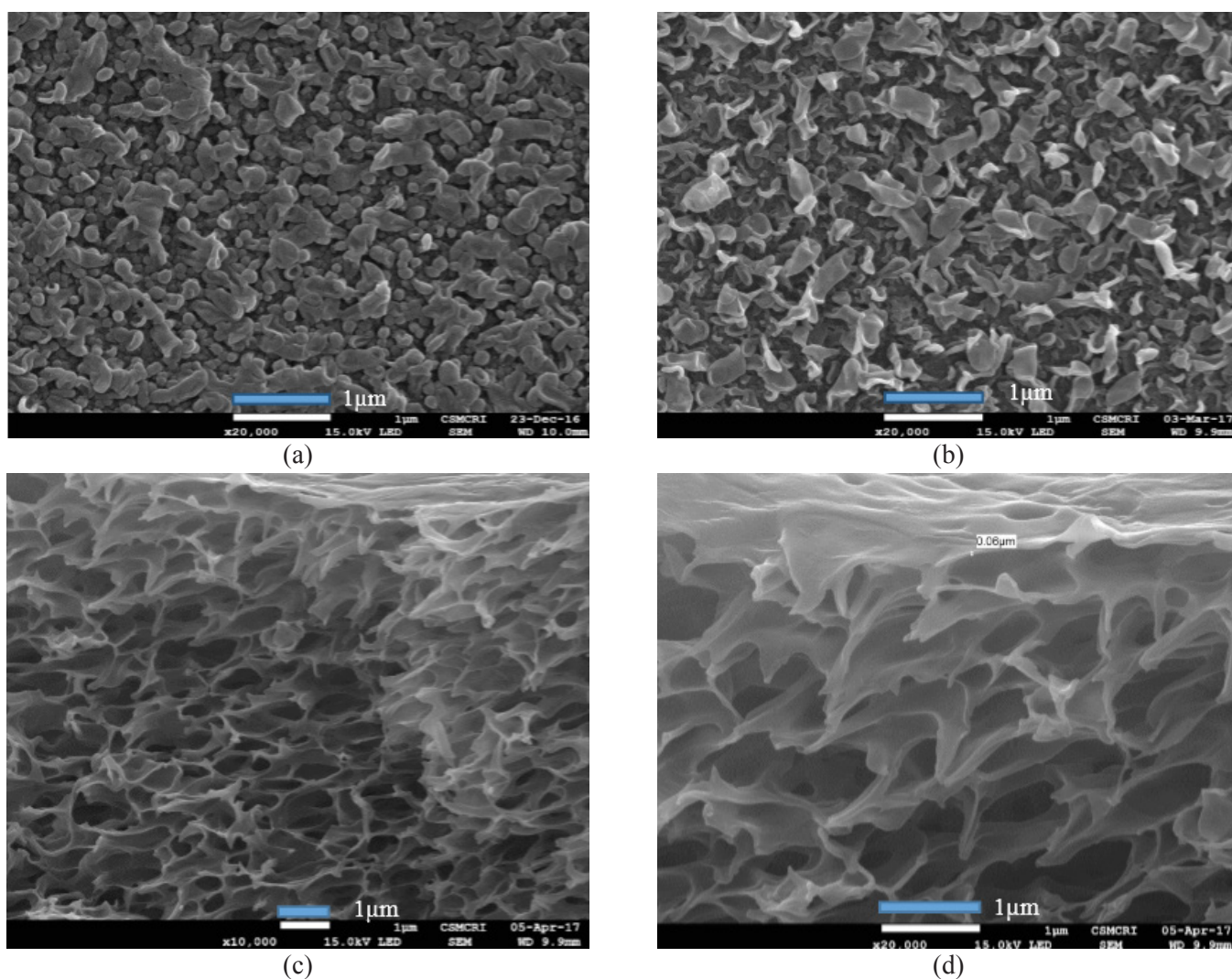
#### Atomic Force Microscope Images (AFM)

The topography and surface roughness properties of the membrane modified by 1-Butyl-

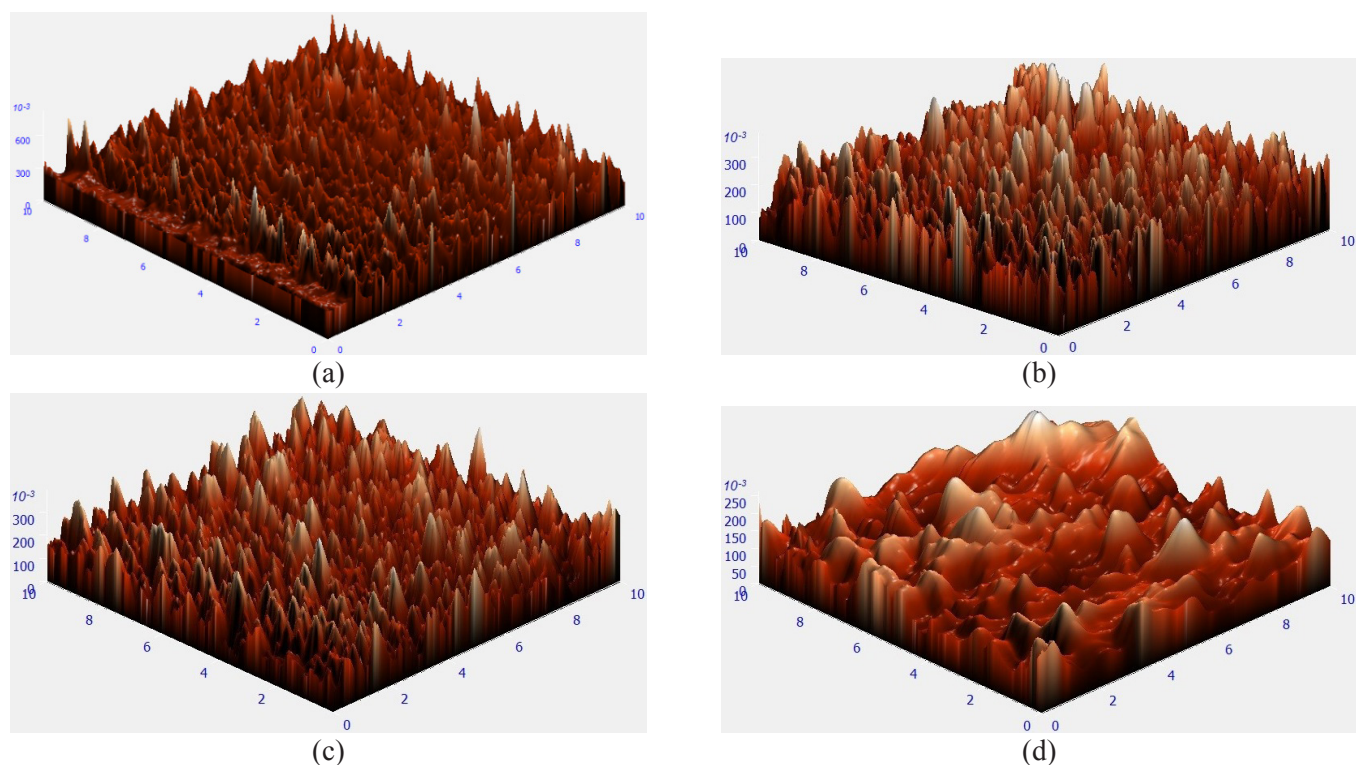
3-Methylimidazolium Chloride [BMIM][Cl] were measured using an atomic force microscope (NTEGRA Aura, NT-MDT Instruments, Russia). The images of virgin thin film composite membrane, and 500 mg/L, 3000 mg/L and 4000 mg/L 1-Butyl-3-Methylimidazolium Chloride [BMIM][Cl] ionic liquid treated membranes are shown in Figure 6. The membrane with the highest water-flux and magnesium sulphate rejection, i.e., membrane treated with 3000 mg/L 1-Butyl-3-Methylimidazolium Chloride [BMIM][Cl], showed the surface area ratio of 21.34% and roughness of 42.7 nm as shown in Table 3. It shows the change in roughness with ionic liquid treatment.

#### ATR – Fourier Transform Infrared spectroscopic analysis

Attenuated total reflectance Fourier transform infrared spectroscopy (Spectrum GX FTIR Spectrometer, PerkinElmer, USA) was used to analyse the chemical structure of the virgin thin film composite



**Figure 5.** SEM images (a) virgin TFC membrane (b) modified membrane with 3000 mg/L [BMIM][Cl] ionic liquid (c) & (d) Cross section of modified membrane with 3000 mg/L [BMIM][Cl] ionic liquid.



**Figure 6.** AFM images (a) TFC membrane (b) 500 mg/L [BMIM][Cl] (c) 3000 mg/L [BMIM][Cl] (d) 4000 mg/L [BMIM][Cl].

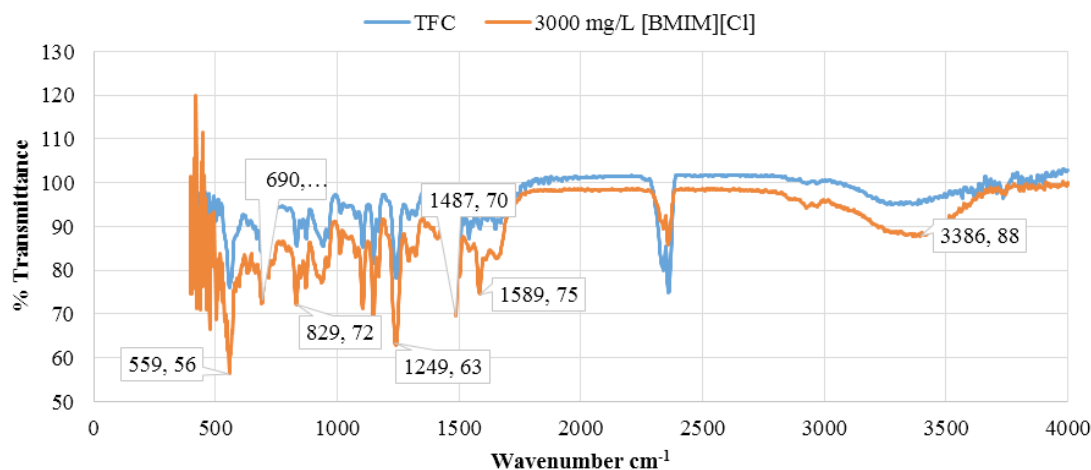
**Table 3.** AFM data of TFC and [BMIM][Cl] treated membranes.

	TFC	500 mg/L [BMIM][Cl]	3000 mg/L [BMIM][Cl]	4000 mg/L [BMIM][Cl]
Roughness Average, Sa (nm)	64.7	44.2	42.7	29.3
Surface Area Ratio, Sdr	21.94 %	29.44 %	21.34 %	18.89 %

membrane and membrane modified with 3000 mg/L 1-Butyl-3-Methylimidazolium Chloride [BMIM][Cl] ionic liquid. The ATR-FTIR spectra of both the membranes are shown in Figure 7. The identifiable peaks are mentioned in Table 4.

It can be seen from Figure 7 and Table 4 that the amide bond of polyamide layer has been modified by ionic liquid treatment. The peaks at 1635  $\text{cm}^{-1}$  and 1589

$\text{cm}^{-1}$  correspond to amide I and amide II, respectively. Amide I is comprised of CO stretching and amide II represents NH in-plane bending. It can be inferred that CC aromatic ring stretching has been affected by the treatment of ionic liquid on the TFC RO membrane. Ether and C=C linkages have been modified by ionic liquid treatment of the polyamide layer. In this way, the chemical structural changes in the polyamide layer



**Figure 7.** ATR FT-IR Spectra of virgin thin film composite and 3000 mg/L [BMIM][Cl].

**Table 4.** Peak identification of spectra of TFC and 3000 mg/L [BMIM][Cl].

Sr. No.	Wavenumber $\text{cm}^{-1}$	Functional Group	Type of Vibration	Intensity
1.	559	Alkyl halides	Stretch	Strong
3.	829	Alkenes (=C-H)	Bend	Strong
4.	1249	Ether (C-O)	Stretch	Strong
5.	1487	Aromatic compound (ring C=C)	Stretch	Medium-Strong
6.	1589	Amide-II	Bend	Weak-Medium
7.	1635	Amide-I	Stretch	Weak-Medium
8.	3386	Amides (R-C(O)-NH <sub>2</sub> )	Stretch	Weak-Medium

are significant with ionic liquid treatment. To further investigate the modification in chemical structure, solid-state Nuclear Magnetic Resonance (NMR) spectra were taken for the virgin thin film composite reverse osmosis membrane and the 3000 mg/L 1-Butyl-3-Methylimidazolium Chloride [BMIM][Cl] ionic liquid treated membrane.

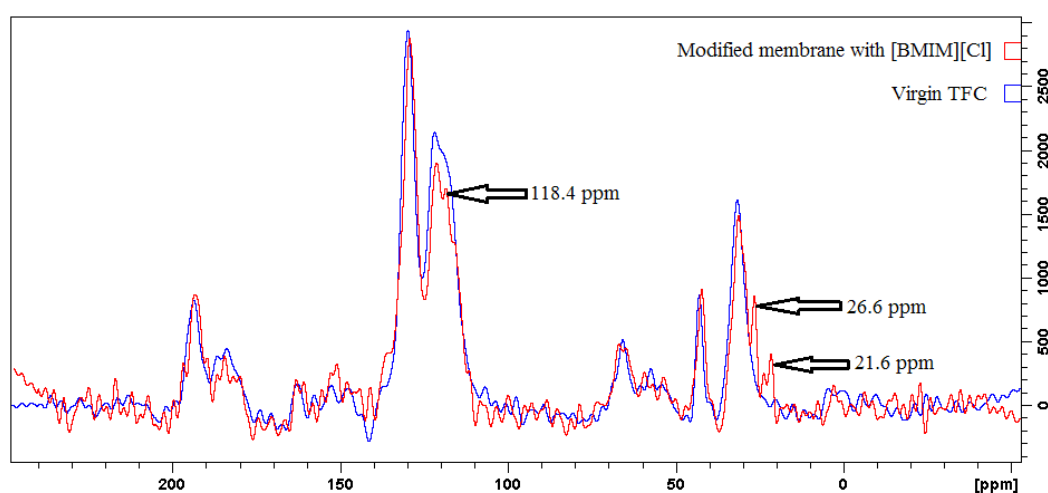
#### Nuclear Magnetic Resonance spectroscopic analysis

Nuclear magnetic resonance spectroscopy (Bruker Avance II 500 MHz FT-NMR equipped with 4 mm

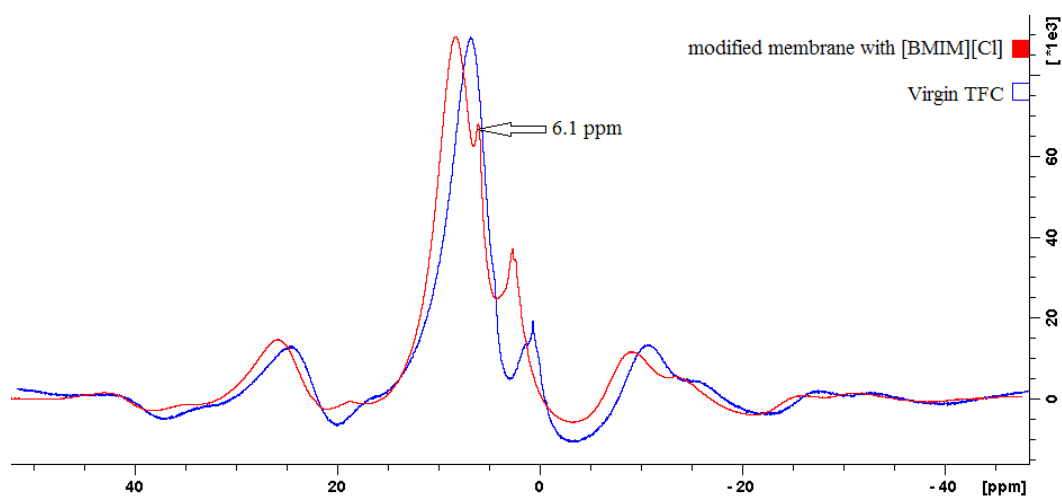
CPMAS probe sample was run at 8000 kHz) was used to analyse the modification in structure of the virgin thin film composite reverse osmosis membrane and modified with 3000 mg/L 1-Butyl-3-Methylimidazolium Chloride [BMIM][Cl] ionic liquid.

Figure 8 shows the <sup>13</sup>C NMR spectra overlay of virgin thin film composite and membrane modified with 3000 mg/L 1-Butyl-3-Methylimidazolium Chloride [BMIM][Cl].

The origin of three new peaks at 21.6 ppm, 26.6 ppm and 118.4 ppm in modified membrane can be attributed to weak interaction with [BMIM][Cl]. The



**Figure 8.** <sup>13</sup>C NMR spectra of virgin TFC and modified membrane with 3000 mg/L [BMIM][Cl] ionic liquid.



**Figure 9.** <sup>1</sup>H NMR spectra of virgin TFC and modified membrane with 3000 mg/L [BMIM][Cl] ionic liquid.



peaks at 21.6 ppm and 26.6 ppm are related to the butyl chain of ionic liquid, where the associated peak at 118.4 ppm can be assigned to aromatic imidazolium carbons. Shifting in the ppm value is due to the change of the chemical environment of carbons from a polymeric aromatic environment.

Figure 9 shows  $^1\text{H}$  NMR overlay spectra of the same. The origin of the new associated peak at 6.1 ppm is related to aromatic imidazolium protons of the ionic liquid.

This clearly demonstrates that the polyamide layer has been modified by the ionic liquid through weak  $\pi - \pi$  or cation- $\pi$  interactions.

Thus, the characterization of the membrane reveals the chemical, surface morphological and roughness changes and hydrophilic surface formation upon addition of ionic liquid into the polysulfone membrane matrix.

## CONCLUSION

Different ionic liquids were used to modify the top polyamide layer of thin film composite reverse osmosis membranes. It was observed that the top polyamide layer of the thin film composite reverse osmosis membrane became thinner. Moreover, the contact angle of such modified membrane declined from  $52.86^\circ$  to  $43.12^\circ$ , which demonstrated increased hydrophilicity. These factors contributed to an increase in water-flux. Divalent salt rejection of the modified membrane (3000 mg/L 1-Butyl-3-Methylimidazolium Chloride [BMIM][Cl] exposed for 2 hours after treatment with 2000 mg/L sodium hypochlorite for 2 hours) increased from 89.3% to 95.10% with an increase in water-flux from 2.08 LMH/Bar to 3.89 LMH/Bar. The performance increment was the highest with 1-Butyl-3-Methylimidazolium Chloride [BMIM][Cl] as compared the other ionic liquids such as 1-Butyl-3-Methylimidazolium Bromide [BMIM][Br] and 1-Methyl-3-Octylimidazolium Chloride [ $\text{C}_8\text{MIM}$ ][Cl]. The modified membrane had decreased surface roughness, with an increased surface area ratio. ATR-FTIR spectra demonstrated the modification in the amide bond and prove the chemical modification of structure with ionic liquid treatment.  $^{13}\text{C}$ - and  $^1\text{H}$ -NMR spectra demonstrated new peaks confirming the chemical structural modification with ionic liquid treatment. Scanning electron micrographs showed the modification in the morphology of the membrane with the treatment. Thus, the present paper demonstrates novel thin film composite reverse osmosis membrane with improved water flux, higher magnesium sulfate rejection and lower thickness of top polyamide layer upon ionic liquid treatment. It opens the possibility of future work in the area.

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