



Evaluation of hydrolyzed poly(isobutylene-*alt*-maleic anhydride) as a polyelectrolyte draw solution for forward osmosis desalination



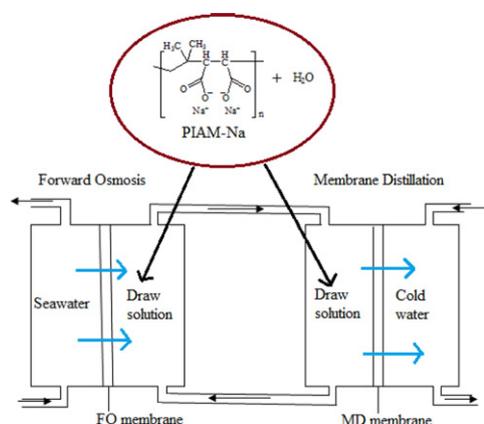
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HIGHLIGHTS

- Hydrolyzed poly(isobutylene-*alt*-maleic acid) (PIAM-Na) was synthesized and used as draw solute for FO desalination.
- Nontoxic PIAM-Na produced high osmotic pressure DS with low viscosity.
- PIAM-Na showed two T_g values well above the operation temperature of FO-MD hybrid process.
- 0.375 g/mL of PIAM-Na produced maximum flux with lowest RSF at AL-DS mode compared to conventional salt.

GRAPHICAL ABSTRACT



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ABSTRACT

The forward osmosis (FO) desalination consisting of polyelectrolyte-based draw solution (DS) is gaining more interest due to low reverse solute flux (RSF) and easy DS recovery. In the current work, a novel solute for the DS was prepared by the base hydrolysis of poly(isobutylene-*alt*-maleic anhydride) (IBMA) using NaOH. The hydrolyzed product, i.e., sodium salt of IBMA (IBMA-Na), was dissolved in deionized (DI) water at various concentrations to obtain the DS. The FO experiments were conducted using commercially available cellulose triacetate (CTA) flat sheet membrane. The cytotoxicity study by the MTT assay method proved nontoxic nature of IBMA-Na and thermogravimetric (TGA) analysis of IBMA-Na showed two glass transition temperature (T_g) values well above the operation temperatures of FO-MD hybrid process. High osmotic pressure (Π) values with low viscosity were obtained for IBMA-Na with a concentration up to 0.375 g/mL, and Π values increased drastically thereafter. The DS consisting of 0.375 g/mL of IBMA-Na showed the flux of 34 L/m² h at active layer facing DS (AL-DS) mode with a RSF of 0.196 gMH at 60 °C. The recycling of the DS was easily achieved using the membrane distillation process and revealed its potential application in the FO-MD hybrid desalination process.

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1. Introduction

The membrane-based forward osmosis (FO) has found a wide range of applications in water purification, seawater desalination, wastewater

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treatment, food processing, and pharmaceutical industry [1,2]. It is an energy efficient process that operates at low pressure or without additional pressure compared to reverse osmosis (RO). The osmotic pressure difference between the draw solution (DS) and the feed solution (FS) will maintain the feed flow (flux) towards the permeate side. The permeating water dilutes the DS, but only to a certain extent, i.e., until an osmotic equilibrium is reached between the DS and the FS [3]. At the same time there is a slow diffusion of solutes through the membrane from the DS to the FS due to the high concentration difference of ions between the two streams called as reverse solute flux (RSF). RSF not only reduces the effective osmotic driving force across the membrane but also increases the replenishment cost and scaling issue [4]. An ideal FO process should produce high water flux with low RSF. This consequently depends on the properties of the DS, membrane characteristics, and other operating parameters used during the FO process [5]. The essential features of a good DS are nontoxicity, high osmotic pressure, low viscosity, low RSF, low cost, and energy efficient methods to recover the draw solute from it [6].

Recently, many efforts have been put towards the development of efficient DS that are found to have their own advantages as well as disadvantages. Ammonium bicarbonate was used as a draw agent that can produce high osmotic pressure and be regenerated by distillation at around 60 °C; however, the removal of ammonia residue in the water product was found to be a problem [7,8]. The conventional salts such as NaCl and MgCl₂ produced high osmotic pressure with high water flux; however, the high reverse solute leakage and the high cost of draw solute recovery increased the total operational costs [9,10]. The large molecular sized water soluble nanoparticles were developed as draw solutes by the coating of hydrophilic molecules. However, the coating resulted in reduced magnetic properties of the solute by particles and resulted in irreversible agglomeration during the recovery process [11,12]. The 2-methyl imidazole-based compounds improved the water recovery rate and its quality by the membrane distillation technique. However, high operating costs unless using low grade heat for membrane distillation (MD) and the high cost of imidazole derivatives are the major disadvantages in the large-scale application [13]. The thermosensitive materials are utilized as draw solutes, which are soluble in water below their lower critical solution temperature (LCST) but precipitates above the LCST. The easy draw solute recovery by membrane distillation was achieved but the development of high osmotic draw solute still remained a problem [14]. The hydrogels contain hydrophilic polymer networks and show high absorption for water. When used as a draw solute, they were found to be energy efficient and environmentally friendly, but they have disadvantages such as the poor liquid water recovery rate and unsuitability for practical application [15].

The polyelectrolyte DS are gaining much interest due to low RSF, high flux, and low cost of the DS recovery. The use of different molecular weights of the sodium salt of polyacrylic acid (PAA-Na) was studied by Ge et al. [16]. They demonstrated that the solute with the lowest molecular weight produces the maximum FO flux due to its low viscosity and high dissociation. PAA-Na DS produced much lower RSF compared to the conventional draw solutes. The sodium salt of polyaspartic acid (PAs-Na) draw solute with an average molecular weight (M_w) of 1,313 Da was studied by Gwak et al. [17]. The higher degree of solvation of PAs-Na compared to PAA-Na resulted in a relatively high osmotic pressure DS even at high concentrations. Henceforth, there is a further scope to explore the novel polyelectrolytic draw solutes with: i) sufficiently low molecular weight which can produce low viscosity solutions, ii) high dissociation to produce more solvating groups which have high affinity towards water to produce high osmotic pressure solutions.

In this work, a novel polyelectrolyte draw solute was prepared by the base hydrolysis of poly(isobutylene-*alt*-maleic anhydride) (PIAM) using NaOH as a base. PIAM is a low cost, synthetic and commercially available polymer built-up of isobutylene and maleic anhydride monomers. The low molecular weight PIAM is commonly used in making low

viscosity solutions for dispersion and emulsification applications [18]. A strong base like NaOH will react with the anhydride of PIAM to produce sodium salt poly(isobutylene-*alt*-maleic acid) (PIAM-Na). Such hydrolysis reaction of PIAM produces sodium salt of dicarboxylic acid (Fig. 1), which may have high affinity for water molecules due to solvation effect to produce high osmotic pressure solutions. The toxicity study of PIAM-Na was carried out by the MTT assay method. The physical properties of the DS relevant to the FO application, such as viscosity and osmotic pressure, were evaluated. Most of the polyelectrolyte-based FO processes have to be coupled with another low cost separation process to separate the final product water [19]. In the current study, for the recovery of DS, membrane distillation (MD) was selected due to its attractive advantages such as high water quality, resistance towards feed salinity, and the ability to operate using low-grade heat.

2. Experimental

2.1. Materials and methods

Poly(isobutylene-*alt*-maleic anhydride, average $M_w \sim 6000$ g/mol) with isobutylene:maleic anhydride = 1:3 was purchased from Sigma Aldrich Co. Sodium hydroxide, and ethanol were purchased from Merck Co. Deionized (DI) water was used for all of the reactions and the DS preparation.

2.2. Preparation of PIAM-Na and its draw solutions

PIAM (100 g) was taken in DI water (500 mL) at 25–26 °C, and NaOH (38 g) was added in three lots over a period of 20 min. An exotherm of ~5–6 °C was observed during each addition; however, the temperature was maintained below 60 °C by controlling the speed of the addition of NaOH. After addition, the reaction mixture was heated at 60 °C for 48 h to obtain the homogeneous solution. The excess NaOH was removed by precipitating the reaction mixture by the addition of ethanol (800 mL) at 25 °C. The solid product obtained was separated by the filtration and dried at 80 °C. Subsequently, the product was dissolved in 500 mL of DI water and purified through a dialysis process till the pH of the solution was neutral, indicating that traces of NaOH was totally removed. The final product of PIAM-Na was dried under vacuum and dissolved in DI water at various concentrations (0.3 g/mL, 0.325 g/mL, 0.35 g/mL, 0.375 g/mL, and 0.4 g/mL) for the characterization and other performance tests.

2.3. Characterization of PIAM-Na

The molecular weight of PIAM-Na sample was measured by gel permeation chromatography using Agilent 1260 Infinity MDS system with refractive index detector, aqueous phosphate buffer as eluent and using 2 mg/mL concentration of PIAM-Na for the analysis. The hydrolysis of PIAM into PIAM-Na was confirmed using ALPHA Attenuated Total Reflectance Infrared (ATR-IR) spectrophotometer (Bruker, Germany). The powder sample was directly placed on the prism and the transmittance was measured in the range of 400 cm⁻¹ to 4000 cm⁻¹. The thermogravimetric analysis of the PIAM-Na sample was carried out using Setaram DSC 131 EVO instrument, France. The sample was heated from -10 °C to +500 °C at a heating rate of 10 °C/min to determine the T_g values. The osmolality of PIAM-Na solutions was measured using WESCOR, 5600 model, USA, and then it was converted into osmotic pressure values. The viscosity of different concentrations of PIAM-Na was measured at various temperatures using Brookfield, CAP-2000 + viscometer (USA) at 20 rpm. Then, it was converted into relative viscosity (η_r) by comparing with the viscosity of DI water.

$$\eta_r = \frac{\eta}{\eta_0} \quad (1)$$

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