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Original Article

Feasibility study of membrane preparation using polycarbonate from waste compact discs by phase separation

Diew Saijun and Watchanida Chinpa*

Department of Materials Science and Technology, Faculty of Science,
Prince of Songkla University, Hat Yai, Songkhla, 90110 Thailand

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Abstract

Using non-solvent induced phase separation (NIPS), asymmetric porous membranes were prepared from polycarbonate (PC) derived from waste compact discs (CDs). The effects of recovered PC (PC-R) concentration (18-24 wt%) on the morphologies and mechanical and permeation properties were investigated. At all casting solution concentrations, PC-R membranes had asymmetric structures that consisted of a skin layer and a marcovoid sublayer. As the PC-R concentration increased, the macrovoid sublayer was suppressed as a sponge-like sublayer formed within the asymmetric structure. The tensile strength and elongation at break of membranes cast from solutions containing 20, 22, and 24 wt% PC-R ranged from 2.60 to 2.97 MPa and 7.69 to 11.45%, respectively. These values were sufficient to resist operating pressures during membrane filtration testing. The pure water flux through the membranes decreased as the PC-R concentration increased, whereas %R presented an inverse trend. These results indicated that PC recovered from waste compact discs could provide an alternative material for porous membrane fabrication.

Keywords: waste CDs, polycarbonate, membrane, phase inversion

1. Introduction

Polycarbonate (PC) is an engineering plastic used in digital media products such as CDs, DVDs, electronic equipment, automobiles, medical devices and containers for liquid storage. It is characterized by its toughness, dimensional stability, optical clarity, thermal and chemical resistance and excellent electrical resistance (Antonakou *et al.*, 2014).

Although the production of compact discs has significantly decreased, huge volumes of used CDs still exist because PC, which is their main component, is a non-biodegradable polymer. Used CDs have either been buried as landfill or incinerated, which contributes to environmental pollution. Previous research has attempted to recycle PC waste by chemical recycling techniques that broke down PC waste into useful products such as phenol and bisphenol A (Achilias, Antonakou, Koutsokosta, & Lappas, 2009; Deepak, 2016). Widely used chemical recycling methods have included hydrolysis (Grause, Sugawara, Mizoguchi & Yoshioka, 2009; Pan, Chou, & Burruss, 2009), aminolysis (Hata, Goto, Yamada, & Oku, 2002), alcoholysis (Hu, Oku, & Yamada, 1998; Liu, Li, Yu, Cui, & Ge, 2010; Pinero, Garcia, & Cocero, 2005) and glycerolysis (Deepak, 2016). Since CDs mainly consist of high quality PC above 95 wt% (Zhu *et al.*, 2017), PC recovered from CDs could provide a cheap source material that may be economically used in other applications. PC recovery techniques were developed using abrasive fluidized bed machining (Coberth & Ceyssons, 1999), dry-ice blasting (Shishkin, Geskin, & Goldenberg, 2002), waterjets (Babets & Geskin, 2000) and chemical etching (Operowsky, 1995).

There are reports of membrane preparation from waste polystyrene (PS), terephthalate (PET) and polyethylene (HDPE). Meireles *et al.* (2008) prepared a blend membrane from cellulose acetate (CA) and polystyrene derived from sugarcane bagasse and plastic cups, respectively. The obtained membrane could be used in a range of ultrafiltration or microfiltration applications. Rajesh *et al.* (2014) studied the preparation of PET ultrafiltration membranes from recycled PET using a phase inversion process and found that the addition of

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*Corresponding author
Email address: watchanida.c@psu.ac.th

high molecular weight polyethylene glycol (PEG) led to membranes with greater pure water flux. Recently, Zukimin *et al.* (2017) successfully synthesized a polymeric membrane by a sintering process from waste HDPE bottles.

PC is an interesting material for membrane technology due to its physical and mechanical properties and easy film forming. Previous works have prepared membranes from PC using vapor induced phase inversion (VIPS) (Bodzek & Bohdziewicz, 1991; Di Luccio, Nobrega, & Borges, 2000, 2002), non-solvent induced phase inversion (NIPS) or immersion casting (Kao *et al.*, 2008; Lee, Teng, Lee, & Lai, 2000; Di Luccio *et al.*, 2000, 2002). Results showed that VIPS produced a sponge-like structure, whereas NIPS produced an asymmetric structure consisting of an upper skin supported on a macrovoid structure. Membrane morphologies were consistent with mechanical properties and water permeation and solute rejection behaviors. Besides depending on the fabrication method, the membrane morphologies depended on other parameters, including casting solution concentration, the type of additive in the casting solution, and exposure time in water vapor before immersion in a coagulant bath. The preparation of PC membrane from waste PC has not been reported and adding value to this material through reuse is an interesting way to potentially reduce the volume of waste PC. The aim of this work was to study the feasibility of using immersion phase inversion to prepare porous membranes for ultrafiltration applications. The recovery of PC from waste CDs was performed first, followed by the study of the properties of the recovered PC in order to evaluate its potential. The effect of recovered PC concentration on morphology, tensile strength and permeability was then investigated.

2. Materials and Methods

2.1 Materials

Used CDs were collected from Department of Materials Science and Technology, Faculty of Science, Prince of Songkla University. Anhydrous 1-Methyl-2-pyrrolidinone (NMP, reagentplus®, 99%, Germany) and bovine serum albumin (BSA, molecular weight 66 kDa, USA) were supplied by Aldrich. Chloroform, analytical reagent grade, was purchased from Labscan (Thailand). All reagents were used as received.

2.2 PC Recovery and characterization

2.2.1 Recovery of PC from CD waste

In order to recover PC from waste CDs, the superimposed layers, comprising the data shield surface, lacquer, metal reflective layer and dye (Barletta, Gisario, & Tagliaferri, 2007), need to be removed. In the present work, the coatings were eliminated by mechanical abrasion. The stripped discs were then washed with water and dried in a hot air oven. After drying, they were cut into small pieces (1 × 1 cm^2) and dissolved in chloroform to obtain a homogeneous solution of 12 wt%. The solution was then precipitated in methanol with a ratio of PC to methanol of 1:3. Finally the precipitant was filtered, washed with methanol several times and dried in a vacuum oven at a temperature of 70 °C until constant weight was achieved. The obtained PC was named PC-R.

2.2.2 Characterization of PC-R

The surface of the used polycarbonate before and after removal of the coating layer was elementally analyzed using a Quanta 400, FEI scanning electron microscope (SEM) operating with a silicon drift detector (X-Max 80 EDX Oxford Instruments). Fourier transform infrared spectroscopy in attenuated total reflection mode (ATR-FTIR, Equinox 55, Bruker) was used to examine the functional groups of PC-R obtained in flake form from the recovery process described in section 2.2.1. Thermal analyses were carried out using thermogravimetric analysis (TGA, Perkin Elmer). The samples were heated from 50 °C to 1,000 °C at a heating rate of 10 °C/min under N_2 atmosphere.

The molecular weight of PC-R was analyzed using the Prominence gel permeation chromatography system (GPC, Shimadzu), comprising an LC-20AD pump and an RID-10A refractive index detector. The mobile phase was tetrahydrofuran (THF) at a flow rate of 1 mL/min at 40 °C. The concentration of PC-R injected was 0.5 wt% with a solution volume of 20 μL. The molecular weight distribution of PC-R was measured relative to linear monodisperse polystyrene standards in a range from 1,200 to 3.73×10^6 g/mol. The viscosities of the casting solutions were determined using a rotating viscometer with a RV-05 spindle rotating at 200 rpm at 25 °C.

2.3 Membrane preparation and characterization

2.3.1 Membrane preparation

Flat sheet membranes were prepared by a wet phase inversion method from PC-R solutions at different concentrations. The PC-R was dissolved in NMP and the homogeneous solutions obtained were cast on a glass plate with a casting knife, maintaining a uniform thickness of 300 μm under environmental humidity of 50%. Once a solution was cast, the glass plate was immediately immersed into the water bath and an opaque membrane soon peeled away from the glass plate. The membrane was immersed in deionized water at 70 °C for 1 h followed by immersion in deionized water at room temperature for 24 h to remove any residual solvent. Membrane fabrication was performed at room temperature. The membranes were prepared from PC-R at concentrations of 18, 20, 22 and 24 wt% and were labeled PC-R-18, PC-R-20, PC-R-22 and PC-R-24, respectively.

2.3.2 Membrane characterization

The surface and cross-sectional morphologies of the membranes were observed by scanning electron microscope (Quanta 400 FEI, JEOL, SEM). Before observation, the membranes were quenched and fractured in liquid nitrogen. They were then attached to the sample supports and coated with gold.

The mechanical properties of the PC membranes were determined using a universal tensile machine (Instron Instrument) at room temperature. The membranes were cut into rectangles 50 mm × 10 mm and put under tension at a constant elongation speed of 5 mm/min. The values of mechanical properties were obtained from the average of five measurements.

The porosity of each membrane was measured by the dry-wet weight method (Ma *et al.*, 2011). The membrane samples were kept in distilled water. After removing a sample from the water, surface water was mopped from the membrane sample with tissue paper and the sample was weighed (W_w). The wet membrane was then dried at 60 °C until constant weight was obtained (W_d). The porosity of the membrane was then calculated using equation (1)

$$P = \frac{W_w - W_d}{dA\delta} \tag{1}$$

where d was the density of water (g/cm^3) and A and δ were membrane area (cm^2) and membrane thickness (cm) in the wet state

The pure water flux (J, L/ m^2 h) was measured at 1.0 bar using a dead-end filtration cell (model 8050, Amicon) with an effective membrane area of 0.00134 m^2 and equipped with a nitrogen gas cylinder and solution reservoir. The value of J was calculated from the following equation:

$$J = \frac{Q}{A\Delta t} \tag{2}$$

where Q was the amount of permeated water (L), A was the membrane area (m^2) and Δt was the permeation time (h),

The rejection property (%R) of the membrane was determined using 1.0 g/L bovine serum albumin (BSA) solution. The protein concentrations in the retentate (C_r) and the permeate (C_p) solutions were measured using a UV–vis spectrophotometer at a wavelength of 280 nm. The BSA rejection ratio was calculated from the following equation:

$$R\ (\%) = (1 - \frac{C_p}{C_r}) \times 100 \tag{3}$$

3. Results and Discussion

3.1 Characterization of polycarbonate derived from waste CDs

The chemical composition of PC from waste CDs before and after removal of the metal-resin coating layer was analyzed using EDX. The peaks of the EDX spectrum of CDs with a coating layer (Figure 1a) indicated the presence of titanium, calcium, silica, sulfur and aluminum contents of 1.9, 1.7, 0.4, 0.2 and 0.2 wt %, respectively. The main components, carbon and oxygen (55.9 and 39.7 wt %, respectively), constitute the PC. The EDX spectrum of waste CD material after mechanical removal of the coating (Figure 1b) exhibited only the peaks of carbon and oxygen with compositions of 72.9 and 27.1 wt %, respectively. The absence of peaks indicating metal elements was evidence of the complete removal of the coating from the CDs.

The FTIR spectrum of PC-R (Figure 2) presents the characteristic peaks of the substituted aromatic ring of PC located at 3,041, 1,602, and 830 cm^{-1} (Antonakou *et al.*, 2014). The peaks at 2,952, 2,232, 1,769 and 1,602 cm^{-1} were respectively assigned to CH_3 groups, aromatic groups connected to CH_3, carbonyl groups and phenoxy groups (Anto nakou *et al.*, 2014). The spectrum of PC-R exhibits similar characteristic peaks to those presented by the spectrum of the model PC. This result confirmed that the impurities were successfully removed by the method outlined in the present work. The recovery of PC from waste CDs by precipitation purification was 80.56 ± 1.19 %.

The thermal properties of PC-R were investigated by TGA and the results show that no mass loss occurred until 450 °C (Figure 3a) and that the major degradation occurred

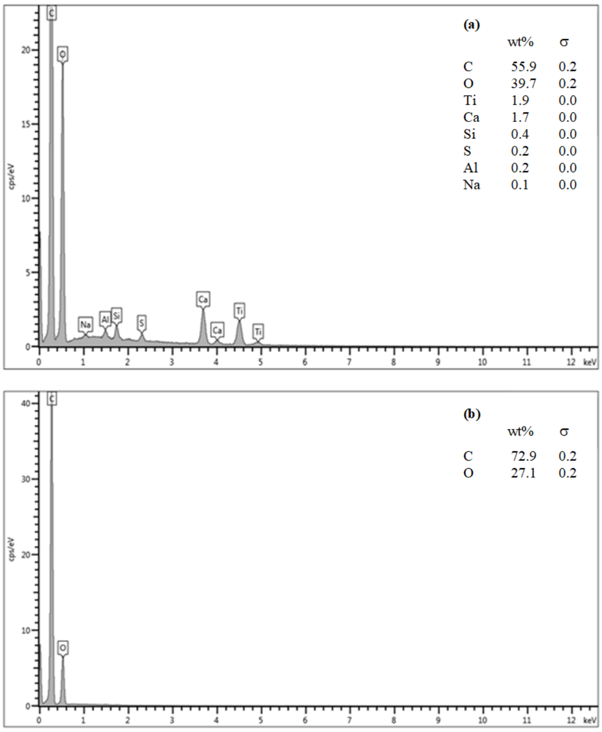

Figure 1. EDX spectra of waste CD sample (a) before and (b) after coating removal.

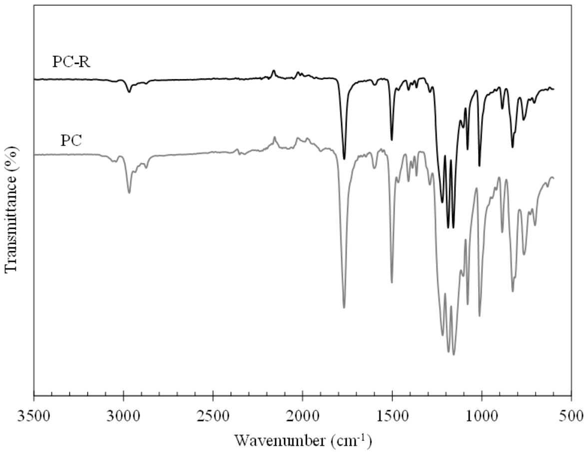

Figure 2. FTIR spectra of model PC (gray) and PC-R (black).

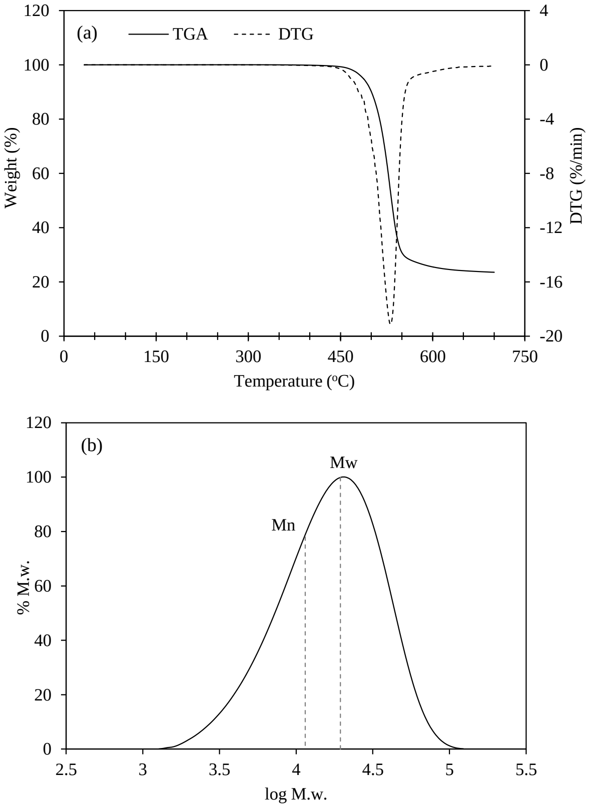

Figure 3. (a) TGA/DTG thermogram of PC-R and (b) GPC curve of PC-R.

between 450 and 550 °C. These results are similar to those reported for PC by Achilias *et al.* (2009).

The molecular weight of PC-R, evaluated by GPC (Figure 3b), was 19,736.63 and 11,458.67 Da for weight average and number average molecular weight, respectively. Di Luccio *et al.* (2002) reported asymmetric porous membranes fabricated by an immersion precipitation method using PC with a molecular weight of 40,000 Da in solution at PC concentrations of 10 to 18 wt%. Although PC-R in the present study possessed much lower molecular weight than PC reported in the literature, it was possible, using higher concentrations (18-24 wt% in the present study), to prepare porous membranes by phase inversion from a PC-R/NMP casting solution. To our knowledge, no report exists of a porous membrane prepared from low molecular weight PC derived from waste CDs. It is expected that the new information from this present study might be useful to the research community with regard to porous material applications and membrane technology.

3.2 Membrane characterization

PC-R membranes produced from solutions at different PC-R concentrations were investigated by SEM. The images of magnified surfaces and cross-sections (Figure 4) show that membrane surfaces were similar whatever the casting solution. The cross-sectional SEMs of all PC-R samples show asymmetric structures consisting of a thin upper skin sup-ported by an open interconnected macrovoid structure. This sublayer has large voids at the base of a finger-like structure clearly visible in images of the PC-R-18 and PC-R-20 membranes (Figures 4a and 4b). This result was indicative of the instantaneous demixing reported for membrane formation in a condition of high mutual affinity between the solvent (NMP) and the non-solvent (water) (Kim, Yoon & Lee, 2002; Leblanc *et al.*, 2001; Wang, Li, & Teo, 1999). Demixing was induced during the exchange by diffusion of solvent from the casting solution and non-solvent from the coagulation bath. The previously homogeneous solution separated into a two-phase system consisting of a polymer-rich phase and a polymer-poor phase (Ronner, Groot Wassink, & Smolders, 1989). These phases form the membrane matrix and the pores, respectively (Ronner, Groot Wassink & Smolders, 1989). The rapid exchange between solvent and non-solvent leads to the formation of a very thin skin layer and macrovoid sublayer (Wienk *et al.*, 1996).

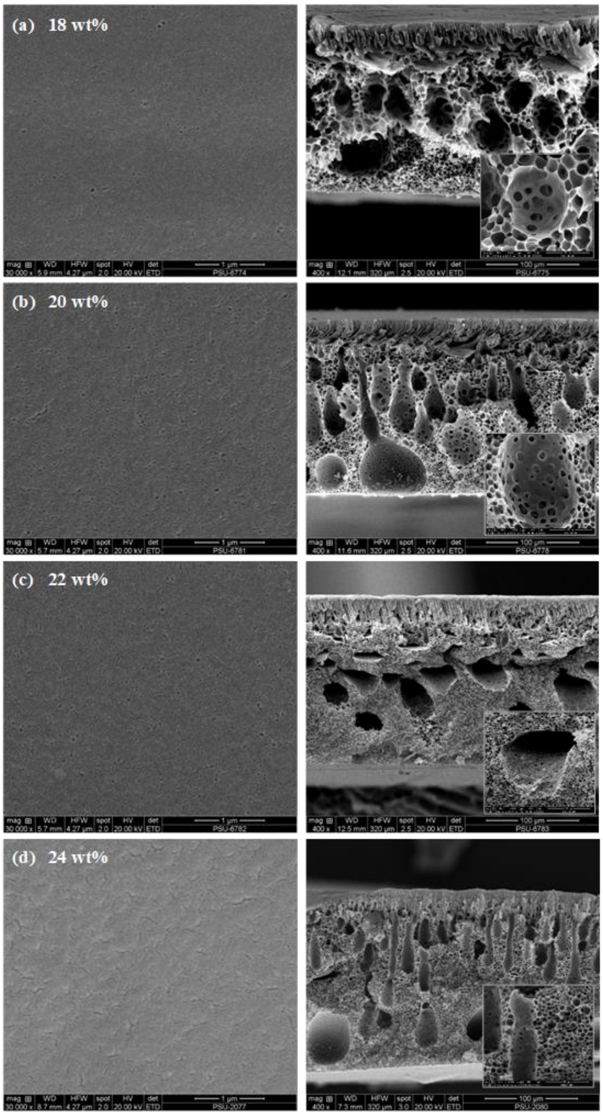

Figure 4. Surface and cross-sectional SEM micrographs of membranes from PC-R casting solutions at concentrations of (a) 18 wt%, (b) 20 wt%, (c) 22 wt% and (d) 24 wt%.

In the sublayers of the asymmetric membranes prepared from casting solutions of 18 and 20 wt% PC-R (Figures 4a and 4b), the numerous large macrovoids were attributed to the lower concentration of the PC-R casting solution: a larger amount of water non-solvent required to solidify the PC-R. This behavior leads to a more porous membrane structure due to the greater amount of non-solvent that diffuses into the cast membrane (Kurdi & Tremblay, 1999). This type of membrane morphology usually provides a high pure water flux. However, in the membranes cast at the higher concentrations of 22 and 24 wt%, the macrovoids were smaller (Figures 4c and 4d) as the tendency to form a sponge-like sublayer increased. At higher concentrations of PC-R, the casting solution was highly viscous (Table 1). Consequently, polymer chain mobility was reduced, resulting in a higher resistance to the coarsening of the polymer-lean phase at the late stage of phase separation (Park, Kim, & Kang, 1999). The effects of polymer concentration on membrane thickness are summarized in Table 1. Usually, the lower the polymer concentration, the thinner the membrane produced (Kurdi & Tremblay, 1999). In the present study, however, due to the large macrovoids present in membranes prepared from the less concentrated casting solutions (Figure 4), membrane thickness did not significantly differ across the range of PC-R concentration.

PC-R concentration, however, did affect the tensile strength and elongation at break of the PC-R membrane. Values for both these properties increased in membranes cast from solutions at higher PC-R concentrations (Figure 5). Membrane morphologies that exhibit larger macrovoids, more porous walls and thinner skin layers are usually indicative of low tensile strength. The present PC-R membranes were derived from low molecular weight PC and, when cast from solutions containing PC-R concentrations of 20, 22 and 24 wt%, these membranes exhibited mechanical properties comparable with those of previously reported asymmetric porous membranes prepared by NIPS from 17.5 wt% of cellulose acetate (CA) (Arthanareeswaran & Thanikaivelan, 2010), 18 wt% of polyethersulfone (PES) (Yu *et al.*, 2013), and 17 wt% of poly(vinylidene fluoride) (PVDF) (Hong & He, 2014). These results support the potential application of the prepared PC-R in the fabrication of polymeric membranes.

It is known that membrane porosity is an important parameter in membrane separation and affects water flux, rejection and mechanical strength of membranes (Jamalludin *et al.*, 2016; Sinha & Purkait, 2013). The porosity of all the prepared membranes was analyzed. The results showed that the PC-R-18 and PC-R-20 membranes had a higher porosity than the PC-R-22 and PC-R-24 membranes (Table 1). Results correlate with the SEM findings shown in Figure 4 and tensile strength data in Figure 5. A similar trend was reported for a membrane prepared from polysulfone modified with rich husk ash (Jamalludin *et al.*, 2016).

Table 1. Effect of PC-R concentration on viscosity of casting solution, porosity and membrane thickness.

Membrane sample	PC-R concentration (wt%)	Viscosity (mPa s)	Porosity (%)	Membrane thickness (μm)
PC-R-18	18	162	69.66 ± 3.76	185.86 ± 4.03
PC-R-20	20	230	68.37 ± 2.78	188.10 ± 1.16
PC-R-22	22	379	62.37 ± 2.00	182.91 ± 1.77
PC-R-24	24	521	62.36 ± 1.26	176.24 ± 2.72

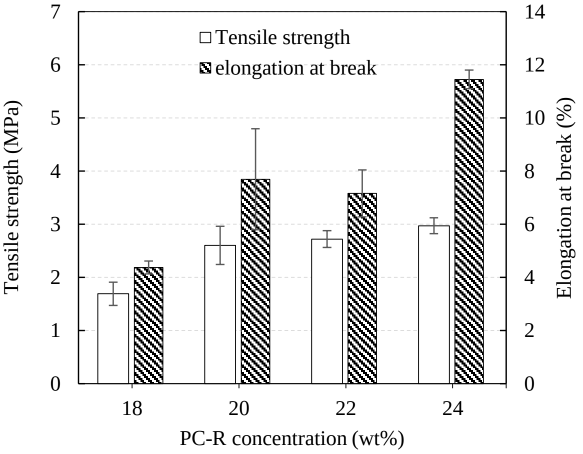

Figure 5. Effect of PC-R concentration on tensile properties of PC-R membranes.

The pure water flux of PC-R membranes was determined under an applied pressure of 1.0 bar at room temperature using a dead-end system with an active surface area of 0.00134 m^2. The morphology of the PC-R-18 membrane (Figure 4a) should have provided the highest pure water flux but the results of filtration testing showed that this occurred through the PC-R-20 membrane (Figure 6). The flux through the PC-R-20 membrane was 90.5% greater than it was through the PC-R-18 membrane. The preponderance of macrovoids in the sublayer of PC-R-18 caused mechanical weaknesses in the membrane (van de Witte, Dijkstra, van den Berg, & Feijen, 1996). Phomdum *et al.* (2018) indicated that the tensile strength of PEGylated poly(ether imide) (PEG-*g*-PEI) was lower than that of neat PEI membrane (UPEI) and that the chance of compaction during filtration was thereby increased, leading to a diminished flux through the PEG-*g*-PEI membrane. It could be implied that the presence of large macrovoids and the low tensile strength of the PC-R-18 membrane caused its pure water flux to be lower than expected. Nevertheless, significantly lower water fluxes were recorded through the PC-R-22 and PC-R-24 membranes. These results were consistent with the morphologies exhibited in Figures 4c and 4d and data for porosity in Table 1. A membrane that had several macrovoids in the sublayer provided higher flux than a membrane that had a sponge-like morphology (Cho, Kim, Nam, & Park, 2011). Fontananova *et al.* (2006) reported that suppressing macrovoid formation by the addition of a concentration of 7.5% LiCl in the polyvinylidene fluoride (PVDF)/ dimethylacetamide dope also reduced permeation flux.

Solute rejection was also evaluated using BSA as the solute model (Figure 6). Rejection depended on the morphological structure of the membrane, involving both the top layer and sublayer (Sinha & Purkait, 2013). A sponge-like morphology had generally low flux and high solute rejection (Guillen, Pan, Li, & Hoek, 2011). The results in Figure 6 showed that protein rejection tended to increase as the polymer concentration increased. This correlation occurred because of the resistance of the dense, sponge-like structure of the membranes (Sinha & Purkait, 2013). The PC-R-24 membrane showed the highest BSA rejection. This might be

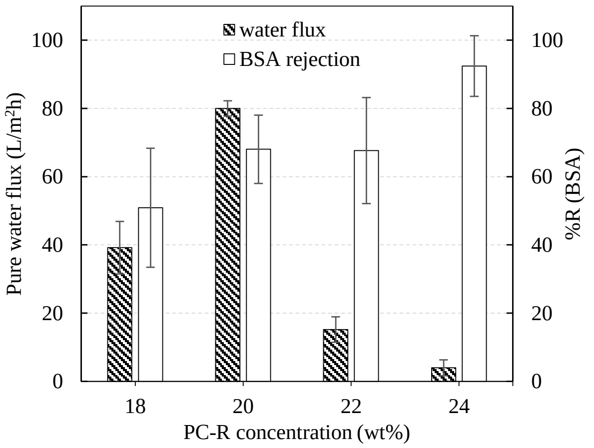

Figure 6. Effect of PC-R concentration on pure water flux and BSA rejection of PC-R membranes.

because the high concentration of polymer in the casting solution tended to increase the strength of the polymer first precipitated during phase inversion. The strength of the first precipitated layer produced a membrane with a less porous top layer (Strathmann, Kock, Amar, & Baker, 1975).

4. Conclusions

Asymmetric porous polycarbonate membranes derived from waste compact discs were successfully prepared using non-solvent induced phase separation. The coating layers on the CDs were removed by mechanical abrasion and the recovered PC (PC-R) was purified following a precipitation method. The effect of PC-R concentration on the membrane morphologies and the correlation of the morphologies to mechanical and permeation properties were investigated. SEM images revealed that, regardless of the concentration of the casting solution, the PC-R membranes consisted of an upper skin layer and a finger-like structure or marcovoid sublayer. At lower PC-R solution concentrations of 18 and 20 wt%, large numbers of large macrovoids were generated in the sublayer. In the membranes cast from solutions of 20 and 22 wt% PC-R, the macrovoid sublayer was suppressed as a sponge-like sublayer was generated. The SEM results were consistent with the improvement in tensile properties and %R of the membranes and deterioration in pure water flux. The results indicate that different PC-R concentrations could be used to prepare membranes with different behaviors in terms of pure water flux and BSA rejection. From the results of our study, we conclude that PC-R derived from cheap, abundant waste CDs offers an alternative material for the preparation of porous membranes for use in ultrafiltration applications.

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