Preparation of poly(butylene succinate)/polyvinylpyrrolidone blend membrane for pervaporation dehydration of acetone

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ABSTRACT

In this research, polybutylene succinate (PBS) was utilized in the fabrication of pervaporation (PV) membranes, and novel PBS/polyvinylpyrrolidone (PVP) blend membranes were prepared and assessed for the dehydration of acetone. The influence of PVP concentration on membrane morphology, chemical structure, relative hydrophilicity/hydrophobicity, and mechanical properties was studied through field-emission scanning electron microscopy (FESEM), attenuated total reflectance Fourier-transform infrared (ATR-FTIR) spectroscopy, measurement of water contact angle, and tensile test, respectively. In addition, the thermal properties of the PBS and PBS/PVP membranes were compared via thermogravimetric analysis (TGA). Finally, membrane swelling was determined, and the performance of the resultant PBS/PVP membranes in dehydration of acetone/water mixture was compared via the determination of acetone and water fluxes, total flux, separation factor, and pervaporation separation index (PSI). The obtained results confirmed the increased hydrophilicity, swelling, and tensile strength of the PBS/PVP blend membranes compared to that of the neat PBS one. Moreover, blending PBS with PVP increased the total flux, separation factor, and PSI of the resultant membranes. Using 3 wt.% PVP in the polymeric solution augmented the mentioned parameters by about 3, 6, and 21 fold, respectively, compared to those of the pure PBS membrane.

1. Introduction

Acetone is extensively used in the rubber, leather, fibers, plastics, explosives, paint, oil, and pharmaceutical industry, but it is reported that the dehydration of acetone generated in the mentioned industries is an important issue (Li et al., 2012). Although it does not form an azeotrope with water, it forms a tangent pinch point with water, and thus, the production of high-purity acetone through simple distillation not only demands high instantaneous reflux ratios but also require a remarkably high number of theoretical trays (Amelio et al., 2018). A pinch point is reported at low water concentrations for the acetone-water mixture, which shows that the relative volatility is close to unity near the pinch point (Kiss, 2013). Pervaporation (PV) is an alternative to distillation for mixtures with tangent pinch point because it is based on the differences in diffusivity and solubility and not the vapor-liquid equilibrium (Barakat and Sorensen, 2006).

Membranes are widely applied in the purification and separation processes owing to their high efficiency and low energy requirement (Jose et al., 2018). As one of the most recent membrane separation technologies, the PV process is more eco-friendly and energy-efficient in comparison with the distillation process and doesn't need the addition of chemicals (Cheng et al., 2017; Mousavinezhad et al., 2020). Also, when compared to the conventional separation processes such as distillation and evaporation, PV provides a higher-quality product as well

Nomenclature

A	Effective membrane area
ATR-FTIR	Attenuated total reflectance Fourier-transform infrared
CA	Cellulose acetate
CBCM	Composite bacterial cellulose membrane
CS	Chitosan
DS	Degree of swelling
DTG	Derivative thermogravimetric
EDA	Ethylenediamine
ETBE	Ethyl tert-butyl ether
FESEM	Field-emission scanning electron microscopy
GA	Glutaraldehyde
HEMA	2-hydroxyethyl methacrylate
IPA	Isopropanol
J	Permeation flux
l	Membrane thickness
MA	Maleic anhydride
M_d	Weight of the dry membrane sample
M_s	Weight of the swollen membrane sample
MTBE	Methyl tert-butyl ether
MWCNT	Multi-walled carbon nanotube
NaAlg	Sodium alginate
NMP	N-methyl-2-pyrrolidone
P84	Co-polyimide of 3,30,4,40-ben zophenone tetracarboxylic dianhydride (BTDA) and 80% toluene diisocyanate (TDI)+20% methylene diphenyl diisocyanate (MDI)
P_i^G and P_j^G	Gas-based permeabilities
p_{io}^{sat}	Vapor pressure of pure component i
p_l	Total pressure in the permeate side
PAN	Polyacrylonitrile
PBS	Polybutylene succinate
PEG	Polyethylene glycol
PES	Polyethersulfone
PLA	Polylactic acid
PMA	Phosphomolybdic acid
PPO	Poly(phenylene oxide)
PPSU	Polyphenylsulfone
PSI	Pervaporation separation index
PV	Pervaporation
PVA	Polyvinyl alcohol
PVC	Poly(vinyl chloride)
PVDF	Polyvinylidene fluoride
PVP	Polyvinylpyrrolidone
Q	Mass of the permeate
SCMS	Sulfonated carbon molecular sieve
T	Duration of the PV experiment
TAEA	Tris(2-aminoethyl)amine
T_{d5}	Temperatures at which 5 wt.% of the samples degrade
T_{dmax}	Temperature at which the highest rate of decomposition occur
TGA	Thermogravimetric analysis
THF	Tetrahydrofuran
x_i	Weight fraction of water in the feed
x_j	Weight fraction of acetone in the feed
y_i	Weight fraction of water in the permeate
y_j	Weight fraction of acetone in the permeate
α	Separation factor
γ_{io}^L	Activity coefficient of component i in the liquid feed

as higher energy and cost efficiency because of the utilization of mild conditions (temperature and pressure) (Rezakazemi et al., 2018). PV applications include dehydration of organic solutions as well as the separation of organics from water and separation of organics from organic mixtures (Manshad et al., 2017). However, market studies show that the majority of commercial PV plants are utilized in the dehydration of organic liquids (Jyoti et al., 2015). PV is currently utilized successfully for the dehydration of highly concentrated organic solvents (Huang et al., 2019). The dehydration of various organic solvents such as isopropanol (IPA) (Huang et al., 2019; Raeisi et al., 2019), ethanol (Dudek et al., 2019), and other organics through PV was studied in the previous literature.

Among different membrane materials, polymers have aroused considerable interest (Matavos-Aramyan et al., 2020). Polymers are primarily used for the fabrication of PV membrane materials because they are easily processed and possess good mechanical stability and adjustable transport properties (Cheng et al., 2017). However, the disposal of plastic waste results in environmental contamination (Nair et al., 2018). The adverse effect of polymers on the environment, as a controversial issue, has been addressed using biodegradable alternatives in recent years, among which is polybutylene succinate (PBS), a semicrystalline polyester with balanced mechanical and outstanding thermal characteristics (Xu and Guo, 2010a,b). An essential feature of biodegradable polymers is their compostability, i.e., these materials will decompose mostly into CO_2 and water after their disposal (Nair et al., 2018). PBS is biodegradable in moist soil, activated sludge, compost, water with activated sludge, and also in the sea (Xu and Guo, 2010b; Fujimaki, 1998). The application of PBS in the preparation of biodegradable filtration membranes has been studied so far in a few numbers of researches (Ghaffarian et al., 2016, 2013; Bahremand et al., 2017). These researches were conducted on blending PBS with polyethersulfone (PES)/polyethylene glycol (PEG) (Ghaffarian et al., 2016), cellulose acetate (CA) (Ghaffarian et al., 2013), and CA/dextran (Bahremand et al., 2017) to prepare biodegradable membranes for filtration of aqueous media.

An ideal PV membrane has the combined characteristics of high permeation and good selectivity (Nawawi, 1997; Jose et al., 2014). According to the solution-diffusion mechanism, the permeability and selectivity of a membrane are controlled by the solubility (sorption) of components and, thus, by the affinity of components to the membrane material (Feng and Huang, 1997). In other words, a high affinity of the species to the membrane material results in their adsorption and diffusion through the membrane, while their low affinity is detrimental to their transport (Jyoti et al., 2015). In the PV dehydration of organic solvents, high solubility and diffusivity of water in the polymeric membrane are essential (Swapna et al., 2019a). Consequently, enhanced membrane performance in PV dehydration can be obtained via the modification of polymeric membranes through a variety of approaches such as copolymerization, grafting, cross-linking, and blending (Feng and Huang, 1997).

Among various modification methods, polymer blending has gained interest from both the scientific and industrial points of view (Nawawi, 1997). It not only provides the intended physical and mechanical characteristics for the blend but also is an economical alternative to the synthesis of new polymers (Mural et al., 2018). As a non-toxic, chemically stable material that is also compatible with various compounds, polyvinylpyrrolidone (PVP) is one of the best polymeric additives that can be utilized in the preparation of membranes for water and wastewater treatment, food processing, and gas separation (Teodorescu and Bercea, 2015). The incorporation of PVP into the PV membranes studied by several researchers in the PV separation of organic-organic mixtures (Wu et al., 2008; Zereshki et al., 2011, 2010) or PV dehydration of

Table 1 – Summary of the literature reported on the application of PVP in preparation of PV membranes.		
Separation field	Membrane material	Detailed application
Separation of organic-organic mixtures	Cellulose acetate (CA)/PVP Wu et al. (2008)	Separation of methanol/Methyl tert-butyl ether (MTBE)
	Polylactic acid (PLA)/PVP Zereshki et al. (2011)	Separation of ethanol/Ethyl tert-butyl ether (ETBE)
	PLA/PVP Zereshki et al. (2010)	Separation of ethanol/cyclohexane
Dehydration of organic-water mixtures	Polyvinyl alcohol (PVA)/PVP/phosphomolybdic acid (PMA) Magalad et al. (2010)	Dehydration of ethanol
	Chitosan (CS)/PVP Anjali Devi et al. (2006)	Dehydration of tetrahydrofuran (THF)
	CS/PVP Zhang et al. (2009)	Dehydration of ethyl acetate/ethanol
	PVA/PVP/PMA Mali et al. (2011)	Dehydration of IPA
	Polyacrylonitrile (PAN)/PVP Nguyen et al. (1985)	Dehydration of THF and ethanol
	Sodium alginate (NaAlg)/PVP on PAN support Zhu et al. (2010)	Dehydration of caprolactam

organic-water mixtures (Magalad et al., 2010; Anjali Devi et al., 2006; Zhang et al., 2009; Mali et al., 2011; Nguyen et al., 1985; Zhu et al., 2010) is summarized in Table 1. It is reported that the affinity of PVP towards both hydrophobic and hydrophilic compounds, together with its strong polar character and hydrophilicity, has made it be considered as a proper additive (Teodorescu and Bercea, 2015).

Zhang et al. (2009) prepared chitosan (CS)/PVP blend membranes for the PV dehydration of ethyl acetate/ethanol. They indicated that the hydrophilicity of the membranes, and thus, the permeation flux was enhanced via blending CS with PVP. In another study on PV dehydration of tetrahydrofuran (THF) using the blend membranes of CS/PVP, blending CS with PVP increased the water permeability and selectivity of the membrane while the membrane crystallinity reduced (Anjali Devi et al., 2006). Zhu et al. (2010) demonstrated that the addition of PVP to the sodium alginate (NaAlg) selective layer enhanced the hydrophilicity of polyacrylonitrile (PAN)/NaAlg composite membrane, and therefore, was beneficial to the permeation flux and separation factor during caprolactam dehydration.

According to the aforementioned advantages of PBS, the objective of the present study was the preparation of biodegradable PBS PV membranes for the dehydration of organic-water mixture. Since the positive influence of PVP introduction into the polymer matrix on PV dehydration performance of the blend membrane was reported in some previous researches, the PBS membrane performance was enhanced for the first time through blending with PVP. Morphology, chemical structure, relative hydrophilic/hydrophobic property, swelling, and mechanical properties of the membranes were studied in detail. Moreover, the influence of PVP on the thermal degradation behavior of the PBS membrane was investigated via thermogravimetric analysis (TGA). Furthermore, PV dehydration of acetone was conducted to compare the performance of the membranes.

2. Materials and methods

2.1. Materials

Poly(1,4-butylene succinate) (PBS) extended with 1,6-diisocyanatohexane (density = 1.3 g/mL and T_m = 120 °C) and polyvinylpyrrolidone (PVP) K 25 (average Mw~24,000) were purchased from Sigma-Aldrich. The chemical structure of these two polymers, which was reported by the supplier, is shown in Fig. 1. N-methyl-2-pyrrolidone (NMP) (density = 1.03 g/mL and purity≥99.5%) and acetone (density = 0.784 g/mL and purity≥99%) were obtained from Daejung and Dr. Mojalali Industrial Chemical Complex Co., respectively.

2.2. Membrane preparation

PBS solution was obtained by dissolving 15.5 wt.% PBS in NMP via stirring at 70 °C for three hours. Blend PBS/PVP casting

Poly(1,4-butylene succinate), extended with 1,6-diisocyanatohexane

Polyvinylpyrrolidone
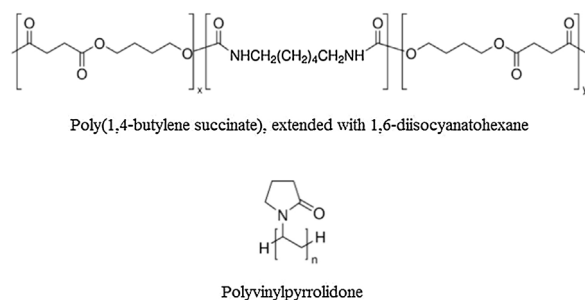
Fig. 1 – Chemical structure of polymers utilized in this study.

solutions were obtained by the addition of a specified amount of PVP in previously obtained PBS solution followed by stirring at 70 °C for another three hours. The concentration of PBS in the resultant solutions was 15.5 wt.% while the PVP concentration was varied at 0, 1, 2, and 3 wt.%. Since one of the aims of this research was to benefit from the biodegradability of PBS, higher PVP concentrations were not utilized. Degassing of the solutions was done by leaving them stagnant for two hours at 70 °C.

After the preparation of the solutions, they were subsequently cast onto glass plates utilizing a film applicator at 250 μm. Before the casting, the film applicator and the glass plate were heated in an (KM-55, Pars Azma, Iran) at 80 °C for 20 min. The resultant film, together with the glass plate, was placed in the oven at 80 °C for 1.5 h for solvent evaporation.

### 2.3. Membrane characterization

2.3.1. Morphological studies

The surface and cross-section of the obtained membranes were studied in terms of their morphology through field-emission scanning electron microscopy (FESEM) using MIRA3-FEG TESCAN apparatus (Czech Republic) after being sputtered with Au particles in a Q150R ES Quorum coater (England). The membranes' cross-section was analyzed after the samples were fractured in liquid nitrogen.

2.3.2. ATR-FTIR spectroscopy

The membrane chemical structure was investigated via attenuated total reflectance Fourier-transform infrared (ATR-FTIR) spectroscopy. The spectrums of PVP, the PBS membrane, and PBS/PVP blend membranes were collected in the wavenumber range of 650–4000 cm^{-1} using a Thermo Nicolet spectrometer
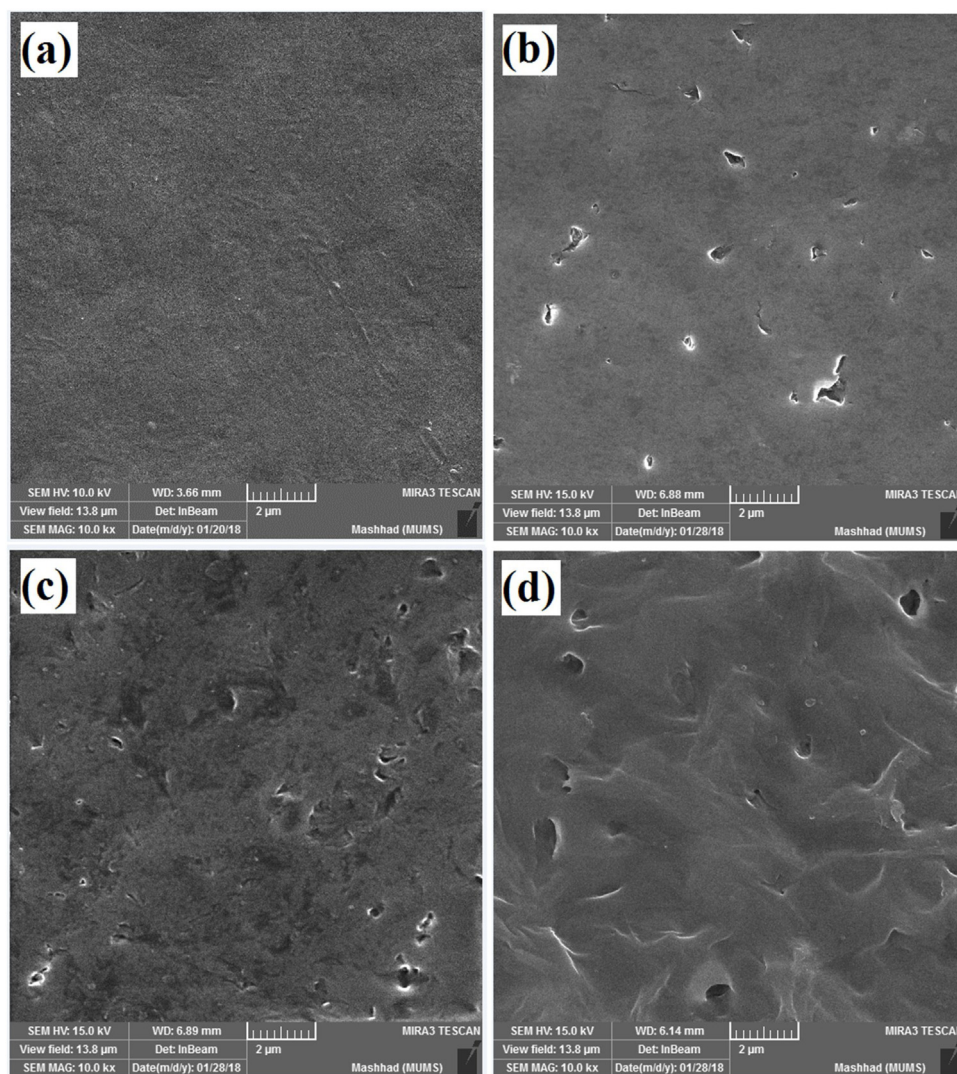
Fig. 2 – Top surface FESEM images of the PBS/PVP membranes versus PVP concentration: a) 0 wt.%, b) 1 wt.%, c) 2 wt.%, and d) 3 wt.%.

(Avatar 370, USA). For each membrane sample, 16 scans with a resolution of 4 cm^{-1} were recorded in transmission mode.

2.3.3. Determination of water contact angle

The water contact angle of the PBS membrane and the PBS/PVP membranes containing various amounts of PVP was measured at room temperature by taking the optical images of 10 μL water droplets immediately after they were put on the membrane surface. The images were taken using a camera and a stereomicroscope (Olympus DP71 and Olympus SZH10, respectively). The test was repeated four times for each sample, followed by the analysis of the images and reporting the average value of the measurements.

2.3.4. Investigation of membrane swelling

The swelling degree of the PBS and PBS/PVP membranes was studied according to the following procedure: membrane sample was dried and weighted (M_d) using an electronic balance and then submerged in acetone/water mixture at 85/15 wt ratio at room temperature. The sample was taken out of the solution every day and weighted after its surface liquid was removed using a filter paper (M_s). Repeating the mentioned procedure for four days confirmed that a constant degree of swelling was reached. The following equation was used for

calculation of the degree of swelling (DS): (Sardarabadi et al., 2016)

$$DS\,(\%) = \frac{M_s - M_d}{M_d} \times 100 \qquad (1)$$

### 2.3.5. Determination of membrane mechanical properties

The tensile mechanical test was performed on rectangular strips of PBS and PBS/PVP membranes with 50 mm gauge length (based on ASTM D882 standard) using an STM-20 Zwick (Korea) universal equipment. During the test, 70 mm × 5 mm samples were stretched at 1 mm/min crosshead speed.

2.3.6. TGA

Thermostability of the PVP additive, PBS membrane, and the PBS/PVP membrane obtained by blending the casting solution with 3 wt.% PVP was assessed by conducting the thermogravimetric analysis (TGA) on a TGA1 METTLER TOLEDO (Switzerland) thermogravimetric analyzer. The samples were heated at a rate of 10 °C/min from 25 to 800 °C under 50 ml/min nitrogen flow.

2.3.7. PV experiments

The schematic of the PV experimental set-up which was utilized in the current study can be seen in our previous paper
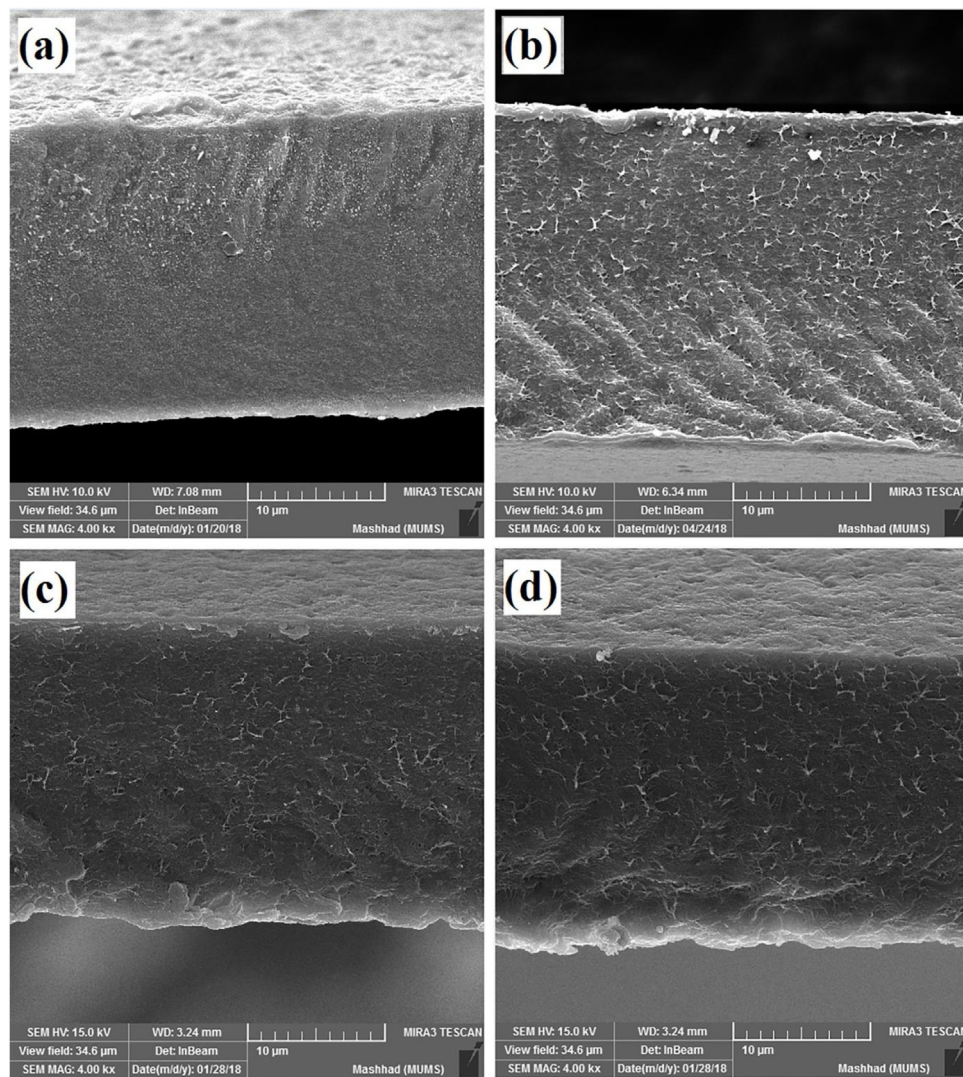
Fig. 3 – Cross-sectional FESEM images of the PBS/PVP membranes versus PVP concentration: a) 0 wt.%, b) 1 wt.%, c) 2 wt.%, and d) 3 wt.%.

(Mousavinezhad et al., 2019). For PV dehydration of acetone, 500 g of the solution containing acetone/water mixture at 85/15 wt. ratio was poured into the feed tank and pumped into the PV cell. The membrane was a circular sample with an effective surface area of 19.6 cm^2. The feed passed in the upstream at 4 L/min flow rate, atmospheric pressure, and 25 °C temperature and circulated to the feed tank. The feed components permeated through the membrane, while their evaporation took place at the downstream at 18 mmHg vacuum pressure, which was induced using a vacuum pump. The cold trap, which was placed in a liquid nitrogen reservoir, was utilized for the collection of permeate. The duration of the PV process was one hour. The permeate weight was calculated considering the cold trap weight before the experiment and also at the end of it. Acetone concentration in the permeate was assessed via ATAGO (Japan) refractometer with the detection limit of 1.3306−1.5284.

To compare the performance of the PBS and PBS/PVP membranes in the PV process, permeation flux (J) and separation factor (α) were determined via the following equations: (Najafi et al., 2018)

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

$$\alpha_{i,j} = \frac{y_i/y_j}{x_i/x_j} \tag{3}$$

where Q is the mass of the permeate, A is the effective membrane area, t is the duration of the experiment, and x and y are the weight fractions of components in the feed and permeate, respectively. Moreover, i and j refer to water and acetone, respectively. A value of unity for the separation factor means that no separation occurs; when the separation factor approaches infinity, the membrane is regarded as perfectly semipermeable (Feng and Huang, 1997).
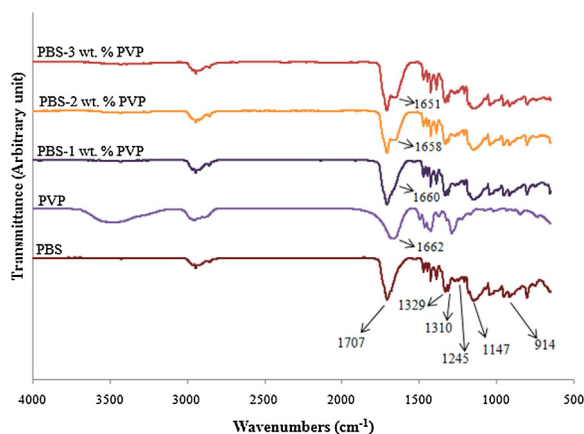
PV separation index (PSI) was calculated using the predetermined permeation flux and separation factor: (Najafi et al., 2018)

$$PSI = J(\alpha - 1) \tag{4}$$

The following equations were applied to study the membrane performance in terms of permeances (P_i^G/l) and selectivity: (Villegas et al., 2015)

$$\frac{P_i^G}{l} = \frac{j_i}{\gamma_{io}^L x_i p_{io}^{sat} - y_i p_l} \tag{5}$$

Fig. 4 – ATR-FTIR spectrums of PVP, PBS membrane, and PBS/PVP blend membranes.

$$Selectivity_{ij} = \frac{P_i^G}{P_j^G} \tag{6}$$

where P_i^G and P_j^G are the gas-based permeabilities, l is the membrane thickness, γ_{io}^L is the activity coefficient of component i in the liquid feed, p_{io}^{sat} is the vapor pressure of pure component i, and p_l is the total pressure on the permeate side. Activity coefficients were estimated using the NRTL equation.

3. Results and discussion

3.1. Membrane morphology

Figs. 2 and 3 illustrate the FESEM images of the membranes' top surface and cross-section, respectively.

According to the images, the pure PBS membrane had a dense morphology on its top surface and also in its cross-section. However, the addition of PVP to the PBS membrane resulted in the formation of pores on the membrane surface. As can be seen in Fig. 3, the cross-section of the PBS/PVP membranes was still non-porous, and thus, these membranes were still structurally suitable for the PV process (as reported in Section 3.7). The formation of the mentioned pores might be due to the absorption of a small amount of water from the environment by the molecules of PVP, and their demixing from the base polymer during the drying process (Zereshki et al., 2010).

3.2. Chemical structure

ATR-FTIR spectroscopy was utilized for investigation of the chemical structure of the PVP additive, the pure PBS membrane, and PBS/PVP blend membranes. The obtained ATR-FTIR spectrums are shown in Fig. 4.

Regarding Fig. 4, the pure PBS membrane exhibited the characteristic peaks of PBS polymer. Concerning the literature (Pérez et al., 2017), the peak at 1707 cm^{-1} is associated with the carbonyl group in PBS, and the bands at 1329 and 1310 cm^{-1} are attributed to the CH$_2$ groups in the main chain of PBS. The peaks at 1147 and 1245 cm^{-1} are assigned to the –COC-stretching bonds in the ester group, while the peak at 914 cm^{-1} is due to vinyl groups (Pérez et al., 2017).

With respect to the results, the addition of 1, 2, and 3 wt.% PVP to PBS resulted in the appearance of a new peak at 1660, 1658, and 1651 cm^{-1}, respectively. As can be seen, The same peak was detected in the spectrum of PVP, which is due to its amide group (Dinari and Mallakpour, 2014). In a study by

Zereshki et al. (2010), the appearance of a new absorption peak at 1655 cm^{-1} was spot as indicative of the presence of PVP in the PLA/PVP membrane.

3.3. Water contact angle

Fig. 5 displays the water contact angles of the membranes in terms of the concentration of PVP in the casting solution. It also provides the images of water droplets dispensed on the membrane surface. According to the literature, the surface with a water contact angle much less than 90 °C is known as a hydrophilic one (Swapna et al., 2019b). Therefore, Fig. 5 shows that the surface of all the obtained membranes was hydrophilic. In addition, the highest contact angle was obtained for the neat PBS membrane at 63.7°. A lower water contact angle is an indication of improved wettability and hydrophilicity of the membrane (Gopakumar et al., 2019). Thus, the neat PBS membrane represented the lowest hydrophilicity.

According to Fig. 5, the reduction of the water contact angle was observed with the addition of 1 wt.% PVP to the PBS membrane and also with increasing its concentration to 2 and then 3 wt.%. For instance, the membrane prepared at 1 wt.% PVP exhibited a water contact angle of 59.4°, which is 4.3° smaller than that of the pure PBS membrane, i.e., an almost 7% decrease. Also, blending the PBS casting solution with 3 wt.% PVP led to a decrease of almost 19° in water contact angle value compared to that of the PBS membrane, i.e., an almost 29% decrease. The observations implied the higher hydrophilicity of the blend membranes compared to that of the neat PBS membrane. The obtained results are compatible with the results of a previous study in which the addition of PVP to the polymeric membrane decreased the water contact angle and thus, increased the membrane hydrophilicity (Zereshki et al., 2010). PVP is generally considered a strongly hydrophilic additive (Zereshki et al., 2010), and the higher hydrophilicity of the blend membranes was mainly attributed to the increased number of free polar carbonyl groups in blend membrane, which form hydrogen bonds with water molecules (Zhu et al., 2010).

3.4. Swelling degree

Membrane samples were immersed in acetone/water mixture containing 15 wt.% water and their swelling degree was determined for 4 days until a constant value was obtained. Fig. 6 shows the variations in the membrane swelling degree in terms of PVP concentration in the casting solution.

With respect to Fig. 6, the pure PBS membrane exhibited the lowest swelling degree at 5.9%. Moreover, compared to that of the pure PBS membrane, the blend membranes experienced a higher degree of swelling, which increased with augmenting the PVP concentration. For example, the swelling degree of the membrane prepared at 1 wt.% PVP was 7.3%, which reveals that blending PBS with a small amount of PVP augmented the degree of swelling. Moreover, the swelling degree of the PBS/PVP membrane prepared at 3 wt.% PVP increased by 2.7 times compared to that of the pure PBS membrane and was determined as the highest value among that of the other samples.

The obtained results can be explained as follows: PVP is known to be a strongly hydrophilic additive (Zereshki et al., 2010). The results of the determination of membrane water contact angles in Section 3.3 revealed that the mem-
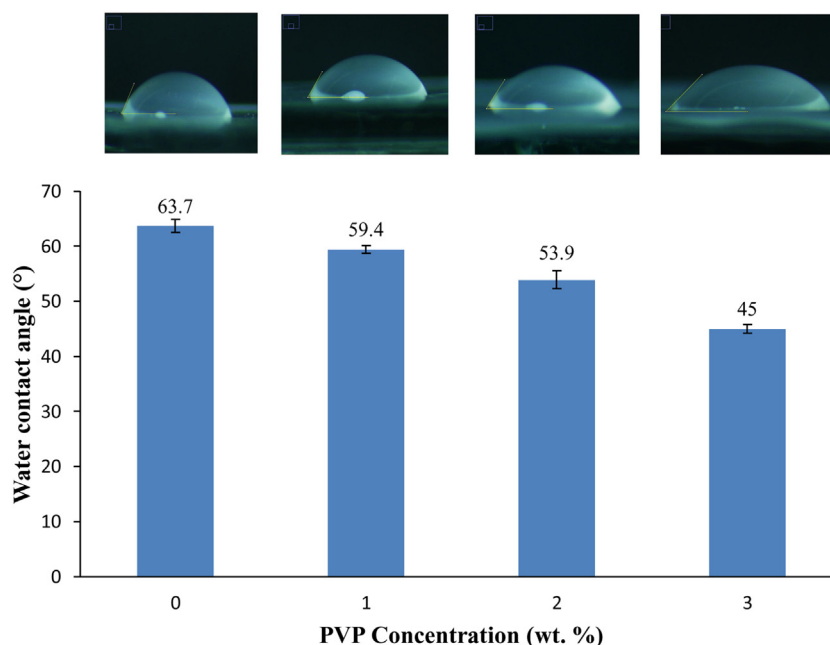
Fig. 5 – Water contact angles of the PBS/PVP membranes and images of corresponding water droplets versus PVP concentration.
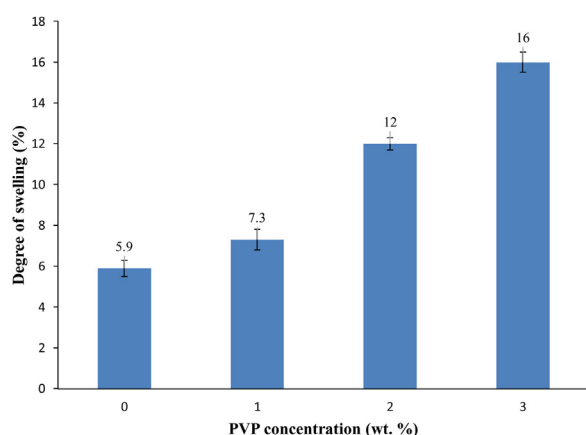

Fig. 6 – Swelling degree of the PBS/PVP membranes versus PVP concentration.

Fig. 7 – Stress-strain curves of the PBS/PVP membranes versus PVP concentration: (a) 0 wt.%, (b) 1 wt.%, (c) 2 wt.%, and (d) 3 wt.%.

brane hydrophilicity increased with PVP addition as well as increasing PVP concentration. It is also reported that a more hydrophilic membrane is more selective towards polar molecules (Zereshki et al., 2010). Hydrogen bonding is formed between the free carbonyl group of PVP and water molecules, and thus, the membrane swells more easily (Zhu et al., 2010). Consequently, a higher membrane swelling is inevitable at increased concentrations of PVP.

3.5. Mechanical properties

Fig. 7 illustrates the stress-strain curves for the PBS and PBS/PVP membranes. In addition, the mechanical properties obtained by analyzing the mentioned curves are tabulated in Table 2.

It was found that the addition of PVP to the PBS casting solution increased the tensile strength of the membrane. Moreover, higher PVP concentrations led to the same result. However, the elongation at the break point had a completely different trend. PVP addition contributed to a drop in the membrane elongation. Furthermore, increasing the concentration of PVP was accompanied by a further reduction of elongation.
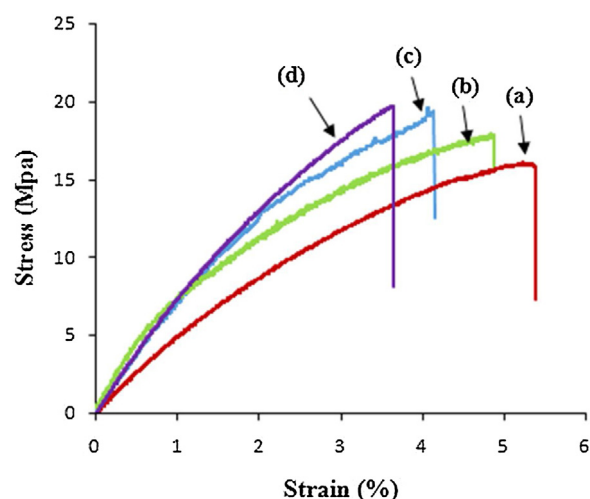
Table 2 – Tensile mechanical properties of the PBS and PBS/PVP membranes.

Membrane	Tensile strength (MPa)	Elongation at break (%)
Pure PBS	15.5 ± 0.5	5.4 ± 0.2
PBS-1 wt.% PVP	17.6 ± 0.3	4.9 ± 0.2
PBS-2 wt.% PVP	19.3 ± 0.1	4.1 ± 0.1
PBS-3 wt.% PVP	19.6 ± 0.2	3.6 ± 0.2

It means that the pure PBS membrane represented the lowest tensile strength and highest elongation at break, at 15.5 MPa and 5.4%, respectively, and the membrane prepared at 3 wt.% PVP exhibited the highest tensile strength and lowest elongation at break, at 19.6 MPa and 3.6%, respectively. The interpretation of the results is only possible by taking the inherent characteristics of PBS and PVP into account.

In the first step, PBS is a macromolecular plasticizer (Boonmahitthisud et al., 2014). Its plasticizing effect on
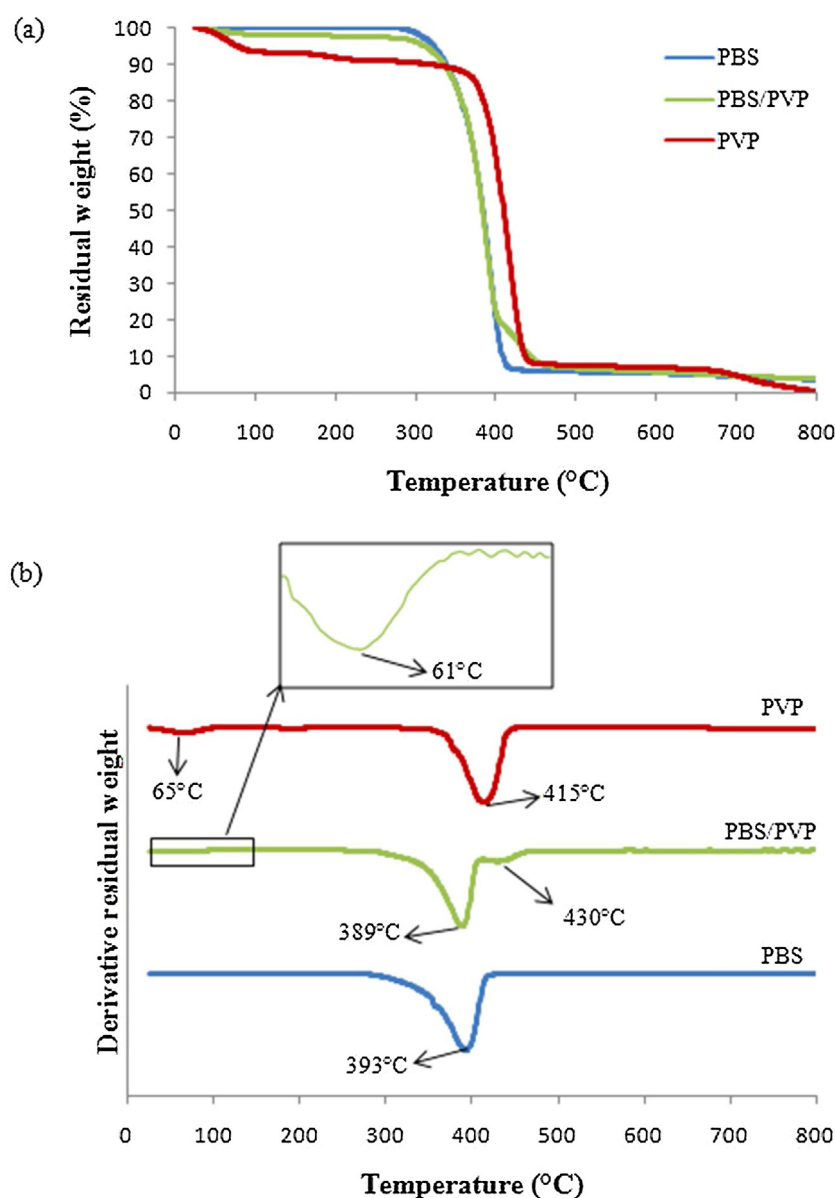
Fig. 8 – Thermal decomposition behavior of the PVP additive, PBS membrane, and PBS/PVP membrane prepared at 3 wt.% PVP: (a) TGA and (b) DTG.

PLA/PBS blends as well as poly(vinyl chloride) (PVC)/PBS blends was reported elsewhere (Hassan et al., 2013; Gao et al., 2016a). On the other hand, PVP is known to function as an anti-plasticizer (Wen et al., 2005) to the extent that the incorporation of plasticizers was proposed for controlling the brittleness of PVP (Teodorescu and Bercea, 2015).

It is widely known that the movement of polymeric chains is enhanced with the addition of a plasticizer to the polymer structure (Kiani et al., 2018). A drop in tensile strength together with higher elongation, was previously observed via blending polyphenyl sulfone (PPSU) with PEG as a plasticizer (Kiani et al., 2015). Moreover, the results of previous literature on the characterization of sheets prepared using PVC/PBS blends indicated that with the increasing PBS content, the tensile strength decreased while the elongation at break increased (Gao et al., 2016a). This observation was explained by the improved flexibility of PVC sheets through PBS addition (Gao et al., 2016a).

According to the explanations mentioned above, it could be concluded that blending PBS as a plasticizer (Boonmahitthisud et al., 2014) with PVP as an anti-plasticizer (Wen et al., 2005)

resulted in decreased flexibility of the resultant membrane and thus, a higher tensile strength in addition to lower elongation at break.
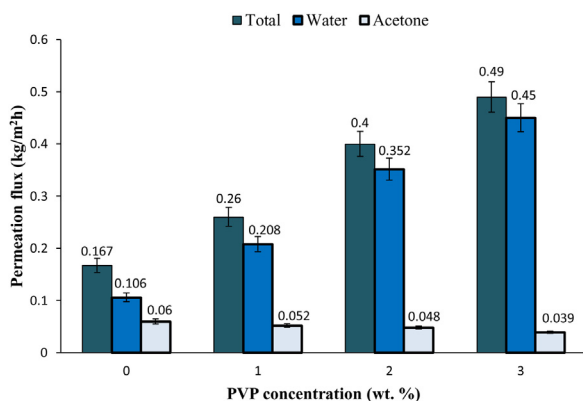
3.6. Thermal stability

Thermal stability of the PVP additive, PBS membrane, and PBS/PVP membrane prepared by blending the polymeric solution with 3 wt.% PVP was studied via performing TGA. Fig. 8 shows the corresponding variation of residual weight and derivative residual weight with temperature through the TGA and derivative thermogravimetric (DTG) curves, respectively. The thermal properties of the PVP additive, PBS membrane, and PBS/PVP membrane are tabulated in Table 3. In Table 3, T_{d5} is associated with the temperatures at which 5 wt.% of samples degrade while T_{dmax} corresponds to the temperature at which the highest rate of decomposition (peaks in the DTG curves) takes place (Phua et al., 2013).

Concerning Fig. 8, PBS underwent a single-step decomposition process, which is consistent with the previous literature (Hassan et al., 2013). Moreover, its weight loss was insignificant

Table 3 – Thermal properties of PVP, the PBS membrane, and the PBS/PVP membrane obtained from TGA curves.

Membrane	T_{d5}(°C)	T_{dmax}(°C)	Char yield(wt.%)
PBS	322	393	3.48
PVP	77	65, 415	0.20
PBS-3 wt.% PVP	312	61, 389, 430	3.72

Fig. 9 – Total permeation flux and water and acetone permeation fluxes through the PBS/PVP membranes versus PVP concentration.

(1.29%) at 300 °C, which shows a relatively good thermal stability of the PBS membrane (Abderrahim et al., 2015). Regarding Table 3, the highest rate of PBS decomposition (T_{dmax}) was located at 393 °C, which is in agreement with previously reported data (Abderrahim et al., 2015).

In the TGA curve of the PVP additive, two steps are observed with the highest rates at 65 and 415 °C (Table 3), respectively. Dinari and Mallakpour (2014) reported that the two-step decomposition process in the TGA curve of PVP is attributed to the decomposition of the carbonic backbone and the pyrrolidine ring, respectively. Fig. 8 indicates a three-step TGA curve for the PBS/PVP sample with the highest rates at 61, 389, and 430 °C, respectively. The peaks at 61 and 430 °C may be ascribed to PVP decomposition while the peak at 389 °C is assigned to PBS degradation, as mentioned above. A comparison of PBS and PBS/PVP decomposition results reveals that PBS exhibited higher thermal stability since the decomposition initiated at 322 °C for PBS while it started at 312 °C for the PBS/PVP sample. In addition, in the DTG curve, the peak associated with PBS decomposition moved to a lower temperature with the introduction of PVP. This result, which shows a destabilizing effect (Márquez et al., 2012), was previously observed with the addition of PVP to CS membranes (Zhang et al., 2009).

3.7. PV performance

The performance of the obtained PBS and PBS/PVP membranes was studied via PV dehydration of acetone/water mixture. The influence of PVP on the PV performance of the prepared membranes can be investigated by comparing the individual flux variation of acetone and water at various PVP concentrations (Fig. 9).

With respect to Fig. 9, the water flux through all of the membranes was noticeably higher than that of acetone. Moreover, it was found that the introduction of PVP to the polymer matrix increased the water flux.

Membrane swelling during PV may lead to the flexible membrane structure with higher free volume through which the permeation of all feed components might be enhanced
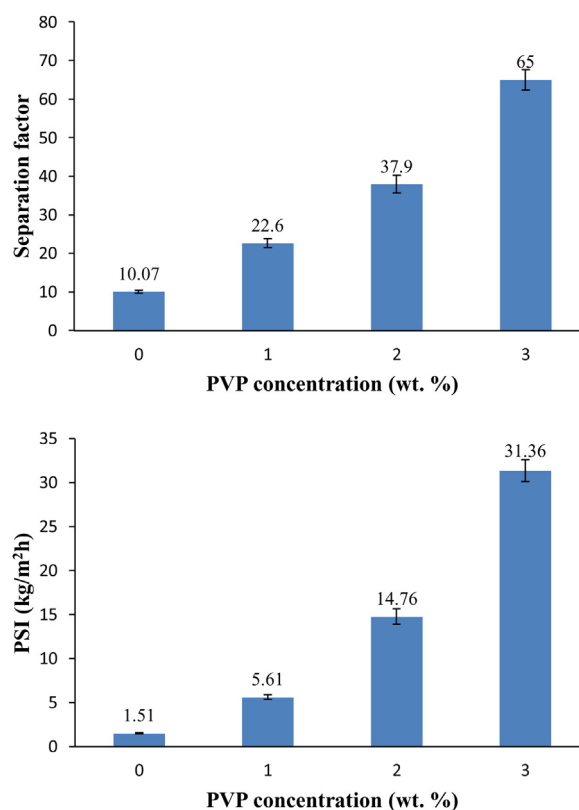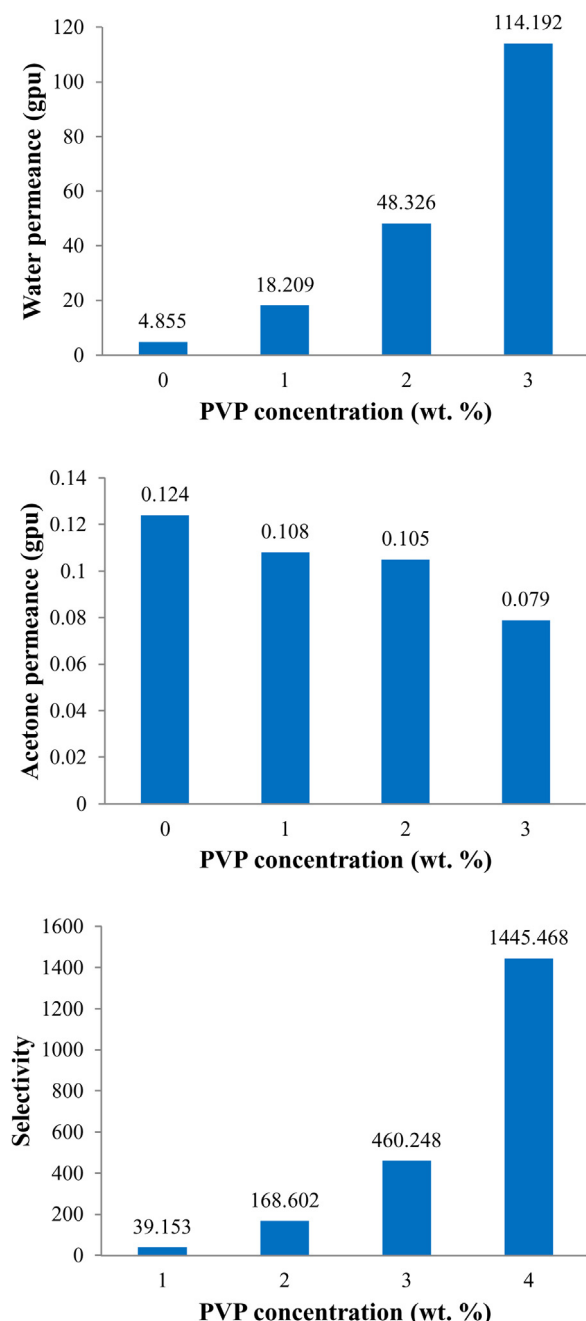

Fig. 10 – Separation factor and PSI of the PBS/PVP membranes versus PVP concentration.

(Sardarabadi et al., 2016). It was previously indicated that the introduction of PVP to the PBS membrane and increasing its concentration resulted in a higher degree of swelling (Fig. 6). Furthermore, the addition of PVP remarkably improved the membranes hydrophilic character because of its hydrophilic groups (Fig. 5). Therefore, the resultant hydrogen bonding between the membrane and water molecules increased the affinity of the membrane to water (Zhang et al., 2009). Increased membrane swelling and the higher affinity of the blend membrane to the water molecules brought about higher water fluxes. Higher flux on account of the increased free volume was reported in the literature (Wilson et al., 2011). Also, the beneficial effect of membrane hydrophilicity on the water permeation rate was reported with the addition of 1 wt.% hydrophilic filler to the PVA membrane (Jose et al., 2014).

However, the introduction of PVP and increasing its concentration was accompanied by a slight reduction in acetone permeation flux despite the increase in membrane swelling. This observation can be explained by increased membrane hydrophilicity at higher PVP loading which inhibited the transport of acetone as an organic molecule through the membrane, and thus, lower acetone fluxes were observed.

Fig. 9 also indicates the total permeation flux at different PVP concentrations. Blending the casting solution with 1 wt.% PVP resulted in an increase in total permeation flux from 0.167 to 0.26 kg/m²h. It is noteworthy that increasing PVP concentration increased the total flux further, and the highest total flux (0.49 kg/m²h) was achieved at 3 wt.% PVP concentration. The significantly higher water flux at increased PVP loading was the reason for the improved total flux.
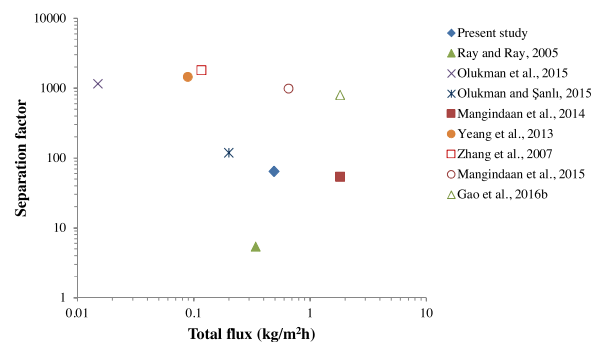
In addition to the membrane flux, the separation factor (α) and the PV separation index (PSI) can also be utilized for investigation of the membrane PV performance. The separation factor of the PBS and PBS/PVP membranes is depicted in

Fig. 12 – **Graphical representation of the data reported in Table 4.**

Fig. 11 – **Permeances and selectivity of the PBS/PVP membranes versus PVP concentration.**

Fig. 10, which shows that the separation factor was significantly higher for all blend PBS/PVP membranes in comparison with that of pure PBS membrane. Regarding Fig. 10, the separation factor increased from 10.07 for the neat PBS membrane to 65 for the PBS/PVP blend membrane obtained at 3 wt.% PVP. The significantly higher water fluxes as well as the slightly lower acetone fluxes of the PBS/PVP blend membranes, increased the numerator of Eq. (3), and thus, increased separation factor.

The PSI values determined for the PBS and PBS/PVP membranes, provided in Fig. 10, shows that the blend membranes exhibited remarkably higher PSI values compared to the neat PBS membrane. Concerning Fig. 10, the neat PBS membrane had a minimum PSI value of 1.51 kg/m^{2}h in comparison with the other membranes. In contrast, the membrane prepared with 3 wt.% PVP provided the maximum PSI (31.36 kg/m^{2}h) among the others, which indicates that the PSI value of this membrane was approximately 21 times higher than the pure PBS membrane. According to the results, the PBS/PVP membrane fabricated by blending the membrane casting solution with 3 wt.% PVP revealed the best combination of permeation flux and separation factor as compared to the other membrane samples.

The PV performance of the obtained membranes in terms of permeances and selectivity is illustrated in Fig. 11. As can be seen, the variation trend of water permeance, acetone permeance, and selectivity is similar to that of water flux, acetone flux, and separation factor, respectively. The PBS/PVP membrane blended with 3 wt.% PVP represented the highest water permeance (114.192 gpu) and selectivity (1445.468), which were almost 23 and 37 times higher than that of the pure PBS membrane, respectively.

To compare the results of the present research with the previous literature, a literature review on acetone dehydration via PV was conducted. The reported data together with the PV results of the membrane with the best performance in the present study (PBS/PVP membrane prepared at 3 wt.% PVP) are tabulated in Table 4. The reported values of separation are also shown graphically in the logarithmic plot in Fig. 12.

According to Table 4 and Fig. 12, some membranes performed worse than the PBS/PVP membrane obtained in the present study, and some others exhibited better performance. However, it should be noted that in the previous literature, different operating conditions (temperature, pressure, and water concentration in the feed solution) were utilized. As can be seen, the PV experiments of the current study were conducted at 18 mm Hg vacuum pressure, which is higher than the vacuum pressure utilized in previous studies and might be the reason for the different performance. Therefore, the comparison of the results of the present paper with that of the literature might not be logical.

In addition, it should be reminded that the PBS/PVP membranes benefit from their biodegradability. Environmental pollution through the disposal of solid waste is often ignored when the efficiency and profitability of a process is under study. But the important fact is that the application of biodegradable polymers is the solution to problems in waste management and is advantageous in the perspective of sustainable development (Rydz et al., 2014).

4. Conclusion

PBS/PVP membranes were fabricated and applied in the PV dehydration of acetone. The influence of PVP additive on the membrane morphology, chemical structure, and relative hydrophilic/hydrophobic property was studied through FESEM

Table 4 – Comparison of the results on the PV dehydration of acetone-water mixtures.						
Membrane material	Temperature (°C)	Pressure (mmHg)	water concentration in the feed (wt.%)	Total flux (kg/m²h)	α	PSI (kg/m²h)
PBS/PVP (present study)	25	18	15	0.49	65	31.36
poly(acrylonitrile-co-maleic anhydride) Ray and Ray (2005)	30	1	10	0.34	5.4	1.496[a]
CBCM[b]- Ramana et al. (2006)	40	–	50	0.235 (L/m²h)	19.8	4.418[a] (L/m²h)
PVA–Fe₃O₄ Olukman et al. (2015)	40	0.5	10	0.015	1166.4	17.481[a]
PVA-g- acrylonitrile/HEMA[c]-Fe₃O₄ Olukman and Şanlı (2015)	40	0.5	20	0.2	120	23.8[a]
P84[d] crosslinked with EDA[e] - Mangindaan et al. (2014)	50	<3.75	15	1.825	53.48	95.78
Three layer CS/PVA–MWCNT[f]/PVDF[g]- Yeang et al. (2013)	30	5	5	0.089	1450.5	129.005[a]
CS-GA[h]-MA[i]- Zhang et al. (2007)	40	0.75–1.5	10	0.117	1791	209.43[a]
P84 crosslinked with TAEA[j]- Mangindaan et al. (2015)	50	<3.75	15	0.658	983	646.15
CS/SCMS[k] on PAN support Gao et al., 2016b	50	<2.25	5	1.81	802	1449.81[a]

[a] Calculated here according to the reported flux and separation factor.
[b] Composite bacterial cellulose membrane.
[c] 2-hydroxyethyl methacrylate.
[d] Co-polyimide of 3,30,4,40-benzophenone tetracarboxylic dianhydride (BTDA) and 80% toluene diisocyanate (TDI)+20% methylene diphenyl diisocyanate (MDI).
[e] Ethylenediamine.
[f] Multi-walled carbon nanotube.
[g] Polyvinylidene fluoride.
[h] Glutaraldehyde.
[i] Maleic anhydride.
[j] Tris(2-aminoethyl)amine.
[k] Sulfonated carbon molecular sieve.

imaging, ATR-FTIR spectroscopy, and measurement of water contact angle. Moreover, the swelling degree and thermal stability of the membrane samples were determined. The degree of swelling of the PBS/PVP membranes in water/acetone mixture, as well as their hydrophilicity and tensile strength, were higher compared to that of the pure PBS membrane. The incorporation of 3 wt.% PVP into the blend solution resulted in almost 2.7-fold increased swelling, approximately 29% lower water contact angle, and about 26% higher tensile strength, respectively. Performance of the obtained PBS and PBS/PVP membranes during PV dehydration of acetone indicated that the water flux through all of the membranes was noticeably higher than that of acetone. The addition of PVP remarkably increased the water flux, while a slight reduction in acetone flux was observed. The total flux, separation factor, and PSI were 0.167 kg/m²h, 10.07, and 1.51 kg/m²h, respectively, using the neat PBS membrane. The addition of 3 wt.% PVP to the casting solution augmented the mentioned parameters by approximately 3, 6, and 21-fold, respectively, compared to the neat PBS membrane. Therefore, the PBS/PVP membrane obtained by blending the membrane casting solution with 3 wt.% PVP exhibited the highest permeation flux (0.49 kg/m²h), separation factor (65), and PSI (31.36 kg/m²h) among the other samples.

Declaration of interests

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Declaration of Competing Interest

The authors report no declarations of interest.

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