Effect of PVP content and polymer concentration on polyetherimide (PEI) and polyacrylonitrile (PAN) based ultrafiltration membrane fabrication and characterization

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ABSTRACT

In this study, four different membranes were fabricated by using polyetherimide and polyacrylonitrile polymers, N-methyl-2-pyrrolidone and polyvinylpyrrolidone (PVP) via phase inversion method to improve the membrane performance in fruit juice wastewater (FJWW) treatment. The addition of PVP to the casting solution increased membrane hydrophilicity, water content, contact angle, porosity, Fourier transform infrared spectroscopy peaks, membrane thickness, average roughness and viscosity of cast solutions compared to the bare membrane. It can be said that the addition of a lower polymer concentration and PVP intensively increases the pure water flux of the membrane. However, as the flux increased, a small decrease in FJWW rejection was observed.

KEY WORDS | fruit juice wastewater, membrane characterization, phase inversion, PVP, ultrafiltration

INTRODUCTION

The choice of suitable polymer in membrane fabrication plays a key role for the ultrafiltration (UF) membrane. Polyetherimide (PEI) and polyacrylonitrile (PAN) polymers are known as a good candidate for forming a good UF membrane structure.

PEI has structural and mechanical properties such as excellent film formation, moderate chemical and good thermal resistance (Trewjumrone & Chinpa 2010).

PAN polymer has a satisfactory chemical stability in filtration implementations. Commercial membranes, which are often marketed commercially, need to be kept wet at all times. If the membranes dry, membrane pores collapse, and the membrane material becomes brittle which causes sudden breaking during the filtration (Scharnagl & Buschatz 2001). PAN membranes with high pure water permeability are generally prepared in the form of flat sheets by selecting a suitable component of the casting solution and the preparation conditions (Wu et al. 2000). However, in the industrial wastewater treatment, pervaporation process, and production of the composite membranes substrate, PAN membranes are being used aplenty because of their tolerance to organic solvents (Qin et al. 2015).

Polyvinylpyrrolidone (PVP) is a water-soluble polymer. It is often used as a pore-forming agent in the preparation of asymmetric membranes by phase inversion method. It is used to modulate the structures of the membranes by affecting the polymer membranes (Wan et al. 2006). PVP provides morphology control in terms of the thermodynamics and the kinetics for polymer membranes. In thermodynamics, it makes the structure of polymer inconsistent and inclined to the phase separation. Kinetically, phase differentiation/discretization of the high viscosity of the polymer solution will provide a lagged and decelerated demixing process by PVP addition (Zhang et al. 2011). Furthermore, PVP addition increases membrane selectivity by reducing relative transport rate or failures at the membrane surface (Rao et al. 2008). The molecular structure of PEI, PAN and PVP polymers is given in Figure 1 (Sigma Aldrich).

The variability of casting solution viscosity, membrane pore size, and membrane porosity are defined as a function of the casting solution constituent (Dal-Cin et al. 1994). There are a lot of studies about blended PEI-PVP (Salleh & Ismail 2011; Bakeri et al. 2015; Hebbal et al. 2015) and blended PAN-PVP (Nouzaki et al. 2002; Jung et al. 2004; Yang & Lin 2002).

The phase inversion method has been widely used (Machado et al. 1999; Kim et al. 2002; Zheng et al. 2006). In this method, the polymer dissolves in a solvent at various times and temperatures to form a viscous solution. This
method is based on the exchange of solvent and nonsolvent on diffusion and causes the cast solution to make a phase transition leading to the formation of the membrane (Young & Chen 1995). Membrane structure and morphology are related to kinetic parameters such as solvent–nonsolvent exchange (Tsai et al. 2002; Li et al. 2008). Furthermore, thermodynamic parameters such as polymer/solvent interactions, solvent/nonsolvent interactions, evaporation time and coagulation bath temperature affect the membrane formation mechanism significantly (Amirilargani et al. 2014). To increase membrane performance and enhance the phase inversion method, additives such as PVP can be added to the base polymer and solvent mixture (Al Malek et al. 2015).

Fruit juice wastewater is formed during the stages of fruit juice production: pressing, container washing, filtration and grinding of the juice. The characteristics of the fruit juice wastewater in the literature are shown in Table 1 (El-Kamah et al. 2010).

Carbohydrates the primary components of juice content: food sugar, food acid and low molecular weight sweetener components. Sugar accounts for up to 20% of the water-soluble components of fruit juices. Organic acid and inorganic salts are present in lower amounts (0.1 to 2%). Many volatile components such as alcohol, aldehyde, ketone and ester are contained in very small quantities in fruit juice. The high amount of sugar present in the solution in the membrane filtration causes a flux decrease due to the high osmotic pressure of the sugar solution. Due to the high viscosity of the feed solution, there is also a reduction in the mass transfer coefficient of the membrane (Rana et al. 2014).

In this study, the asymmetric membrane fabrication was performed using PEI and PAN polymers, PVP as pore forming agents and N-methyl-2-pyrrolidine (NMP) as a solvent. For membrane fabrication, wet phase inversion method was used. After preparation, the performances of the membranes were tested by treating fruit juice wastewater (FJWW). According to the results, the effect of polymer and PVP concentrations on water flux and rejection were investigated and evaluated. Fourier transform infrared (FTIR) spectroscopy, scanning electron microscopy (SEM), atomic force microscopy (AFM), water contact angle, viscosity, porosity and water content methods have been used for characterization of membrane structure.

### EXPERIMENTAL

#### Materials

PEI (CAS Number 61128-46-9) is a polymer which has 30,000 g/mol average molecular, and PAN (CAS Number 25014-41-9) is a polymer which has the average molecular weight of 150,000 g/mol. They were purchased from Sigma Aldrich. NMP (8060722500) was purchased from Merck. In the preparing of PEI and PAN membranes, in order to increase pore size and improve permeability of membranes PVP10 was used as an additive. PVP10 (CAS Number 9003-39-8) was purchased from Sigma Aldrich.

#### Preparation of membranes

For preparing PEI0 membranes, a casting solution of 20 wt.% of PEI polymer and 80 wt.% of NMP was used. PVP-added membrane casting solution was prepared including 12 wt.%
of PEI, 8 wt.% of PVP and 80 wt.% NMP. For the casting solution of PAN membranes, the solution of PAN₀ membrane comprised 15 wt.% of PAN and 85 wt.% of NMP, and the solution of the PAN₈ membrane comprised 10 wt.% of PAN, 8 wt.% of PVP and 82 wt.% of NMP (Table 2).

For the preparation of the PEI₀ casting solution, PEI polymer was added to NMP, and then the mixture was stirred for 24 h at 40 °C with 110 rpm to obtain a homogeneous solution. During PEI₈ membrane preparation, firstly PVP was added to NMP; after the dissolution of PVP, the solution was added with PEI and the new solution was stirred for 24 h at 40 °C in 110 rpm centrifuge velocity. Similarly, PAN₀ and PAN₈ membrane mixtures were stirred for 24 h at room temperature at 90 rpm. After the solution reached a homogeneous phase, before membrane solution casting to a glass plate, we ensured that air bubbles were removed.

Membrane casting solutions were cast on the glass plate and using knife gap of 200 μm, were cut properly. After 10 seconds, the glass plate was immediately immersed in distilled water for coagulation at room temperature. After this process, prepared membranes were kept in water and a cold room until their use in the membrane bioreactor (MBR) system.

Membrane characterization

Viscosity

To measure the viscosity of PEI₀, PEI₈, PAN₀ and PAN₈, a Brookfield DV-E viscometer was used. The measurement was carried out at 100 s⁻¹ shear rate for 2 min at 20 °C using cone/plate geometry.

Porosity measurements

For PEI₀, PEI₈, PAN₀, and PAN₈, porosity measurements were carried out by the dry–wet method and calculated using the expression in Equation (1).

\[
\text{Porosity} = \frac{w_{\text{w}} - w_{\text{d}}}{v.d_{\text{water}}} \times 100\%
\]  

(1)

\(w_{\text{w}}\): weight of membrane (after dipping into distilled water) (g),
\(w_{\text{d}}\): weight of dry membrane (g),
\(v\): volume of the membrane in the wet state (cm³),
\(d_{\text{water}}\): density of distilled water at room temperature (g/cm³).

Water content

Water content measurements for all membrane types were done by the dry–wet method. In this method, unlike the porosity calculation method, the membrane, which was left in distilled water for 24 hours, was dried with drying paper and then the wet weight was measured. For dry weight, the measurement was carried out after the membrane was kept in a 45 °C incubator for 48 hours. It was calculated by the expression given in Equation (2):

\[
\text{water content (\%)} = \frac{W_{\text{w}} - W_{\text{d}}}{W_{\text{w}}} \times 100
\]  

(2)

\(w_{\text{w}}\): weight of membrane (after dipping into distilled water for 24 h at room temperature and drying by filter paper) (g),
\(w_{\text{d}}\): weight of membrane (after being in the incubator at 45 °C for 48 h) (g).

Contact angle measurements

To characterize the membrane surface polarity, an Attension T200 Theta was used. At room temperature, using a droplet of 5 μL on the membrane surface, the measurement was carried out. After three different measurements on the membrane surface, the mean of three values was used.

Fourier transform infrared spectroscopy

FTIR spectra for each membrane were recorded with a Perkin Elmer Spectrum 100 TIR spectrophotometer. The spectral range of this spectrophotometer is 40–4,000 cm⁻¹ and resolution is 4 cm⁻¹ scan. The probes used were 1,2-dichloroethane, 1,1,2,2-tetrabromoethane, and 1,2-diphenylethane. The procedures for the preparation of polymer films,
determination of their thickness, the introduction of probes into them, and the estimation of the concentration of probes are similar to those described in Kamalova et al. (2008).

**Scanning electron microscopy**

The membranes were examined by SEM using an FEI Quanta 450 FEG-EDS. The emission cathode was operated at 0.2–30 kV. Before SEM analyses, membranes were cut by cold nitrogen.

**Atomic force microscopy**

AFM of PEI, PVP-added PEI, PAN and PVP-added PAN membranes was carried out using a Digital Instruments atomic force microscope.

**Pure water flux**

Water flux measurements were performed using a membrane bioreactor. The effective area of all membranes was 49 cm² and the operation was carried out at 20 °C and 600 mbar pressure. The water flux was calculated by Equation (3).

\[ J = \frac{Q}{A \Delta t} \]  

(3)

\( J \): water flux (L/(m² h)),  
\( Q \): quantity of permeate collected after the membrane reached the steady state (L),  
\( \Delta t \): sampling time (h),  
\( A \): membrane area (m²).

**Flux and percentage rejection**

A series rejection model was used for rejection calculations (Chang & Lee 1998). Firstly, in order to determine the rejection of membranes due to their structure, distilled water was passed through the clean membranes. The flux–time graphs were drawn to determine the rejection value \( R_m \) of the membranes, and the total rejection \( R_t \) value was obtained by filtering FJWW through the membranes. In Figure 2 a schematic diagram of the flow is given.

**RESULTS**

**Viscosity measurements**

The viscosity of casting solutions of PEI₀, PEI₈, PAN₀ and PAN₈ was measured to control membrane morphology. If the phase inversion process is used for the membrane, polymer solution viscosity becomes an important factor for the membrane morphology. Solutions with higher viscosity are more compatible with a slower diffusion between the phase conversion components, because there is a delayed exchange between solvent and nonsolvent.

Casting solution viscosity of membranes was decreased with the PVP addition. Although the amount of PVP added to the polymers is the same, the viscosity of the membrane casting solutions is reduced. This may be due to a reduction in the amount of polymer in weight (wt.%) in the casting solution. But many researchers have reported that adding PVP into the casting solution increased the viscosity (Torrestiana-Sánchez et al. 1999; Yuan & Dan-Li 2008; Alpatova et al. 2013). Han & Nam (2002) have found that when the amount of polymer is kept constant and the
content of NMP in the solution decreases, the viscosity of the casting solution increases with increasing PVP amount. In this study, the decrease in the viscosity of the casting solution was considered to be due to the decrease in the amount of polymer, since the amount of polymer was reduced and PVP was added to the casting solution. When PVP is added and PVP-free membranes are compared, the viscosity of the membrane decreases as more polymer is reduced, while the viscosity of the membrane decreases as less polymer is reduced in proportion to the amount of polymer (Table 3).

In addition, increased water content and increased hydrophobicity of PVP-added membranes can be considered as factors that lead to decreased viscosity.

It is known that as the viscosity of membrane casting solution increases, the thickness of the initial membrane increases. However, in this study, as the viscosity decreases due to PVP addition, the porosity-causing membrane thickness increases (Table 4) (Fathizadeh et al. 2012). In addition, the weight reduction of the main polymer amount, but the increase in the total amount of polymer by weight with the addition of PVP, may have resulted in an increase in membrane thickness (Mustaffar et al. 2004). The viscosity value of PEI0 was first measured as 2.82 Pa.s, but after the addition of PVP, the viscosity of PEI8 decreased to 1.19 Pa.s. The thickness of the PEI0 membrane was 2.80 Pa.s, but after the addition of PVP, the viscosity of PEI8 decreased to 2.04 Pa.s. The thickness of the PAN0 membrane was 1 mm while it increased to 1.7 mm by the addition of PVP.

**Water content and porosity**

When all membranes were analyzed, membrane water content and membrane porosity increased with decreasing weight of the main polymer (Table 4). In addition, there was a significant decrease in PAN0 porosity with increasing main polymer amount. When PVP was added and polymer concentration was decreased, values of contact angle were increased. However, there was no significant change in pore size for all membranes with the addition of PVP. While the water content of the PEI0 membrane was 72%, the water content of the PEI8 membrane increased to 84% as a result of the PVP addition. The contact angle increased from 19% to 78% and the porosity increased from 74% to 77%.

**Contact angle measurement**

According to Table 4, the contact angle of membranes increased by using PVP as an additive. In addition, in the absence of PVP, pure membranes presented less hydrophilicity (Wan et al. 2006). The contact angle for PEI0 membrane was 19° and for PEI8 was 78°. The contact angle for PAN0 was 16°, and for PAN8 was 50°. Table 4 shows that the addition of PVP to PEI and PAN polymers increases contact angle values and decreases membrane hydrophilicity. When polymer PVP is added in membrane fabrication the contact angle increases significantly. Since PVP polymer is more hydrophobic compared to PEI and PAN polymers, PVP presence in the membrane structure causes membrane hydrophilicity to be reduced. In addition, the contact angle value is reduced due to the reduction of the main polymer concentration (Table 2) (Ochoa et al. 2003).

However, the molecular weight of PVP used in this study is low. For this reason, it can be said that a part of PVP is retained in the membrane pores and affects the structure of the membrane (Saljoughi & Mohammadi 2009).

**Fourier transform infrared spectroscopy**

Figure 3 depicts the FTIR absorption spectra of PEI0, PEI8, PAN0, and PAN8. FTIR spectra of pure PEI fibers have many peaks which are related to the existence of C=O, C–N, C–N–C and C–O bonds. The typical imide bands can be detected at 1,717 cm⁻¹ (symmetric and asymmetric imide C = O stretching, respectively). C–N–C absorption of...
the imide ring was observed at 1,357 cm\(^{-1}\). The C–O stretching appears at 1,236 cm\(^{-1}\) and the C–N stretching appears at 743 cm\(^{-1}\) (Trewjumrone & Chinpa 2010). After adding PVP to PEI membrane, many peaks occurred at 1,444, 1,478, 1,496, 1,600, 1,667, 1,722 and 1,777 cm\(^{-1}\). The C–O stretching at 1,236 cm\(^{-1}\) has disappeared. The peak at 1,444 cm\(^{-1}\) may be related to C–H and O–H deformation vibrations (Lamsal et al. 2015). The absorption bands at 1,478 cm\(^{-1}\) correspond to the C–N vibration of the quaternary ammonium (Sánchez et al. 2004). The reason for the peak formation at 1,496–1,600 cm\(^{-1}\) is the presence of –C = N bonds, and the peak at 1,667 cm\(^{-1}\) is due to –C = N bond stretching (Farsani et al. 2004). At 1,722 cm\(^{-1}\) and 1,777 cm\(^{-1}\), C = O and –C = N stretching was observed (Mungali et al. 2015). And at these peaks, the FTIR spectrum showed the appearance of characteristic imide bands.

PAN\(_0\) showed two peaks at 1,452 and 2,243 cm\(^{-1}\); the characteristic nitrile (C ≡ N) peak at 2,243 cm\(^{-1}\) and deformation at 1,452 cm\(^{-1}\), respectively (Majee et al. 2012). After adding PVP to PAN membrane, many peaks occurred at 1,071, 1,289, 1,371, 1,441, 1,494, 1,668, 2,942 and 3,407 cm\(^{-1}\). But the peak which is at 1,452 cm\(^{-1}\) disappeared. The absorption peak around 3,407 cm\(^{-1}\) is related to O–H, NH and COO–H bonds. Another peak was observed around 2,942 cm\(^{-1}\) (nitrile bonds, C ≡ N). C = N and C = C bonds are situated at 1,668 cm\(^{-1}\). The strongest peak at 1,668 cm\(^{-1}\) arising from the carbonyl group (C = O) stretching vibration in PVP can be regarded as the typical peaks of PVP (Wan et al. 2006; Farsani et al. 2009).

Absorption in the range of 1,441 and 1,494 cm\(^{-1}\) is related to tensile vibration and peaks in the range of 1,289–1,371 cm\(^{-1}\) are related to vibration in a different situation. Another peak was observed at 1,071 cm\(^{-1}\) which is related to presence of C = O or C–O bonds and is caused by the co-monomers’ presence (Farsani et al. 2009).

**SEM analyses**

Membrane thickness and cross-section structure were measured by SEM. To dissolve the precise section of the membrane, a prepared membrane was cut in the shape of a rectangle and immediately immersed in liquid nitrogen and used for sectioning. The surfaces and cross-sectional shapes of PEI\(_0\), PEI\(_8\), PAN\(_0\), and PAN\(_8\) membranes are shown in Figure 4(a)–4(d) respectively.

Depending on the different chemical properties, it can be observed that the size of the microporous structure decreases when blending PEI and PAN with PVP (Figure 4(b) and 4(d)). According to these images, finger-like structures can be said to be present due to the addition of PVP in PEI membranes (PEI\(_8\)) (Yoo et al. 2004). All membranes have characteristic asymmetric structures consisting of layers with cellular morphology added to the polymer matrix, as well as strata engaging with asymmetric finger-like pores (Balta et al. 2012). From the SEM analysis, it is seen that the pore size of PEI\(_0\) and PEI\(_8\) membranes is larger than the pore size of PAN\(_0\) and PAN\(_8\) membranes (Table 4).
Atomic force microscopy

The surface roughness of the membranes was determined using AFM. Surface roughness parameters, expressed as mean roughness (RMS), the mean square of the Z data (Ra) and mean difference of the five highest peaks and the lowest five peaks (Rz), were calculated (Table 5).

According to the measurement results, the roughness of PVP-added membranes was markedly higher than PEI0 and PAN0. The three-dimensional topography is given in Figure 5(a)–5(d). The surface of PEI8 and PAN8 appears intensely and finely dispersed under gravity. This means that PEI8 and PAN8 have a rougher surface. PEI0 and PAN0 have a smoother surface. While all membranes, especially PEI0, were examined, it was observed that the nodule structure of all membranes exhibited a heterogeneous nodule agglomerate distribution (Kanagaraj et al. 2014). In addition, as shown in Figure 5(a) and 5(c), the size of the nodules of PEI0 and PAN0 is slightly smaller than that of PEI8 and PAN8.

Because of membrane structural nodules formed from polymer aggregates, surface roughness often affects membrane performance (Shehzad et al. 2015). Studies indicate that the roughness of the membrane affects the increase in flux and permeability as the surface roughness increases (Johnson & Hilal 2015).

Pure water permeability

The pure water fluxes (PWFs) of PEI0, PEI8, PAN0, and PAN8 were measured at 0.6 bar transmembrane pressure. Figure 6 shows the PWF of the PEI0, PEI8, PAN0 and PAN8.

Initially, PEI0 presented the lowest value of PWF (26 L/(m² h)) and PAN8 presented the highest value of PWF (545 L/(m² h)). When the PVP was added to the casting solution, the PWF values of the membranes increased (for PEI0 from 26 to 160 L/(m² h) and for PAN0 from 281 to 545 L/(m² h)). This can be explained by the increased number of pores and size of pores due to the presence of PVP, because PVP acted as a pore-forming agent during the phase inversion. PVP is a hydrophilic polymer but when the characterization of PVP-added membranes is examined, contact angle values of membranes show that they become hydrophobic.
The highest membrane permeability is obtained for PAN8. When PEI0 and PEI8 are compared, it is possible to see that PEI0 provides less permeability. It may be the result of decreasing of PEI polymer concentration. The same situation is encountered in the membranes PAN0 and PAN8 (The PAN polymer in the membrane casting solution for PAN8 membrane fabrication was reduced by 33%) (Kanagaraj et al. 2013).

Figure 6 shows the effect of different formulations on PWP with different polymer concentrations. According to Figure 6, when the polymer concentration was increased from 10% to 15% by weight for the PAN membrane and from 12% to 20% by weight for the PEI membrane, the average PWF was reduced.

**Flux and percentage rejection**

In this study, FJWW was used to determine flux and rejection values of membranes. The sludge age of the juice wastewater is 30 days and the COD value is 1,000 mg/L. Figure 7 shows the total rejection, membrane rejection and
Figure 7 | Effect of polymer concentration on percentage rejection.

FJWW treatment flux values. FJWW treatment flux was similar to PWP behavior. As the wt.% of the PEI and PAN polymers in the casting solution decreased, the FJWW treatment flux increased. According to Figure 7, a mean flux of 65 L/(m² h) was obtained for the PAN polymer containing 10 wt.% polymer (PAN8). In the case of a membrane containing 15 wt.% polymer (PAN0), the average flux value was determined to be 49 L/(m² h). For PEI, when the polymer concentration was 12% by weight, the mean flux value was 25 L/(m² h), whereas for polymer concentration of 20% it was 20 L/(m² h). However, the PWF values were increased by PVP addition to the polymers.

The effect of different polymer concentration on total rejection and membrane rejection is shown in Figure 7. The total rejection percentage of the PEI membrane was 8.55 × 10¹⁴, while it decreased to 4.28 × 10¹⁴ after PVP was added. The total rejection percentage of the PAN membrane was 2.09 × 10¹⁴ but decreased to 1.23 × 10¹⁴ after PVP was added.

When all of the PEI₀, PEI₈, PAN₀, and PAN₈ membranes were examined, there was a decrease in the percent membrane rejection by decreasing the main polymer concentration (PEI and PAN). It is also possible to say that PVP addition increases the percentage of membrane rejection, because the concentration of the main polymer, with respect to PEI₀ and PAN₀, was reduced in PEI₈ and PAN₈ membranes. With the addition of PVP, the rejection of the PEI membrane increased from 6.32 × 10¹³ to 11.2 × 10¹³ and the rejection of the PAN membrane increased from 0.1 × 10¹³ to 1.1 × 10¹³. Thus, PVP addition has an adverse effect on total rejection and membrane rejection.

CONCLUSIONS

In this study, membranes were prepared with PVP as an additive, NMP as a solvent and water as a coagulation bath. The permeability of the membranes was checked by PVP addition. When the results are examined, it has been found that PVP addition and reduction of polymer concentration increase membrane permeability and membrane water content. The chemical structure, morphology, and surface roughness were investigated using FTIR spectroscopy, SEM, and AFM. Adsorption peaks and membrane surface roughness increase with the addition of PVP and lowering of the main polymer concentration. SEM analyses showed that the pore size of PEI₀ and PEI₈ was larger than the pore size of PAN₀ and PAN₈. Thus, it can be said that PVP does not affect the membrane pore size. This result can be explained by the PEI polymer structure (the molecular weight of the PEI polymer is lower than the molecular weight of the PAN polymer). PVP-added membranes were observed to be rougher by the AFM. In particular, there was a significant increase in PAN₀ porosity (121%). A decrease in solution viscosity was observed with the addition of PVP. This can be explained by the fact that the main amount of polymer is not kept constant but reduced. In addition, pure membranes provide a hydrophilic surface when PVP is not present. However, the hydrophilicity of the membrane surface was reduced by PVP addition and the membrane showed a hydrophobic tendency. The PWF of the membranes increased when the PVP addition was sufficient and the polymer concentration was reduced.

To determine the resistance of the membranes to wastewater, FJWW with a 30-day sludge age and a concentration of 1,000 mg/L COD was used. When the performances obtained from the membranes in the wastewater treatment system are examined, it is determined that, when PVP is added and the main polymer concentration decreases, the FJWW treatment flux increases and the total rejection decreases. However, the membrane rejection increases with the PVP addition.

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