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PII: S0376-7388(17)33471-3
DOI: <https://doi.org/10.1016/j.memsci.2018.02.055>
Reference: MEMSCI15980

To appear in: *Journal of Membrane Science*

Received date: 11 December 2017
Revised date: 20 February 2018
Accepted date: 24 February 2018

Cite this article as: Liliana Pérez-Manríquez, Pradeep Neelakanda and Klaus-Viktor Peinemann, Morin-based nanofiltration membranes for organic solvent separation processes, *Journal of Membrane Science*, <https://doi.org/10.1016/j.memsci.2018.02.055>

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Morin-based nanofiltration membranes for organic solvent separation processes

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Abstract

In this work we demonstrate the successful optimization of the interfacial polymerization reaction for the manufacture of organic solvent nanofiltration membranes by replacing the toxic amines commonly used for this method with the natural occurring bio-polyphenol morin. For the manufacture of this type of OSN membrane a crosslinked PAN support was coated by interfacial polymerization using morin as the monomer of the aqueous phase and terephthaloyl chloride as the monomer of the organic phase. These membranes showed an exceptional performance and resistance to NMP by having a permeance of 0.3 L/m² h bar in NMP with a rejection of 96% of Brilliant Blue dye which has a molecular weight of 825.97 g/mol, making these membranes attractive for harsh industrial separation processes due to their ease of manufacture, low cost, and excellent performance .

1. Introduction

In the last decade there has been a trend towards bio-inspired approaches for the formation of nanocoatings as well as to accomplish energy-intensive industrial separations in a more sustainable fashion. Organic solvent nanofiltration (OSN) is a pressure driven process, where the operation conditions are moderate and additional waste streams are minimized [1]. Compared to distillation OSN is an energy efficient approach for challenging molecular separations such as purification of active pharmaceutical ingredients [2-4], production of specialty chemicals [5-7] and separations in the petrochemical industry [8-10].

The overall performance of OSN membranes is determined by solute/solvent interactions with the membrane top layer [11]. Therefore, the modification of the membrane surface becomes crucial in order to obtain high-performance OSN membranes. The first commercial OSN membranes had an integral-asymmetric structure made from cellulose acetate or crosslinked polyimides; but recently thin-film composite membranes with superior properties have been developed. Karan et al. described coatings as thin as 10 nm with

ultrahigh solvent fluxes [12], Jiminez-Salomon et al. used contorted monomers, which are known for the manufacturing of polymers with intrinsic microporosity, for the fabrication of solvent resistant nanofilms by interfacial polymerization [13]. Sorribas et al. introduced metal-organic framework based nanocomposite films for OSN [14].

. Recently a growing interest exists to use “green approaches” and less toxic monomers for the preparation of OSN membranes without sacrificing their permeation properties. One “green” option for the fabrication of the thin-films in OSN membranes proposed in this paper is the use of bio-polyphenolic molecules. Among the many classes of phenolic biomolecules, plant phenols are capable of binding and cross-linking due to their strong interfacial activity. Therefore the use of plant-derived polyphenols as precursors for spontaneous, multifunctional, and substrate-independent nanocoatings has been recently reported [15-20].

In this paper we decided to use morin hydrate, a naturally occurring polyphenolic compound that can be isolated from the common guava plant (*Psidium guajava*) [21], as one of the monomers to fabricate thin-film composite membranes through interfacial polymerization to replace the commonly applied toxic aromatic amines. The use of this particular bio-polyphenol has never been reported before for OSN membrane fabrication, which makes this particular molecule quite attractive as a new natural approach to form thin-films.

Here, we report the application of morin hydrate/terephthaloyl chloride for the manufacturing of organic solvent nanofiltration membranes. For the membrane support, we used our previously reported crosslinked PAN support [22] as it exhibits exceptional stability in organic solvents. The interfacial polymerization reaction was optimized in order to obtain competitive permeation and rejection performance in NMP.

2. Experimental

2.1. Materials

Polyacronitrile (PAN) polymer with a molecular weight of approximately 100,000 g/mol was supplied by Kelheim Fibers GmbH, Germany. Methanol, hexane, N,N-dimethylformamide, N-Methyl-2-pyrrolidone, hydrazine hydrate, terephthaloyl chloride, morin hydrate, Rose Bengal dye (purity 90%) and Congo Red dye, were all

acquired from Sigma-Aldrich. Brilliant Blue R250 was acquired from Fisher Bio Reagents and polypropylene nonwoven material was acquired from Sojitz Europe, Germany.

2.2. Fabrication of polyacrylonitrile membrane support

The PAN ultrafiltration support was fabricated via water induced phase separation process. First, the PAN powder was dissolved in DMF under stirring at 70 °C to obtain a 12 wt.% solution. The clear PAN solution was set aside for about 12 hours for degassing and was then cast on a polypropylene nonwoven support using a continuously operating casting machine. Knife air gap was adjusted at 150 μm , the casting speed was 2 m min^{-1} . The coagulation bath was tap water at 23°C. Then, after polymer precipitation on the nonwoven support and after washing the membranes were treated in a hot water bath for 45 min at 90 °C.

Finally the wet membranes were cross-linked by immersing them for 6 h in a 20% (v/v) aqueous solution of hydrazine hydrate in deionized water at 85 °C; these were the optimum conditions previously reported [19] in order to produce solvent-stable membranes with good mechanical stability. The obtained ultrafiltration membranes showed excellent stability in both DMF and NMP. The crosslinked membranes were carefully washed to remove excess hydrazine and prior to be used, they were stored in deionized water.

2.2.1. Thin-Film composite membrane manufacturing by interfacial polymerization

Since morin has a poor solubility in water, methanol was selected as solvent. Composite membranes were manufactured by interfacial polymerization using morin hydrate in methanol [2wt%] as the polar phase and terephthaloyl chloride (TPC) in cyclohexane [0.2wt%] as the organic phase (Fig. 1). The wetted crosslinked PAN membranes were soaked with the methanol solution of morin hydrate for 5 min. After removal from this bath excess solution was removed from the surface by placing the membrane between 2 filter papers and gently rolling a rubber roller over the backside of the membrane. Morin hydrate treated

membranes were then mounted into a Teflon frame (Fig. 1). The purpose of placing the membranes in this Teflon frame is to avoid the contact of the organic phase with the back of the membrane and just letting it react with the top side of the membrane so the interfacial polymerization reaction can take place. The membrane surface was contacted with a solution of TPC in cyclohexane (0.2 wt%) for 2 min. Then the TPC/cyclohexane solution was drained, the Teflon frames were removed and the membranes were put in the oven at 85 °C for 10 min. This annealing step is crucial for the formation of the thin film when we use this combination of monomers for the interfacial polymerization process. . The bio-polyphenolic coated membranes were stored in water before performing filtration experiments.

Fig 1. Digital images of the fabrication by interfacial polymerization of morin hydrate / terephthaloyl chloride thin-film composite membranes. 1) Contacting the crosslinked PAN support with the morin hydrate solution (2 % w/v) in methanol for 5 min, 2) removal of residual morin hydrate solution droplets on the membrane surface using a rubber roller, 3) fixing the membrane between the plate and frame assembly, 4)

contacting the membrane with the organic phase of 0.2 % (w/v) TPC/hexane for 2 mins,
4) annealing the membrane at 85°C for 10 min under atmospheric pressure.

2.3. Membrane characterization

2.3.1. Fourier transform infrared spectroscopy (FTIR)

The membrane surface was examined by Fourier transform infrared (FTIR) analysis, spectra were taken on a FTIR spectrometer model Nicolet iS10 in the range of 3700–900 cm^{-1} . All membrane samples were dried at 80 °C 24h in a laboratory oven prior analysis.

2.3.2. X-ray photoelectron spectroscopy (XPS)

The surface chemical compositions of the composite membrane were investigated by X-ray photoelectron spectroscopy (XPS) in a Kratos Axis Ultra DLD spectrometer equipped with a monochromatic Al $K\alpha$ x-ray source at 1486.6 eV. Charge neutralization was required for the analysis of the membrane sample. Binding energies were referenced to the aromatic sp^2 hybridized (C=C) carbon for the C1s peak set at 284.5 eV.

2.3.3. Scanning electron microscopy (SEM)

The membrane surface and cross-section was imaged using a Helios™ G4 DualBeam microscope with a voltage of 1 kV. Low voltage and a high resolution microscope was needed to image these membranes as they were beam sensitive. In order to analyze the morphology of the membranes, samples were fractured in liquid nitrogen to obtain a sharp and well defined cross-section and then they were mounted on aluminum stubs. The samples were sputtered with a 2 nm thin iridium layer to avoid electrostatic charges.

2.3.4. Membrane performance

A dead-end stainless steel cell with a magnetic stirrer was used for the filtration

experiments. The applied pressure was 20 bar, filtration was performed at 23 °C with a membrane area of 12.6 cm². Various samples of permeate and retentate were taken after reaching a steady state flux. Each dye solution of Rose Bengal (Mw: 1017.64 g/mol), Brilliant Blue R250 (Mw: 825.97 g/mol) and Congo Red (Mw: 590.71 g/mol), was tested at least with 3 different membrane coupons. The concentration of all dye solutions used for this study was 35 µmol/L in NMP in order to be able to determine the molecular weight cut-off of the membranes.

50 ml of feed solution was poured into the filtration cell, which contained one of the dyes mentioned above. The first 10 ml of permeate were discarded and samples of 3 ml were collected by triplicate. Retentate and permeate concentrations were then used to calculate the dye rejection using the equation:

$$\text{Rejection (\%)} = \left(1 - \frac{C_p}{C_r}\right) \times 100$$

C_r and C_p are the solute concentrations of retentate and permeate, respectively. A UV VIS/near IR (Perkin Elmer Lambda 1050) spectrophotometer was used to determine the dye concentration using dye's different wavelengths shown in Table 1.

3. Results and discussion

3.1 Stability in NMP

Non-crosslinked and crosslinked PAN membranes were immersed for a prolonged period of time in NMP. The base polyacrylonitrile membranes rapidly dissolved in NMP as expected, given the fact that polyacrylonitrile is known to be soluble in NMP. However, the crosslinked PAN membranes did not change in NMP even after 3 months storage time; they still showed the same shiny appearance characteristic of a PAN membrane.

3.2. Fourier transform infrared spectroscopy (FTIR)

FTIR membrane analysis was used to corroborate that the interfacial polymerization reaction between morin hydrate and terephthaloyl chloride on the cross-linked PAN support was successfully performed as shown in Figure 2. The FTIR spectra (Fig. 3) show the characteristic bands of the crosslinked PAN (blue spectrum) and characteristic additional peaks for the coating. The multiple peaks found in the region $1000\text{--}1300\text{ cm}^{-1}$ are characteristic for the C-O group found in the ester groups, that were formed by the reaction of morin hydrate OH groups with terephthaloyl chloride. The peak at 1450 cm^{-1} indicates the presence of aromatic C=C groups which are not found in the cross-linked PAN membrane but they are present in both the morin hydrate and the acyl chloride reactants. The elongated peak at 1680 cm^{-1} is also an indication for a higher amount of C=C groups present in the composite membrane due to the interfacial polymerization reaction. At last, the strong broad band in the $3200\text{--}3600\text{ cm}^{-1}$ region indicates the presence of OH groups from the morin hydrate natural polyphenol. It is important to mention again that these membranes were dried in a laboratory oven for 24 h at $80\text{ }^{\circ}\text{C}$ prior analysis to avoid any water residues in the samples so this last strong brand indeed corresponds to the OH functional group present in the bio-polyphenol and is not due to water traces in the membrane cupon.

Fig 2. Chemical reaction between the crosslinked PAN support, morin and terephthaloyl chloride.

Fig 3. FTIR spectra of the crosslinked PAN membrane (CL PAN) and the composite membrane with morin acid and terephthaloyl chloride (Morin/TPC).

3.3. X-ray photoelectron spectroscopy (XPS)

The high-resolution C1s core-level XPS spectrum of the composite membrane shown in Figure 4 shows peaks at the binding energy of 284.5, 285, 286.3, 287.8, and 288.7 eV that correspond to C=C, C-C, C-N and C-O, O=C-N, and O=C-O functional groups, respectively. The peaks of the O=C-N and O=C-O species at 286.3 and 287.8 eV indicate the formation of amide and ester groups [23], the peak at 286.3 corresponds to C-N and C-O species. The O=C-O group is formed by the reaction between terephthaloyl chloride and morin, whereas the O=C-N group formation is due to the interaction between the nitrogen groups present in the crosslinked PAN support with both interfacial polymerization reactants, morin and the acyl chloride. The C-N and C-O species shown in the spectra are a result of the reaction of the crosslinked PAN support and the morin. The C=C peak at 284.5 eV is another indicator of the introduction of the aromatic bio-polyphenol into the

membrane structure. All these XPS data confirm, that the morin /TPC thin-film layer was successfully formed as shown in Fig. 2.

Fig 4. XPS spectra of the PAN/morin-TPC composite membrane

3.4 Scanning electron microscopy (SEM)

The membrane surfaces of both, crosslinked PAN and morin/TPC thin-film composite membranes, were observed by SEM. Fig. 5A shows the crosslinked PAN membrane surface, where many large pores are clearly visible. Due to the beam sensitivity of the morin/TPC coating the SEM image of the coating is not perfect, but it can be seen, that the porosity and pore size decrease drastically after the coating by interfacial polymerization (Fig. 5B).

Fig 5. Surface of the crosslinked PAN membrane (A) and the morin/TPC thin-film composite membrane (B).

3.5. Membrane permeance and rejection performance

The morin-based composite membranes have been tested for permeance and rejection using three dyes dissolved in NMP. Permeance and rejection are summarized in Table 1. Prior to the interfacial polymerization reaction, the crosslinked PAN support membranes showed an average NMP permeance of 110 L/m² h bar and a low or zero rejection for dyes in the feed solutions. After the interfacial polymerization using morin as one monomer the flux decreased drastically and the rejection for all dyes increased to above 90%. This is a clear indicator for a successful film formation on top of the crosslinked PAN membrane.

Table 1. Permeance and rejection of morin /TPC thin-film composite and other membranes tested with different molecular weight dyes in NMP.

Solute	Permeance in NMP L / m ² h bar	Rejection %	Source
Rose bengal (Mw: 1017.64 g/mol) Abs at 563nm, (-) charge	0.2 $\sigma = 0.004^*$	97%	this work
Brilliant blue R250 (Mw: 825.97 g/mol) Abs at 592nm, (-) charge	0.3 $\sigma = 0.008^*$	96%	this work
Congo Red (Mw: 590.71 g/mol), Abs at 406nm, neutral	0.5 $\sigma = 0.005^*$	89%	this work
Polystyrene (Mw: 500 g/mol)	0.06	93%	Dutczack et al.[25]
Solvent Blue 35 dye (Mw: 350 g/mol)	0.1	62%	Fontananova et al. [26]
Remazol Brilliant Blue R (Mw: 626 g/mol)	0.2	94%	Anokhina et al. [27]
Rose Bengal dye (Mw:1017.6 g/mol)	0.2	98%	Vanherck et al. [28]

- Standard deviation for each of the different solvent/solute mixtures tested (3 coupons per solvent/solute mixture were tested).
- NMP permeance of PAN support: 110 L/m² h bar

The work presented here is focused on the solvent NMP, which is a special challenge for OSN using polymeric membranes, because NMP is good solvent for many polymers. NMP is an important solvent in the chemical and petrochemical industry. A relatively small number of publications describe the application of polymeric

nanofiltration membranes for NMP containing solutions. The literature data are summarized in Table 1. This comparison shows, that the morin-based membranes can compete with the best published performances in NMP solutions. It is also important to mention, that only a small flux variation was observed, when different membranes were tested. This small flux variation shows that the manufacturing of this membrane was reproducible; the high reactivity of the morin with the terephthaloyl chloride created a smooth and thin layer on top of the crosslinked PAN support.

We reported recently another membrane for solvent resistant nanofiltration, where tannic acid was used as monomer for the interfacial polymerization. Permeances up to 0.09 L/m² h bar with a molecular weight cut-off of approximately 800 g/mol (determined with dyes of different molecular weights) were obtained [24]. The replacement of tannic acid with morin resulted in membranes with a 3 times higher permeance with a similar MWCO of approx. 800 Dalton. The new reactant morin in the current work was dissolved in methanol and the crosslinked PAN support was conditioned with methanol prior the interfacial polymerization. This might have contributed to the increase in permeance.

Conclusion

With this work we demonstrated that morin, a bio-polyphenolic molecule abundant in nature in the guava plant (*Psidium guajava*), can be used as a monomer for the successful fabrication of solvent resistant nanofiltration membranes through interfacial polymerization using as a support a crosslinked PAN ultrafiltration membrane. The method of fabrication is simple and reproducible and therefore easy to upscale. This type of OSN membranes can withstand the harsh organic solvent NMP with an excellent performance, showing a permeance of 0.3 L/m² h bar with a rejection of 96% of Brilliant blue dye, which has a molecular weight of 826 g/mol. This permeance is superior to previously reported NMP resistant membranes and it is evident, that morin is a “green” alternative to be used as a monomer for the

fabrication of OSN membranes via interfacial polymerization. The cost of morin is low due to the abundance of this bio-polyphenol in nature, making this type of OSN membranes attractive for industrial harsh solvent separation processes.

Acknowledgements

The authors acknowledge Dr. Muhammed Nejb for the XPS analysis as well as Dr. Ali R. Behzad for the SEM images. This work was supported by King Abdullah University of Science and Technology (KAUST).

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Highlights

- Morin was used as a monomer for interfacial polymerisation.
- Solvent-resistant nanofiltration membranes with competitive fluxes were manufactured.
- Cross-linked PAN was applied as support membrane.