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Metallized membranes for membrane distillation

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Key words: membrane distillation, polymer membrane, magnetron sputtering, metal coating

Abstract

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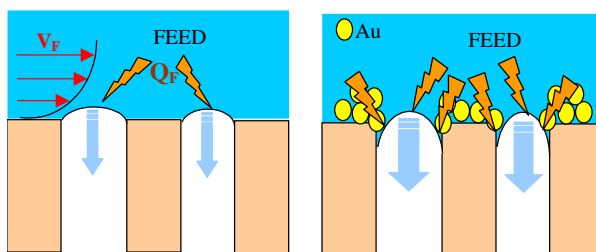


Fig. 1. Schematic model illustrating how metal deposition alters the meniscus shape and enhances heat transfer to the evaporation surface.

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Porous polypropylene capillary membranes were coated with nanoscale metallic layers (Au, Ag, and Cr) using magnetron sputtering and applied in membrane distillation (MD). The metallized membranes were assembled into submerged modules and their performance was experimentally evaluated. In submerged MD, heat and mass transfer are mainly controlled by a macroscopic boundary layer surrounding the membrane surface, which determines the overall transport resistance. In addition, a local near-wall boundary layer exists at the surface of individual membrane capillaries. The nanoscale metallic surface features introduced in this study primarily affect this near-wall region, enhancing thermal energy delivery to the evaporation interface while leaving the macroscopic hydrodynamics of the submerged module unchanged. As a result, a moderate increase in permeate flux, reaching 20–30%, was achieved for the metal-coated membranes compared to unmodified ones, demonstrating the potential of controlled surface metallization for improving membrane distillation performance.

Introduction

Membrane distillation (MD) is a thermally driven separation process in which volatile components of the feed evaporate and are transported through a porous hydrophobic membrane [1,2]. In systems containing non-volatile solutes, such as NaCl, only water vapour passes through the membrane pores. As a result, saline wastewater is separated into high-purity water (permeate) and a concentrated retentate containing the non-volatile components originally present in the feed solution [1-3].

Since water evaporation requires a substantial amount of energy, achieving high thermal efficiency is essential for the industrial implementation of MD [4,5]. The process is characterized by low single-pass water recovery, typically below 10%, and therefore requires feed recirculation, which further increases energy consumption [6]. However, the use of MD membrane modules that

enable heat recovery from the distillate stream can significantly reduce the overall energy demand [7]. In addition, MD can operate using low-grade thermal energy, making its integration with waste heat sources particularly attractive [8].

The performance of the MD process is strongly influenced by the design of the membrane module. Polypropylene capillary modules specifically developed for MD have demonstrated thermal efficiencies in the range of 70–80% [9]. During the concentration of highly saline solutions, fouling and salt crystallization frequently occur on the membrane surface. Under such conditions, submerged membrane modules, in which the feed flows on the shell side of the capillaries, are commonly applied to mitigate scaling and fouling effects [2,3].

The driving force for mass transfer in MD is the vapour pressure difference across the membrane, which arises from the temperature gradient and the composition of the boundary layers adjacent to the membrane surface [2]. Evaporation of water at the membrane interface leads to a reduction in surface temperature, a phenomenon known as temperature polarization, which limits process performance. This effect can be alleviated by increasing feed flow turbulence [2,10].

Under laminar flow conditions, a stationary boundary layer develops at the surface of capillary membranes, and heat transfer to the evaporation interface is governed primarily by conduction across this layer. The presence of nanoscale porous metallic surface features (e.g. 30–60 nm) can locally disturb the near-wall region of the boundary layer, thereby reducing combined heat and mass transfer resistances close to the evaporation interface. Furthermore, the nanoscale roughness introduced by metal coatings may promote the formation of a more stable evaporation interface at the pore mouths and enhance the local energy supply to the phase-change region, particularly under laminar flow conditions [11].

Therefore, the objective of this study was to investigate whether a nanostructured metallic layer deposited around the pore inlets of a hydrophobic polypropylene membrane can enhance permeate flux in the membrane distillation process.

Experimental

Capillary polypropylene membranes Accurel PP S6/2 (Membrana GmbH, Germany) were used in this study. The membranes had an outer diameter of 2.6 mm and a wall thickness of 0.4 mm. The maximum and nominal pore diameters were 0.6 μm and 0.22 μm , respectively, and the membrane porosity was 73%.

The membranes were modified by depositing nanoscale metallic layers using magnetron sputtering. The coating process was carried out with a Q150T sputtering system (Quorum Technologies, USA) equipped with a turbomolecular pump. Sputtering was performed under an argon atmosphere at a pressure of 1×10^{-2} mbar. The process conditions were as follows: ambient temperature (293–295 K), argon flow rate of 30 cm^3/min , discharge power of 40 W, DC power supply, and a discharge current of 100 mA.

Membrane samples were mounted on a rotating holder positioned 10 cm below the metallic target. Targets with a diameter of 57 mm and a metal purity of 99.99% were used for sputtering.

The deposition rate ranged from 5 to 7 nm/min. After completion of the coating on one side, the membranes were inverted and the sputtering process was repeated, which enabled the formation of a continuous metallic coating over the entire circumference of the capillary membranes.

The surface morphology of the modified membranes was examined using a scanning electron microscope (SEM) SU8020 (Hitachi Ltd., Japan). In addition, a laser scanning microscope (LSM) LEXT OLS5100 (Evident Europe GmbH, Germany) was employed to analyze the surface topography.

Submerged membrane modules were fabricated by potting the ends of three capillary membranes into glass tubes, resulting in an effective membrane length of 12 cm. Each module was placed in a feed tank with a total volume of 4 L. The feed solution was heated and continuously mixed using a magnetic stirrer operating at 750 rpm.

The distillate, maintained at a temperature of 291–293 K, was circulated through the lumen side of the capillaries using a peristaltic pump at a flow rate of 0.2 L/min. A schematic diagram of the experimental setup is shown in Fig. 1.

For comparison, a second submerged module fabricated from unmodified (non-coated) membranes was simultaneously immersed in the same feed tank and connected to an identical, independent distillate circulation loop.

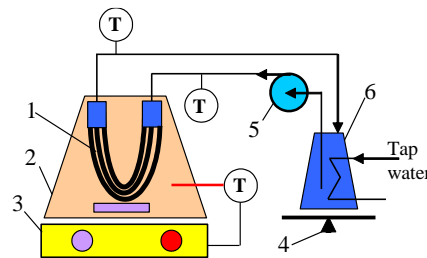


Fig.1. Experimental MD set-up: 1 – submerged module, 2 – feed tank, 3 – magnetic stirrer with heating plate, 4 – balance, 5 – peristaltic pump, 6 – distillate tank, T – thermometer.

The permeate flux (water vapour) transported through the membrane was determined by measuring the increase in water volume on the distillate side. The permeate flux was calculated according to the following equation:

$$J = \frac{V_D^2 - V_D^1}{A \cdot t} \quad [\text{L/m}^2 \text{ h}] \quad (1)$$

where: V_D^1 is the volume of water in the MD system on the distillate side at the start of MD process, L; V_D^2 the volume of water in the MD system on the distillate side at the end of MD process, L; A the working area of membranes in the MD module, m^2 ; and t the time of the MD permeate collection, h.

The electrical conductivity and pH of the examined solutions were measured by a 6P Ultrameter (Myron L). The calibration was made according to manufacture's instructions using pH calibrating buffer solutions (± 0.01 pH at 298 K). The conductivity was calibrated using the KCl standard solution (accuracy $\pm 1\%$ at 298 K).

Results

Metals are impermeable to water vapor; therefore, it is crucial that the deposited layer does not block the pore openings of the membrane. SEM images of LSM membranes coated with a 50 nm thick layer are shown in Fig. 2. Regardless of the metal used, a porous structure was obtained on the membrane surface. The resulting layer consists of nanograins with sizes ranging from 30 to 80 nm, as well as their agglomerates with an average size of up to 200-300 nm (Fig. 3).

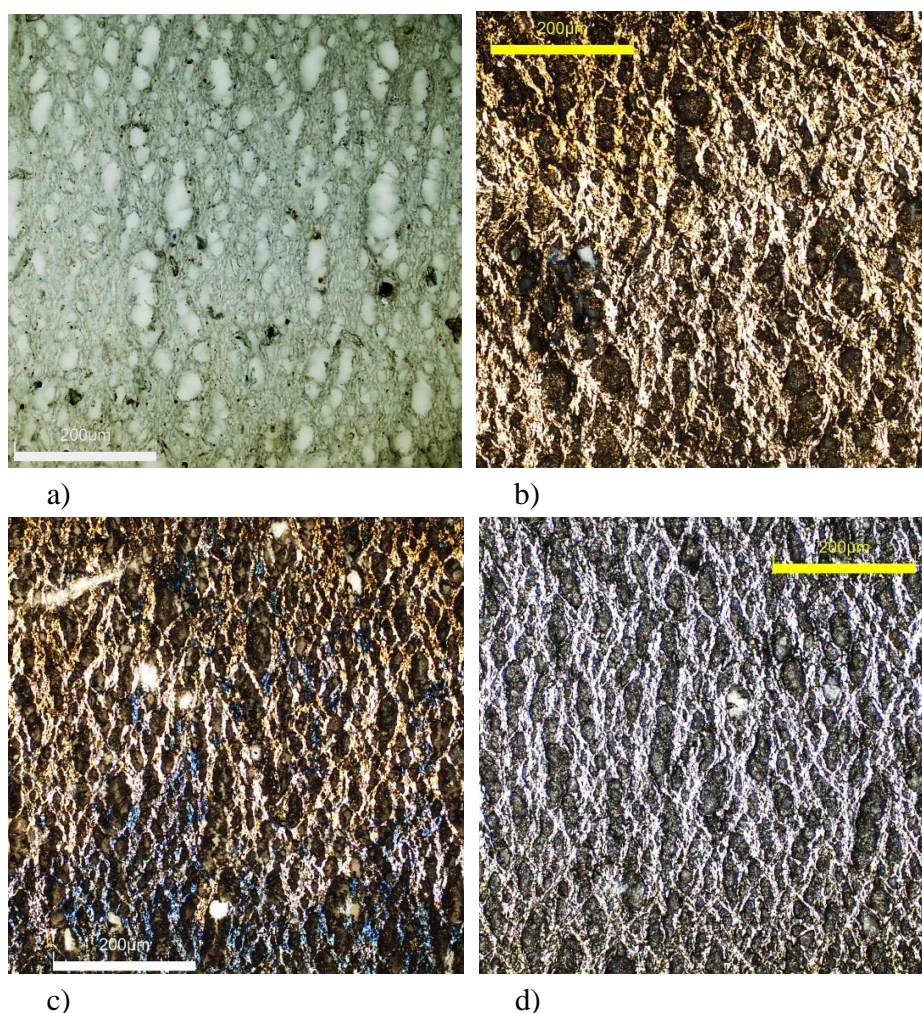


Fig. 2. LSM images of polypropylene membrane: a) unmodified, and metal-coated: b) Au, c) Ag, d) Cr.

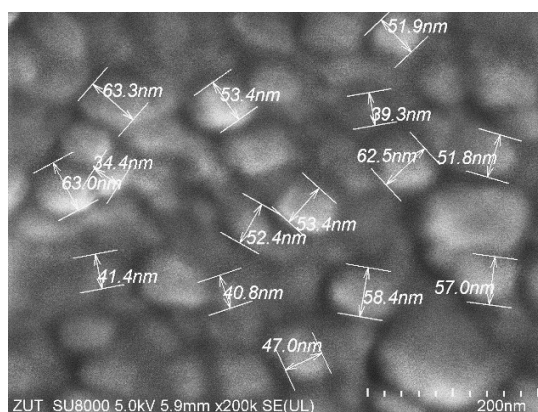


Fig. 3. SEM image of Au nanograins formed on the surface of a polypropylene (PP) membrane after metal deposition.

SEM studies confirmed that the deposited metals did not block the pores and predominantly covered the membrane surface around the pore entrances, as exemplified in Fig. 4 for the sputtered Au layer. The image was acquired using a low-angle backscattered electron (LA-BSE) detector; thus, brighter regions correspond to areas with a higher Au content. The arrangement of metal nanograins and their agglomerates around the membrane pores enables the transport of vapor or feed toward the pores of the underlying hydrophobic membrane.

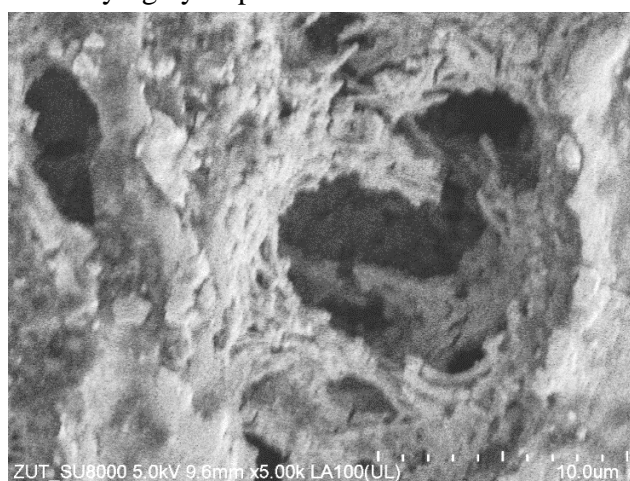


Fig. 4. SEM image of a membrane pore entrance showing an Au layer deposited at the pore edges, visible as brighter structures.

The obtained modified membranes were tested using distilled water as the feed, and the results are presented in Fig. 5. The driving force for mass transport in membrane distillation (MD) is the vapor pressure difference, which increases exponentially with the feed temperature [2, 10]. The observed changes in permeate flux confirmed this relationship. However, significantly higher permeate fluxes were obtained for membranes coated with a metal layer (up to 30%). This indicates that the metallic coating increases both the effective meniscus surface area from which water

evaporates and the intensity of heat transfer to this surface. This effect is schematically illustrated in Fig. 6.

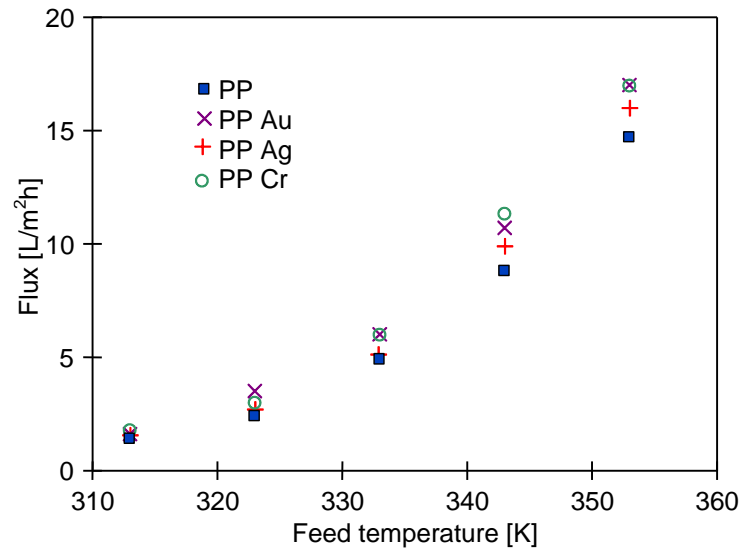


Fig. 5. Changes in permeate flux as a function of feed temperature and the type of metal layer deposited on the surface of the PP membrane.

Under laminar flow conditions, the effective thermal and mass transfer boundary layers adjacent to the membrane surface are estimated to be on the order of several micrometers. Although the metallic surface features are much smaller (30–100 nm), their presence within the near-wall region may locally reduce interfacial transport resistances and facilitate energy delivery to the evaporation interface. Moreover, as reported by Ojha et al. [11], the evaporation rate depends not only on the meniscus surface temperature but also on its shape. A more pronounced convex curvature of the meniscus, which is associated with hydrophilic pore walls, can enhance evaporation. Such an effect may occur as a result of depositing hydrophilic metals at the pore edges, as schematically illustrated in Fig. 6.

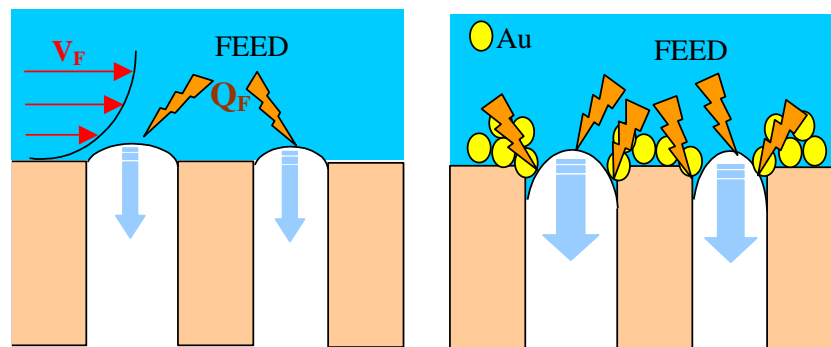


Fig. 6. Schematic model illustrating how metal deposition alters the meniscus shape and enhances heat transfer to the evaporation surface.

However, the deposition of hydrophilic metals onto the surface of hydrophobic membranes may promote partial water penetration into the pore interior and, consequently, accelerate pore wetting. This effect arises from the local modification of surface wettability at the pore entrances, where the presence of hydrophilic metallic islands can reduce the capillary resistance opposing liquid intrusion. Progressive wetting of membrane pores may ultimately lead to pore blockage, vapor transport suppression, and a reduction in permeate flux, which is commonly regarded as a critical limitation in membrane distillation processes.

Nevertheless, despite this potential risk, no deterioration in membrane performance attributable to metal deposition was observed during several days of continuous operation of the investigated membranes (Table 1). The permeate flux remained stable, and no indications of pore wetting or wetting breakthrough were detected. This suggests that the applied deposition conditions resulted in a metallic layer mainly confined to the membrane surface and pore edges, without significant penetration into the pore interior. As a result, the hydrophobic character of the membrane matrix was preserved, allowing the beneficial effects of enhanced heat transfer and increased evaporation area to be achieved without compromising long-term operational stability.

Table 1. Variation of permeate flux during long-term membrane distillation operation.

Membrane	Peremate flux [L/m ² h] after period [h]:								
	2	7	15	19	24	31	38	43	50
PP Au	16.95	16.80	16.83	16.80	16.80	17.1	16.91	16.81	16.78
PP	15.45	15.43	15.31	15.50	15.50	15.49	15.51	15.40	15.21

Conclusions

This study demonstrates that the deposition of thin metallic layers onto hydrophobic polymer membranes used in membrane distillation can significantly influence heat and mass transfer processes occurring at the membrane–fluid interface. While the presence of hydrophilic metals on a hydrophobic membrane surface may potentially promote water penetration into the pores and accelerate membrane wetting, the experimental results obtained during several days of operation showed no deterioration in membrane performance. In particular, no pore blockage or decline in permeate flux was observed for the investigated metal-coated membranes under the applied operating conditions.

The results indicate that magnetron sputtering enables the formation of porous metallic coatings composed of nanograins and their agglomerates, preferentially deposited around pore entrances rather than inside the pores. Such a coating architecture preserves pore accessibility while simultaneously modifying the near-wall region. The metallic layer enhances heat transfer toward the evaporation interface and increases the effective evaporation area by altering the meniscus shape at the pore entrances. Both effects contribute to a noticeable increase in permeate flux compared to unmodified membranes, especially at elevated feed temperatures.

Overall, magnetron sputtering appears to be a promising and controllable technique for tailoring membrane surface properties for MD applications. By carefully optimizing deposition parameters, it is possible to combine improved thermal performance with maintained membrane integrity and operational stability. These findings suggest that metallic surface modification may offer a viable pathway for enhancing the efficiency of membrane distillation systems without compromising long-term performance, although further studies under extended operation times and with different feed compositions are recommended.

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