Powdered activated carbon (PAC) – vacuum-assisted air gap membrane distillation (V-AGMD) hybrid system to treat wastewater containing surfactants: Effect of operating conditions

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ABSTRACT

Membrane distillation (MD), which uses hydrophobic porous membranes with a temperature gradient to produce pure water, has the potential to treat high-salinity wastewater. However, it cannot directly treat wastewater containing surfactants, which lower the surface tension and thus result in membrane wetting. To overcome this limitation, this study proposed a hybrid process consisting of powdered activated carbon (PAC) adsorption and MD, where PAC removes the surfactants in the wastewater to alleviate the wetting of the MD membranes. A bench-scale vacuum-assisted air gap MD (V-AGMD) equipment was adopted for the treatment of synthetic wastewater containing inorganic salts and surfactants. The conductivity of the permeate from V-AGMD was continuously monitored to detect membrane wetting. Without the use of PAC, the MD membrane was wetted within a short period, which decreased as the surfactant concentration increased. On the other hand, the addition of PAC retards the onset of wetting even at higher surfactant concentrations. The effectiveness of the PAC addition to the MD system on wetting control was examined under various conditions to elucidate its mechanism.

Keywords: High-salinity wastewater, Membrane distillation (MD), Surfactant, Vacuum-assisted AGMD (V-AGMD), Wetting

1. Introduction

Demand for freshwater is rapidly increasing worldwide due to population growth, industrialization, and urbanization while the amount of available freshwater is limited. [1-3]. To resolve the imbalance between the water resource and demand, water reclamation has drawn attention as a sustainable way of providing additional water supply. Water reclamation is the processing of wastewater to meet the water quality requirement for its reuse [4]. Membrane technology such as reverse osmosis (RO) is a key technique for water reclamation due to its high removal efficiency of target contaminants [5]. However, the operation of RO is restricted by the barriers including the osmotic pressure, concentration polarization, and membrane fouling [6]. This is especially problematic when industrial wastewater containing high concentrations of ionic species should be reclaimed as the process water.

Such limitations of RO has motivated the development of novel technologies that allow the treatment or reuse of high salinity

wastewater. Being one of them, membrane distillation (MD) holds the potential to allow the production of high-purity water [7]. MD is one of the membrane contactors and can separated vapors from liquid-phase water using hydrophobic porous membranes [8-11]. There are four typical MD configurations, including direct contact MD (DCMD), air gap MD (AGMD), sweeping gas MD (SGMD), and vacuum MD (VMD) [12-14]. Among them, AGMD is a preferred configuration for practical implementation due to its high thermal efficiency and the capability of stable operation [13]. Nevertheless, AGMD has drawbacks associated with relatively low flux and productivity compared with DCMD or VMD [12, 15, 16]. Accordingly, a handful of works have been done to overcome this shortcoming by increasing the flux of AGMD with the application of vacuum pressure [12, 17, 18].

While AGMD is applicable for the reclamation of high-salinity wastewater, it may suffer from critical problems caused by membrane wetting [19, 20]. Membrane wetting is regarded as a primary barrier to the widespread industrial use of MD technology. Membrane wetting involves three stages: surface wetting, partial

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wetting, and fully wetting [19, 20]. Surface wetting means that only the membrane surface is wetted by the liquid and does not affect membrane performance. Partial wetting can lead to a reduction in salt rejection as the membrane pores are partially filled with liquid. When the membrane pores are fully filled with liquid, the membrane becomes fully wet. A fully wetted membrane will lose its separation through MD [19].

Membrane wetting may occur when feed water contains surfactants that can reduce surface tension [21, 22]. Surfactants are widely used in household goods, detergents, industrial processes, and pesticides [23]. Surfactants are usually amplified organic compounds, meaning they contain both hydrophobic and hydrophilic groups. This structure of the surfactant causes the hydrophobic tail to adhere to the membrane surface and pore, resulting in hydrophilization of the membrane. Therefore, special attention must be paid to wastewater containing surfactants in the MD process. Various techniques, such as physical, chemical, biological, and membrane treatment, have been used to remove surfactants as a part of wastewater treatment [24]. To the best of the authors' knowledge, however, few works have attempted to remove surfactants from the feed water to mitigate the wetting of MD membranes.

In this paper, a technique based on activated carbon (AC) adsorption was proposed to mitigate the wetting of MD membranes during the treatment of high-salinity feed water containing surfactants. A bench-scale vacuum-assisted AGMD (V-AGMD) system was operated without and with added powdered activated carbon (PAC) and the wetting properties were compared. Experiments were conducted to analyze the effect of operating conditions and to elucidate the different observed wetting behaviors. The novelty of this work lies on the combination of PAC adsorption and V-AGMD for the treatment of high-salinity and low surface tension wastewater. Although many studies have been done in the integration of PAC with pressure-driven membranes [25–28], there have been few works on the application of PAC in the V-AGMD process especially for wetting control.

2. Materials and Methods

2.1. Materials

The hydrophobic polyvinylidene fluoride (PVDF) membranes were purchased from Merck Millipore (GVHP, Merck Millipore, U.S.A.). The average pore size, thickness, and porosity of the membranes are 0.22 μm , 125 μm , 75%, respectively. The PAC was obtained from Samchun Pure Chemical Co. Ltd. Korea. Sodium dodecyl sulfate (SDS) and NaCl were purchased from Sigma Aldrich and they were used to prepare the synthetic wastewater for MD experiments.

2.2. Batch adsorption Experiments

Batch adsorption was carried out in a 1,000 mL flask to investigate the adsorption of SDS by the PAC. SDS solutions with the initial concentrations ranging from 1.71 mg/L to 80 mg/L were prepared. The temperature of the solution was set to 60°C and the PAC dose was 0.6 g. It took about 1 h to reach adsorption equilibrium in this experiment. After equilibrium, the PAC was separated from

the solution by filtration using 0.45 m Whatman filter papers. Then the concentrations of SDS were measured using a spectrophotometer (DR4000, Hach). The adsorption capacity was determined based on the difference between the initial and final SDS concentrations.

2.3. V-AGMD

A schematic diagram of the experimental setup for V-AGMD is shown in Fig. 1. The membrane cell was made of acrylic resin and its depth, width, and length were 2 mm, 20 mm, and 60 mm, respectively. The effective membrane area was 0.0012 m^2 . The temperature of the feed and coolant was maintained at 60°C and 20°C, respectively. The flow rates were set to 0.6 L/min for the feed and 0.4 L/min for the coolant. Deionized (DI) water was used as the feed solution to measure the pure water flux as a function of vacuum pressure.

Fig. 1. Schematic diagram of laboratory-scale V-AGMD system.

2.4. Effect of SDS on Membrane Wetting

The high-salinity synthetic wastewater of varied SDS concentration was prepared to investigate the effect of SDS on membrane wetting in the V-AGMD process. The compositions of the synthetic wastewater are presented in Table 1. The V-AGMD experiments were carried out with the vacuum pressure of 0.5 bar. The temperatures and flow rates were the same as those of the pure water tests. In the V-AGMD system, the flux increases due to the pressure difference between the feed and permeate if wetting occurred. The permeate conductivity also increases with the progress of wetting because the feed water penetrates the wetted pores and mixed with the permeate. Accordingly, both flux and permeate conductivity were continuously monitored to examine the progress of fouling and wetting.

2.5. Control of Membrane Wetting by PAC Adsorption

To mitigate the wetting by SDS in the synthetic wastewater, the adsorption by PAC was applied to the V-AGMD system. Two different methods of PAC adsorption were compared. In the first method, the PAC was directly added to the feed tank of the V-AGMD system to let the adsorption occur during the MD operation. In the second method, the PAC was mixed with the feed water for 1 hour and

Table 1. Summary of Experimental Conditions

Configuration	PAC	Feed solution	Vacuum pressure
V-AGMD	-	NaCl 50,000 mg/L SDS 3 mg/L	0.5 bar
	-	NaCl 50,000 mg/L SDS 5 mg/L	0.5 bar
	-	NaCl 50,000 mg/L SDS 7 mg/L	0.5 bar
V-AGMD with PAC adsorption	0.6 g (No prefiltration)	NaCl 50,000 mg/L SDS 3 mg/L	0.5 bar
	0.6 g (No prefiltration)	NaCl 50,000 mg/L SDS 3 mg/L	0.65 bar
	0.6 g (No prefiltration)	NaCl 50,000 mg/L SDS 3 mg/L	0.8 bar
	0.6 g (Prefiltration)	NaCl 50,000 mg/L SDS 3 mg/L	0.8 bar

then removed by the filter paper before the MD operation. In both cases, the initial SDS concentration was fixed at 3 mg/L and the vacuum pressure was adjusted from 0.5 bar to 0.8 bar, as shown in Table 1.

The permeate flux was presented in terms of time and volume concentration factor (VCF). The VCF, defined as a ratio of the feed volume to concentrate volume, indicates the extent of concentration:

$$VCF = \frac{V_f}{V_c} = 1 + \frac{V_p}{V_c} \quad (1)$$

where V_f , V_c , and V_p are defined as the volume of feed, concentrate, and permeate, respectively. The solute concentrations of the permeate were measured at different concentration factors.

2.6. Contact angle Measurement

The liquid contact angle (CA) was measured after the V-AGMD experiments. The CA of the membranes was measured by the sessile drop contact angle technique using a CA measurement device (Smart Drop, South Korea). First, membrane samples were placed on a plate and then water droplets (3~4 mL) were dropped onto the membrane surface. In a stationary state, the camera in the device captured an image of the droplet and the CA was automatically determined by the software. At least seven measurements were made for each sample and the average CA value was recorded.

2.7. Liquid entry Pressure Measurement

The liquid entry pressures (LEPs) were also measured after the V-AGMD experiments. LEP is the minimum pressure required to enable water to penetrate a membrane's pores. LEP is affected by the hydrophobicity of the membrane, the maximum pore size, and the shape of the pores [29]. For the current study, the LEP of the membranes was measured using an in-house LEP apparatus. First, 50 mL DI water was added to the water chamber, then a

dry membrane sample (with an effective surface area of 7 cm²) was placed on the apparatus. Then nitrogen gas was supplied to the bottom of the water chamber. The pressure of the nitrogen gas was increased in a stepwise manner until the first droplet of water was observed on the membrane surface. To confirm reproducibility, at least three measurements were taken.

2.6. Scanning Electron Microscopy

The samples were coated with Pt. The surfaces and cross-sections of the membranes were then examined by FE-SEM (FE-SEM 7800F Prime, JEOL Ltd. Japan).

3. Result and Discussion

3.1. Effect of SDS Concentration on Membrane Wetting

Since wetting is related to the reduction of the surface tension of the feed solution, the addition of SDS is expected to accelerate it [30]. Moreover, SDS may also adsorb on the membrane surface, reduce its surface tension, and thus result in wetting. To examine how SDS affects the wetting, a series of MD experiments were carried using feed solutions with different SDS concentrations. In Fig. 2(a), where there was no SDS added to the feed solution, the flux and permeate conductivity were stable up to the operation time of 4,500 min. After 5,000 min, the permeate conductivity began to increase, indicating that wetting started. Without SDS, wetting occurred due to membrane fouling due to salt precipitation [19]. Nevertheless, it was observed only after a long period. The flux and permeate conductivity with the addition of 3 mg/L SDS are shown in Fig. 2(b). The flux increased from the beginning and the conductivity increased after 200 min, suggesting that wetting happened from the early stage of the MD operation.

The extent of wetting became more significant when SDS concentration increased. As shown in Fig. 2(c), the flux increased from the beginning at the SDS concentration of 5 mg/L. Moreover, the conductivity increased up to 1,376 S/cm within 600 min. A further increase in the SDS concentration to 7 mg/L resulted in a higher increasing rate of the flux and conductivity as depicted in Fig. 2(d). To quantitatively compare the effect of SDS concentrations on wetting, NaCl rejection was calculated at 600 min in each case. Without SDS, the NaCl rejection by MD was 99.98%. However, the NaCl rejections at SDS concentrations of 3, 5, and 7 mg/L were 98.94, 98.24, and 96.12%, respectively. Compared with the case without SDS, the salt passage increased by 45.4, 75.9, and 167 times, respectively, at the SDS concentrations of 3, 5, and 7 mg/L.

After the MD experiment, the liquid entry pressure (LEP) and the contact angle of the membranes were measured. The intact membrane had the LEP of 1.91 ± 0.22 bar and the contact angle of 126.78 ± 1.0°. After the MD experiments, the LEP and contact angle were reduced to zero in all cases. These results confirmed that wetting occurred during the MD operation.

3.2. Adsorption of SDS by PAC

It is evident from Fig. 2 that a more severe wetting was induced with an increased SDS concentration. This suggests that the wetting may be mitigated by reducing the SDS concentration.

Fig. 2. Effect of SDS concentration in the synthetic feed water on flux and permeate conductivity in V-AGMD process. (Conditions: feed temperature; 60°C; coolant temperature; 20°C, feed flow rate; 600 mL/min; vacuum pressure, 0.5 bar; feed solution: NaCl 50,000 mg/L and SDS 0~7 mg/L) (a) SDS 0 mg/L (b) SDS 3 mg/L (c) SDS 5 mg/L (d) SDS 7 mg/L.

Accordingly, PAC was considered to reduce SDS concentration by adsorption. First, the adsorption properties of SDS onto PAC was analyzed in batch experiments. Two isotherm models, including Langmuir and Freundlich isotherms, were fitted to the experimental data for the analysis of the mechanism of SDS adsorption by the PAC [31]:

Langmuir equation:

$$\frac{c_e}{q_e} = \frac{1}{q_m K_L} + \frac{c_e}{q_m} \quad (2)$$

Freundlich equation:

$$\log q_e = \log K_F + \frac{1}{n} \log c_e \quad (3)$$

where c_e is the equilibrium concentration of the adsorbate (SDS) on the adsorbent (PAC), q_e is the amount of the adsorbate at equilibrium, K_L is the Langmuir constant related to the free adsorption energy and the reciprocal of the concentration at which half-saturation of the adsorbent is reached, q_m is the monolayer adsorption capacity, and K_F and $1/n$ are the Freundlich constants, which correspond to adsorption capacity and adsorption intensity, respectively.

Fig. 3. Adsorption isotherm of SDS by PAC.

The parameters determined by the Langmuir equation and Freundlich equation were listed in Table 2. The Freundlich model fit shows a higher R^2 than the Langmuir model. The Freundlich isotherm of SDS adsorption was plotted in Fig. 3. The K_F and $1/n$ for the Freundlich model were 1.1514 mg/g and 0.8988, respectively.

Table 2. Comparison of Adsorption Isotherm Model Fits

Adsorption model	Estimated model parameters	R ²
Langmuir	KL = 1.407 L/mg qm = 13.42 mg/g	0.8944
Freundlich	KF = 1.1514 mg/g 1/n = 0.8988	0.9629

3.3. Control of Wetting by PAC Adsorption

3.3.1. Effect of PAC addition

After the batch adsorption tests, MD experiments were carried out to investigate the effect of PAC on membrane wetting by SDS. The feed solution containing 50,000 mg/L NaCl and 3 mg/L SDS was prepared for the MD experiment. PAC was directly added to the feed tank and recirculated during the MD operation. The results are presented in Fig. 4. Compared with Fig. 2(b), which is the results of the MD experiment using the same feed solution without PAC addition, the flux and permeate conductivity were maintained almost constant for a longer period of operation time. In Fig. 2(b), the flux and conductivity increased from the beginning but they were stable up to 5,700 min in Fig. 4. Since these results are similar to those without SDS (Fig. 2(a)), likely, the increases in flux and conductivity in Fig. 4 are also attributed to the fouling due to NaCl precipitation.

Fig. 4. Effect of PAC addition on flux and permeate conductivity in V-AGMD process. (Conditions: feed temperature; 60°C; coolant temperature; 20°C, feed flow rate; 600 mL/min; vacuum pressure, 0.5 bar; feed solution: NaCl 50,000 mg/L and SDS 3 mg/L).

3.3.2. Effect of vacuum pressure on wetting mitigation by PAC adsorption

In V-AGMD, the application of vacuum improves the flux and productivity. As shown in Fig. 5, the pure water flux in V-AGMD increase from 5.5 L/m²-h to 28 L/m²-h with vacuum pressure. However, an increase in the vacuum pressure increases the risk of wetting because of the hydraulic pressure difference across the membrane. This is because only the vacuum pressure of 0.5 bar was considered in Fig. 2. However, it was expected that a higher

Fig. 5. Correlation with vacuum pressure and pure water flux in V-AGMD process. (Conditions: feed temperature; 60°C; coolant temperature; 20°C, feed flow rate; 600 mL/min; vacuum pressure, 0 ~ 0.8 bar; feed solution: deionized water).

vacuum pressure could be applied in V-AGMD with PAC because of a reduced risk of the wetting due to PAC adsorption.

Fig. 6(a) shows the changes in flux and permeate conductivity in V-AGMD at 0.65 bar vacuum. The initial flux was maintained at 18 L/m²-h until 3,500 min and then abruptly increased. The permeate conductivity also increased after 3,600 min. When the vacuum pressure was 0.5 bar (Fig. 4), the flux and conductivity were almost constant before 5,700 min. Compared with these results, the wetting occurred more rapidly at higher vacuum pressure. Nevertheless, the addition of PAC is effective to mitigate the wetting by SDS. Since the wetting occurred after 200 min at 0.5 bar without PAC (Fig. 2(b)), more severe wetting should occur at 0.65 bar without PAC. In Fig. 6(b), the vacuum pressure was set to 0.8 bar in V-AGMD with PAC. The flux was initially 22 L/m²-h and increased after 2,700 min. At the similar time, the permeate conductivity increased, indicating the occurrence of wetting. Again, the addition of PAC was found to be effective to mitigate the wetting by SDS but the effect became lower at the higher vacuum pressure.

When the initial flux is different, it is sometimes misleading to analyze the changes in fouling and wetting in terms of operation time. This is because the feed concentration in MD systems may be different even at the same operation time in such cases. Accordingly, the results in Fig. 4, Fig. 6(a), and Fig. 6(b) were further analyzed in terms of VCF. As shown in Fig. S1(a) in the supplementary material, the flux increased due to the wetting at VCF of 3.3, 2.5, and 1.8, respectively, when the vacuum pressures were 0.5, 0.65, and 0.8 bar. The permeate conductivity also increased at 3.2, 2.5, and 2.4, respectively, as depicted in Fig. S1(b). Based on these results, it can be concluded that both the time and VCF for the wetting occurrence decrease by increasing the vacuum pressure. In V-AGMD, the wetting occurs when the applied vacuum is higher than the liquid entry pressure. Accordingly, an increase in the vacuum pressure should accelerate the wetting if all the other conditions are the same.

Fig. 6. Dependence of flux and permeate conductivity on time in V-AGMD process with PAC adsorption under different vacuum pressures. (Conditions: feed temperature; 60°C; coolant temperature; 20 °C, feed flow rate; 600 mL/min; feed solution: NaCl 50,000 mg/L and SDS 3 mg/L; PAC dose, 0.6 g). (a) 0.65 bar (b) 0.8 bar.

3.3.3. Comparison of PAC adsorption methods

In the previous experiments, PAC was directly added to the feed tank and recirculated during the MD operation. It was confirmed by visual observation that some PAC was deposited on the membrane. Accordingly, it appears that SDS was continuously adsorbed onto PAC in the feed solution as well as on the membrane surface. In Fig. S2 in the supplementary material, a set of MD experiment was carried out with a different PAC adsorption method. Here, the feed solution was mixed with PAC for 60 min and filtered using a 0.45 μ m microfilter. As a result, no PAC remained during the MD experiment. Although the initial SDS concentration was 3.0 mg/L, the SDS concentration in the feed solution after the PAC adsorption was 0.3 mg/L. However, the flux increased from the beginning and the permeate conductivity started to increase after 400 min. Unlike the case with the PAC addition without prefiltration (Fig. 4), the effect of PAC adsorption on wetting miti-

gation is not significant. In fact, the results were similar to the those without PAC addition (Fig. 2(b)).

The flux and permeate conductivity of the MD experiments with different PAC adsorption methods were compared in Fig. S3 in the supplementary material. The results are presented as a function of VCF. When PAC was not prefiltered, the flux and conductivity remained almost constant up to the VCF of 2.4. According to the isotherm model previously obtained from the batch tests, the equilibrium concentration of SDS at VCF = 2.4 was calculated to be 0.53 mg/L, which is higher than the initial concentration of SDS in the case of MD with PAC prefiltration (0.3 mg/L). This implies that there is an additional mechanism of wetting control in the case of MD without PAC prefiltration or continuous PAC adsorption during MD operation.

To further investigate the mechanisms of the wetting control, the SEM images of the MD membranes were compared. Fig. S4(a) and S4(b) in the supplementary material show the intact MD membrane and the membrane after the MD operation with PAC prefiltration. No clear difference was found from these two images although the initial wetting was found in Fig. S4(b). This is because the wetting by SDS resulting from the reduction of surface tension cannot be observed using SEM. In Fig. S4(c), the surface of the membrane was covered by PAC deposits. Accordingly, a substantial fraction of the membrane surface is not directly exposed to the feed solution and the PAC deposits seem to cover it to protect from SDS in the feed solution. Since the size of PAC is sufficiently large (1 ~ 20 μ m), it does not seem to provide additional resistance to mass transfer. On the other hand, it may act as a protective “coating” against the wetting by SDS.

The proposed mechanism of the PAC effect on the wetting is illustrated in Fig. S5 in the supplementary material. When there was no PAC during MD operation, SDS in the feed solution causes wetting. If SDS concentration is very low, wetting may not occur. However, based on the result in Fig. S2, wetting occurred at even the SDS concentration of 0.3 mg/L. When PAC exists during the MD operation, the deposition of PAC led to the formation of the PAC layer on the membrane surface (Fig. S5(b)). Accordingly, the PAC concentration at the membrane surface is much higher than that in the feed solution, resulting in very low SDS concentration near the membrane surface. In fact, the adsorbed amount of SDS was 4.5 mg/g when the initial concentration of SDS was 3 mg/L. This corresponds to 1.4 times higher adsorption compared with the results of the batch adsorption experiments, which can be explained by the PAC layer on the MD membrane surface.

4. Conclusion

In this study, a laboratory-scale V-AGMD was used to treat synthetic wastewater containing NaCl of 50,000 mg/L and SDS. The PAC adsorption was suggested and applied to mitigate membrane wetting by SDS. Based on the results, the following conclusions were drawn:

- (1) When SDS exists in the high-salinity feed solution, wetting began within 200 min, leading to rapid increases in the flux and permeate conductivity. Without SDS, wetting occurred due to salt precipitation after 5,000 min, which is

more than 25 times longer than that with SDS.

- (2) The adsorption isotherm of SDS by the PAC used in this study could be described by the Freundlich isotherm with K_F of 18.30 mg/g and $1/n$ of 0.7833.
- (3) The addition of the PAC into the feed tank resulted in the dramatic retardation of wetting by SDS. The increase in flux and conductivity were observed after 5,700 min, suggesting that the wetting occurred not by SDS but by salt precipitation.
- (4) The effect of PAC on the wetting mitigation was also found at higher vacuum pressures, indicating that a higher degree of vacuum may be used for V-AGMD. Nevertheless, an increase in vacuum reduced the time of wetting occurrence.
- (5) When PAC adsorption was implemented prior to MD operation, wetting still occurred within 200 min at the reduced SDS concentration (0.3 mg/L). This is attributed that PAC deposits provide additional protection against wetting by SDS. This suggests that PAC adsorption should be continuously done during the MD operation.

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Author Contributions

Y.K. (Master Student) conducted all the experiments and wrote the manuscript. S.L. (Professor) revised the manuscript. Y.C. (Associate Professor) revised the manuscript. J.C. (Ph.D. student) conducted the experiments.

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