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Ammonia recovery from high concentration wastewater of soda ash industry with membrane distillation process

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ABSTRACT

Vacuum membrane distillation was applied in the treatment of high ammonia content wastewater for the recovery of ammonia. Hydrophobic poly(vinylidene fluoride) hollow fiber membranes were used as the separation media. The initial ammonia concentration and pH value of the feed solution were destined owing to the composition of the wastewater. The effects of feed temperature, velocity, and operation time on the ammonia removal efficiency, separation factor, and water content in produced ammonia vapor were studied. The results showed that feed temperature was the most important factor affecting ammonia removal efficiency, which was improved from 78 to 99% as the temperature enhanced from 30 to 60˚C. The feed velocity and operation time also affected the ammonia removal efficiency and separation factor obviously. A pilot-scale experiment was carried out using a membrane module of 20 m^2 membrane area. Preliminary results showed that proper ammonia removal efficiency and product property were obtained.

Keywords: Ammonia removal and recovery; Vacuum membrane distillation; Poly(vinylidene fluoride); Hollow fiber membrane; Hydrophobic membrane

1. Introduction

As a common pollutant, dilute ammonia wastewater exists in many industries, such as chemical fertilizer plants, chlorine alkali industries, chemical plants, meat packing plants, etc. Excessive ammonia will cause the eutrophication of water and further more harm to animals and human beings. So it is necessary to remove ammonia from the water [1].

Researchers have developed many methods or processes for ammonia removal. Pagans et al. utilized biofiltration to remove ammonia from composting exhaust gases [2]. Zhang et al. used an aerobic cathode microbial fuel cell to remove ammonia from wastewater [3]. Degermenci et al. and Zhang et al. used air stripping for ammonia removal [4,5]. Tanaka and Matsumura applied ozone for ammonia removal [6]. Gendel and Lahav comparesd ion exchange and electrochemical regeneration for ammonia removal [7]. Membrane contactor, especially membrane adsorption, was recently adopted for ammonia removal using acid, especially vitriol as drawing solution [1,8–11]. But there are still many problems needed to be overcome, which include energy consumption, secondary pollution, the complexity of equipment, and the

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dependent of biological methods on the activity of micro-organisms.

As a new type of membrane separation technology, membrane distillation (MD) has gained worldwide attention owing to its advantages comparing to traditional separation technology [12]. The principle of separation in the MD process is based on the volatility difference of each substance, and the difference of vapor pressures on the two sides of the membrane is the driving force of the process [13]. The membranes used in the MD process are hydrophobic microporous membranes. Theoretically, liquid can neither wet the membrane nor penetrate through the membrane pores; only vapor can penetrate through the membrane.

In recent years, many researchers have applied MD process to treat solutions containing volatile substances such as ammonia and ethanol, and obtained respectable achievements [14–16]. Qu et al. comparatively investigated the ammonia removal efficiency of direct contact membrane distillation (DCMD), hollow fiber membrane contactor, and modified direct contact membrane distillation (MDCMD) [17]. The results showed that the ammonia removal efficiency of DCMD, HMC, and MDCMD were 52, 88, and 99.5% within 105 min. Xie et al. adopted sweeping gas membrane distillation (SGMD) in ammonia removal from simulated wastewater with a concentration of 100 mg/L [18]. Ding et al. adopted three MD configurations in ammonia removal experiment, and the results showed that vacuum membrane distillation (VMD) had the highest mass-transfer coefficient (Ka) but the lowest selectivity (β); DCMD gave the highest β and moderate Ka; SGMD showed the moderate β and the lowest Ka [19]. Duong et al. studied the efficiency of VMD and SGMD in ammonia removal process [20], and their results supported that of Ding's work. EL-Bourawi et al. also selected VMD for ammonia removal from water solution owing to the higher flux of the configuration [21].

Despite massive literatures on ammonia removal, most of them focused on wastewater with relative low ammonia content and mainly aimed at removing ammonia from the water instead of reclamation and reuse of ammonia. Seldom report could be found on the treatment of solutions with high ammonia content, even less on the reclamation of ammonia from the solution.

While there are many industries, such as soda ash production by Solvay method (ammonia-soda process), using ammonia as raw material and discharging wastewater with high ammonia concentration. At present in China, most of these plants employ evaporation or distillation method at about 95℃ to recover the ammonia from the solution. However, evaporation at 95℃ not only means high-energy consumption but also results in high water content in the produced ammonia and affects the subsequent production efficiency. So, it is really necessary to develop effective ammonia recovering method from high ammonia content solution.

In this work, VMD process was employed for the recovery of ammonia gas from near-saturated ammonia solution. It is important to understand the mass-transfer efficiency of ammonia and the competition between ammonia and water vapor during the VMD process. Literatures showed that, for the removal of ammonia from dilute solution, there were four key factors affecting the ammonia removal efficiency. The factors included the initial ammonia concentration, pH value of the solution, feed temperature, and feed velocity [20]. These factors may also play an important role in the treatment of high concentration ammonia solution, but real experience is still lack and needs further detection. This work mainly focused on the recovery of ammonia from the solution from soda ash plant. So the initial ammonia concentration and pH value were fixed. The effect of operating parameters such as feed temperature and velocity on the ammonia recovery performance, together with the variation of the performance during VMD experimental process were thoroughly studied.

2. Experimental

2.1. Reagents

All chemical reagents used in the experiment are analytical grade reagents. The feed ammonia solutions were prepared by commercial ammonia solution. The water used in the study is deionized water. A dilute sulfuric acid solution was used as the tail gas absorption solution. The ammonia concentration of the samples was measured by an ammonia-gas-sensing electrode.

2.2. Membrane and membrane module

Polyvinylidene fluoride (PVDF) hollow fiber hydrophobic membrane developed in our lab was used in the experiment. Cylinder membrane module was prepared and used in VMD process. The parameters of membrane and membrane module were listed in Table 1.

Table 1
Characteristics of the membrane modules

Membrane		Membrane module	
Outer diameter (μm)	500	Module diameter (mm)	50
Inner diameter (μm)	750	Length (cm)	20
Pore size (μm)	0.16	Membrane number	300
Porosity (%)	80	Membrane area (m^2)	0.0942

2.3. Experimental procedure

The VMD experimental setup used in this work was shown in Fig. 1. The temperature of the feed solution was controlled by a super thermostatic water bath (501A, Shanghai Jingke Co. Ltd. China). When the temperature of the feed solution reached the destined temperature, the solution was pumped into the membrane module by a magnetic pump (MP55, Shanghai Xin Xi Shan pump Co. Ltd. China) and circulated through the lumen of the hollow fiber membrane. The vacuum on the shell side of the module was given and controlled by a water circulating vacuum pump (SHB-Ⅲ, Shanghai Jingke Co. Ltd. China). The vacuum degree was measured by a mercury manometer at the shell side of the membrane module. The tail gas was absorbed by sulfuric solution. The ammonia concentration was detected by an ammonia-gas-sensing electrode.

2.4. Ammonia removal efficiency and separation performance

The ammonia removal efficiency, η, is determined by Eq. (1) [10,11,20]:

$$\eta\% = \frac{(C_0 - C_t)}{C_0} \times 100 \tag{1}$$

where C_0 is the ammonia concentration in the initial feed solution (g/L) and C_t is the ammonia concentration at time t (min).

Separation factor, S_t, represents the measure of the preferential transport of ammonia, which is defined by Eq. (2) [11,13].

$$S_t = \frac{(C_{\mathrm{NH_3}}/C_{\mathrm{H_2o}})_p}{(C_{\mathrm{NH_3}}/C_{\mathrm{H_2o}})_f} \tag{2}$$

where numerator and denominator separately are the ammonia molar concentration and the water molar concentration at the permeate side (p) and feed side (f).

In this study, the aim is to recover the ammonia from the solution, and the quality of products should be considered. As is mentioned above, S_t can represent the quality of products but it is not enough. Water content in ammonia ($t_{\mathrm{H_2O}}/t_{\mathrm{NH_3}}$), which is

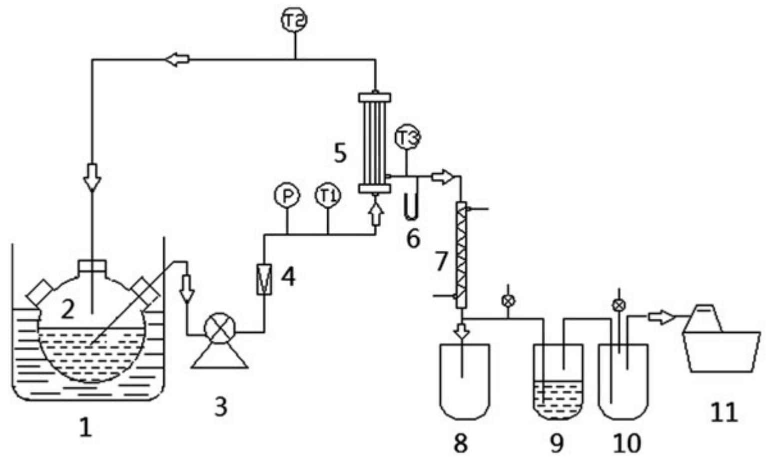

Fig. 1. VMD experimental setup for ammonia removal from wastewater. (1) thermostatic water bath, (2) feed tank, (3) magnetic circulating pump, (4) flow meter, (5) membrane module, (6) mercury manometer, (7) condenser pipe, (8) fluid reservoir, (9) tail gas tourie, (10) surge flask, and (11) vacuum pump.

concerned by the soda ash plant, is presented to measure the quality of the ammonia:

$$t_{H_2O}/t_{NH_3} = \left(\frac{m_{H_2O}}{m_{NH_3}}\right)p \tag{3}$$

where m_{H_2O} and m_{NH_3} are the ammonia and water mass at the permeate side, respectively.

3. Results and discussion

3.1. Effect of feed temperature

The effect of feed temperature on the ammonia removal process was studied using 18.0 wt.% ammonia solution with feed velocity of 0.21 m/s and vacuum pressure of 88 kPa. The results were shown in Fig. 2.

From Fig. 2, we could see that the variation of ammonia removal efficiency showed the same trend though the feed temperature was different (in the range of 30–60℃). It increased rapidly in the beginning, then the increasing rate slowed down and at last the ammonia removal efficiency remained at a relatively stable level.

The figure also showed that feed temperature affected the ammonia removal process greatly. As the temperature increased from 30 to 60℃, both the ammonia removal efficiency and the increasing rate of the efficiency is enhanced. For example, when the VMD process was operated with a feed temperature of 30℃, the ammonia removal efficiency increased from 0 to about 60% in 60 min, then upto 80% as the experiment continued 150 min and became stable then. When the feed temperature was 60℃, the ammonia removal efficiency increased from 0 to 84% in 40 min, then upto 92% at 60 min and 99% at 120 min, then became stable at last.

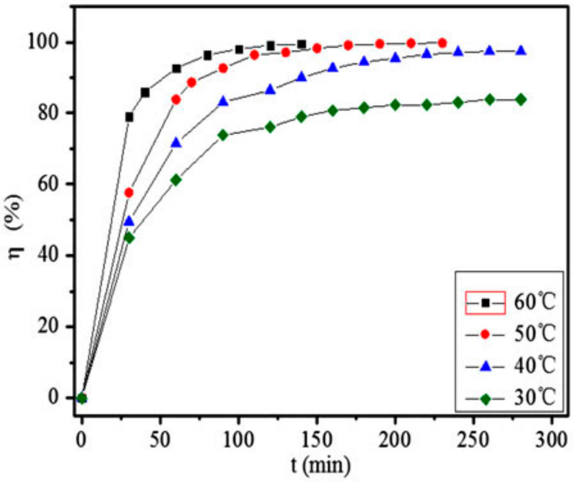

Fig. 2. Effect of feed temperature on the ammonia removal efficiency. The pressure at the vacuum side was 88 kPa, the feed velocity was 0.21 m/s.

The driving force of the VMD ammonia removal process was the vapor pressure difference of ammonia on the two sides of hydrophobic membranes. The ammonia vapor pressure on the hot side (theoretically at the interface of the feed solution and membrane surface) mainly relied on the concentration and temperature of the feed solution.

For the VMD ammonia removal experiment carried out at fixed temperature, the concentration of ammonia in the feed solution was highest at the beginning of the experiment, and the driving force for ammonia removal was highest. As the experiment progressed, the ammonia content in the feed solution declined, so the ammonia vapor pressure difference between two sides of membranes declined, thereby, the mass-transfer driving force declined. This might be the reason for the variation of ammonia removal efficiency during VMD process.

From Fig. 2, we could also see that the ammonia removal efficiency reached a stable state in the end of each process operated at different temperatures. This indicated that the adsorption and desorption of ammonia reached equilibrium in the end of each process. Now the concentration of ammonia in water (i.e. C_t) was close to the solubility of ammonia. Since the solubility of ammonia in water decreased with the increase of temperature, so the removal efficiency increased with the increase of temperature according to Eq. (1). This was quite similar with other studies for dilute ammonia solution treatment [7,10,11,21].

Fig. 3 showed the variation of the separation factor during the VMD experiments which were operated at different temperatures.

From Fig. 3, it could be seen that the separation factor showed a declining trend at each temperature. While the separation factor and its declining rate were quite different for the experiments operated at different feed temperatures. The separation factor increased as the feed temperature increased from 30 to 40℃, and then decreased as the temperature further increased to 50 and 60℃. When operated at 40℃, the separation factor at the beginning was 13.4 and declined to about 3.3 after 275 min of the experiment. When operated at 60℃, the separation factor was only 4.5 at the beginning, and declined to 1.3 after 140 min of experiment.

As we know, the distillation of ammonia is inevitably combined with the evaporation of water during the ammonia removal process by distillation or VMD

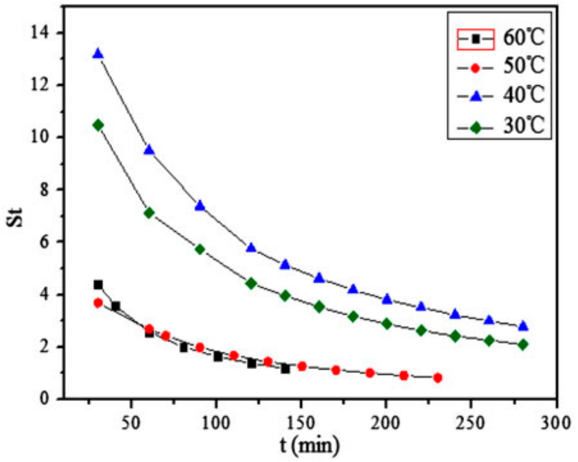

Fig. 3. Effect of feed temperature on separation factor. The pressure at the vacuum side was 88 kPa, the feed velocity was 0.21 m/s.

method. The variation of separation factor during the VMD process, and the difference of separation factor obtained at different feed temperatures may be mainly because of the competition of evaporation of ammonia and water.

Fig. 4 illustrated the saturated vapor pressure of water and ammonia at different temperatures. The figure showed that the saturated vapor pressure of water and ammonia both increased with the increase of temperature. The increasing speed of ammonia saturated pressure is much higher than that of water. When the temperature is lower than 42℃, the saturated vapor pressure of ammonia is lower than that of water. This indicated that the evaporation of water was relatively small and the mass transfer of ammonia was dominant. Now, the separation factor was mainly affected by the evaporation of ammonia. With the increase of feed temperature, the driving force of ammonia transfer enhanced and it made the separation factor increase. This was the reason that separation factor at 40℃ was higher than that of 30℃. But when the temperature reached 50℃, the saturated vapor pressure of water was higher than that of ammonia and the mass transfer of water became dominant. Now, the separation factor was mainly affected by the evaporation of water, and the increase of water in the permeate side made the separation factor decline. This was the reason that separation factor at 50 and 60℃ was lower than that of 40℃.

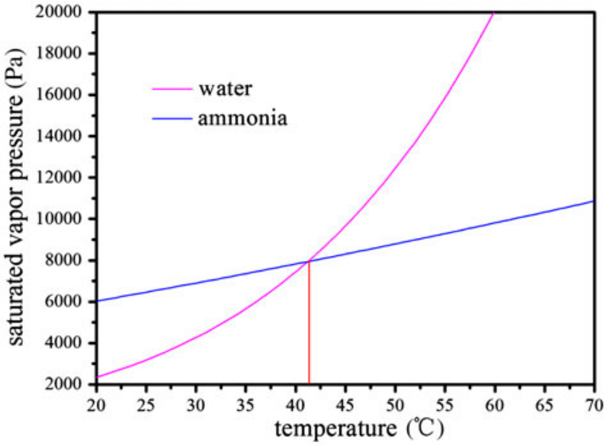

Fig. 4. Effect of temperature on the saturated vapor pressure of water and ammonia.

When the feed temperature was fixed, the evaporation driving force of water was destined, so water evaporated with a relatively steady speed. While that for ammonia evaporation decreased owing to the decrease of ammonia concentration as the VMD experiment went on. This might be the reason for the declination trend of separation factor at each temperature.

When the feed temperature increased from 30 to 60℃, both the water and ammonia vapor pressure enhanced, and increased mass-transfer speed of them was resulted. The saturated water vapor pressure at the four temperatures was presented in Table 2. The overall fluxes of ammonia and water during the VMD experiments were calculated using the following equations and the results were also listed in Table 2.

$$N_{\mathrm{NH_3}} = \frac{(C_0 - C_t) \cdot V}{A \cdot t} \tag{4}$$

where NH_3 was the total flux of ammonia (g/(m^{2}h)) during the experimenting time t (h), C_0 and C_t were the concentration of ammonia in the feed solution at the beginning and at the time t (g/L), V was the initial volume of feed (L), A was the active area of membrane module (m^2).

$$N_{\mathrm{H_2O}} = \frac{m_0 - m_t - (C_0 - C_t) \cdot V}{A \cdot t} \tag{5}$$

where H_2O was the total flux of water during the experimenting time t (h), m_0 and m_t were the mass of feed solution at the beginning and time t (g).

From Table 2, it could be seen that NH_3 was larger than H_2O when operated at relatively lower temperature (30–40℃). This was mainly because that the evaporation of water was relatively small and the mass transfer of ammonia was dominant in the range

Table 2
The values of NH_3, H_2O, and the saturated vapor pressure of water at different temperatures

T (℃)	Operating time (min)	Saturated vapor pressure of water (kPa)	NH_3 (g/(m^{2}h))	H_2O (g/(m^{2}h))
30	275	4.24	606	489
40	275	7.38	924	528
50	225	12.3	1,045	2,233
60	135	19.9	1,013	2,708

of 30–40℃. But when the feed temperature exceeded 50℃, H_2O was much larger than NH_3. It was mainly caused by two factors: The rapid increase of saturated vapor pressure of water and the total amount limitation of ammonia in feed solution. When the temperature was higher than 50℃, the saturated vapor pressure of water was much higher than that of ammonia and the mass transfer of water was dominant. In addition, the removal efficiency at 50 and 60℃ was both 99%. This indicated that almost all of ammonia was removed and the ammonia fluxes had approached their limit values owing to the initial concentration. But for the solvent water, the flux increased all along with the increase of temperature and it exceeded the limit value of the ammonia flux when the operating temperature was higher than 50℃.

As mentioned before, the aim of this study was to recycle the ammonia from the feed solution, so the water content in the produced ammonia vapor was calculated to represent the quality of the production and the results were shown in Fig. 5.

From Fig. 5, we could see that as the experiment prolonged, the water content increased. When the feed temperature fixed, the evaporation driving force of water was destined, so water evaporated with a relatively steady speed. While that for ammonia evaporation decreased owing to the decrease in ammonia concentration as the VMD experiment went on. This might be the reason for the increasing trend of water content at each operating temperature.

In Fig. 5, it could also be seen that the water content decreased firstly and then increased when the feed temperature increased from 30 to 60℃. When the feed temperature was between 30 and 40℃, the saturated vapor pressure of ammonia was higher than that of water. This indicated that the evaporation of water was relatively small and the mass transfer of ammonia was dominant. This was the reason that water content at 40℃ was lower than that of 30℃. But when the temperature reached 50℃, the saturated vapor pressure of water was much higher than that of ammonia and the mass transfer of water was dominant. Now the water content was mainly affected by the evaporation of water, and the increase of water in the permeate side made the water content increase. This was the reason that water content at 50 and 60℃ was higher than that of 30 and 40℃. When operated at 40℃, the water content in produced ammonia vapor was the least, so the quality of the production was the best.

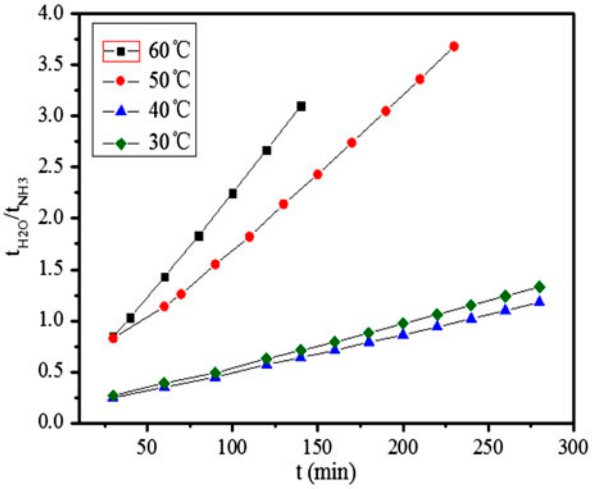

Fig. 5. Effect of feed temperature on the water content in produced ammonia vapor. The pressure at the vacuum side was 88 kPa, the feed velocity was 0.21 m/s.

3.2. Effect of feed velocity

The effect of feed velocity on the ammonia removal process was studied using 18.0 wt.% ammonia solution with a feed temperature of 40℃ and vacuum pressure of 88 kPa. Considering the character of the hollow fiber hydrophobic membrane, the flowing resistance and the pressure loss along the hollow fiber (especially when operated with the pilot scale module in latter part), even the energy consumption and membrane serving time, relatively lower feed velocity (0.14–0.21 m/s) was studied in this work.

Fig. 6 illustrated the variation of ammonia removal efficiency during VMD process when operated with different feed velocities.

Fig. 6 showed that the ammonia removal efficiency increased as experimental time prolonged. But the increasing rate declined slowly at the same time. This might be mainly because of the declination of ammonia concentration during VMD experiment. For the VMD ammonia removal experiment carried out at fixed feed velocity, the concentration of ammonia in the feed solution was highest at the beginning of the experiment, the driving force for ammonia removal was highest. As the experiment went on, the ammonia content in the feed solution declined. So the ammonia vapor pressure difference between two sides of membranes declined, thereby, the mass transfer driving force declined. This was the reason that the increasing speed of ammonia removal efficiency declined with the increase of experimental time for each feed velocity.

Fig. 6 also illustrated that the ammonia removal efficiency was improved when the feed velocity increased from 0.14 to 0.21 m/s. The overall mass-transfer coefficient at each feed velocity was calculated by Eq. (6) and listed in Table 3.

$$K_{OV} = \frac{V}{At} \ln\left(\frac{C_0}{C_t}\right) \tag{6}$$

where K_{OV} was the overall mass-transfer coefficient (10^{-6} m/s) during the experimenting time t (s), C_0 and C_t were the concentration of ammonia in the feed solution at the beginning and at time t (g/L), V was the initial volume of feed (L), and A was the active area of membrane module (m^2).

Table 3

Effect of feed velocity on the overall mass-transfer coefficient of ammonia

Feed velocity (m/s)	K_{OV} (10^{-6} m/s)
0.14	1.39
0.18	1.72
0.21	3.01

The increase of feed velocity might promoted the turbulence and eddied in the bulk feed solution, especially in the boundary layer at the interface of the feed solution and the membrane surface. So the temperature and concentration polarization effect might be alleviated, the heat- and mass-transfer from the bulk feed to the membrane surface was improved. As a result, higher ammonia mass-transfer coefficient and ammonia removal efficiency were obtained at higher feed velocity (as illustrated in Table 3), in the range of this study.

Fig. 7 showed the variation of separation factor during VMD process when operated at different feed velocities. The figure showed that the separation factor declined gradually as the VMD experiment went on. The trend was similar to that obtained at different operating temperatures. The declination of ammonia concentration in the feed solution might also be the reason for this phenomenon.

The figure also illustrated that the separation factor was improved by the increase of feed velocity. This result was similar to those obtained in the treatment of dilute ammonia solutions [19–21]. As the feed

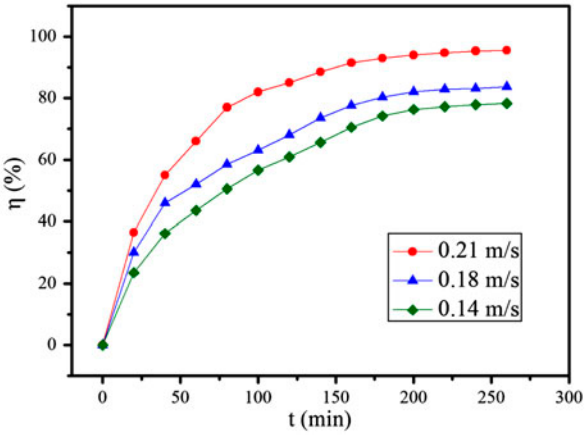

Fig. 6. Effect of feed velocity on ammonia removal efficiency. The pressure at the vacuum side was 88 kPa, the feed temperature was 40˚C.

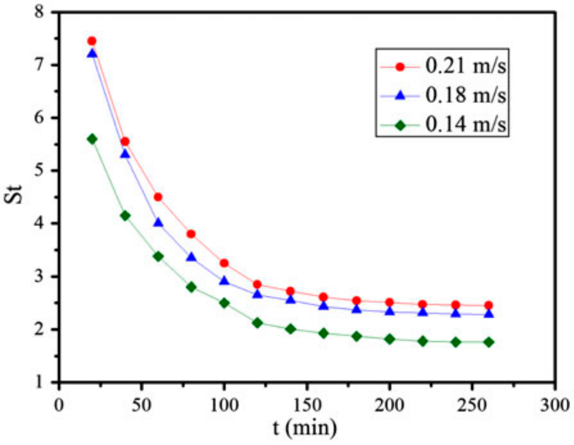

Fig. 7. Effect of feed velocity on separation factor. The pressure at the vacuum side was 88 kPa, the feed temperature was 40˚C.

Table 4
Effect of feed velocity on the overall fluxes of ammonia and water

Feed velocity (m/s)	Operating time (min)	NH_3 (g/(m^{2}h))	H_2O (g/(m^{2}h))
0.14	260	370	1,112
0.18	260	518	1,145
0.21	260	655	1,190

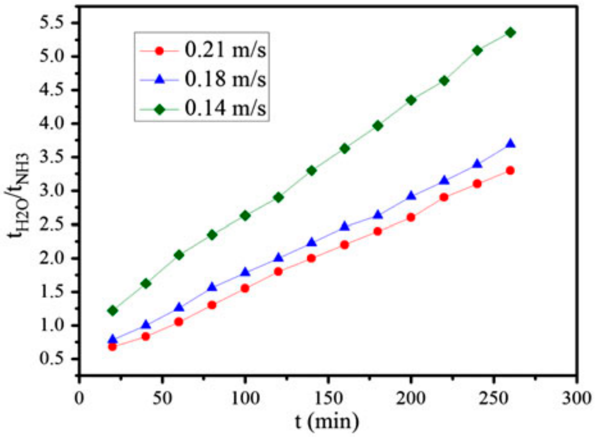

Fig. 8. Effect of feed flow rate on the water content in ammonia. The pressure at the vacuum side was 88 kPa, the feed temperature was 40℃.

velocity increased, the heat and mass-transfer resistance in the liquid phase decreased. So the fluxes of both ammonia and water increased (as shown in Table 4). The flux of ammonia increased as the feed velocity increased from 0.14 to 0.21 m/s, while that of water only showed marginal increase when operated at 40℃. So the separation factor showed an increasing trend with feed velocity.

Fig. 8 showed that as the experiment went on, the water content increased. The effect of feed velocity on the water content in produced ammonia vapor was opposite to that on the separation factor. The reason was that when the process operated at 40℃, the mass transfer of ammonia was dominant. The water content was mainly affected by the evaporation of ammonia. When the feed velocity increased, the mass transfer of ammonia increased more compared with the mass transfer of water. So the water content decreased with the increase of feed velocity. When operated with a feed velocity of 0.21 m/s, the water content in produced ammonia vapor was the least, so the quality of the production was the best.

3.3. Preliminary result of pilot scale experiment

Experiment with a pilot scale membrane module (about 1.70 m length, effective membrane area of 20 m^2) was carried out using the high concentration ammonia solution from soda ash plant (saturated solution at 40℃) with a feed velocity of about 0.20 m/s.

Preliminary results from the experiment showed that the ammonia removal efficiency could reach 82–85% with a separation factor at about 4.6 in about 70 min of VMD experiment, both of the distillation capacity and product quality were better than traditional distillation method.

4. Conclusions

The removal and reclamation of ammonia vapor from high ammonia concentration wastewater was efficient by VMD method using PVDF hydrophobic hollow fiber membrane. The water content of the produced ammonia vapor was lower than that produced by traditional distillation process and would improve the overall efficiency of soda ash production process by Solvay method.

The feed temperature and velocity were key factors for this ammonia removal process when the feed ammonia concentration and pH value destined. Both of the ammonia removal efficiency and water content in the produced ammonia vapor were the best when operated at optimized conditions (feed temperature of 40℃ and feed velocity of 0.21 m/s). As VMD operation prolonged, the feed ammonia concentration decreased, which resulted in higher water content in produced ammonia vapor and lower ammonia removal efficiency. It was important to decide a proper operation time and final ammonia concentration by considering both the removal efficiency and product property (water content).

Preliminary results from pilot plant tests further testified the efficiency of the method in near saturated ammonia removal.

Acknowledgments

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List of symbols

η	—	ammonia removal efficiency (%)
t	—	time (min)
C_0	—	ammonia concentration in the initial feed solution (g/L)
C_t	—	ammonia concentration in feed solution at time t (g/L)
S_t	—	separation factor
f	—	feed side
p	—	permeate side
C_{NH_3}	—	ammonia molar concentration (mol/L)
C_{H_2O}	—	water molar concentration (mol/L)
t_{H_2O}/t_{NH_3}	—	water content in ammonia
m_{H_2O}	—	water mass (g)
m_{NH_3}	—	ammonia mass (g)
N_{NH_3}	—	flux of ammonia (g/(m^{2}h))
V	—	initial volume of feed (L)
A	—	active area of membrane module (m^2)
N_{H_2O}	—	flux of water (g/(m^{2}h))
m_0	—	mass of feed solution at the beginning (g)
m_t	—	mass of feed solution at time (g)
K_{OV}	—	overall mass-transfer coefficient (10^{-6} m/s)

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