Experimental study on the concentration of ethylene glycol aqueous solution by metal powder sintered porous capillary wicks

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ABSTRACT

In industry, the ethylene glycol concentration is realized by flashing and distillation and a series of processes. These processes with high energy consumption are complicated. To solve this problem, a method for concentrating ethylene glycol aqueous solution with metal powder sintered capillary wick is proposed. The capillary wick with length 50 mm, outer diameter 30 mm and inner diameter 10 mm sintered by copper powder is used as an evaporator for ethylene glycol solution in this study. The effects of inlet flow rate, heating temperature, inlet concentration, as well as the pore size of the capillary wicks on the concentration of ethylene glycol are all analyzed experimentally. It is found that the ethylene glycol solution can be concentrated from the mass fraction of 10% at inlet to 16% at outlet through the capillary wick, which indicates that the concentration method proposed in this paper is feasible. Moreover, the evaporated water is generally positively correlated with inlet flow rate and evaporation temperature within a certain range, however, the evaporation will not increase when the inlet flow rate and heating temperature reaches a certain level due to the permeation and evaporation limitation effect. Increasing the inlet concentration reduces the volume of the condensate, but the corresponding increase in outlet concentration is not noticeable. It is also found experimentally that the capillary wick sintered with particles with 56.3 μm diameter has the best concentration performance for the ethylene glycol solution. The concentration method proposed by this study is expected to be helpful for the dehydration of organic solvents or the separation of binary non-azeotropic liquid mixtures in industrial production.

1. Introduction

Ethylene glycol(EG) is a kind of important organic chemical raw material for the manufacture of polyester fiber, rubber and other products. At the same time, ethylene glycol itself is also used as antifreeze and lubricants. The main method of producing ethylene glycol in the industry is the direct hydration of ethylene oxide [1–4]. This method does not use the catalyst and its process is mature, but about 85% water in the products must be evaporated by using massive energy, which brought the disadvantages, such as long process flows, large equipments and enormous waster of energy and seriously affected the cost of production of EG [5].

In order to achieve the purpose of energy-saving and emission reduction, the ethylene glycol refining system need to be optimized. Many new studies have been reported. For example, pervaporation is widely used for the separation of organic solvents such as ethylene glycol aqueous solutions [6–13]. Feng et al. [14] reported that the neutralized chitosan/polysulfone composite membrane was used to separate the aqueous ethylene glycol solution by PV. They

demonstrated that PV has the potential to replace conventional distillation. Shaheverdi et al. [15] investigated the PV performance of EG dehydration with PVA/zeolite 4A composite membranes. Nik et al. [16] studied the PV of EG/water mixture through NaA Zeolite membranes. Yan et al. [17] studied the pervaporation dehydration of ethylene glycol with dual-layer polybenzimidazole(PBI)/polyetherimide(PEI) membranes. According to those literatures, the optimization of ethylene glycol separation is mainly focused on the development of new membrane materials. Metal sintered porous materials are not reported for ethylene glycol separation.

Sintered metal powder porous materials have many advantages, such as lightweight, large specific surface area, large capillary force, heat and corrosion resistance. It is widely used in the efficient evaporation device. Loop heat pipe capillary core is one of them [18,19]. Metal powder sintered capillary wick is the key of the loop heat pipe because of its excellent evaporation performance. The capillary wick has two main functions in the heat pipe. The first is as a capillary pump. Using liquid surface tension between the evaporator and the tank to form a capillary pressure difference, and driving the working fluid to

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cycle. The second is to provide more nucleation sites for evaporation. The higher specific surface area of the capillary core and the internally connected pores can enhance the evaporation efficiency. At present, the study of the porous structure on sintered metal powder for heat pipe is detailed, but the research of metal sintered capillary core for solution concentration separation is relatively rare. Zhang et al. [20] applied the loop heat pipe capillary structure to the seawater desalination system. The results showed that original seawater with total dissolved solid (TDS) of 16,900 mg/L can be concentrated to 23,000 mg/L by using the capillary wick evaporation system, and the TDS of the condensate fresh water is 30 mg/L, which is far lower than that of drinking water standard and is suitable for drinking. This work proves the feasibility for the application of capillary wick in solution concentration.

For the liquid evaporation and separation process in metal sintered porous capillary wick, capillary distillation is one of the theoretical basis, which is firstly proposed by Yeh G C [21]. Abu Al. Rub F A et al. [22–24] investigated the separation of the two-component mixture by capillary distillation. It is found that the effect of separation depends on the characteristics of the microporous plate. Generally, the porous capillary stainless steel sieve is better than the ordinary sieve plate. Rub et al. [25,26] have successfully separated several binary liquid mixtures with capillary porous trays, including ethanol-water, ethanol-benzene and acetone-ethanol. They found that the main factor affecting the separation efficiency is the relative polarity difference between the solution component and solid material.

The above studies show that separation of the binary non-azeotropic liquid mixture by metal powder sintered capillary structure is more effective than the conventional method of distillation and multi-effect evaporation under some specific conditions. On the one hand, the capillary wick can provide more nucleation site for gasification, strengthen the boiling evaporation process. On the other hand, as the solid-liquid interface force between the different liquid components and the capillary wick is different, the saturation vapor pressures and activity coefficients of the component will be changed, leading to a different vapor-liquid equilibrium relationship, thereby increasing the relative volatility and strengthening the separation process. Taking the separation of the ethylene glycol aqueous solution for example, since the metal sintered porous wick has a non-polar surface, while the dielectric constant of the ethylene glycol is smaller than that of the water, thus the interaction between the metal wall and the ethylene glycol molecule is more intense. This makes the ethylene glycol molecules be more difficult to escape from the bond of solid-liquid interaction than water molecules, then to some extent increasing the relative volatility of ethylene glycol and water, thus strengthening the separation process.

Based on the above analysis, the dehydration effect of ethylene glycol solution by the metal sintered porous capillary wick is studied in this paper. The effects of the feed rate, the heating temperature, the initial concentration of ethylene glycol inlet concentration, as well as the pore size of the capillary wicks on the separation efficiency are all analyzed, and the mechanism is also explained from the perspective of heat transfer process and capillary distillation. A new idea for the separation of binary non-azeotropic liquid mixtures is proposed in this paper, which is expected to be helpful for the dehydration of organic solvents in industrial production.

2. Experimental principle and apparatus

2.1. Experimental principle

Based on the principle of the open loop heat pipe, the experimental system for this study is designed and shown in Fig. 1. When the external heat is applied to the surface of the evaporator, the ethylene glycol aqueous solution will undergo phase change in the capillary core and produce steam. As the boiling point of water is 97 °C lower than that of ethylene glycol, a large amount of water will evaporate firstly. The steam will then flow out of the evaporator through vapor channel of the

capillary wick, and then became water in the condenser. Since the water component near the outer surface of the capillary wick evaporates faster, the ethylene glycol is concentrated in the outer surface of the capillary wick, resulting in concentration difference in the radial direction of the capillary wick. According to the Fick's diffusion principle, ethylene glycol near the outer surface of capillary wick will diffuse to the inside wall of wick and discharged from the evaporator under the gravity to the concentrate tank. Since the condensed water and the concentrated solution flow in different pipelines, the purpose of concentrating the aqueous ethylene glycol solution can be achieved.

The advantage of separating the ethylene glycol aqueous solution with metal sintered porous capillary wick is that the porous structure can use the solid-liquid interface force difference to change the saturation vapor pressure and activity of the components, thereby changing the gas-liquid equilibrium and relative volatility, then improving the separation effect. Moreover, the gas-liquid equilibrium can also be changed by the interfacial curvature in the metal sintered porous capillary wick. According to the Kelvin equation [16], the saturated vapor pressure of each phase in the pores is lower than that outside, which is described by:

$$RT \ln \frac{p_r}{p_o} = \frac{2\gamma M}{\rho r} \tag{1}$$

where R is the gas constant, T is the thermodynamic temperature, p_r is the actual vapor pressure in porous wick, p_0 is the saturated vapor pressure, γ is the surface tension, M is the molar mass, ρ is the density, and r is the radius of the liquid drop.

Since the liquid in the metal sintered porous wick is in the form of a concave liquid surface, so $r < 0$, which means:

$$\ln \frac{p_r}{p_o} < 0, \ p_r < p_o \tag{2}$$

It shows that the vapor pressure in the capillary pore is smaller than the saturated vapor pressure at the same temperature. As the reduction degree of vapor pressure is different for different liquid composition, which leads to better separation effect for liquid mixture.

2.2. Test facility and method

The experimental system consists of evaporator, advection pump, compensation chamber, reservoir and pipeline. The role of the compensation chamber is to prevent the capillary core being dry. The heating tape is wrapped around the outer wall of the evaporator, and the evaporation temperature can be adjusted through the temperature controller. There are four temperature testing points are set in the system, where T_{in} measures the inlet temperature, T_{out} measures the outlet temperature, T_v measures the steam temperature, and T_w measures the evaporator wall temperature. Four temperature measurement points all use T-type thermocouple with the calibration error ± 0.5 °C and temperature measurement frequency 0.5 Hz. In order to ensure the temperature accuracy, the outer wall of the evaporator is wrapped by glass asbestos insulation material with thickness of 8 mm.

2.3. Evaporator design and capillary pore analysis

The material of the evaporator shell is brass, and grooves are manufactured in its inner wall as the steam flow channel. The copper powder sintered capillary wick, which is shown in Fig.2, is stuffed into the evaporator shell, and the outer wall of the capillary wick and the inner wall of the evaporator shell is tightly fitted. The center hole of the wick is used as the concentrate flow channel. Fig. 3 shows the assembled evaporator.

In order to study the effect of capillary pore size on the separation process, four different capillary wicks, named type A, B, C, and D, are sintered by copper powder with diameters range from 32 μm to 256.3 μm, the average pore size, porosity and permeability of the four
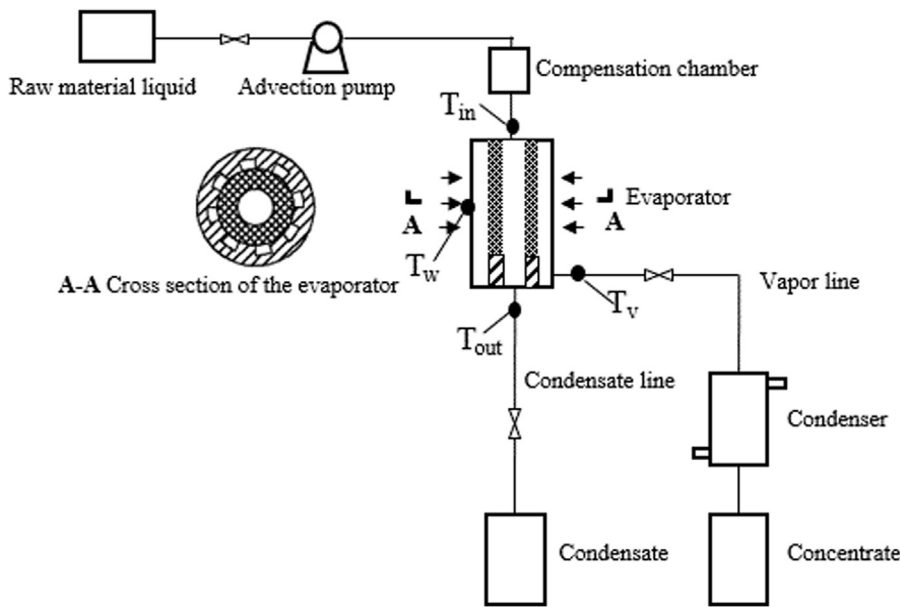
Fig. 1. Schematic of the experimental system.

Fig. 2. The copper powder sintered capillary wick.

Fig. 3. The assembled evaporator with brass shell and copper powder sintered capillary wick.

Table 1
Capillary wick properties used in the experiments.

Capillary wick	Copper particle average diameter (μm)	Average pore radius (μm)	Porosity ε(%)	Permeability $\kappa_p \times 10^{-11}$(m^2)
Type-A	256.3	65.37	54.26	61.63
Type-B	99.5	30.54	50.13	6.67
Type-C	56.3	25.64	48.52.	1.77
Type-D	32.0	14.68	43.32	0.40

wicks used in the experiment are listed in Table 1.

For the convenience of measuring the particle diameter, spherical particles are selected and measured by microscope, it is observed that the diameters are in a relatively narrow diameter distribution. The pore size and porosity of the capillary wicks are measured by mercury porosimeter, while the pore size distribution is obtained by the PoreMaster software, through which it is found that the pore size is in a normal distribution. The average pore size is derived using the following equation:

$$r_p = \frac{1}{2}d_p = \frac{1}{2}\sum_1^N (V_i d_{pi})/\sum_1^N V_i \tag{3}$$

where V_i is the volume of the pore with diameter d_{pi}. The permeability of the wick K_p is calculated from the Blake–Kozeny equation [21]:

$$K_p = \frac{d^2\varepsilon^2}{150(1-\varepsilon)^2} \tag{4}$$

Where d is the mean particle diameter, and ε is the porosity. The metal powder morphology is demonstrated by the scanning electron microscope (SEM) image, which is shown in Fig. 4(a), SEM of the porous pore after sintering is shown in Fig.4(b). It can be seen that the wick surface is made of roughly spherical particles with uniform sizes. Besides, highly populated reentrant-type cavities are observed.

2.4. Testing procedures

Adding the pre-configured ethylene glycol aqueous solution to the raw material liquid tank, opening the metering pump, the ethylene glycol aqueous solution is sucked from the raw material tank and sent into the evaporator passage. After heating and evaporating, the
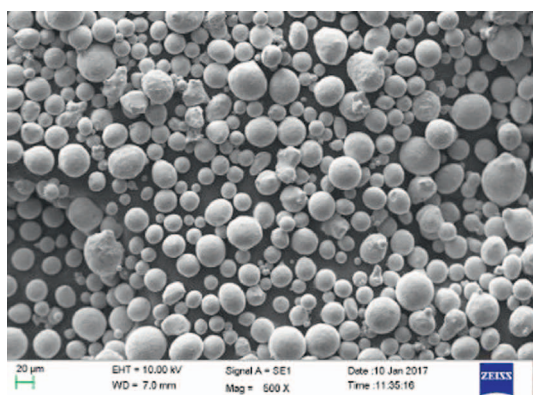
(a)
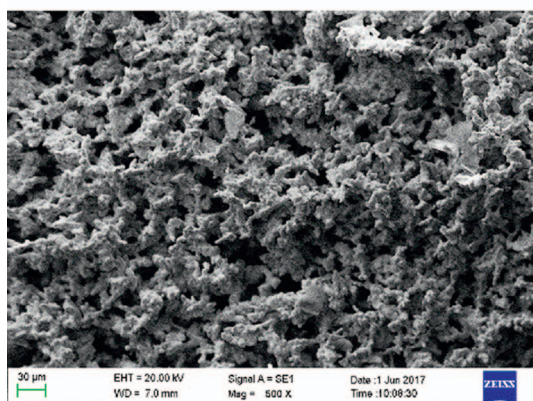
(b)

Fig. 4. SEM image of the sintered porous wick
(a) Metal powder before sintering; (b) Porous structure of the sintered porous wick.

concentrated liquid flows into the concentrated tank, while the evaporated steam flows into the steam line and is condensed by the condenser, and then collected in the condensing tank.

Fig. 5 shows the temperature variation for each test point within 2 h after starting the system. It can be seen that after about 700 s, the temperature measured by the four temperature test points will no longer changes, indicating that the system evaporation process has reached a stable running state.
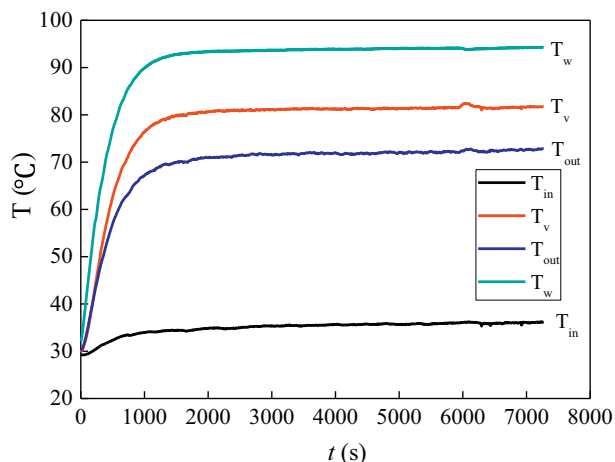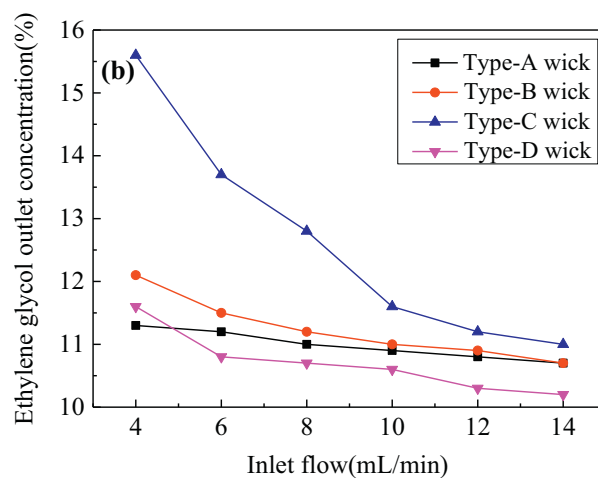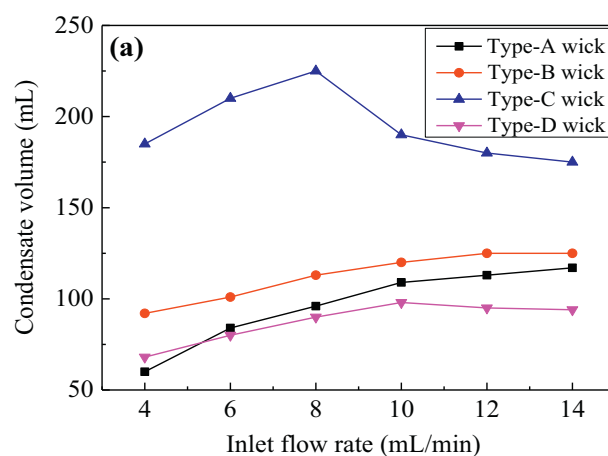

Fig. 5. Temperature at four test points at the initial 2 h of the evaporation process.

Fig. 6. The effect of inlet flow rate on the concentration of ethylene glycol solution in four capillary wicks (a)The condensate volume; (b) The outlet concentration of ethylene glycol solution.

For the results listed in the bellowing analysis, all of them are obtained in a steady state. The condensate volume is obtained after the experiment has continuously collected for 2 h in a stable condition. Based on the above preparation work, the effects of the pore size of the capillary wick, ethylene glycol inlet flow rate, heating temperature as well as the inlet concentration on ethylene glycol concentration process are analyzed in the following work.

3. Results and discussion

3.1. Effect of inlet flow rate

The effects of different inlet flow rates on the concentration of ethylene glycol are investigated in the four wicks when the heating temperature is 160 °C and the mass fraction of the inlet ethylene glycol is 10%. Fig. 6(a) shows the collected condensate volume at different inlet flow rates. It is clear that the condensate volume increases with the increase of inlet flow rate initially. However, there exists a peak value, the condensate volume reaches its peak value when the flow rate is 8 mL/min for type C wick, while it is 10 mL/min for type D wick. The condensate volume start to decrease with the increase of inlet flow rate after the peak value. Furthermore, it is clear that the largest condensate volume is obtained by type C wick at the same inlet flow rate. This can be explained as the metal particle diameter and pore size of correspondingly sintered capillary wick have significant effect on the

capillary force and nucleation site density for the diffusion and evaporation processes. The particle size used for capillary wick C has the most suitable pore size, porosity and permeability characteristics for the diffusion and evaporation of ethylene glycol aqueous solution at the experimental conditions, thus the evaporated vapor or the condensate volume is the most at the same inlet flow rate. However, the amount of evaporated vapor will reach its maximal limit due to the fixed external heat source with the increase of inlet flow rate, while more heat will also be carried out and lost by the liquid flow in the wick center if the inlet flow rate continues to increase, which leads to the decrease of evaporated vapor or the condensate volume. As the capillary wick C has the best evaporation capability in the four wicks, thus its condensate volume 'peak value' is the largest among four wicks, and also appears firstly. The evaporated vapor from wick C decreases more significantly because of its good heat transfer capability compared with wick A and B, which leads to more serious heat loss at large inlet flow rate conditions. For wick D, as its heat conduction capability is the largest, the most heat is carried out and lost by the liquid flow in wick center, which leads to the worst evaporation effect among four wicks. The 'peak value' of wick D is also lower than the other three, thus the presented decrease of condensate volume is also smaller with the increase of inlet flow rate. It can be predicted that the condensate volume of capillary wick A and B will appear at an inlet flow rate larger than 14 mL/min, which is not included in the figure. The experimental results in this part agree well with the results of numerical simulation of capillary evaporator [17].

Fig.6(b) shows the outlet concentration of ethylene glycol solution at different inlet flow rates. It can be seen that larger inlet flow rate leads to lower outlet concentration, which also because of the evaporation limitation at the fixed heating temperature as explained above. Furthermore, it can be seen the largest outlet concentration also appears at type C wick. Although more nucleation sites and larger capillary force can be provided by smaller particle diameter and pore size in capillary wick, which is good for the evaporation and boiling processes. However, smaller pore size also leads to reduced permeability, which will increase the liquid flow resistance. Moreover, more serious heat leakage phenomenon also occurs for wick with smaller particle and pore size due to the correspondingly formed smaller porosity, which means larger heat conductivity, thereby reducing the concentration effect.

3.2. Effect of heater temperature

Heater temperature is an important factor for the concentration process. Fig. 7(a) and (b) show the variation of the condensate volume and the outlet ethylene glycol concentration at different heater temperatures when the flow rate is fixed at 6 mL/min and the inlet mass fraction is 10% in four capillary wicks. It can be seen that the condensate volume and the mass fraction of the concentrated ethylene glycol both increase with the increase of heater temperature. However, the condensate volume and ethylene glycol outlet concentration no longer change when the temperature exceeds 165 °C at the setting operation conditions. The reason is that more permeated water will be evaporated into vapor with the increase heater temperature, however, the liquid permeated to the outer surface of the capillary wicks has an upper limit due to the fixed inlet flow rate and the diffusion and permeation limitation of the porous structure. Thus the evaporated vapor will get to the maximum value when the temperature reaches a certain level. In addition, with the increasing of heater temperature, more ethylene glycol in the solution will be evaporated into vapor channel, this is also one of the reason why the higher wall temperature cannot improve the concentration effect anymore.

It can also be seen from Fig. 7 that the reduction of the wick pore size can improve the separation effect, but it does not mean that the smaller the pore size is, the better the separation effect. As shown in the figures, although the pore size of type-D wick is the smallest, but the
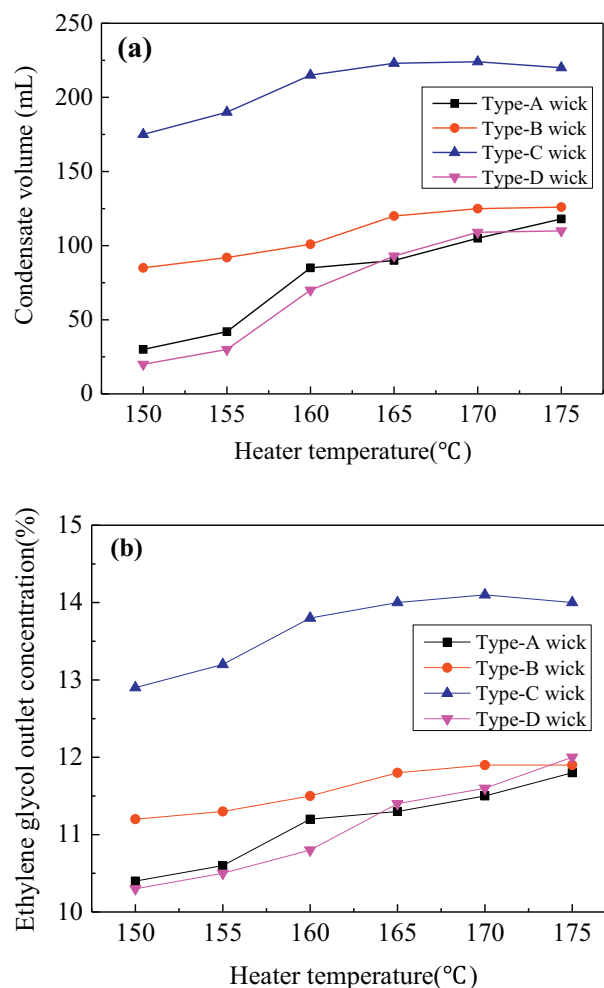

Fig. 7. The effect of heater temperature on the concentration of ethylene glycol solution (a)The condensate volume; (b) The outlet concentration of ethylene glycol solution.

separation effect is worse than the other three samples. Because the appropriate reduction of pore size can increase the capillary force, resulting in increased suction speed and evaporation nucleation sites, thereby enhancing the separation, however, smaller permeability and more serious heat leakage will also be caused, thereby reducing the concentration effect.

3.3. The effect of ethylene glycol inlet concentration

As the concentration of ethylene glycol in the industry varies at different processes, it is of great practical significance to study the effect of the inlet concentration on the concentration of ethylene glycol solution in four capillary wicks. Fig. 8 (a), (b) and (c) show the condensate volume, outlet concentration as well as the concentration difference between the inlet and outlet at different inlet ethylene glycol concentrations, respectively. The heating temperature is 160 °C while the inlet flow rate is 6 mL/min. It can be seen that the condensate volume decreases slowly with the increase of inlet concentration when it varies in the range of 6%–14%, which means less water is evaporated from the liquid mixture. The reason is that as the inlet ethylene glycol concentration increases, the amount of water permeated to the outer wall surface of capillary wick will be reduced accordingly due to its less mass fraction, meanwhile, the solution flow resistance in the porous structure also increases as the viscosity of eghylene glycol is larger than water. Both of the above reasons lead to the decrease of the amount of condensed water.
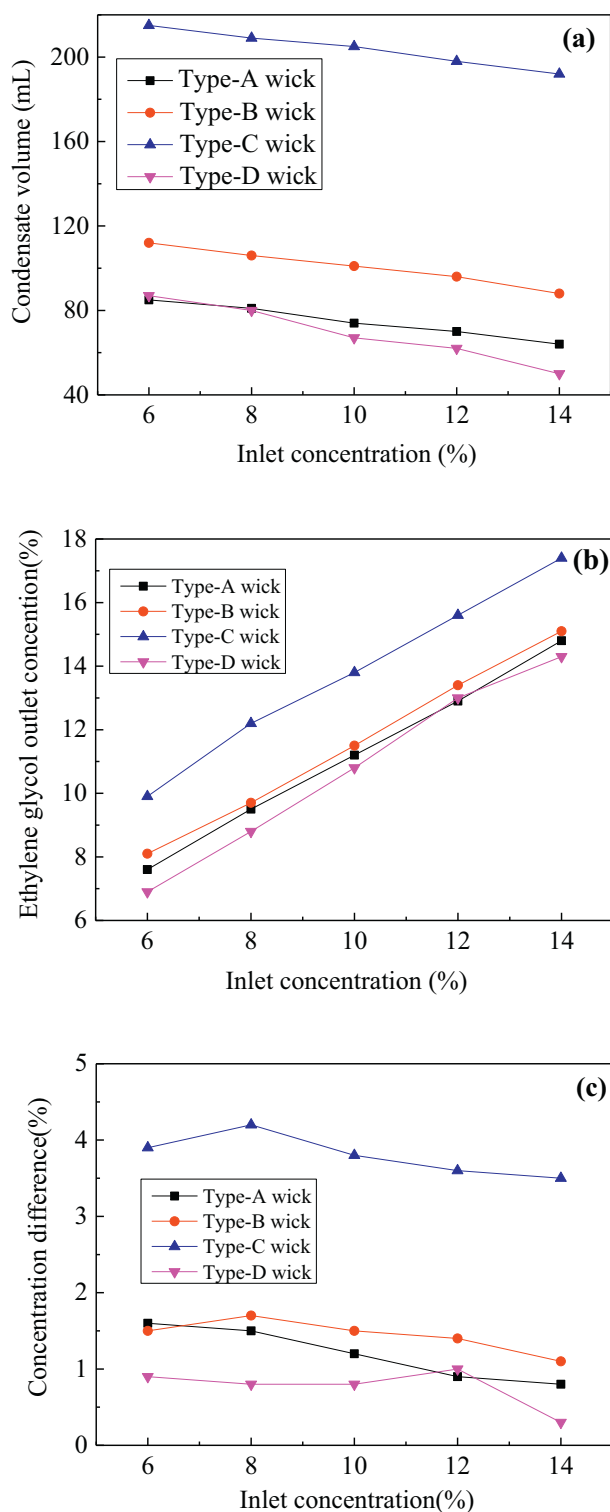
Fig. 8. The effect of inlet concentration on the concentration of ethylene glycol solution (a)The condensate colume; (b) The outlet concentration of ethylene glycol solution; (c) The outlet and inlet concentration difference of eghylene glycol.

It is also clear,from Fig.8(b) and (c), that the ethylene glycol outlet concentration is positively correlated with the inlet concentration, and the difference between the outlet concentration and the inlet concentration remains stable at the condition of low concentrations, because the reduction of condensate is consistent with the reduction of water content in the inlet solution.

Furthermore, the effect of capillary pore diameter on the concentration effect at different inlet concentrations keeps consistent with the previous conclusion, the explanation will not be repeated here.

4. Conclusion and prospect

A new method for concentrating ethylene glycol aqueous solution with metal powder sintered capillary wicks is proposed is this paper. The effects of inlet flow rate, heating temperature, inlet concentration, as well as the pore size of the capillary wicks on the concentration of ethylene glycol are all analyzed experimentally. The conclusions are as following:

(1) The ethylene glycol solution can be concentrated from the mass fraction of 10% at inlet to 16% at outlet through the capillary wick with a length of 50 mm, which indicates that the concentration method proposed in this paper is feasible.
(2) The evaporated water is generally positively correlated with inlet flow rate and evaporation temperature within a certain range, however, the evaporation will not increase when the inlet flow rate and heating temperature reaches a certain level due to the permeation and evaporation limitation effect.
(3) Increasing the inlet concentration reduces the volume of the condensate, but the corresponding increase in outlet concentration is not noticeable.
(4) Appropriate reduction in pore size can increase the capillary force, resulting in increased capillary force and nucleation sites, thereby enhancing the evaporation and concentration effect. However, smaller pore size also leads to reduced permeability, which will increase the liquid flow resistance. Moreover, more serious heat leakage phenomenon also occurs due to the correspondingly formed smaller porosity and larger heat conductivity, thereby reducing the concentration effect. It is experimentally observed that the capillary wick sintered with particles with 56.3 μm diameter has the best concentration performance for the ethylene glycol solution.

Acknowledgements

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