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Research Article

Experimental Design and Optimization of the Utilization of Vacuum Membrane Distillations Lithium Cloride-Water Absorption Refrigeration System

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Abstract: Response surface methodology (RSM) has been applied for modeling and optimization in utilization of vacuum membrane distillation on Lithium Cloride-water absorption refrigeration system (ARS). The effect of the operational parameter is initial feed concentration, feed inlet temperature, feed flow rate and interaction on the permeate flux. The developed model has been statistically validated by analysis of variance (ANOVA) and further used to predict the permeate flux. The result for the given factors is a saddle point one, which meant for the range given, there is no optimal value. This saddle point parameter is outside the data range which is concentration of 24.41 %, feed temperature at 65.03°C and flow rate 0.88 $L.min^{-1}$ while its predicted flux value is 166.9 $g.m^{-2}.s^{-1}$.

Keywords*:* Response surface methodology; lithium Cloride-water; vacuum membrane distillation, optimization.

INTRODUCTION

Regeneration process in vapor absorption refrigeration process (VARS) happens in generator. In here, the solution (absorbent and refrigerant) which has been in low concentration (weak solution) re-concentrates usually by means of heat energy[1]. After re-concentrates process, the absorbent which now have a higher concentration (strong solution), then will send back to absorber while the refrigerant vapor will flow to the condenser and continue to make refrigeration effect happen. The process in generator includes a heat transfer phenomena in which make it easy to have some inefficiency[2].

Based on second law based thermodynamic analysis, Kilic and Kaynakli[3] stated that due to the temperature difference between the heat source and the working fluid, the energy loss in the generator give a significant amount to the system, made the generator should be regarded as a system component that needs improvement.

Lithium chloride is chosen as absorbent for safety and cost consideration. LiCl is a monovalent ion and the separation process in regenerator should only result water as its product. Heat in form of low grade thermal energy (solar energy, heat from boiler) usually applied as separation process motive energy. Large contact area is needed so that the separation process is efficient. This requirement for large area made conventional generator seems bulky and difficult to be fitted in small area.

Membrane usage has advantage of having small dimension while keeping large contact area. For VARS, one of the choices is using vacuum membrane distillation (VMD). VMD usage for VARS will have benefit since the result of VMD is pure water (theoretically)[4,5]. VMD is also preferable than other membrane process since the nature of the absorbent itself. Applied vacuum then will enhance the resulted flux thus will enhance the regeneration process.

Coupling VMD in regenerator of VARS will raise some questions and problems, since each process has its own limitations and considerations, such as pressure and temperature. Some of them will be on operating parameters (and the optimal ones), energy required and application aspect[6]. Using statistical study to identify the optimal parameter in membrane process has been conducted by some author. However, there is no publication for regeneration of salt solution using VMD. The objective of this study then is to define the parameters crucial in the process.

EXPERIMENTAL

Material used and VMD set – up: A commercial hollow fiber Microza PVDF membrane was used in this research. The hollow fiber has large area for regeneration while remaining compact. PVDF membrane is hydrophobic so that it suitable for MD application even though it is NF membranes. The membrane properties are shown in **Table 1**.

Table 1: Specification material constructions of membrane module

Specifications material constructions :		
	:	Polyvinyldenefluoride (PVDF)
Housing	:	Native clear polysulfone (P) or filled polysulfone (W)
Potting Material	:	Epoxy Resin
Gasket	:	Silicon (P)
Bacteriostat	:	Glycerin 65%, Ethanol 2 %, Water balance

The aim for this study is to study the VMD application on VARS. In this study, the working fluid for VARS is lithium chloride solution. The solution prepared by dissolved anhydrous lithium chloride 99% bought from Alfa Aesar with de-ionized water. A refractometer (N.O.W Refractometer, Japan) were used to determine the LiCl concentration after and before experiment and collected permeate in each run. The measurement of LiCl concentration was based on previous calibration carried out on different concentration.

Table 2: Part number and technical specification of membrane module

Part Number and Technical Specification :		
Part Number	:	UMP-0047R
Number of membrane	:	21
Fiber ID/OD (mm)	:	1.4/2.2
Nominal Length(mm)	:	310
Membrane Area (m^2)	:	0.02
Clean Water Flux ($Lh^{-1}bar$, 25°C)	:	19
Crossflow Rate for 1 ms^{-1} Fluid velocity	:	116
Maximum Pressure (barg) Feed/TMP/Permeate at 50°C	:	3/3/1.5
Volume Feed	:	23
Volume Permeat (mL)	:	30

A schematic diagram of the VMD experimental set up used is shown in **Figure 1**. During the experiment, LiCl solution from the feed bath was pumped (Flojet 2130) through the tube lumen and vacuum was applied on the shell side. A rotatometer placed before the solution entering membrane module to measure the feed flow. Temperature and pressure before the feed entering the membrane was also measured, as well as after entering membrane module. Retentate flowed back to the feed bath, while the permeate, forced with vacuum, flowed to permeate collector. The vacuum on the shell side was created using vacuum pump GAST DAA V 503 EB. The feed temperature was kept constant using thermostat Maxthermo MC – 2438. Water-cooled condenser was used as cold trap to condensate the permeates.

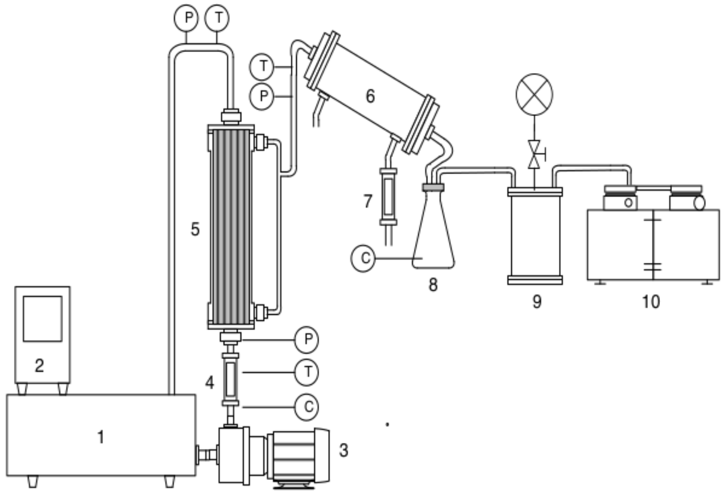

Figure 1: Experimental VMD set-up: 1. water bath; 2. thermostat water bath; 3. Pump; 4. solution flowmeter; 5. VMD module; 6. Condenser; 7. Cooling water flowmeter; 8. Receiving tank; 9. Cold trap; 10. Vacuum pump

Experimental design of VMD process: The statistical experiment design (DoE) employed has been carried out considering three factors (controllable variables), namely the feed inlet temperature (T_{in}), the feed flow rate (U), and the initial LiCl/feed concentration (C_o). **Table 3** shows the controllable variable and their levels in coded and actual values. The vacuum pressure for each experiment was at the maximum, and its value is between –715 to –730 mbar.

Table 3: Designed variables and their coded and actual values for experimental design

Designed variables (factors)	Symbol	Actual values of coded levels		
		-1	0	+1
Initial feed concentration, C_o (%)	X_1	36	38	40
Feed inlet temperature, T_{in} (^{0}C)	X_2	60	65	70
Feed flow, U (L/min)	X_3	1.1	1.5	1.9

Experimental design and analysis of data were done using a commercial statistical package, JMP version 7. By using the software, a Box – Behnken design with 3 center points was employed with three factors and three levels. This design is not randomized. The Box – Behnken design contains of 15 experiments with three center points. The experimental design matrix is summarized in **Table 4**. Each experimental run was performed for 2 hour and VMD permeate flux were collected after every run.

Table 4: Box – Behnken design

Run	Pattern	Factors		
		C_o (%)	T_{in} (^{0}C)	U (L/min)
1	--0	36	60	1.5
2	-0-	36	65	1.1
3	-0+	36	65	1.9
4	-+0	36	70	1.5
5	0--	38	60	1.1
6	0-+	38	60	1.9
7	000	38	65	1.5
8	000	38	65	1.5
9	000	38	65	1.5
10	0+-	38	70	1.1
11	0++	38	70	1.9
12	+-0	40	60	1.5
13	+0-	40	65	1.1
14	+0+	40	65	1.9
15	++0	40	70	1.5

The permeate flux was determined gravimetrically by weighting the distillate collected in the permeate tank for a predetermined time. For every run, the permeation flux achieved is calculated with

$$J = \frac{W}{S.t} \tag{1}$$

With J is permeate flux (kg/m^{2}s), W is quantity of water (kg), S is membrane area (m^2), and t is time (s). This permeation flux data and its running parameter are then used as input for RSM analysis.

RESULTS AND DISCUSSION

The DoE (design of experiment) and Response Surface Methodology (RSM) were employed in this study for maximizing and optimizing VMD process. The procedure of DoE and RSM consists of following steps [2]: (a) designing and conducting a series of experiment to obtain the process response, (b) developing mathematical model of first or second order response surface with best fittings, (c) finding out optimal set of process variables that guarantee an optimum value of the selected response, and (d) studying and representing the main and interaction effects of the process variables on the response. **Table 5** shows the responses of each run conducted. The range of concentration is chosen to resemble the applied solution in practical operation. The temperature range is chosen so that the system could be applied on low heat input energy.

Table 5: Box – Behnken design and experimental responses

Run	Pattern	Factors			Response
		X_1, C_o (%)	X_2, T_{in} (^{0}C)	X_3, U (L/min)	Y, J (g/m^{2}h)
1	--0	36	60	1.5	91.760
2	-0-	36	65	1.1	123.748
3	-0+	36	65	1.9	115.468
4	-+0	36	70	1.5	180.833
5	0--	38	60	1.1	58.148
6	0-+	38	60	1.9	68.373
7	000	38	65	1.5	113.440
8	000	38	65	1.5	114.448
9	000	38	65	1.5	109.065
10	0+-	38	70	1.1	169.993
11	0++	38	70	1.9	162.219
12	+-0	40	60	1.5	37.040
13	+0-	40	65	1.1	93.990
14	+0+	40	65	1.9	93.125
15	++0	40	70	1.5	161.080

Based on obtained experimental results, the response surface model has been developed for the given response (permeate flux) by applying JMP software. JMP use standard least square method to fit experimental and predicted one. **Figure 2** gives the plot of experimental and predicted value. **Table 6** shows the summary of fit and analysis of variance. The obtained second order RS – model with coded variables (with four scientific numbers) is:

$$Y = 112.3175 – 15.82156X_1 + 52.3505\ X_2 – 0.8366\ X_3$$
$$+8.7418X_1X_2 +1.8537X_1X_3 – 4.4995X_2X_3$$
$$–1.3699X1X1 +6.7305X_2X_2 – 4.3651X_3X_3 \quad (2)$$

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From both figure and table, the statistical validation gives high value of R^2, 0.99. It means that the model could predict99.58% of the variability of this response. The experiment is running in continuous process and each process has the same resting time. The consistency in process made the system seems like running in continuous order.

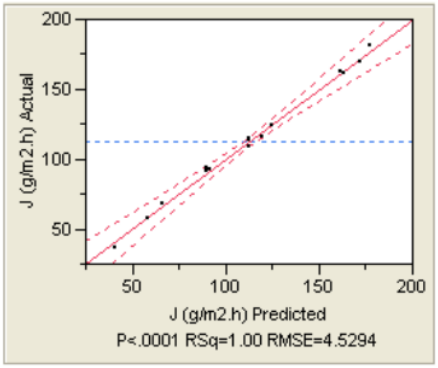

Figure 2: The actual result and its predicted value plot

Table 6: Summary of fit and analysis of variance for the experiment

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Summary of fit		Analysis of variance				
RSquare	0.996	Source	DF	Sum of squares	Mean square	F ratio
RSquareadj	0.988	Model	9	24597.870	2733.100	133.145
Root mean square error	4.531	Error	5	102.636	20.53	Prob>F
Mean of response	112.849	C. Total	14	24700.505		<.0001*
Observations	15					

From the predicted equation, a single effect of X_2, that is the initial feed temperature, has a positive strong effect on the response and it has the most considerable constant in the equation, means that it is the most significance parameter. X1 is the second significance parameter but its effect is negative. The positive of the temperature is showed in most VMD equation. This phenomenon is in accordance with the mass transfer explanation. The mass transfer happen in membrane process in highly depends on the temperature. Besides, having a higher temperature means having a less temperature polarization so that it will enhance the heat transfer process which in turn will also increase the permeate flux.

While for the initial concentration, its trend is also shows accordance with another VMD experiment. Higher concentration means that the solution has more solutes than solvent. In case of water, it will decrease the partial water vapor pressure of the solution. Less value of water vapor pressure then will cause a decrease in heat transfer process so that the permeate flux will also decreasing. Higher concentration is also prone to have higher concentration polarization and the effect on the permeate flux is the same with the experiment result.The interaction factor between X_1 and X_2 also shows that the initial feed temperature indeed is the most significance factor since the value of their interaction is positive.

The significance of the regression coefficient of the models written as function of the coded variables could also be tested with statistical Student *t* – test. **Table 6** shows the t – test result for the experiment. From the test result, it shows that the feed initial temperature is the most significance. Furthermore, its interaction effect with the feed initial concentration and temperature itself is still significant. The feed flow shows its effect does not affect permeate flux in significant way. In relate with this, a lower flow rate then will be favorable so that it will reduce power consumption of the pump.

Table 7: Parameter estimates and t-test results

Term	Estimate	Std Error	t ratio	Prob>|t|
Intercept	112.3175	2.615795	42.94	<.0001*
X_1	-15.82156	1.601841	-9.88	0.0002*
X_2	52.35031	1.601841	32.68	<.0001*
X_3	-0.836656	1.601841	-0.52	0.6238
X_1*X_2	8.741875	2.265345	3.86	0.0119*
X_1*X_3	1.85375	2.265345	0.82	0.4504
X_2*X_3	-4.499563	2.265345	-1.99	0.1037
X_1*X_1	-1.366906	2.357846	-0.58	0.5864
X_2*X_2	6.7305312	2.357846	2.85	0.0356*
X_3*X_3	-4.365094	2.357846	-1.85	0.1233

The response surface for the given factors and response is a saddle point one, which meant for the range given, there is no optimal value. This saddle point parameter is outside the data range which set is concentration of 24.41%, feed temperature at 65.03^{0}C and feed flow 0.88 L/min while its predicted value is 166.9 g/m^{2}h. The confirmation for this result could not be conducted for the system scheme could not support the feed flow rate in the above set of parameters.

To have a more understanding on the effect of each parameter and their interaction, a prediction profiler of the experiment is shown at **figure 3**. As can be seen, the feed inlet temperature shows an obvious increasing curvature since its significance. The initial feed concentration show a slight decreasing curvature which shows its effect on the response. The feed flow shows a slightly maximum point but since it is not significant enough, its prediction profiler is not so clear.

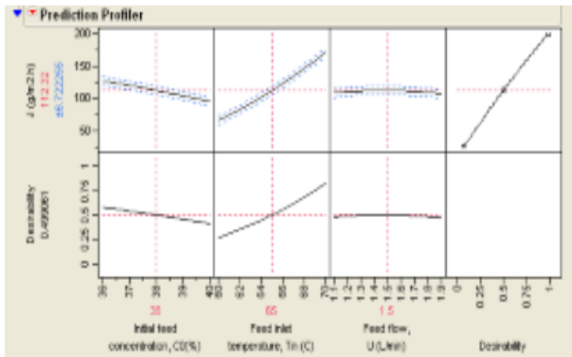

Figure 3: Prediction profiler of experiment result

Following figures and explanation will show the mutual effects of factors and the influence upon the response in two-dimensional plots. **Figure 4** shows the influence of initial feed concentration and feed inlet temperature variables on the permeate flux. As can be seen, the increase of initial feed concentration will leads to a decrease of the permeate flux while the increment of feed inlet temperature will leads to an increase of the response. At high concentration, the effect of temperature is stronger than at low concentration. The graph also confirms the strong effect of the feed inlet temperature over the initial feed concentration.

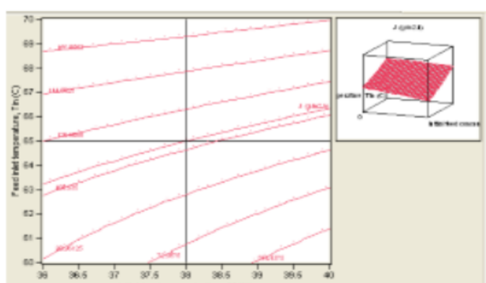

Figure 4: Contour lines showing the response as function of initial feed concentration and feed inlet temperature for feed flow rate = 1.5 L/min

Figure 5 shows the effects of feed flow rate and initial feed concentration on the VMD permeate flux for a fixed value of feed inlet temperature, feed inlet temperature = 65^{0}C. As can be observed, for all feed flow rate values, there is a decrease of the permeate flux with the increase of initial feed concentration, whereas the increase of feed flow rate value will increase the permeate flux until its maximum, then it will decrease. The figure also implies that the concentration has a stronger effect on permeate flux than feed flux has.

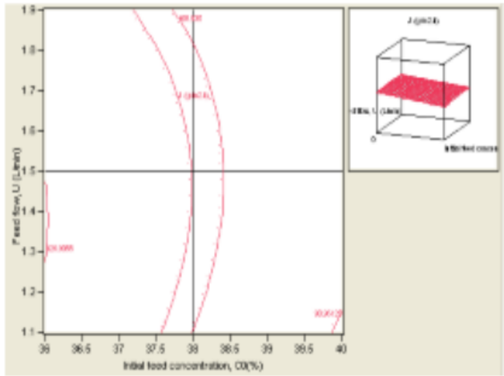

Figure 5: Contour lines showing the response as function of initial feed concentration and feed flow rate for feed inlet temperature = 65^{0}C

Figure 6 shows the effects of feed inlet temperature and feed flow rate on the VMD permeate flux for a fixed value of initial feed concentration = 38%. From the figure, the increase of feed flow will increase the permeate flux until maximum is reached. The increase in temperature also make the permeate flux increased. The effect of temperature increasing is more profound than feed flow increasing.

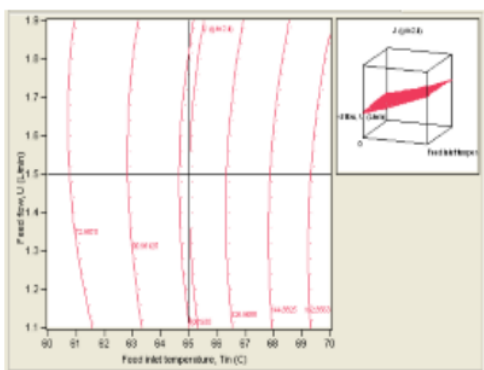

Figure 6: Contour lines showing the response as function of feed inlet temperature and feed flow rate for initial feed concentration of 38%

CONCLUSIONS

In this research, VMD experiment for VARS application has been conducted by using RSM regard with three parameters: initial concentration, feed inlet temperature, and feed flow. The summary and conclusion of above-mentioned process are:

1. Feed inlet temperature is the most significant factor in this experiment. It has positive effect. Furthermore, its effect strong enough so that the interaction factor with initial feed concentration is also positive.
2. Initial feed concentration has negative effect on the permeate flux. Compared to the feed inlet temperature significance, the initial feed concentration in low showed that temperature polarization has no effect compared to the concentration polarization.
3. The suggested solution is outside the data range and could not be performed for the system scheme limitations.

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