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Taguchi optimization approach for phenolic wastewater treatment by vacuum membrane distillation

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ABSTRACT

In the present research, treatment of phenolic wastewater via vacuum membrane distillation (VMD) was studied. VMD experiments were performed using polytetrafluroethylene membranes. Taguchi method was applied in order to design the experiments and optimize the experimental results. Effect of different operating parameters, including temperature, vacuum pressure, feed pH and phenol concentration on permeate flux, and separation factor, was studied. The results showed that increasing temperature, decreasing vacuum pressure, decreasing phenol concentration, and decreasing feed pH, enhance permeate flux. Temperature was found to be the most effective factor for permeate flux. Furthermore, it was observed that with decreasing temperature, increasing phenol concentration, and increasing feed pH separation factor increase. The experiments showed that water separation factor is approximately independent of vacuum pressure. A temperature of 45˚C, a pressure of 60 mbar, a concentration of 1,000 mg/L, and a pH of 13 were found as the best condition for separation factor determined by the Taguchi method. At this condition, separation factor was found to be 63.63.

Keywords: Wastewater treatment; Membrane distillation; Taguchi method; Phenol

1. Introduction

Phenol is a toxic substance which normally exists in wastewater of many chemical plants such as pesticides, paper, and pulp, dyes, and chemical manufacturing industries. Besides these, wastewater originating from other industries such as gas, resin manufacturing, and coke manufacturing, textile, tanning, plastic, pharmaceutical, petroleum, rubber and also contains different types of phenols [1–3]. Phenols are considered as pollutants since they are harmful to organisms at low concentrations and many of them are classified as hazardous pollutants because of their potential harm to human health [4]. Wastewater containing phenolic compounds presents a serious discharge problem due to its poor biodegradability, high toxicity, and ecological aspects [5]. The utilization of phenol-contaminated waters causes protein degeneration, tissue erosion, and paralysis of the central nervous system and also damages kidney, liver, and pancreas in human bodies [6]. According to the recommendation of World Health Organization, the permissible concentration of phenolic contents in potable waters is 1 μg/L [7], and the regulations by the Environmental Protection Agency call for lowering

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phenolic contents in wastewater less than 1 mg/L [8]. Therefore, removal of phenols from waters and wastewater is an important issue in order to protect public health and environment. There are a variety of treatment methods that have been applied for phenol removal. Popular among these are activated carbon adsorption [9], chemical oxidation [10], membrane process [11], and biological treatment [12]. Such problems as high cost, low efficiency, and generation of toxic byproducts are associated with the above methods [13].

Biological treatment is not normally suitable for wastewater with high phenol concentration such as those come from refinery, petrochemical, and pharmaceutical operations [14]. Chemical oxidation requires a large amount of oxidizing agents under high operating conditions [15] with a risk of incomplete oxidation resulting in more toxic products [10]. Meanwhile, activated carbon adsorption can effectively remove organic compounds such as phenol [9,16] but this method has a drawback in that it is expensive and difficult to regenerate due to chemisorptions of phenol and the degradation of carbon [15]. Membrane separation methods, including reverse osmosis, ultrafiltration, and pervaporation, have attracted more attention for phenolic wastewater treatment, with cellulose acetate membranes employed mostly [17].

Vacuum membrane distillation (VMD) is a membrane operation where microporous hydrophobic membranes are used for removing water vapor and volatile compounds from aqueous solutions. Being the membrane hydrophobic, the liquid cannot permeate through the pores and it is blocked at one side of the membrane in correspondence of the pores' mouths. By applying vacuum at the other side of the membrane, a difference of partial pressure is created across the membrane, and both the water vapor and the volatile species start to permeate through the membrane pores (see Fig. 1). VMD is strongly dependent on the temperature, because of its exponential relation with vapor pressure, and it is not limited by the osmotic pressure of the feed. The main advantages of MD over conventional separation processes are [18]:

- Complete separation (in theory) of ions, macromolecules, colloids, cells, etc. in other words, it produces high-quality distillate.
- Water can be distilled at relatively low temperatures.
- Low-grade heat (solar, industrial waste heat, or desalination waste heat) may be used.
- The water does not require extensive pretreatment as in pressure-based membrane treatment processes.

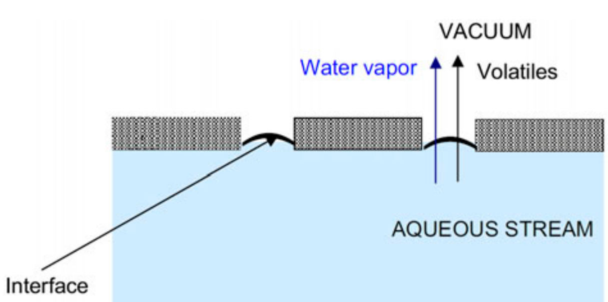

Fig. 1. Permeation of water vapor and volatile species by VMD [18].

The Taguchi method which was established by Genichi Taguchi has been generally adopted to optimize the design variables because this approach can significantly minimize the overall testing time and the experimental costs. The Taguchi crossed array layout consists of an inner array and an outer array. The inner array is made up of an orthogonal array (OA) selected from all possible combinations of the controllable factors. Using the OA specially designed for the Taguchi method, the optimum experimental conditions can be easily determined [19]. After the optimum conditions are chosen and predicted, the confirmation experiments should be performed with the prediction. This confirmation experiment is necessary and important as it provides direct proof of the methodology [20].

Accordingly, an analysis of the signal-to-noise (S/N) ratio is needed to evaluate the experimental results. Usually, three types of S/N ratio analysis are applicable: (1) lower is better, (2) nominal is best, and (3) higher is better (HB) [21]. Because the target of this study is to maximize water separation factor and permeate flux, the S/N ratio with HB characteristics is required, which is given by the following equation:

$$S/N = -10\log\left(\frac{1}{n}\sum\frac{1}{y_i^2}\right) \quad (1)$$

where n is the number of repetitions under the same experimental conditions, and y_i is the performance value of ith experiment.

The main disadvantage of the Taguchi method is that the results obtained are only relative and do not exactly indicate what parameter has the highest effect on the performance characteristic value. Also, since OAs do not test all variable combinations, this method should not be used with all relationships between all variables needed. The Taguchi method has been criticized in the literature for difficulty in accounting for interactions between parameters. Another limitation is that the Taguchi method is offline and therefore inappropriate for a dynamically changing process such as a simulation study. Furthermore, since Taguchi methods deal with designing quality rather than correcting for poor quality, they are applied most effectively at early stages of process development [19].

In the present paper, treatment of phenolic wastewater using VMD is discussed and tried to determine (1) the optimum conditions for separation factor (2) percent of contribution (P, %) of each factor on separation factor and permeate flux, and (3) the influence of each investigated factor including feed concentration, feed pH, feed temperature, and vacuum pressure, on separation factor and permeate flux.

2. Experimental

2.1. Materials and methods

The phenol crystals and sodium hydroxide were supplied by Merck. The phenolic solutions were prepared by dissolving certain amounts of phenol crystals according to the run numbers in distilled water, and then for complete mixing, the solutions were mixed by magnetic stirring. The feed pH was adjusted by addition of 10 N NaOH solutions. Concentrations of phenol were determined by UV–Vis spectrophotometry (Labomed UVS-2,800) at 270 nm [22].

The permeate flux was calculated by the following equation:

$$J = \frac{W}{S \times t} \tag{2}$$

where J is the permeate flux (kg/m^2 h), W is the quantity of permeate (kg), A is the effective membrane area (m^2), and t is the sampling time (h).

The water separation factor, β, was calculated according to the following equation:

$$\beta = \frac{x_{o,w}/x_{o,p}}{x_{i,w}/x_{i,p}} \tag{3}$$

where $x_{o,p}$ and $x_{o,w}$ are the fractions of the amounts of phenol and water in the permeate, respectively, and $x_{i,p}$ and $x_{i,w}$ are the fractions of the amounts of phenol and water in the feed, respectively.

2.2. Experimental system

Experiments were carried out using a flat sheet polytetrafluroethylene (PTFE) membrane from Ningbo Changqi Co (China). A cross-flow membrane module made from steel was used in the experiments (Fig. 2). Effective area of the membrane in the module was 16.61 cm^2.

The membrane properties are reported in Table 1. The schematic representation of VMD setup is shown in Fig. 3.

The feed was continuously fed to the membrane module from the feed tank, sufficiently large (3 L) to keep the feed concentration nearly constant. The membrane flux was measured by collecting the

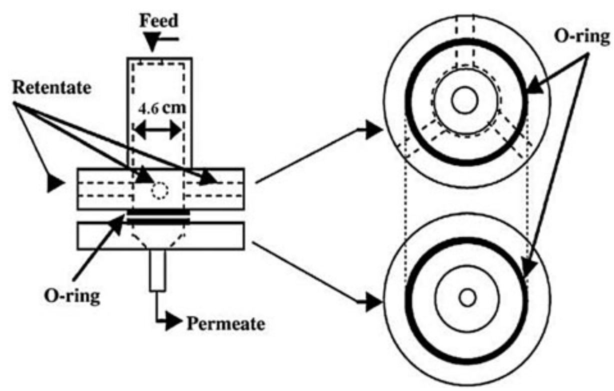

Fig. 2. Membrane module.

Table 1
Properties of the flat sheet PTFE membrane

Type	CHQISTEX®e-PTFE
Pore size (μm)	0.22
Porosity (%)	85
Thickness (with support, μm)	230

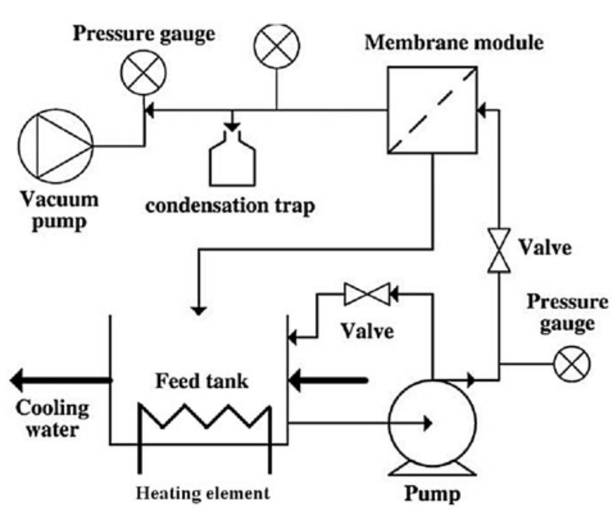

Fig. 3. Schematic diagram of VMD set-up.

permeate in the condensation trap (liquid nitrogen). The feed composition and temperature were considered constant within the module.

One important consideration in the setup was that the feed pump was not able to flow the small required flow rates in this research, so the excess flow was bypassed to approximately keep the feed volume constant. The bypass flow which was heated by the pump had a significant influence on the feed temperature. As a result, it was needed to cool it to control the feed temperature, so the feed tank was equipped with the cooling water coil. In other words, for the lower temperatures, the cooling water coil and for the higher temperatures, the heating element was employed to control the feed temperature.

Prior to permeate collection, the cell was kept running for 1 h to reach a steady state and each experiment was done for 30 min.

2.3. Design of experiments

Factors such as feed temperature, vacuum pressure, feed pH, and feed concentration influence the VMD process. In addition to these factors, feed flow rate is also effective. However, in this work, to reduce the number of experiments, the last one considered constant (70 L/h) and the effects of the others were investigated. According to the Taguchi parameter design methodology, one experimental design should be selected for the controllable factors. A L_9 OA (four factors with three levels each in nine runs, L_9 (3^4) OA) was employed [23]. Each row of the OA represents a run, that is, a specific set of factor levels to be tested. Experimental parameters and their levels are given in Table 2. As shown, the levels of the factors are as follows: feed temperature (45, 55, and 65℃); vacuum pressure (30, 60, and 90 mbar); feed pH (12, 12.5 and 13); feed concentration (20, 510, and 1,000 mg/L). The performance of VMD can be affected by some factors known as controllable or uncontrollable (noise sources). In order to observe the effects of uncontrollable factors on this process, each experiment was repeated twice at the same conditions.

The mean response for each run and also the appropriately chosen S/N ratio were used to analyze the results [20]. Tables 3 and 4 present the results of the experiments and the S/N ratio for each response.

2.4. ANOVA analysis

ANOVA is used to estimate the error variance and to determine the relative importance of various factors. It indicates the effect of each investigated factor on the optimization criterion. ANOVA also demonstrates whether the observed variation in the response is due to the alteration of level adjustments or

Table 2
Taguchi L9 (3^4) OA

Run	Operating parameters			
	T (℃)	Pv (mbar)	*C* (mg/L)	pH
1	45	30	20	12.0
2	45	60	510	12.5
3	45	90	1,000	13.0
4	55	30	510	13.0
5	55	60	1,000	12.0
6	55	90	20	12.5
7	65	30	1,000	12.5
8	65	60	20	13.0
9	65	90	510	12.0

Table 3
Results of the experiments and the S/N ratios for permeate flux

Run	Permeate flux (kg/m^2 h)		Mean permeate flux (kg/m^2 h)	Permeate flux (*S/N*)
	1	2		
1	58.005	59.34	58.6725	35.3670
2	37.908	37.76	37.8340	31.5576
3	14.440	15.55	14.9950	23.5011
4	79.420	78.81	79.1150	37.9650
5	69.550	67.59	68.5700	36.7200
6	51.025	53.35	52.1875	34.3449
7	110.47	112.5	111.485	40.9432
8	111.61	113.4	112.535	41.0249
9	98.200	99.81	99.0050	39.9123

Table 4
Results of the experiments and the S/N ratios for separation factor

Run	Separation factor		Mean separation factor	Separation factor (*S/N*)
	1	2		
1	2.11864	3.81679	2.967719	9.44846
2	46.7461	61.3718	54.05897	34.6574
3	55.4324	63.9230	59.67772	35.5162
4	52.5232	41.1955	46.85932	33.4159
5	18.9000	18.2548	18.57743	25.3797
6	2.19539	2.66312	2.429253	7.70945
7	25.7467	29.6033	27.67498	28.8417
8	6.04047	3.86100	4.950738	13.8934
9	9.95316	9.72540	9.839281	19.8593

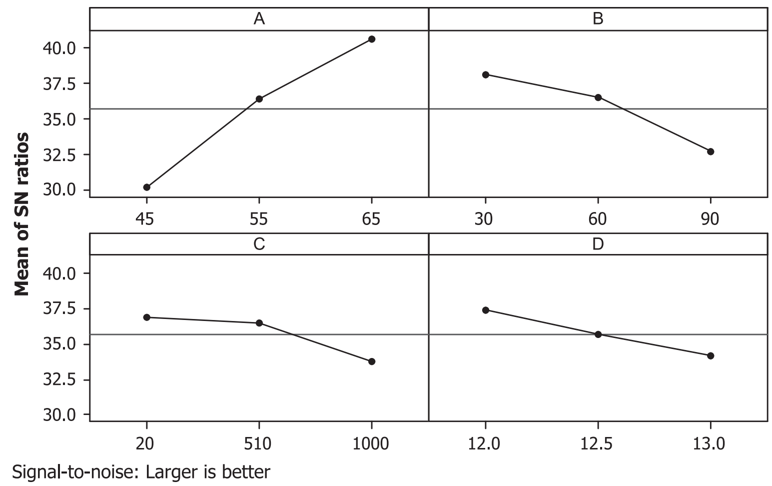

Fig. 4. Main effects plots for S/N ratios on permeate flux. (A) Feed temperature, (B) vacuum pressure, (C) feed concentration and (D) feed pH.

experimental standard errors. In ANOVA analysis, the values of sum of squares (SS), degree of freedom (DOF), mean square (variance), and associated F-test of significance (F) were calculated. SS of factor A was calculated as follows:

$$SS_A = \left[\sum_{i=1}^{k_A}\left(\frac{A_i^2}{n_{A_i}}\right)\right] - \frac{T^2}{N} \tag{4}$$

k_A is the number of levels for factor A ($k_A = 3$ for all factors in this study), n_{A_i} is the number of all observations at level i of factor A, A_i is the sum of all observations of level i of factor A, N is the number of all experiments, and T is the sum of all observations. P is the percent of contribution of each factor on the response ($P_A = (SS_A/SS_T) \times 100$), where SS_T is the sum of squares for all factors [24].

3. Results and discussion

3.1. The influence of operating conditions on permeate flux

3.1.1. Effect of feed temperature

Fig. 4(A) shows the effect of feed temperature on the S/N ratio on permeate flux. It is observed that the flux increases with increasing feed temperature. Increasing the MD flux with increasing temperature for the commercial PTFE membranes is due to the higher vapor pressure at the higher temperature based on the Antoine equation for vapor pressure of water [25]. Besides, both the feed viscosity and the boundary-layer thickness reduce with increasing feed temperature, and this enhances the mass transfer coefficient [17,26,27].

3.1.2. Effect of vacuum pressure

Fig. 4(B) presents the S/N ratio for vacuum pressure. As can be observed, reduction in the downstream pressure increases permeate flux through the membrane. This is due to the fact that the driving force in MD process in general and VMD in particular is a vapor pressure difference across both sides of the membrane pores. Therefore, working at lower downstream pressure usually results in higher transmembrane flux [28,29].

3.1.3. Effect of feed concentration

The effect of feed (phenol) concentration on permeate flux is presented in Fig. 4(C). The experimental results show that the S/N ratio decreases with increasing feed concentration. At feed concentration of 20 and 510 mg/L, permeate flux is higher within the range of feed concentrations tested, and permeate flux does not decrease significantly. When feed concentration is higher than 510 mg/L, permeate flux reduces dramatically. This can be attributed to the fact that addition of phenol reduces water activity of the feed. Since water vapor pressure is the driving force of MD process, and it relates to water activity, reduction in permeate flux with further increasing feed concentration can be due to the reduction in driving force [30,31].

3.1.4. Effect of feed pH

Phenol in aqueous solutions exists in two forms: volatile phenol molecules (C_6H_5OH) and phenolate ions ($C_6H_5O^-$). Herein, considering the following reaction of phenol in an aqueous solution [32]:

$$C_6H_5OH + OH^- \rightarrow C_6H_5O^- + H_2O \quad (5)$$

It is reasonable to infer that addition of OH^- into wastewater enhanced formation of phenolate ions, the volatility of which is much lower than that of phenol molecules. As a result, phenol can be easily separated when some basic agents such as NaOH are combined with MD system, this causes that phenol remains in feed as phenolate ions that can be recovered easily [15]. The change with pH of the relative concentrations of both species, phenol and phenolate ion, with respect to the total concentration of phenol in solution can be observed in Fig. 5 [33]. As can be seen, phenolate ions increase with increasing feed pH. The maximum level of phenolate ions is obtained at pH value higher than 12, as a result in this work, for effective separation, pH was adjusted to higher than 12.

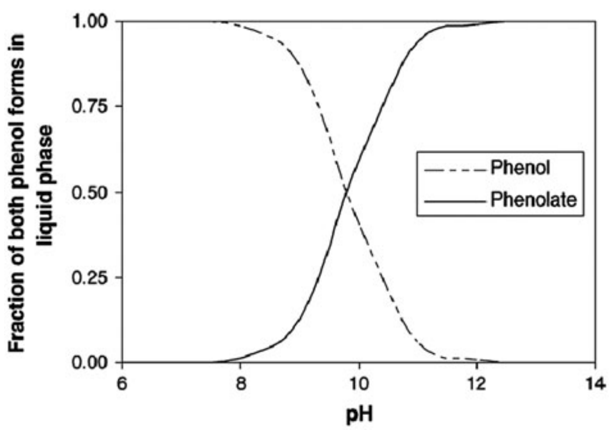

Fig. 5. Effect of the pH on phenol dissolution [33].

The effect of feed pH on permeate flux is presented in Fig. 4(D). As observed, flux decreases with increasing feed pH. Sodium hydroxide was used to increase feed pH. Addition of this material to the feed reduces the water activity in the feed solution, and thus due to decreasing the driving force of MD process, permeate flux decreases.

3.2. The influence of operating conditions on separation factor

3.2.1. Effect of feed temperature

The effect of feed temperature on water separation factor is shown in Fig. 6(A). As observed higher water selectivities were obtained at lower feed temperatures. This may be due to the presence of the phenol molecules in the feed solution that are not fully converted to the phenolate ions. With increasing temperature, evaporation rate of volatile phenol molecules, increases and phenol molecules can easily enter the vapor phase, thus separation factor decreases with the presence of phenol in permeate.

3.2.2. Effect of vacuum pressure

Fig. 6(B) illustrates the effect of vacuum pressure on water separation factor. According to Figure, it is

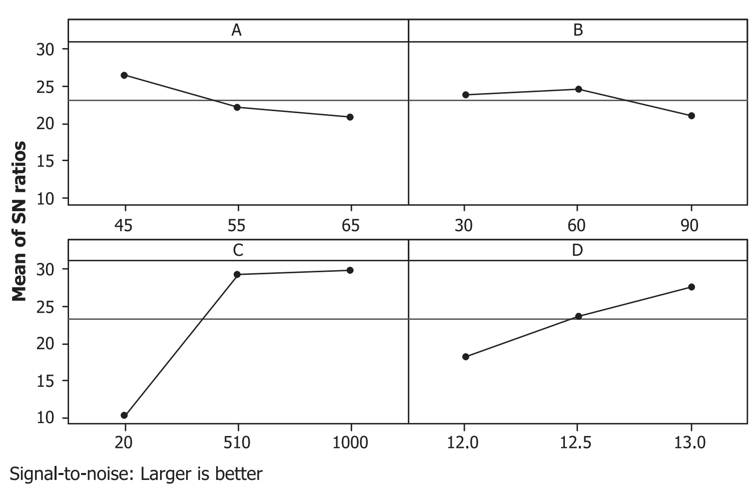

Fig. 6. Main effects plots for S/N ratios of separation factor. (A) Temperature, (B) vacuum pressure, (C) feed concentration and (D) feed pH.

clear that water separation factor is approximately independent of vacuum pressure, because the changes in S/N ratio with variations of vacuum pressure are insignificant. As will be explained later in ANOVA section, the factors, which have the lowest influence on water separation factor, is vacuum pressure (0.161%) which confirms previous statements.

3.2.3. Effect of feed concentration

Among the factors that most affect on water separation factor is feed (phenol) concentration. Fig. 6(C) shows the effect of feed concentration on water separation factor. According to the results increasing feed concentration from 20 to 510 mg/L significantly increases water separation factor, however, further increasing feed concentration to 1,000 mg/L increases water separation factor slightly. This can be attributed to the separation factor definition. According to Eq. (3), separation factor is directly proportional to feed concentration. Concentration ratio from 20 to 510 mg/L is approximately 25, while from 510 to 1,000 mg/L, is about 2. As a result the separation factor increases with increasing feed concentration.

3.2.4. Effect of feed pH

As mentioned earlier, one of the parameters affecting water separation factor is feed pH. According to Fig. 5, when pH < 7.5, phenol in the wastewater exists mostly in molecular form, while when pH > 12, is in ionic form. As a result, the main volatile component is phenol when pH < 7.5, while water acts as the volatile component when pH > 12. Fig. 6(D) shows water separation factor variation with feed pH. The results indicate that increasing feed pH increases water separation factor. This is due to increasing phenolate ions concentration with increasing feed pH, which has less volatility than phenol molecules.

3.2.5. Optimized conditions for separation factor

The optimization of conditions for water separation factor was performed as the treatment goal. The higher average $(S/N)_{HB}$ response represents the best level of each factor and can be interpreted as the optimized water separation factor. Considering that the water separation factor is independent of vacuum pressure, 60 mbar were chosen as an optimum point. This is because in lower vacuum pressure, the risk of membrane pores wetting increases. Furthermore, in higher vacuum pressure, permeate flux through the membrane decreases [28,29]. Therefore, the optimum treatment conditions are as follows: temperature 45°C, feed concentration 1,000 mg/L, feed pH 13, and vacuum pressure 60 mbar. At these conditions, the predicted water separation factor using its mean value is 61.55.

Table 5
ANOVA analysis for permeate flux

Factor	DOF	SS	Variance	*F*	*P* (%)
Temperature	2	15,048.5	7,524.2	5,942.52	83.91
Vacuum pressure	2	2,356.9	1,178.4	930.71	13.14
Concentration	2	288	144.0	113.71	1.606
Feed pH	2	227.3	113.6	89.75	1.267
Error	2	11.4	1.3		0.063
Total	9	17,932			100

Table 6
ANOVA analysis for water separation factor

Factor	DOF	SS	Variance	*F*	*P* (%)
Temperature	2	1,898.2	949.1	35.05	22.02
Vacuum pressure	2	13.9	7.0	0.29	0.161
Concentration	2	4,275.9	2,137.9	87.96	49.61
Feed pH	2	2,210.8	1,105.4	45.48	25.65
Error	2	218.8	24.3		2.538
Total	9	8,617.6			100

Table 7
Results of confirmation experiments and statistical model at optimum conditions

Run	Operating parameters				Experimental mean flux (kg/m^{2} h)	Predicted mean flux (kg/m^{2} h)	Experimental separation factor	Predicted separation factor
	T (°C)	Pv (mbar)	*C* (mg/L)	pH				
1	45	60	1,000	13	31.854	32.578	63.635	61.558
2	65	60	510	12.5	109.257	108.342	28.689	29.312
3	45	30	20	12	56.225	58.672	2.7792	2.9677

3.2.6. ANOVA results

The results of ANOVA obtained from the experiments are presented in Tables 5 and 6. The last column in the both tables shows the percent of contribution (*P*, %) of each factor to the response. The percent of contribution shows the influence of one factor on the total observed variance in the experiments. A higher value of the percent of contribution means that the factor affects more the response. According to Table 5, the factors, which have the most influence on permeate flux, are feed temperature and vacuum pressure. Furthermore, in the case of water separation factor as observed in Table 6, the effect of feed concentration is much higher than the other factors and the factor, which has the lowest influence on water separation factor, is vacuum pressure (0.161%). However, validations of these results are sensitive to choice of the levels as mentioned earlier about the disadvantages of the Taguchi method.

After determination of the optimum conditions using the statistical analysis, confirmation experiments were carried out at these conditions in order to evaluate the predicted results. The results are presented in Table 7. Comparing the results of these experiments with those of the statistical model shows a very good consistency. This means there is a good agreement between the predicted values and the experimental values and confirms the experimental design is very effective for the MD process.

4. Conclusions

An experimental study of VMD process to treat phenolic wastewater was carried out. Effects of feed temperature, vacuum pressure, feed concentration, and feed pH on water separation factor and total permeate flux using Taguchi method were also studied. For all the experiments, a commercial polytetrafluoroethylene membrane with a pore size of 0.22 µm was employed. VMD performance was measured in terms of water separation factor, and it was observed that it increased with decreasing feed temperature and increasing feed concentration and feed pH. Furthermore, with increasing feed temperature and decreasing feed concentration, vacuum pressure and feed pH, total permeate flux through the membrane increases. Optimum operating conditions for maximizing water separation factor are as follows: temperature, 45°C; vacuum pressure, 60 mbar; pH, 13, and concentration, 1,000 mg/L. Confirming experiments were also carried out. Errors of statistical model were calculated, and it was found that the errors are in the range of 2–6%. This confirms that there is a good agreement between the predicted values and the experimental data.

Acknowledgement

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