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Prediction of maximum permeate flux (%) of disc membrane using Response Surface Methodology (RSM)

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Abstract

The paper investigates increasing permeate flux (%) of the disc membrane which can improve the quality of rubber industrial effluent of Tripura. Response surface methodology (RSM) was used to optimize the independent influencing parameters to improve the permeate flux. The effect of different influencing parameters like operating pressure, membrane pore size and inlet feed velocity on membrane permeate flux were studied to determine the optimum operating conditions within the predefined boundary. The experiments were pre-planned and designed according to Central Composite Rotatable Design (CCRD), and second-order polynomial regression model was developed for regression and ANOVA study. Results show the membrane has maximum permeate flux (%) when the operating pressure is 14.50 Pa, pore size is 0.20 μm and inlet feed velocity is 2.10 m/sec. The Pareto analysis in the study established that the inlet velocity was the most influential parameter in the model equation.

KEYWORDS: Membrane, Optimization, Response surface methodology, Pareto analysis, ANOVA analysis, Central Composite Design.

INTRODUCTION

Tripura is a landlocked state of the Republic of India, situated in the North-eastern part of the country. The suitable agro-climatic conditions like fertile soil, rainfall etc. helps in the large-scale production of rubber, boosting the state economy. Due to these conditions Tripura has become the second largest producer of rubber in the country after Kerala. In the state, approximately 37558 hectares of land have been used for rubber plantation, where 29581 hectares of land are used as trapping area and the remaining 7977 hectares have been identified as newly cultivated areas. During 2013-2014, Tripura used to have an average annual production of 20000MT (MT=metric tonnes) of rubber. It has been predicted that the output may cross the 25000MT mark within 5-6 years as trapping areas are increasing with the passing days (Jamatia et al. 2014). The growth of rubber based industries play a vital role in attaining financial stability of the state. However, the environmental problems related to the rubber industries cannot be ignored.

The commercial processing and production of rubber goods requires a large quantity of water and chemicals as additives for manufacturing. These produce a significant amount of industrial effluents and discharge of untreated effluents to the local water bodies leads to the water pollution, water scarcity, and other problems. The alleviation of environmental degradation due to these untreated effluents is one of the significant challenges faced by the state. This research article deals with improving the quality of effluents from the rubber industry using membrane as green technology.

Over the last decades, membrane separation technique has attracted many environmental scientist and engineers for its ability to produce high quality permeate flux and without the

addition of chemicals making the process a green one. The membrane separation technique finds broad application in Petro-Chemical Industry (Ravanchi et al. 2009), Papermaking Industry (Zhou et al. 2012; Li and Zhang 2011), Distillery (Shivajirao 2012; Ambrosi et al. 2014), and Rubber Industry (Mokhtar et al. 2015; Banik et al. 2017). The latest development in the field of the membrane separation has found limited application due to rapid fouling tendencies which reduces the permeate flux. Different methods for overcoming the fouling problem has been developed and can be broadly classified as pre-treatment of feed, optimizing the operating conditions, cleaning procedures and membrane modification (Hilal et al. 2005). Optimization of the operating conditions can be useful to mitigate the fouling problem and thus reducing the cost of experimentation. Many studies implement Artificial Neural Network (ANN) and Genetic Algorithm (GA) for optimizing the parameters of the membrane module for increasing the permeate flux. Though these methods have numerous advantages like robustness, adaptability, etc. but the ways require large data set for preparing the model increasing the cost and time of experimentation (Yuen et al. 2000; Soleimani et al. 2013). It is crucial that an experimental design methodology be economical for extracting maximum complex information, saving materials, personal and experimental time (Myers and Montgomery 2002). So, Response Surface Methodology (RSM) is used for optimization with pre-design limited experimental run reducing the cost and time of experimentation. Central Composite Design (CCD) and Box Behnken and Doehlart designs (BDD) are among the principal response surface methodologies used for experimental design (Anupam et al. 2011b). CCD is widely used in experimental design over BDD for its ability to produce results with minimum error percentage, sequential construction of higher order design from more straightforward design, constant variance check, etc. (Anupam et al. 2011a). Thus, CCD was used for optimization of the membrane module. The quantity and quality of the permeate flux of the membrane generally depends upon pH, pressure, feed velocity, temperature, pore

size, membrane surface porosity, volumetric concentration factor, and membrane thickness (Lin et al. 1997; Kuo and Cheryan 1983; Cassano et al. 2007; Xu et al. 2016). The parameters like temperature and volumetric concentration remain constant as the effluents are collected only from the Rubber Industry of Tripura. Due to the constraint of the experimental module, the membranes of a particular thickness of different pore size were procured from the local market. Thus, from the parameters mentioned above, pressure, feed velocity and pore size of the membrane were selected for optimizing the membrane process. However, the optimization of operating conditions of the disc membrane using RSM based model for improving the quality of Rubber Industry effluents has not been reported earlier.

The objective of the study is to increase the permeate flux (%) of the disc membrane used for improving the quality of rubber industrial effluent. RSM was used to search the optimum operating conditions and to analyze the effect of the working conditions like operating pressure, pore size and inlet feed velocity on permeate flux of the membrane. The Central Composite Rotatable Design was used to obtain the design matrix of experimentation. Second order regression model was developed using MINITAB 18 software. ANOVA and Pareto analysis were conducted to determine the significance or influence of each parameter.

EXPERIMENTAL DESCRIPTIONS

Cellulose Acetate disc membrane was utilized for improving the quality of the rubber industrial effluent of Tripura. The pore size of the membrane bed was varied from 0.20 μm to 3 μm , operating pressure of the membrane was varied from 14.50 Pa to 931Pa and inlet velocity of the feed was varied from 0.179m/sec to 5.126 m/sec. The parameters were studied for their influences in the membrane permeate flux.

Many Samples of the rubber industrial effluent were collected from the common effluent treatment plant of the Bodhjung Nagar complex of TIDC. The samples were tested in the laboratory to find the characteristics of the feed. The parameters like pH, Total Dissolved Solids (TDS), Total Suspended Solids (TSS), Sulfide, Oil and Grease, Total Nitrogen, BOD were tested in the laboratory, and average values of these parameters of 3 to 6 samples were considered as the characteristic of the feed. Table 1 illustrates the components of the feed used as the inlet of the disc membrane module.

Figure 1 shows the schematic diagram of the experimental setup of the disc membrane installed in the laboratory. The raw feed was allowed to flow through the neutralizing tank to maintain the operating pH (as any deviation from the optimum pH of the membrane may affect the lifespan of the membrane module) since raw rubber industrial effluent is acidic. From the neutralizing tank, the feed flowed into the feed tank from which the water was allowed to flow inside the module with the help of a centrifugal pump. The concentrate of the membrane module was re-circulated to the feed tank, and the permeate flux of the membrane was collected in the permeate tank.

RESPONSE SURFACE METHODOLOGY

Design of Experiment (DOE)

The Design of Experiment (DOE) is a method of finding the cause and influence of the parameter by using a pre-planned approach. The purpose of the statistical method is to collect or gather information regarding the relationship between the various parameters affecting the process towards finding the optimum results. The experimental design methodology is very crucial, and it must be an economical way of extracting the maximum complex information, minimizing the experimentation time, material and personal cost (Kincl et al. 2005). Central Composite Design (CCD) or Central Composite Rotatable (CCRD) is one of the principal

response surface methodologies used for experimental design and developing a second-order or quadratic model for response variable using the pre-design limited experimental run. CCD has wide application in designing the experimentation. The most suitable approach for fitting the quadratic surface is the CCD method which can optimize the operating parameters with the help of minimum number of experiments, and also evaluate the relationship between the variables. The CCD comprises of 2^k ($2^3 = 8$) factorial run with $2k$ [$2(3) = 6$] axial run and n_0 ($n_0 = 6$) center runs (Anupam et al. 2011b). Investigation of each variable in CCD are conducted in two levels and when the number of the parameter (k) increases the number of runs for replicating total design increases. Such kind of design provides desirable predicted values at equidistant points from the centers which is one of the advantages of the Response Surface Methodology (RSM). Center point is essential for calculating the error in experimentation and reproducibility of the data. The input parameters are coded to avoid any error due to the difference in units. The maximum and minimum value of the inputs are coded as $\alpha = 1.68179$ and $-\alpha = -1.68179$ respectively. From the point of recommendation and efficiency for input parameter ($k=3$), the rotatable design is preferred because Orthogonal and Hartley's design properties are the worst even if their requirements of the experimentation are quite low. The later methods are useful only when design points are to be kept minimal. Rotatable designs of second order is the most suitable design method as it does not minimize the variance of estimates of regression coefficients. This method of the design is the most efficient method of solving research problems which aims to find the optimum solution. To evaluate the effect of various operating variables CCRD has been incorporated. The operating parameters that are selected for conducting the study are operating pressure (X_1), pore size (X_2) and inlet feed velocity (X_3). For the Present study, the number of independent parameters are three so for each categorical variable like full factorial points, axial points and replicated center points can be found by using the equation 1:

$$M = 2^k + 2k + n_0 \quad (1)$$

Where M is the total number of required experiments and k is the number of the independent parameter, each trial coded in the form of $-\alpha$ (-1.68179), -1, +1, α (1.68179). The encrypted value of α depends on the number of independent parameters in the factorial portion of the design. For the three independent parameters, the α can be defined as,

$$\alpha = 2^{\frac{k}{4}} \quad (2)$$

For k=3

$$\alpha = 2^{\frac{3}{4}} = 1.682 \quad (3)$$

The relation of the coded and un-coded parameters can be defined as,

$$\text{coded value} = \frac{X_m - \overline{X_m}}{\Delta X} \quad (4)$$

Where X_m represents the actual data points of the m^{th} factor in the un-coded form, $\overline{X_m}$ is the average value of the minimum and maximum data point for the m^{th} factor, and ΔX denotes the change in step.

Empirical model development

Response Surface Methodology is the suitable approach of approximation which has been introduced to search the right existing relationship between the influencing dependent variables and the set of independent parameters. This single response model has been developed using RSM to correspond to the independent parameters. The mathematical regression model of second order polynomial is developed to predict the maximum permeate flux response depending on the function of the independent variables including their interactive relationship (Dutta 2013). The general behaviour of the method can be broadly defined by using the following equation of quadratic nature.

$$Y = B_0 + \sum_{i=1}^N B_i X_i + \sum_{i=1}^N B_{ii} X_{ii}^2 + \sum_{i=1, j=1}^N B_{ij} X_i X_j \quad (5)$$

In equation 5, Y denotes the output of the model or predicted response of the model, B_0 shows the offset term of the model, B_i illustrates the linear effect, B_{ii} shows the square impact and B_{ij} denotes the interactive terms of the model, lastly X_i and X_j denotes the coded value of the influencing independent variables. In the concerned study, a second order polynomial equation has been developed by using uncoded values of the independent parameters and multiple regression techniques has been utilized for calculating the coefficient of the model. The second order polynomial equation can be defined as,

$$Y = B_0 + B_1X_1 + B_2X_2 + B_3X_3 + B_{11}X_{11}^2 + B_{22}X_{22}^2 + B_{33}X_{33}^2 + B_{12}X_1X_2 + B_{23}X_2X_3 + B_{13}X_1X_3 \quad (6)$$

RESULT AND DISCUSSION

Model equation development

From the experimental study, it was found that parameters like operating pressure, pore size and inlet feed velocity of the membrane module affects the percentage permeate flux. Regression model equation was constructed by considering parameters like operating pressure, pore size, and inlet velocity of the feed. CCRD was adopted to develop the correlation between the selected influencing parameters to maximize the permeate flux of the membrane. The complete design value matrixes along with the response matrix values were calculated from the experiments as provided in Table 2. The center runs were repeated six times as it helps in estimating the quadratic terms in the developed model. Calculated values of all the coefficient of the regression equation (equation 6) are provided in Table 3. The values of the experiments in Table 3 were calculated from the experimental data points using MINITAB® 18 to develop the model equation for percentage permeate flux of the disc membrane for improving the quality of the rubber industrial effluent (equation 7).

$$\begin{aligned} \text{Permeate Flux (\%)} = & 64.72 - (1.647 \times \text{Operating Pressure}) - (2.340 \times \text{Pore size}) + (2.897 \times \text{inlet} \\ & \text{velocity}) - [0.544 \times (\text{Operating Pressure})^2] + [0.444 \times (\text{pore size})^2] - [0.505 \times (\text{inlet velocity})^2] + \\ & [2.875 \times (\text{operating pressure}) \times (\text{Pore size})] + [2.608 \times (\text{Operating pressure}) \times (\text{inlet velocity})] - \\ & [1.480 \times (\text{pore size}) \times (\text{inlet velocity})] \end{aligned} \quad (7)$$

From equation 7, it was found that percentage permeate flux of the disc membrane showed linear behaviour, square and 2-way interaction between the parameters (operating pressure, membrane pore size and inlet velocity of the feed).

ANOVA Study

The Analysis of Variance (ANOVA) study was conducted to determine the most significant parameter to test the acceptability of the model (Suresh et al. 2016). ANOVA study comprises of classifying and cross-classifying results of statistics and was evaluated by using specified classification difference, which was conducted by Fisher's Statistical test (F-value test). F-value can be defined as the ratio of the mean square of regression to the error. $F = \text{MRR}/\text{MRe}$ can define the F-value. Where MRR denotes the mean square regression and MRe denotes the noise. The parameters are said to be more significant if the F-value is greater than F-critical value and the P-value is found to be less than 0.05. The F-value is said to be the least significant one under the following conditions: i) if the F-value is greater than F-critical but p-value is greater than 0.05 and ii) if the F-value is less than F-critical value. F value represents the significance of each variable in the tested model. It is used to check the agreement of the regression model with the experimental data (Dutta 2013). For determining the coefficient of R^2 , the regression equations were subjected to the F-value test. Lack of fit (LOF) is one of the model evaluation tests for evaluating the significance or acceptability of the model. In the concerned study, Table 4 shows that the regression model, linear and 2-way interaction of the model developed is of high significance and it can be concluded from the calculated F value of 6.95, 12.56 and 7.66 respectively as the values were greater than their respective F-critical values and corresponding P-values were less than 0.05. The square

interaction of the parameters was considered to be the least significant one. In statistical hypothesis testing, P-value or probability value is a statistical method for evaluating the significance of the parameters. The P-values less than 0.05 were assumed to be significant, as it is the default parameter of the developed model. From Table 4, p-value of the regression model, linear and 2-way interactions of the model were found to be 0.003, 0.001 and 0.006 respectively. The predicted R^2 of 73.81% showed good concurrence with the adjusted R^2 of 89.62%. From the ANOVA study, it was found that all the terms in the model equation were not of same significance. ANOVA study helps to sort out the most significant parameters from the least ones (Dutta 2013; Anupam et al. 2011a). Smaller the value of P more significant is the term in the model equation. From table 3, based on the p values the crucial parameters are sorted out and least significant ones were removed. So, the new model equation is defined by equation 8,

$$\begin{aligned} \text{Permeate Flux (\%)} = & 64.72 - (1.647 \times \text{Operating Pressure}) - (2.340 \times \text{pore size}) + \\ & (2.897 \times \text{inlet velocity}) + (2.875 \times \text{Operating pressure} \times \text{Pore size}) \\ & + (2.608 \times \text{Operating pressure} \times \text{Inlet velocity}) \end{aligned} \quad (8)$$

Figure 2 illustrates the experimental results versus the calculated results of the permeate flux (%) of disc membrane. The R^2 value showed the acceptability of the model and the value closer to unity is the most acceptable one. The value of R^2 was calculated from the Figure 2 and is found to be 84% and a typical value greater than 50% was considered to be acceptable (Liew et al. 2003; Santhi et al. 2002; Moriasi et al. 2007). As the value of R^2 is closer to unity, it was concluded that the calculated results had a good agreement with the experimental results and the model was thus considered as the best one. Table 4 shows the analysis of the influencing variance for permeate flux (%) of the disc membrane.

Response surface plots

Utilization of 3D regression plots is recommended by Aktas (Aktaş 2005) for the graphical interpretation of the interaction of the influencing parameters. 3D surface plots are useful for gathering information regarding the system behaviour within the experimental boundary; it evaluates the effects of the parameters of testing on the response and contour plot between the influencing parameters of the model equation. The nature of the plot demonstrates that interactive results between the independent influencing parameters which are not significant and the optimum values of the test variables are also not easy to find. Figure 3(a-c) illustrates the contour plot of the percentage permeate flux of the disc membrane. Figure 3(a) illustrates that when the operating pressure increased the percentage permeates flux decreased, as the high pressure compelled the solutes to block the membrane pores. The increase in the inlet velocity of the feed increased the percentage permeates flux initially but became constant after few operations. Figure 3(b) illustrates that percentage permeate flux increased initially with the increase in the operating pressure but gradually showed a declining trend at constant inlet feed velocity. Figure 3(c) demonstrates that at constant operating pressure, permeate flux increased with the improvements in the inlet feed velocity. Figure 4(a-c) illustrates the surface response plots of permeate flux (%) of the disc membrane for improving the quality of the rubber industrial effluent. The peak of the surface response plot denotes the maximum design point of the response which is calculated from the independent influencing parameters operating pressure, pore size, and inlet feed velocity. Figure 4 (a) shows the surface plot of the permeate flux (%) to the operating pressure and pore size where the inlet velocity is kept constant. The plot shows some increasing trends of permeate flux with the gradual increase of pressure but have declining trend with the pore size as reducing of pore size increased the membrane resistance thus affecting the flux. Figure 4(b) is the surface plot of the permeate flux to the operating pressure and inlet velocity when the pore size of the membrane bed is kept constant. The plot shows the increase of permeate flux with the increase in operating

pressure and feed velocity; as high kinetic energy of the fluid particles and high trans-membrane pressure facilitate the high permeate flux. But beyond a particular point, the surface plot illustrates declining trend due to the partial and complete pore blocking. The surface plot of permeate flux to the inlet velocity and pore size of the membrane have decreasing trend due to the high fouling tendency of the particle having high kinetic energy and increase of membrane resistance due to blocking of the pores.

Pareto analysis

Pareto analysis is used to determine the most significant terms having the highest cumulative effect in the outcome. The study comprises of a bar chart where the height or length of the bar chart denotes the impact of the parameters. The parameter which has the maximum height or length is the most significant one (Anupam et al. 2011a). Figure 5 illustrates that all the parameters are not equally important. It is the inlet feed velocity that has maximum effect on the percentage permeate flux of the membrane.

Optimization process and optimum parameter

From the study, it was found that maximum permeate flux of the disc membrane was subjected to operating pressure, pore size, and inlet feed velocity. Figure 6 illustrates the optimization plot of the disc membrane, where the parameters were optimized to maximize the permeate flux and thus reducing the fouling. The plot shows the affect of each parameter at the top of the figure on the resulting response. The vertical red line in the plot illustrates the current factor setting of the model. The numbers in red displayed at the top of the column show the optimal parameters. The horizontal dashed blue line and number in blue shows the response (percentage permeate flux) for the optimal parameters. From the calculation it was found that when the membrane is operating at a pressure of 14.50 Pa, membrane pore size of 0.20 μm and inlet feed velocity was 2.10 m/sec then the membrane module exhibit maximum

permeate flux of 79.77%. When the experiments were conducted using the predicted optimal operating parameters, the membrane exhibited the maximum permeate flux (%) of 78.96%. The experimental and predicted value were found to be compatible, thus justifying the use of the RSM based model for accurate model development.

CONCLUSION

The Present study was conducted to increase the permeate flux (%) of the Disc membrane. Response surface methodology (RSM) was implemented in the study to optimize the process and to find the optimum value of the independent influencing parameters. The research shows membrane used for treating the effluents; the process gets affected by the parameters like operating pressure, pore size, and inlet feed velocity. The Central composite rotatable design (CCRD) was developed to determine the effect of these independent influencing variables. Responses obtained from the model were thoroughly analyzed. ANOVA study showed that interaction like operating pressure to operating pressure, pore size to pore size and inlet velocity to inlet velocity was not essential for developing the regression model. The Pareto Analysis showed the terms having more significance or influence on the permeate flux (%) of the Disc membrane. The experimental results hold good concurrence with predicted results of the regression model. The optimum value of permeate flux (%) was calibrated by using independent parameters like the operating pressure, the pore size and inlet feed velocity of the membrane. And the values of these influencing parameters were 14.50Pa, 0.20 μ m and 2.10 m/sec respectively resulting in maximum permeate flux of 79.77%. The above mentioned parameters were the optimum operating conditions required for treating the rubber industrial effluent and reducing the fouling of the disc membrane.

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Table 1. Feed Characteristics (Rubber Industrial Effluent)

Sl. No.	Parameters	Value 1	Value 2	Value 3	mean	Standard Deviation
1.	pH	4.80	4.80	4.70	4.76	0.047
2	Total Dissolved Solid (TDS) (mg/l)	3954	3953	3955	3954	0.27
3	Total Suspended Solid (TSS) (mg/l)	393	393	395	393.6	0.32
4	Sulphide (mg/l)	25	26	24	25	0.27
5	Oil and Grease (mg/l)	10	11	10	10.34	0.47
6	Total Nitrogen (mg/l)	196.20	195.90	196.40	196.16	0.45
7	BOD ₅ (mg/l)	725	725	726	725.33	0.47

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Table 2. Experimental Coded, Un-Coded and Response Matrix

Sl. No.	Coded values			Un- coded values			Response (Permeate Flux) (%)
	Operating pressure	Pore Size	Inlet velocity	Operating Pressure (Pa)	Pore Size (μm)	Inlet Velocity (m/sec)	
1	1	1	-1	505	1.70	2.50	58.60
2	1	1	1	505	1.70	2.80	68.60
3	0	1.68179	0	473	3.00	2.65	61.23
4	0	0	-1.68179	473	1.60	0.179	59.23
5	-1	-1	-1	441	1.45	2.50	65.61
6	0	0	1.68179	473	1.60	5.126	70.23
7	0	0	0	473	1.60	2.65	64.64
8	-1	-1	1	441	1.45	2.80	71.10
9	-1	1	1	441	1.70	2.80	57.12
10	0	0	0	473	1.60	2.65	64.64
11	1	-1	1	505	1.45	2.80	66.12
12	-1.68179	0	0	14.5	1.60	2.65	68.97
13	-1	1	-1	441	1.70	2.50	62.51
14	1	-1	-1	505	1.45	2.50	55.16
15	0	0	0	473	1.60	2.65	64.64
16	0	-1.68179	0	473	0.20	2.65	73.60
17	0	0	0	473	1.60	2.65	64.64
18	1.68179	0	0	931	1.60	2.65	60.27
19	0	0	0	473	1.60	2.65	64.64
20	0	0	0	473	1.60	2.65	64.64

Table 3. Calculated coefficient of regression for percentage permeate flux of the disc membrane

Terms	Coefficient	SE Coefficient	T-Value	P-value
Constant	64.72	1.00	64.73	0.000
Operating pressure	-1.647	0.663	-2.48	0.032
Pore size	-2.340	0.663	-3.53	0.005
Inlet velocity	2.897	0.663	4.37	0.001
(Operating pressure) × (operating pressure)	-0.544	0.646	-0.84	0.419
(Pore size)×(Pore size)	0.444	0.646	0.69	0.507
(Inlet velocity)×(Inlet velocity)	-0.505	0.646	-0.78	0.452
(Operating pressure)×(Pore size)	2.875	0.867	3.32	0.008
(Operating pressure)×(inlet velocity)	2.608	0.867	3.01	0.013
(Pore size)×(Inlet velocity)	-1.480	0.867	-1.71	0.119

Table 4. Analysis of variance for percentage permeate Flux of the Disc Membrane

Source	DF	Adj. SS	Adj. MS	F-value	F-critical	P-value
Model Regression	9	375.971	41.775	6.95	2.42	0.003
Linear	3	226.445	75.482	12.56	3.13	0.001
Square	3	11.486	3.829	0.64	3.13	0.608
2-way interaction	3	138.041	46.014	7.66	3.13	0.006
Lack of fit	5	60.100	12.020	-		-
Pure error	5	0.00	0.00	-		-
Total	19	436.071				

Figure1. Schematic diagram of Experimental setup of Disc Membrane

Figure 2. Experimental Vs Calculated results of percentage permeate flux of the Disc membrane

Figure 3. Contour plots for permeate flux of Disc Membrane for improving the quality of rubber industrial effluent of Tripura (a) Constant Pore Size (b) Constant inlet feed velocity (C) constant operating pressure.

Figure 4. Response surface plot for percentage permeate flux of the Disc membrane (a) constant inlet feed velocity (b) constant pore size (c) constant operating pressure.

(b)

(C)

Figure 5. Pareto graph of the percentage permeate flux of the disc membrane

Figure 6. Optimization plot of parameters for percentage permeate flux of the Disc membrane

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