

Treatment of Coloured Waste Water by Using Spiral Wound Nanofiltration Membrane and Parameter Evaluation

R. R. Marlar¹, Sanjeevani Hooda², Sangeeta Garg³, Vidusi Bajpai¹, S. Bajpai³

PhD¹, M. Tech², Associate Professor³, Department of Chemical Engineering,
^{1,2,3}Dr B R Ambedkar National Institute of Technology, Jalandhar, 144011, India

Abstract: In this work, efficacy of nanofiltration (NF) process treating coloured waste water was investigated. The NF membrane in spiral wound (polyamide) configuration is having molecular weight cut-off (MWCO) 600 Da was used for the removal process. Experiments were conducted using synthetic waste water. The various membranes parameters such as hydraulic permeability (L_p), rejection co-efficient (σ) and solute permeability (P_s) was estimated using Spiegler-kedem (SK) model. The membrane performance was examined by changing initial feed concentration, pH, temperature and external pressure. It was found that NF process can effectively treat coloured waste water.

Keywords: NF process, SK model, Polyamide membrane, waste water, textile industry.

1. INTRODUCTION

Most of the industries are using synthetic dyes like textile, printing, paper, leather industries etc., [1]. Dyes are toxic in nature [2]. Dye waters are rich in colour, suspended solids and COD [3]. Physical, chemical and biological methods are commercially available to treat textile effluents. Dyes are naturally complex aromatic molecular structures and are stable so it's difficult to biodegrade them. Dye wastewaters are easily affecting the nature of the waters when it's mixed with the streams or some other water sources. Once it get mixed with natural waters, it affects the aquatic life. Photosynthesis process is significantly effected because of the restriction in light penetration. Meanwhile, it's toxic also due to the presence of aromatics, metals, chlorides etc. [6]. Other than water streams, dye wastewaters are also less concentration affecting solid and air environmental conditions. Dye staining is possible, if 1 ppm of dye or even less concentration value in water is enough to produces coloured wastewater [5]. So the dye wastewaters are sufficiently well treated before it leaves from the treatment plant in order to minimize water pollution.

The available techniques to treat dye effluents are photochemical reaction [3, 4], coagulation-flocculation [8], Ozonation [10, 11, 12], integrated treatment process of activated sludge process with chemical coagulation [13], adsorption [9], biodegradation [14], and membrane separation [21]. Most process has its own advantage and disadvantage. Among these techniques adsorption is the common technique, but it has the limitation of equilibrium and also it's a slow process [15]. Advance oxidation process (AOP) need further scale up and need research with the concern of cost effectiveness. The formation of by products is one of the main disadvantage of AOP. Marmagne [16] et al., did experimental studies with coagulation and flocculation and concluded that it's not a suitable method for reactive dye removal. Tzitzis et al. [11] reported that ozonation can give the best results in short ozone contact time. To increase the performance of ozonation, either combined process-like coagulation-precipitation/ozonation are to be used or increase the ozone dosage which will affect the operation cost. Even after the combination of two processes, the COD reduction is less compared to other processes [12]. Traditionally, integration of two processes were followed to treat textile wastewater, for example activated sludge process (ASP) and chemical coagulation. The integrated techniques are efficient in the terms of

removal, but it has the limitations of its own performance and still need further research and development [10, 13]. Biodegradation methods need fermentation process, which is difficult due to large volumes of textile effluents. The continuous base is critical in biodegradation process due to the consumption of time [7]. So the most promising technique is membrane technology to treat dye wastewater. It has the potential to remove the dye stuff, to concentrate the auxiliary chemicals and to produce purified water.

Membrane Technology has multi advantages like clarification, concentration etc. continuous feeds are also possible in membrane for dye effluent. The limitations in temperature, chemical environment and microbial disturbance for other techniques is surpassed by membrane technology [17, 18].

Chakraborty [15] et al., compared the available filtration techniques. Microfiltration (MF), Ultrafiltration (UF) and Reverse Osmosis (RO) are suitable for colloidal dyes, secondary textile wastewater and dye bath effluents respectively. Nano filtration (NF) is the best for low molecular weight (<1000 Dalton) organic compounds. In dye wastewater treatment better results can be achieved by membrane filtration technique [19]. Ulson et al., [20] did experimental studies on various membranes with respect to flux, COD, reduction in color and conductivity. Optimized results concluded that nano membrane is suitable for color rejection. NF lies in between RO and UF, which is a well-known fact in membrane research. The process of NF include, high permeate flux, low osmotic pressure difference, with retention of molecular weight compounds (>300 dalton) are, low investment, low operational and maintenance costs [22].

In this present work, a self-designed test plant cell having NF membrane was used to optimize various parameters. The colour removal of methylene blue (MB) dye was optimized by evaluating different parameters like pH, concentration, transmembrane pressure, flow, observed rejection etc. In this experimental study, MB was used as a synthetic dye; parametric studies were performed and results were discussed to evaluate the performance of the membrane. The various membranes parameters such as hydraulic permeability (L_p), rejection co-efficient (σ) and solute permeability (P_s) were estimated using Spiegler-Kedem (SK) model [24, 25].

2. MATERIALS AND METHODS

2.1. Materials:

Methylene Blue ($C_{16}H_{18}ClN_3S$; M.W. 319.86) was purchased from Nice Chemicals Pvt Ltd. Kerala, India. Stock solutions of various concentrations were prepared using RO water. RO water was obtained from Aquelix 5 which was procured from Millipore Asia Ltd., Taiwan. To maintain the pH level concentrated sulphuric acid and sodium hydroxide were used which were purchased from S.D. Fine Chemicals Limited, Mumbai (India).

2.1.1. Membrane:

A hollow fiber membrane with a molecular weight cut off of 600 Da was used for the experimental study. The configuration of the membrane is spiral wound. The membrane was obtained from Permionics Membrane Pvt Ltd., India. Characteristics are mentioned in Table.1

Table 1: Membrane Properties

MWCO	pH Tolerance	Temperature tolerance	Membrane module	Membrane type	Area
600 Da	2 - 12	Up to 80°C	Spiral-wound	Polyamide	0.24 m ²

2.2. System configuration:

The test cell purchased from PMI India, Ghaziabad. Fig. 1 shows the flowchart of NF test cell. The test cell is equipped with pressure gauges, flow meters, feed tank (10 litre capacity), pump, stirrer, retentate tank (10 litre capacity), prefilter, temperature indicator and NF spiral wound membrane. Feed flow was controlled either by rpm of the pump or by restricting the flow of the channel path. Batch and recycle experiments were controlled by solenoid valve 1 & solenoid valve 2. The NF membrane is configured with flow meters, pressure gauges and valves for the measurement of flow, pressure and for controlling the flow respectively. NF test cell configured with automatic time settings which is used for the automatic machine off.

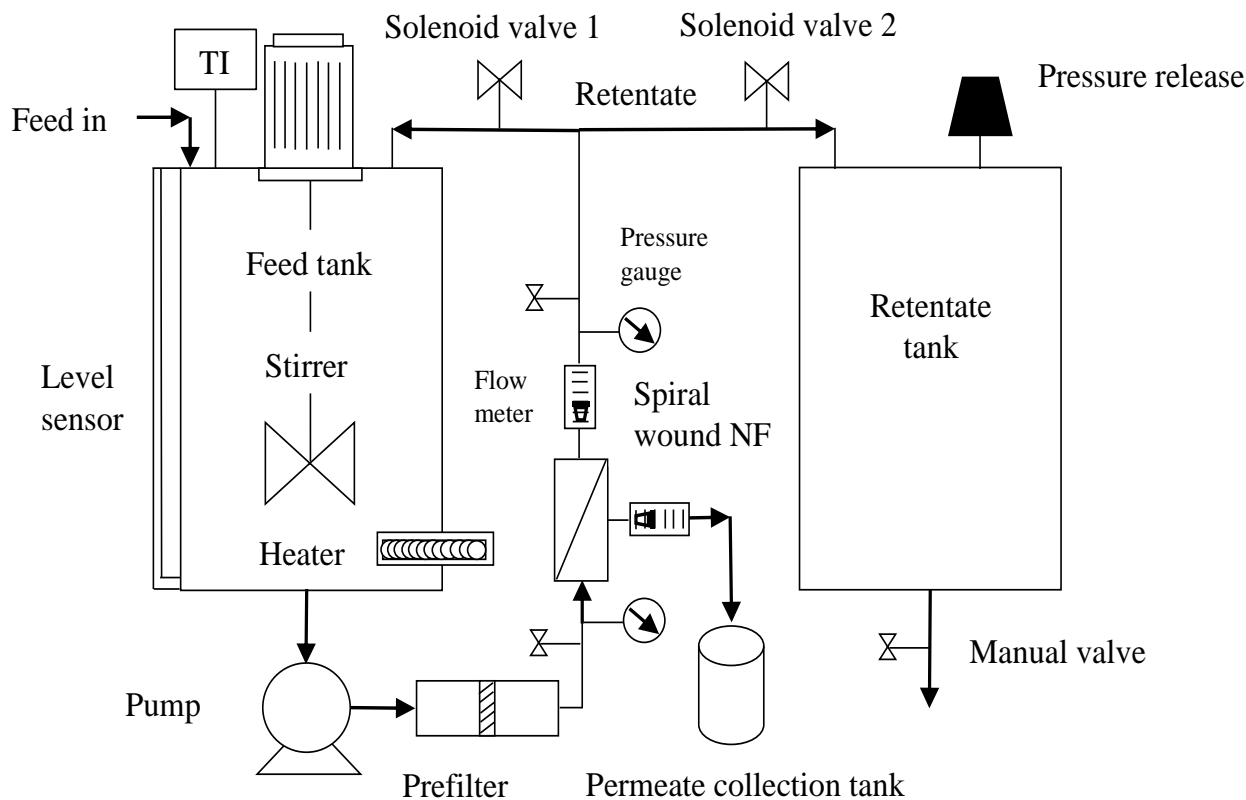


Figure 1: Flowchart of NF Test cell

2.3. Experimental Methods:

Several experimental works were involved in the parametric study which is described as follows:

2.3.1. Membrane Compaction & Flushing:

The fresh membrane was maintained at a pressure of 690 kPa for 3 hours. For compaction, RO water was used as feed. Water flushing was given to the membrane for 1 hour after each process.

2.3.2 Feed solution:

The feed water was prepared by mixing a known amount of MB with RO water. The sample concentrations were in the range of 20, 40 and 60 mg/l.

2.3.3. Hydraulic permeability:

Hydraulic permeability was measured by using RO as feed. The feed pressure and retentate pressure were measured from pressure gauges. The permeate pressure was 1 atmospheric pressure (1 atm). The flux values were measured for different operating pressures. Graph was plotted between transmembrane pressures to the respected flux values. The average permeability value is $2 \times 10^{-5} \text{ L h}^{-1} \text{ m}^{-2} \text{ atm}^{-1}$.

2.3.4. Experimental conditions:

The prepared feed concentrations (20, 40 and 60 mg/l) were given as a feed. By changing the pump's rpm and by restricting the valve, the flow rate was maintained. The change in pressure was measured for different inlet pressures (1, 2, 3 & 4 kgf/cm²). The pH of the feed solution was adjusted by using concentrated sulphuric acid and sodium hydroxide, like 4, 6, 7 & 8. Solenoid valve 1 was used for the experiment. It is allowed retentate water to the feed tank again. Solenoid valve 2 was used to remove water from feed tank and allowed in retentate tank or in other words it can be used for batch study without recycling the retentate. All the experiments were conducted with solenoid valve 1 only for better efficiency.

2.3.5. Permeability after the experiments:

After conducting each experiments the NF membrane was water flushed with by RO water for 1 hour between each experiments. Then the same procedure was repeated to find the permeability again. It was found that membrane permeability was almost constant before and after the experiments.

2.3.6 Sample analysis:

2.3.6.1 pH:

P^H of the samples at different stages (feed, permeate and retentate) were measured by a pH meter, supplied by Thermo Fisher scientific company, America.

2.3.6.2. Dye concentration:

Inlet and outlet concentration of the sample was analyzed by using Shimadzu UV vis spectroscopy 2700 which was procured from Shimadzu Corporation, Japan.

2.3.6.3. Reflection co-efficient (σ) determination:

To estimate the mass transfer co-efficient (k), Murthy [31] et. al. classified three measurement techniques which is 1. Optical or micro electrode (direct measurement), 2. Through the calculation of true rejection (Indirect measurement) 3. By combining concentration polarization model and membrane transport model (Indirect measurement). In this paper the second techniques was used to evaluate the reflection co-efficient. Jonsson [33] et al, calculated equation co-efficient by indirect measurement with the use of concentration and polarization model. The assumptions of this calculation are true retention (R_t) is a function of a membrane surface concentration and membrane concentration varies due concentration polarization. Extending circulation to infinity leads to the measurement of true retention. From the concentration polarization model [25],

$$\varphi = \exp\left(-\frac{J_v}{k}\right) = \frac{c_m - c_p}{c_f - c_p} \quad (1)$$

Jonsson [33] et al rearranged the equation as follows,

$$\ln\left(\frac{1-S}{S}\right) = \ln\left(\frac{1-R_t}{R_t}\right) + \text{const}\left(\frac{J_v}{U^a}\right) \quad (2)$$

Now the assumption of mass transfer value (k),

$$k = U^a \quad (3)$$

where, U is circulation velocity and “a” is constant (0.8 for turbulent flow & 0.33 laminar flow) [31].

From the graph plot of, $\ln\left(\frac{1-S}{S}\right)$ vs $\left(\frac{J_v}{U^a}\right)$, the obtained intercept is as follows;

$$\text{Constant} = \ln\left(\frac{1-R_t}{R_t}\right) \quad (4)$$

The true retention can be calculated by using formula [24],

$$R_t = 1 - \frac{c_p}{c_m} \quad (5)$$

The concentration at the membranes was calculated by using the above formula. Osmotic pressure can be calculated by using Van't Hoff Equation [25],

$$\Delta\pi = iCRT \quad (6)$$

It can be written as follows,

$$\Delta\pi = iRT(C_m - C_p) = \alpha(C_m - C_p) \quad (7)$$

To calculate the reflection co-efficient, combined model of Spiegler-Kedem/film theory model was used [30, 31]. The expressions are as follows,

$$J_v = L_p * (\Delta P - \sigma\Delta\pi) \quad (8)$$

It can be re-written as follows by using equation 7,

$$J_v = L_p * \{\Delta P - \sigma \alpha (C_m - C_p)\} \quad (9)$$

Now the expression for volumetric flow for σ value,

$$\sigma = \frac{\left(\Delta P - \left(\frac{J_v}{L_p}\right)\right)}{\alpha(C_f - C_p)} \quad (10)$$

where, J_v = Volumetric flux (m/s), ΔP = Osmotic pressure (atm), i = Vant Hoff factor, C_f = feed concentration, R = Universal Gas constant (0.0821 L.atm/mol.K), T = absolute temperature (K), True retention (R_t) = $1 - C_p/C_m$, C_f = feed concentration, C_p = Permeate concentration, C_m = concentration at the membrane surface, L_p = Hydraulic Permeability = $2 \times 10^{-5} \text{ L h}^{-1} \text{ m}^{-2} \text{ atm}^{-1}$ (from experimental data).

2.3.6.4. Solute Permeability determination:

The observed rejection (R_o) for the membrane can be calculated by the following formula [24]:

$$R_o = 1 - \left(\frac{C_p}{C_f}\right) * 100 \% \quad (11)$$

where, C_f and C_p are the concentrations of feed and permeate respectively.

The simplified form of solute flux equation [32] is as follows,

$$J_s = P_s * (C_m - C_p) + (1 - \sigma) * J_v * C_s \quad (12)$$

where, J_s = Solute flux, P_s = Solute permeability. C_s = logarithmic mean concentration of the solute between the feed and permeate. According to the SKK theory the observed rejection (R_o) can be explained as follows;

$$R_o = \frac{\sigma(1-F)}{1-\sigma F} \quad (13)$$

where, F = parameter that is related to solvent flux as follows,

$$F = \left(-\frac{1-\sigma}{P_s} * J_v\right) \quad (14)$$

The Spiegler-Kedem-Katchalsky model [30] simplified a relation between reflection co-efficient, solute permeability and solvent flux as given below,

$$\ln(X) = -\frac{1-\sigma}{P_s} * J_v \quad (15)$$

Now the solute permeability can be written as follows,

$$P_s = \frac{-(1-\sigma)}{\ln(X)} * J_v \quad (16)$$

To calculate the parameter F the following equations used [30],

$$F = \frac{1}{1-\sigma} - \frac{1}{1-R_o} * \frac{1-\sigma}{\sigma} \quad (17)$$

3. RESULTS AND DISCUSSIONS

Parametric study of treatment of the textile effluents is the main objective of this work. Synthetic wastewater was prepared by using MB for various concentrations (20, 40 & 60 mg/l). For the same concentrations, pH (4, 6, 7 and 8) was adjusted by using acid and base. By changing the operating pressures the permeate flow and concentration of MB was measured in feed, permeate and retentate. The effect of concentration, pH and transmembrane pressure were also discussed. Spiegler-Kedem model was used to evaluate the rejection co-efficient (σ) and solute permeability (P_s).

3.0 Hydraulic Permeability:

The membrane hydraulic permeability was analyzed by using RO water. Flux values for different operating pressures were measured and plotted. Average permeability value is $2 \times 10^{-5} \text{ L h}^{-1} \text{ m}^{-2} \text{ atm}^{-1}$.

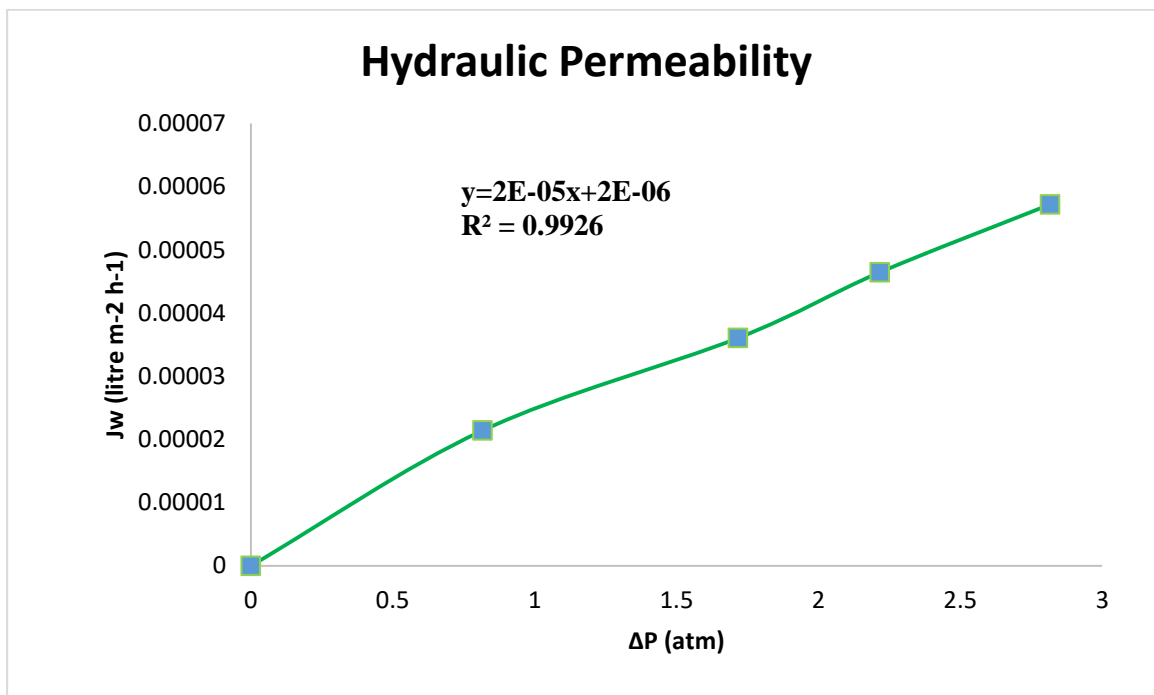


Figure 2: hydraulic permeability

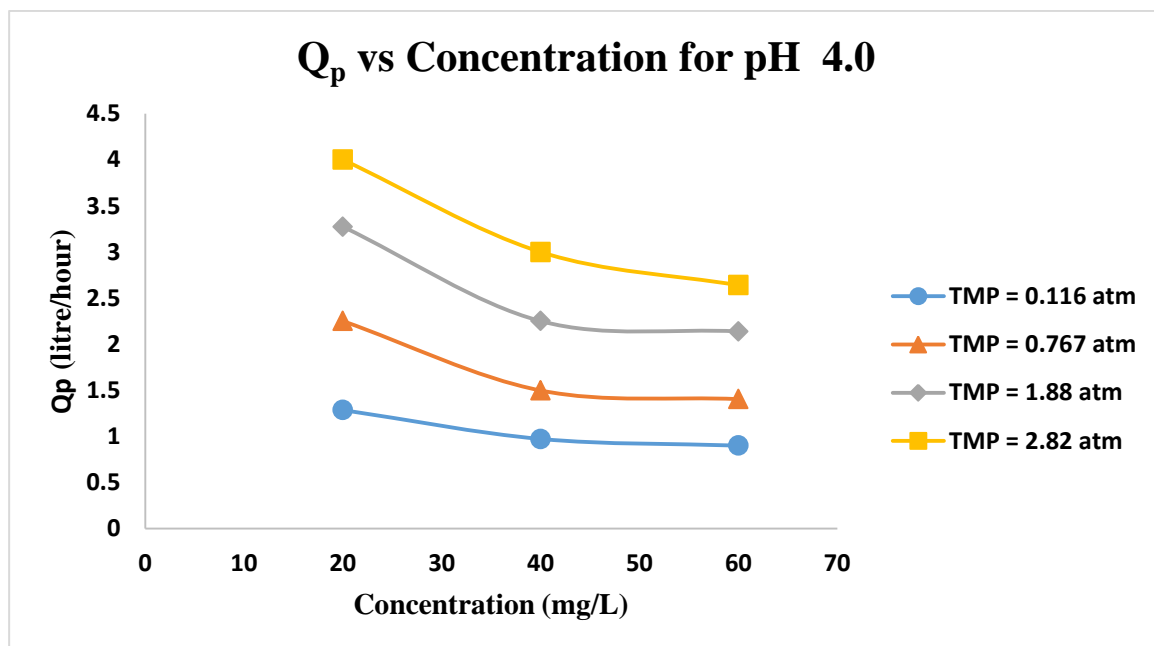


Figure 3: Concentration with respect to Permeate flowrate (Q_p) for pH 4.0

3.1. Effect of concentration:

3.1.1. Effect of concentration on permeate flow:

The effect of concentration with respect to permeate flow is shown in figure 3, 4, 5 & 6 for pH of 4, 6, 7 & 8 respectively. From the graph, it's very clear that permeate flow is decreasing with increasing concentration. Variation in pressure also effects permeate flow (Q_p). In figure. 3, the maximum permeate flowrate occurs at 20 mg/l at the transmembrane pressure (TMP) of 2.82. Similar treatments observed for other pH also. As pressure increases the flow rate also. The same treatments was observed for higher concentration also. The graphs have the same trend for all pH (4, 6, 7 & 8). For pH 7, the flow is maximum as compared to other pH levels. For example at 6mg/L the maximum flow rate is at pH 7 and at a TMP of 2.8 atm. It shows that the pH level of the feed water has little effect on permeate flow. The pressure applied on the membrane is acting across the membrane, which leads to increase in permeate flow.

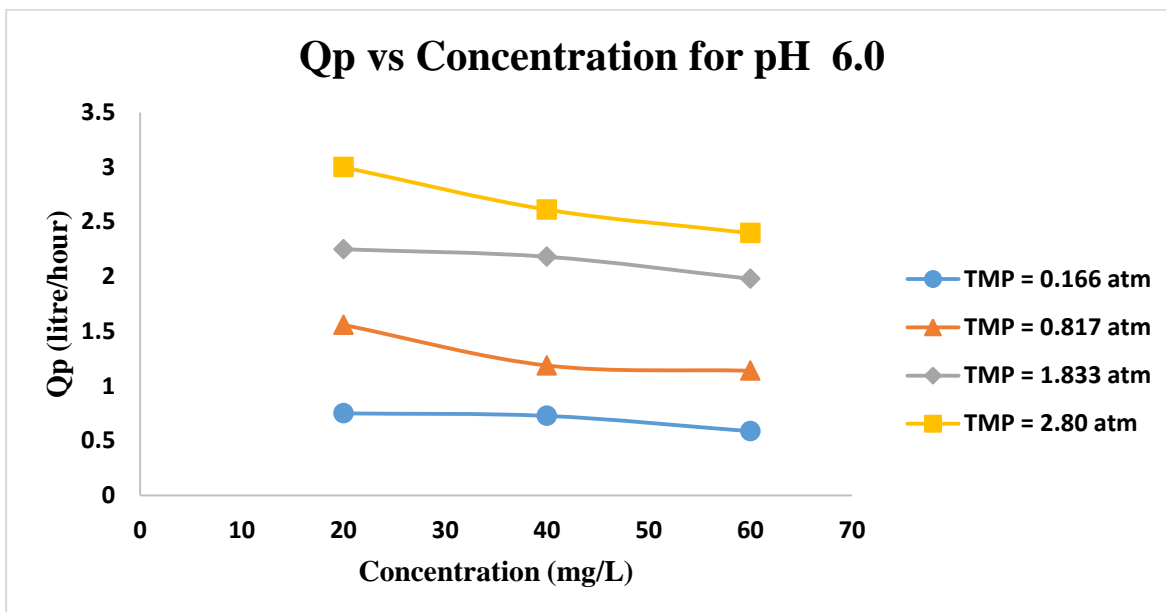


Figure 4: Concentration with respect to Permeate flowrate (Q_p) for pH 6.0

3.1.2. Effect of concentration on observed rejection (R_o):

Observed rejection or separation efficiency is compared in figure 7, which is fairly reasonable. The observed rejection is linearly decreasing with increase in concentration. The observed rejection was calculated by using equation 1. At 60 mg/l for all the pH levels, the observed rejection is low as compared to other concentrations like 20 and 40 mg/l. In pH 7 the observed rejection is high. It is again showing that pH has the little effect on the membrane. At pH 7 also, with respect to concentration the observed rejection is linearly decreasing linearly. It proves the results were in the expected range.

3.1.3. Effect of feed concentration on Permeate Concentration:

The effect of concentration with respect to permeate concentration is shown in figure 8. As shown in the figure, permeate concentration increase as the feed concentration is increasing 20 and 40 mg/l is giving effective results compared to 60 mg/l. pH is having lies effect on permeate concentration. That's shown in the pH 7 the permeate concentration is low. Increasing feed concentrations will affect the work efficiency of the membrane. At the same time the higher concentrations gave lies the 2.9 mg/l of permeate concentration. It shows that in dye treatment nano spiral wound membrane has a significant role.

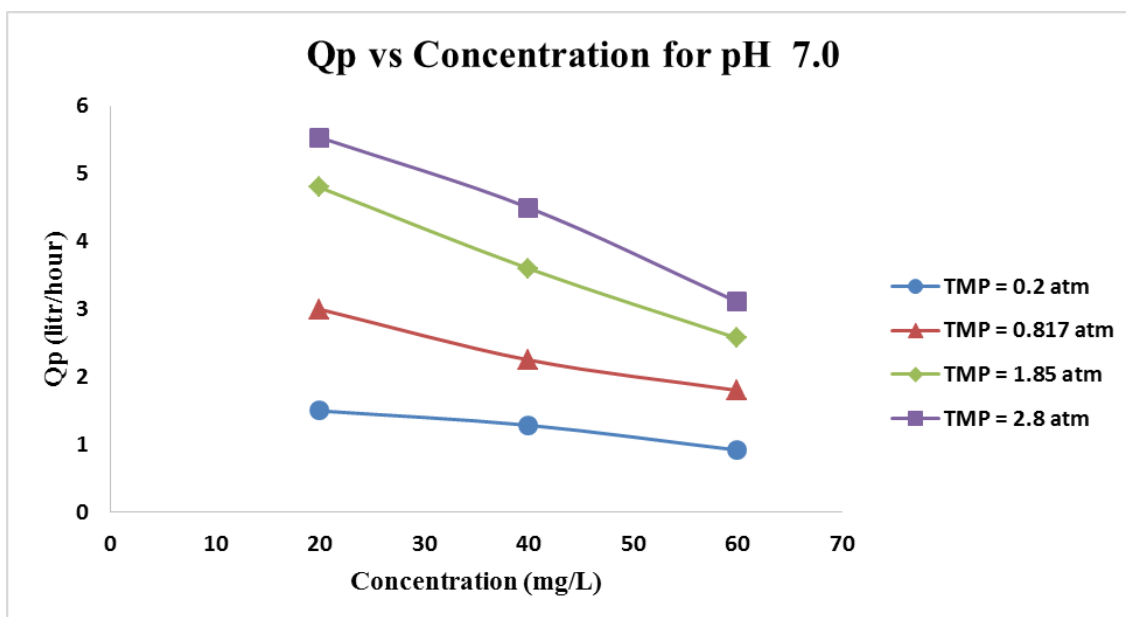


Figure 5: Concentration with respect to Permeate flowrate (Q_p) for pH 7.0

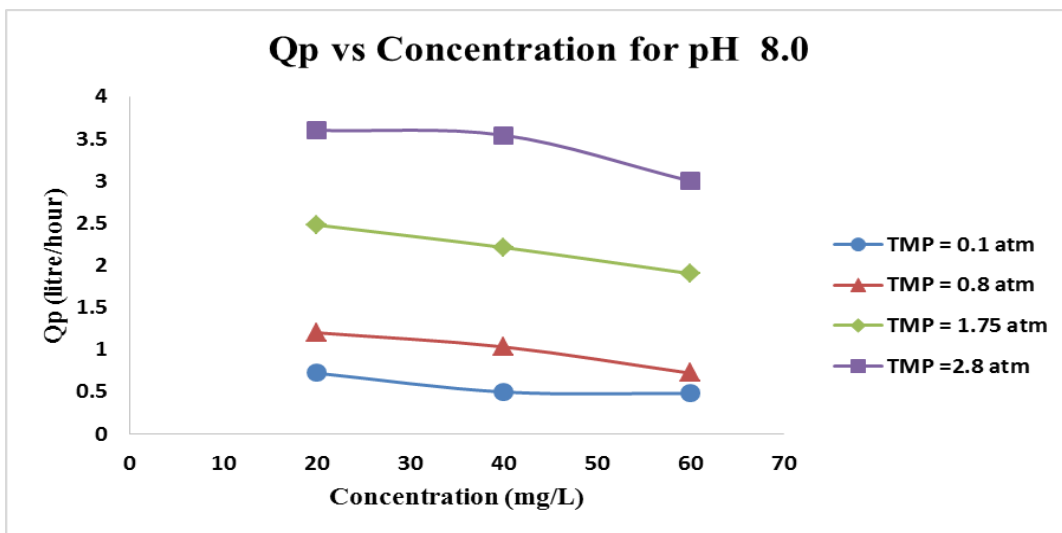


Figure 6: Concentration with respect to Permeate flowrate (Q_p) for pH 8.0

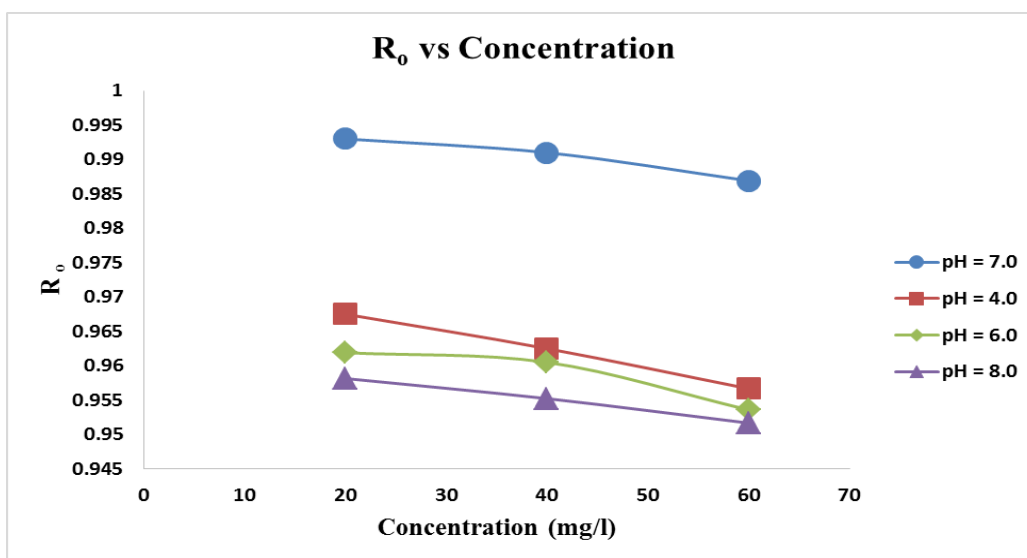


Figure 7: Effect of Concentration in terms of observed rejection (R_o)

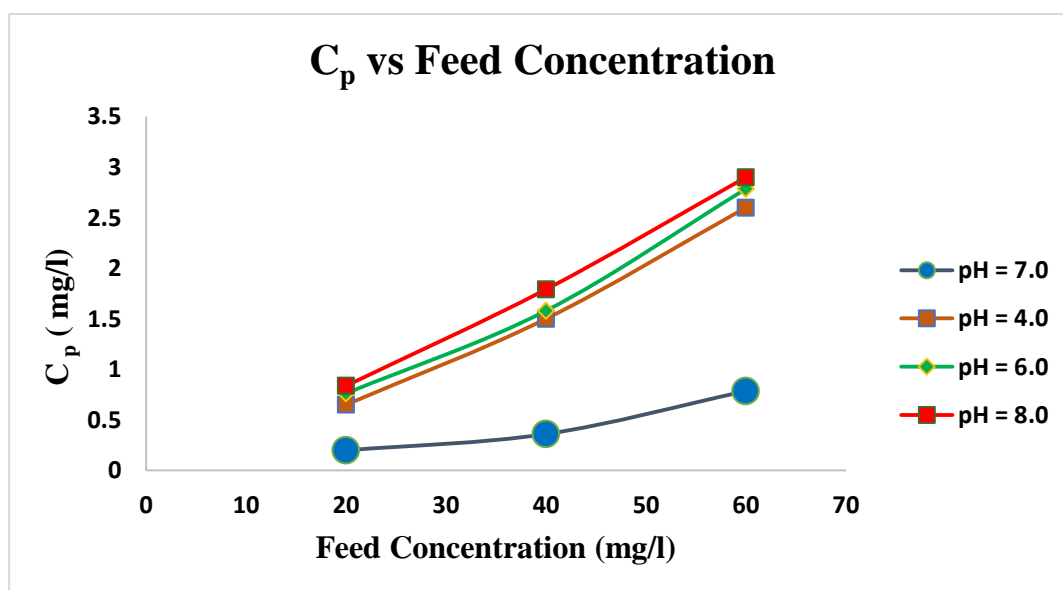


Figure 8: Effect of Concentration towards the Permeate concentration (C_p)

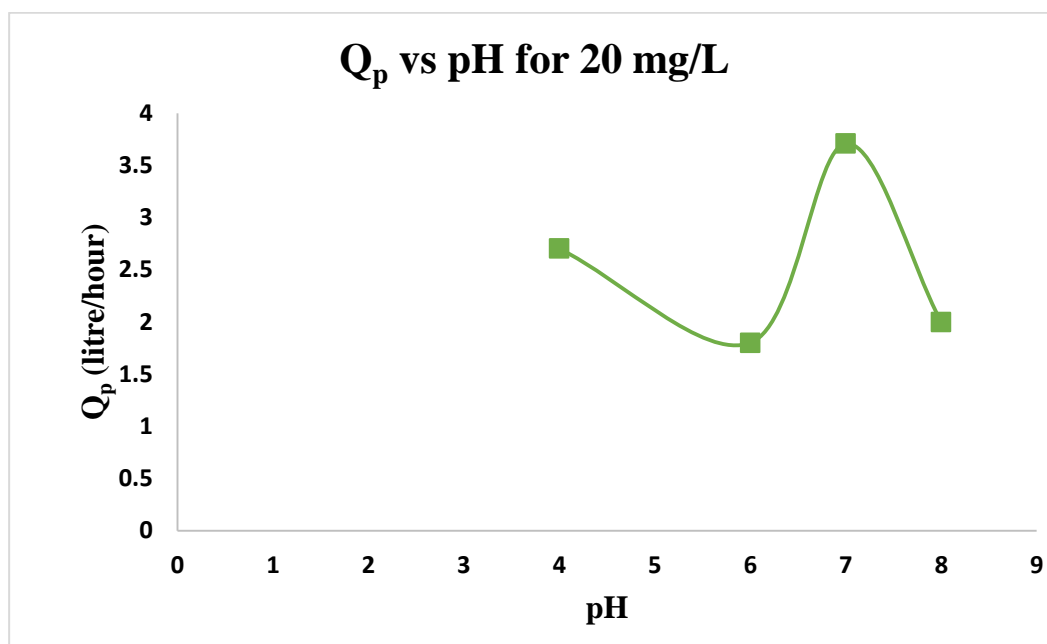


Figure 9: Effect of pH with respect to Permeate Flow (Q_p)

3.2. Effect of pH:

3.2.1. Effect of pH on permeate flow and permeate concentration:

The effect of pH on permeate flow rate is shown in the figure 9, 10 & 11 for different concentrations. MB is a cationic dye. So MB ionizes when it is dissolved in water and it is forming ionic linkages if the water is acid range. Rizzo [26] et. al experimentally proved that pH value is significantly affecting the process. The temperature was maintained at room temperature (25±2°C) throughout the process. Ulson [23] et al mentioned that pH variations may affect the permeate flow of the membrane due to the interactions between the polymer chain. Increase or decrease in polymer chain distance will increase or decrease the permeate flow respectively. At pH 7 the permeate flow rate was maximum. Due to the time variance the flow rate is changing. After compaction and of membrane, a nearly constant permeate flow rate was achieved.

In figure 13, effect of pH with respect to permeate concentrations (C_p) was plotted for various feed concentrations. At low pH of feed solution, the permeate concentration was low and simultaneously increasing with increase of pH. In aqueous solution, basic dyes are developing a net positive charge [34]. Graph shows the C_p for pH 6 is low as compared 8. At pH 4, the permeate concentrations are good as expected.

3.2.2. Effect of pH on observed rejection:

The effect of, with respect to observed rejection for different concentrations pH is shown in figure 13. For various concentrations the observed rejection is showing different values. At the low pH the observed rejection value is high and in basic its low. The results were similar to graph 12.

3.3. Effect of Transmembrane Pressure:

3.3.1. Effect of Transmembrane Pressure on observed rejection (R_o):

The TMP values were varied from 0.05 to 3 atmospheric pressure. In this pressure range, totally four permeate samples were collected and analyzed for various pH. The studied concentrations were 20, 40 and 60 mg/l and pH 4, 6, 7 & 8. The effect of transmembrane pressure with respect to observed rejection for different pH levels was explained in figures 14, 15, 16 & 17.

3.3.2. Effect of Transmembrane Pressure in Permeate Concentration (C_p):

The effect of transmembrane pressure with respect to permeate concentration shown in figures 18, 19, 20 & 21 for various pH levels. The feed concentrations of 20 mg/L and 40 mg/L showed almost similar lower concentrations of of permeate.

The plot is almost a straight line in all cases. This parametric study proves that membrane has a constant output. TMP affects the permeate flow of the membrane. But as per the plotted graph, permeate concentration is almost a constant.

3.3.3. Effect of Transmembrane Pressure in Permeate Flow (Q_p):

The effect of TMP with respect to permeate flow is mentioned for various pH levels in figure 22, 23, 24 & 25. From the figures it can be observed that permeate flow is increasing proportionally with respect to TMP for all concentrations and all pH values. Fersi [28] et al., compared the graph for distilled water and textile effluents with respect to TMP for permeate flux. In that comparison the minimum TMP needed was 2.9 bar to get the permeate flow if its textile effluent. It varies for distilled water which needs less TMP to get the permeate flow and also for the same TMP permeate flux is higher for distilled water. In similar way, in the current experimental study lower concentration of feed having higher permeate flux and vice versa. It can be concluded that membrane permeate flow for NF is giving better results for higher TMP for all the pH. Izadpanah [29] et al., mentioned that permeate flux/flow is changing linearly with TMP. As per the expectation, in this study also permeate flow is changing linearly with respect to TMP and also the permeate flow is decreasing as feed were increasing.

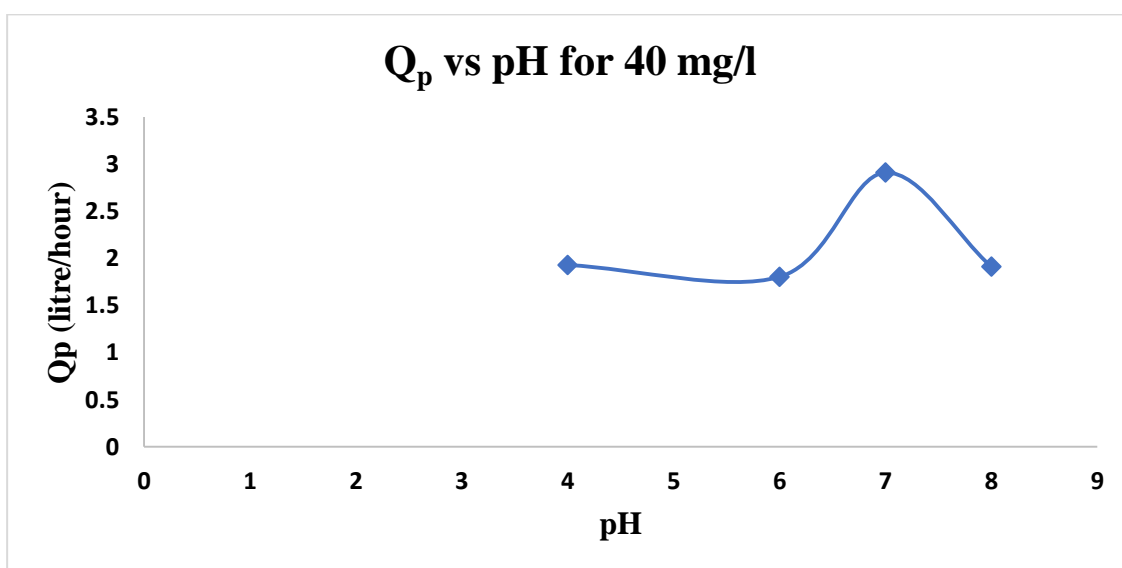


Figure 10: Effect of pH with respect to Permeate flow (Q_p) for 40 mg/L

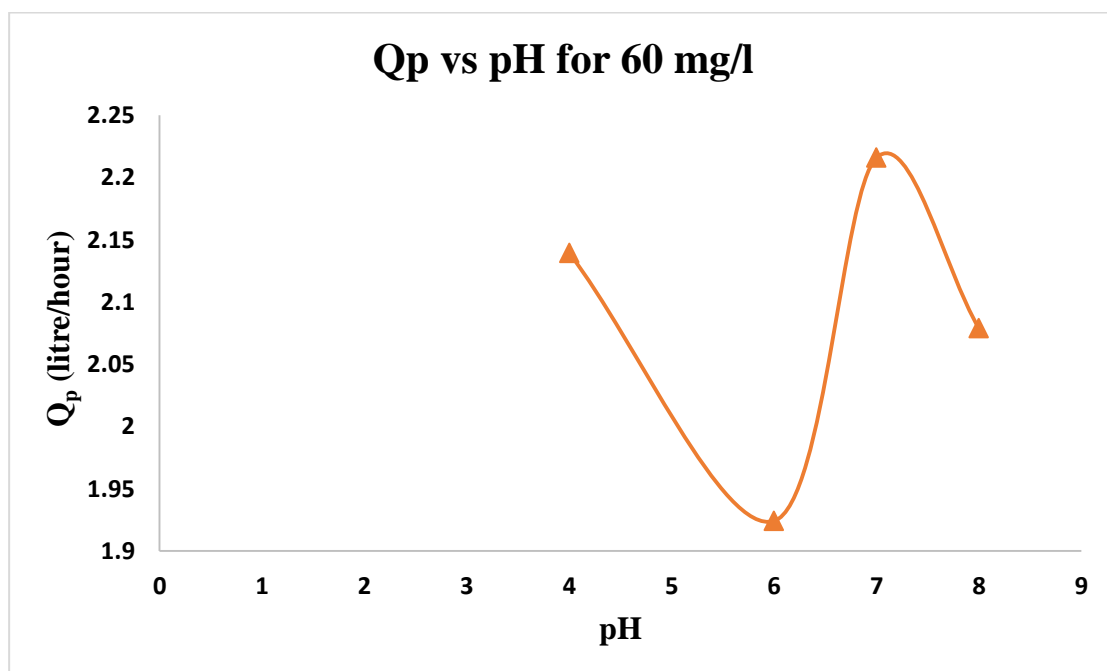


Figure 11: Effect of pH with respect to Permeate flow (Q_p) for 60 mg/L

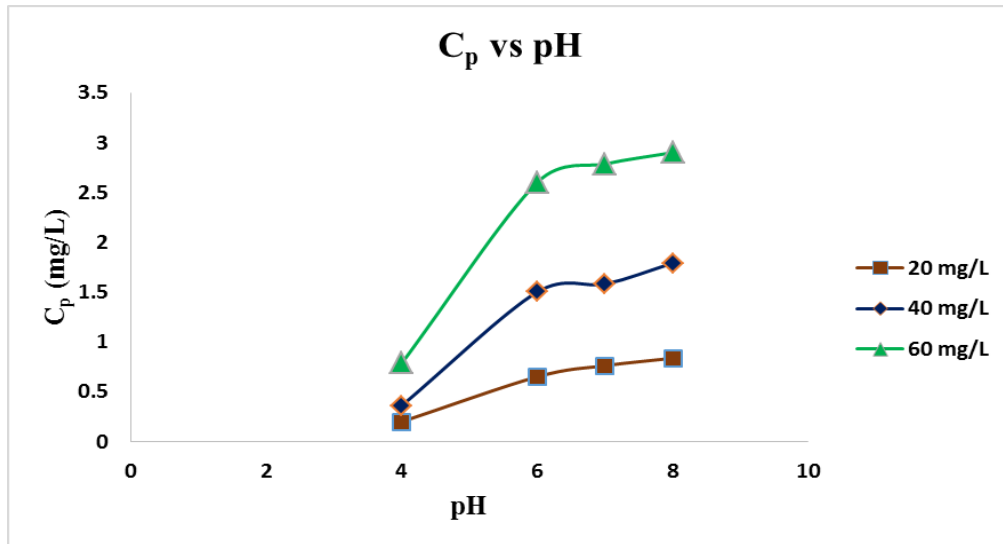


Figure 12: Effect of pH with respect to permeate concentrations for various concentrations (20, 40 & 60 mg/l)

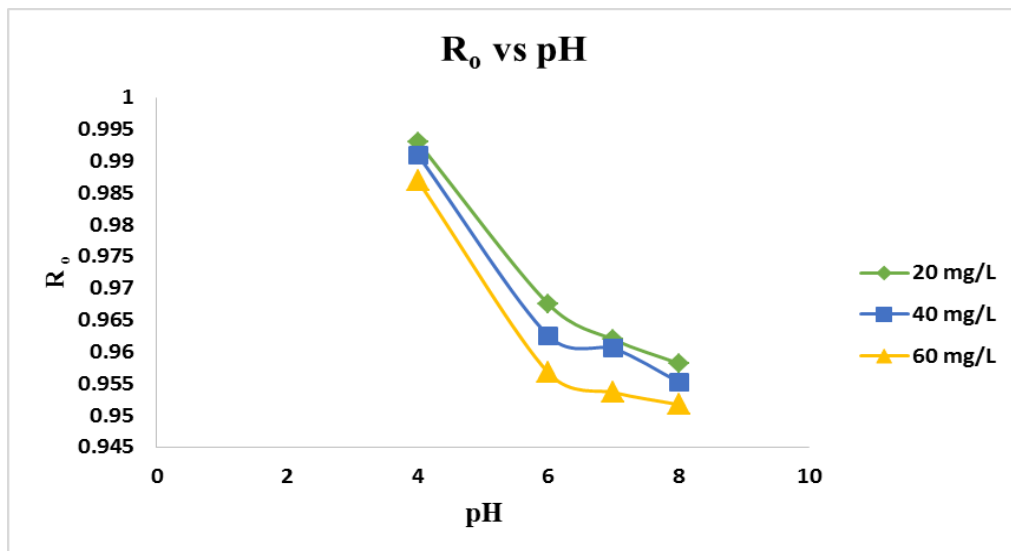


Figure 13: Effect of pH with respect to observed rejection for various feed concentrations (20, 40 & 60 mg/l)

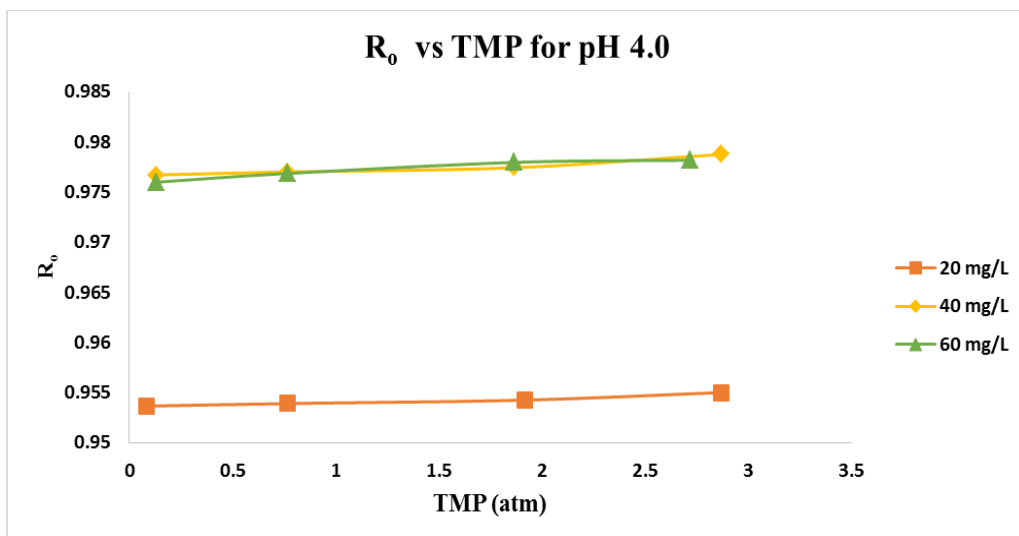


Figure 14: Effect of Transmembrane Pressure with respect to observed rejection at pH 4.0 for various concentrations (20, 40 & 60 mg/l)

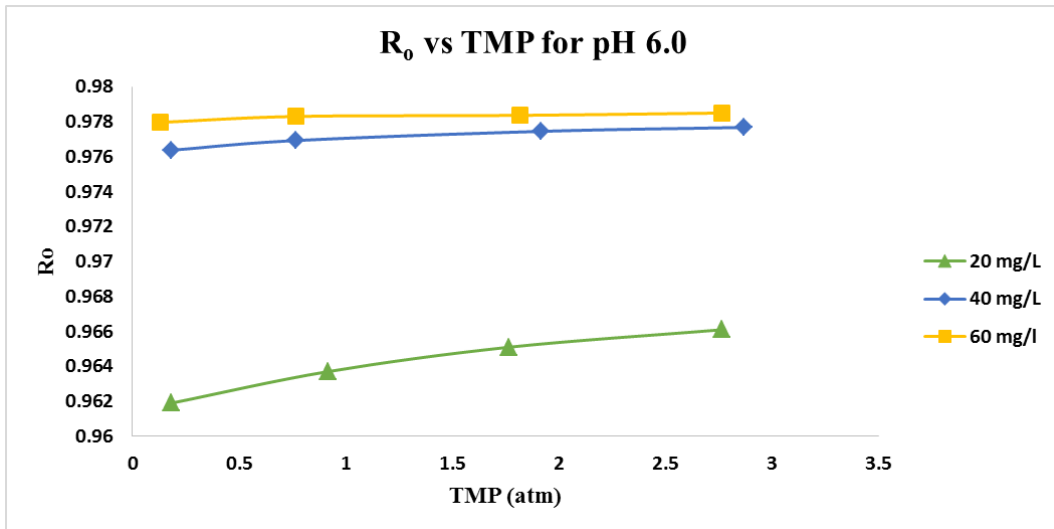


Figure 15: Effect of Transmembrane Pressure with respect to observed rejection at pH 6.0 for various concentrations (20 40 & 60 mg/l)

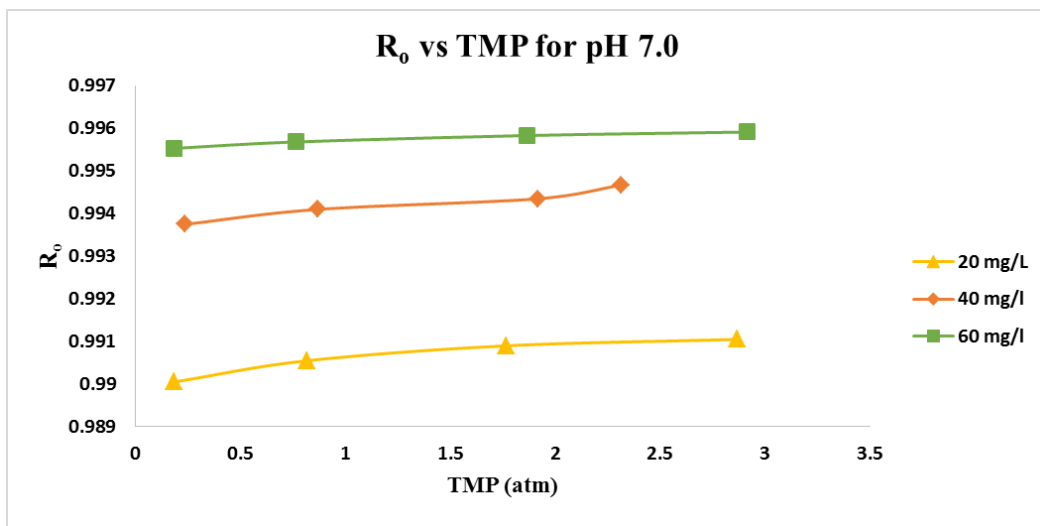


Figure 16: Effect of Transmembrane Pressure with respect to observed rejection at pH 7.0 for various concentrations (20, 40 & 60 mg/l)

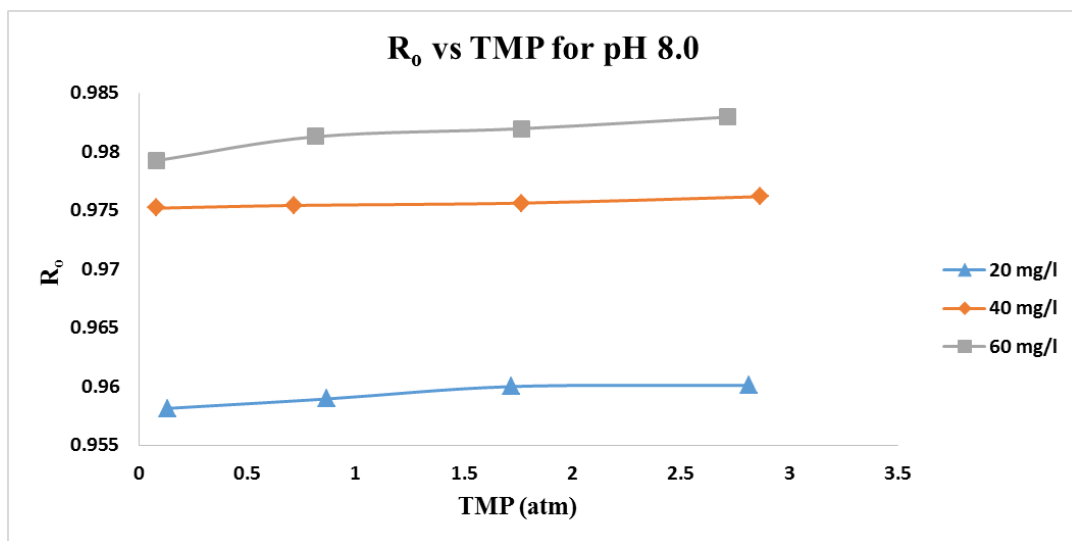


Figure 17: Effect of Transmembrane Pressure with respect to observed rejection at pH 8.0 for various concentrations (20, 40 & 60 mg/l)

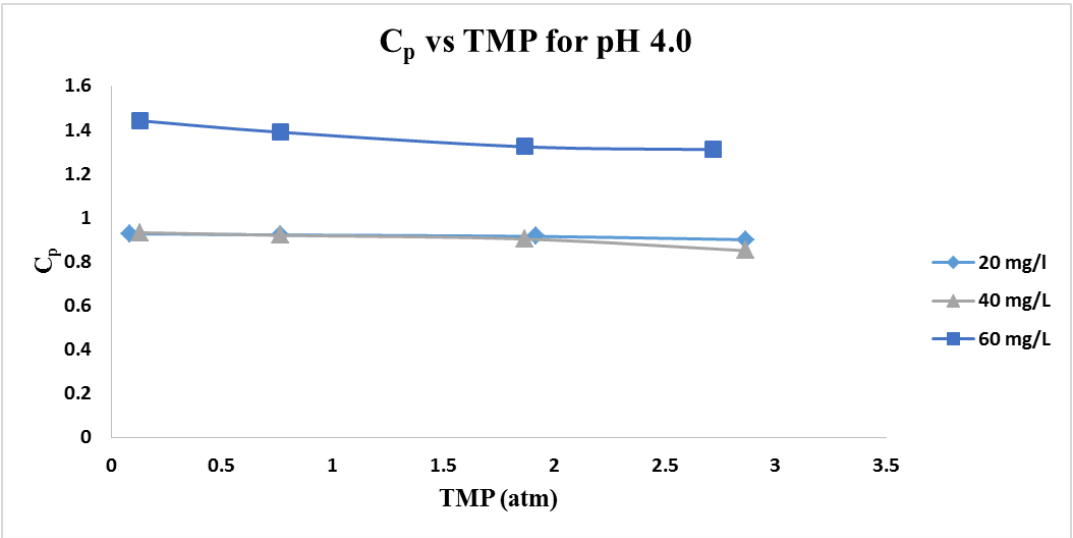


Figure 18: Effect of Transmembrane Pressure with respect to permeate concentration for pH 4.0

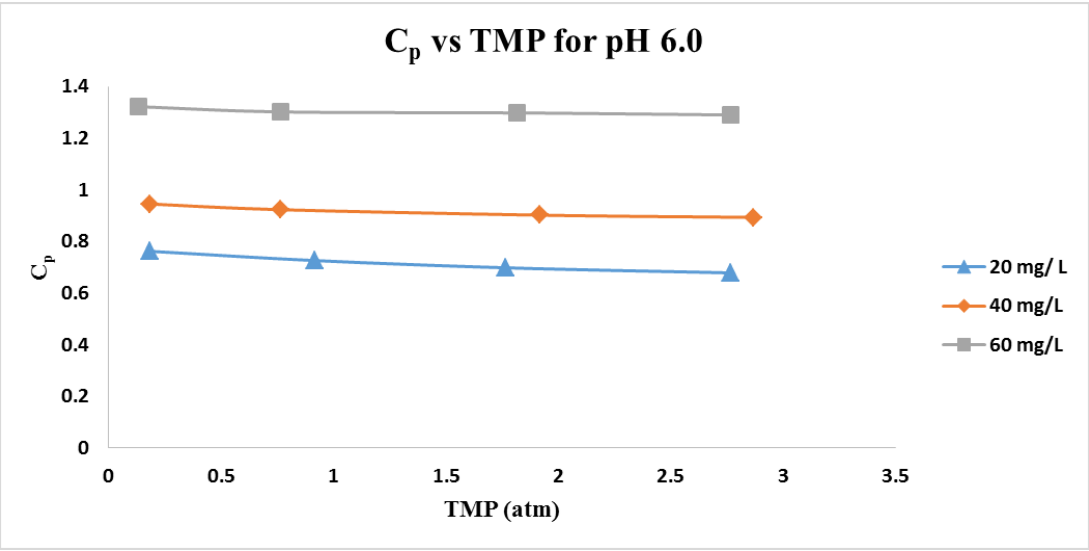


Figure 19: Effect of Transmembrane Pressure with respect to Permeate concentration for pH 6.0

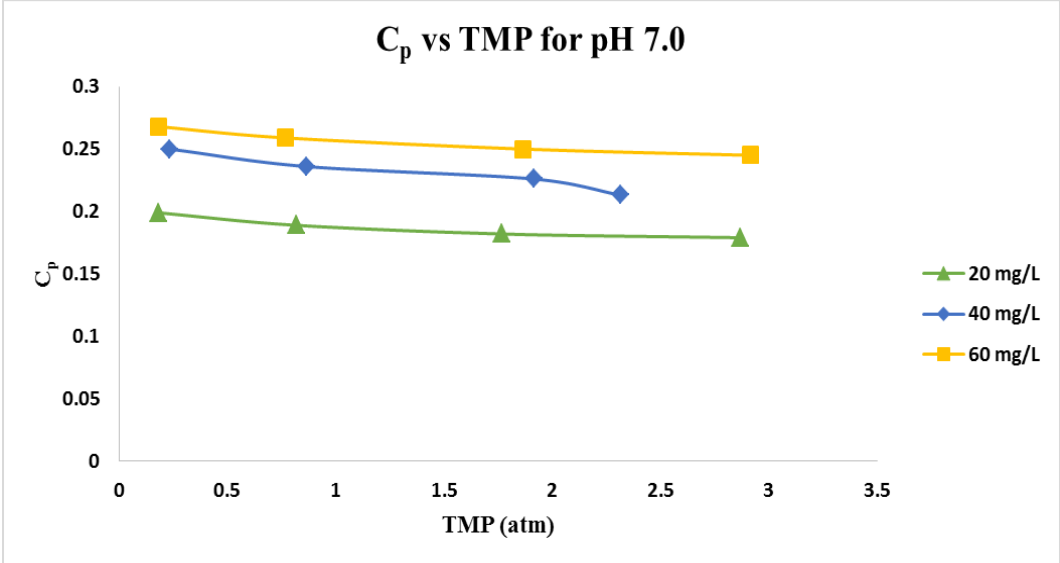


Figure 20: Effect of Transmembrane Pressure with respect to Permeate concentration for pH 7.0

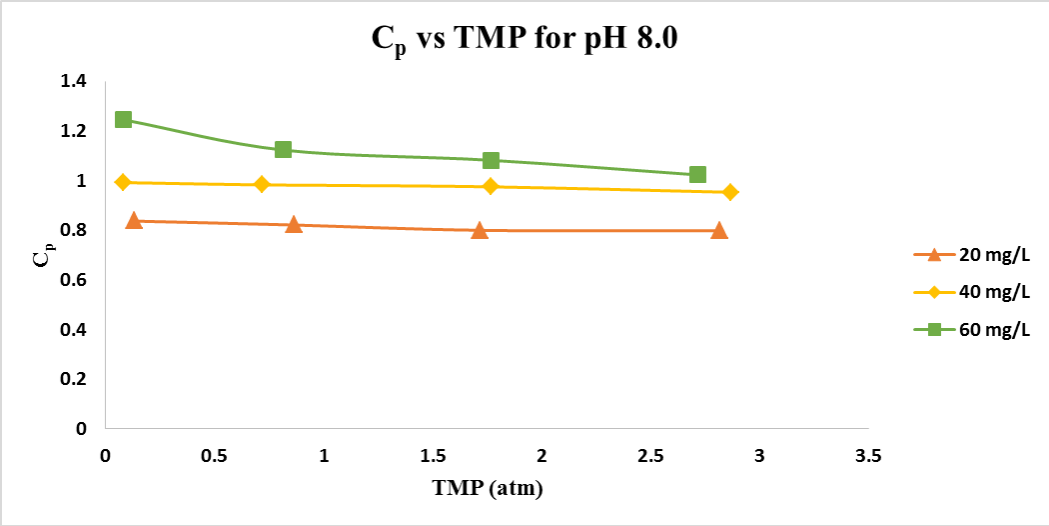


Figure 21: Effect of Transmembrane Pressure with respect to Permeate concentration for pH 8.0

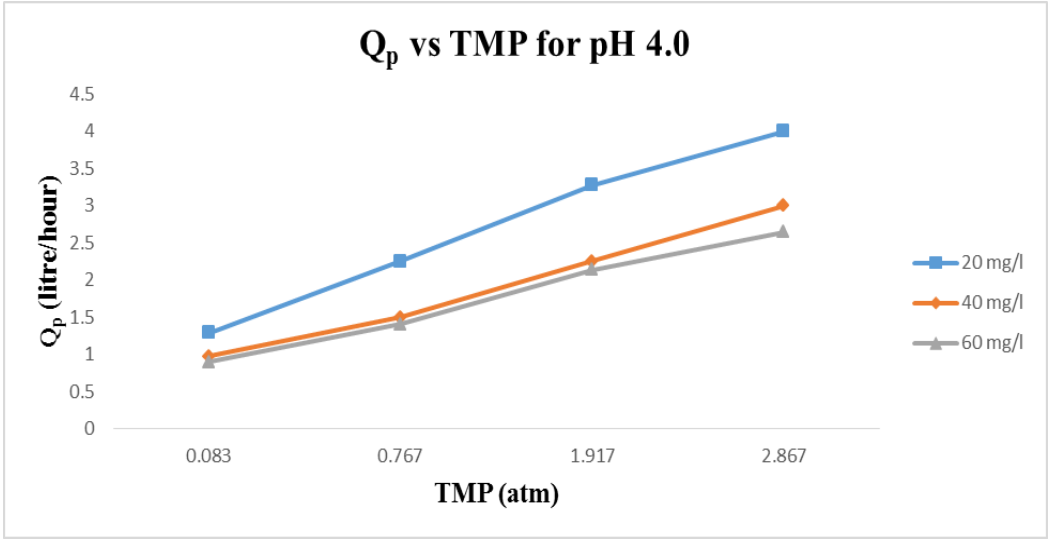


Figure 22: Effect of Transmembrane Pressure with respect to Permeate flowrate for pH 4.0

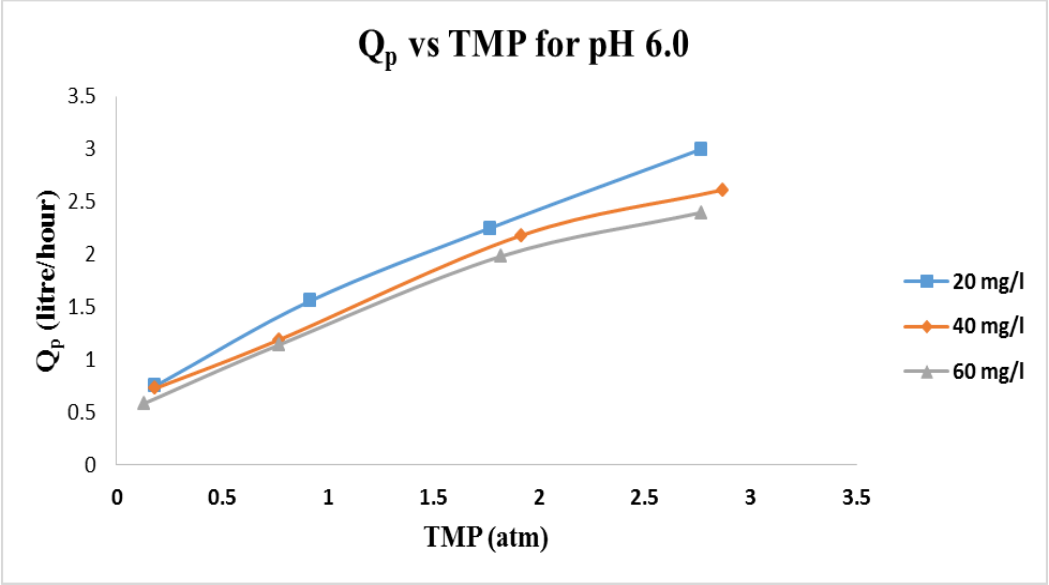


Figure 23: Effect of Transmembrane Pressure with respect to Permeate flow for pH 6.0

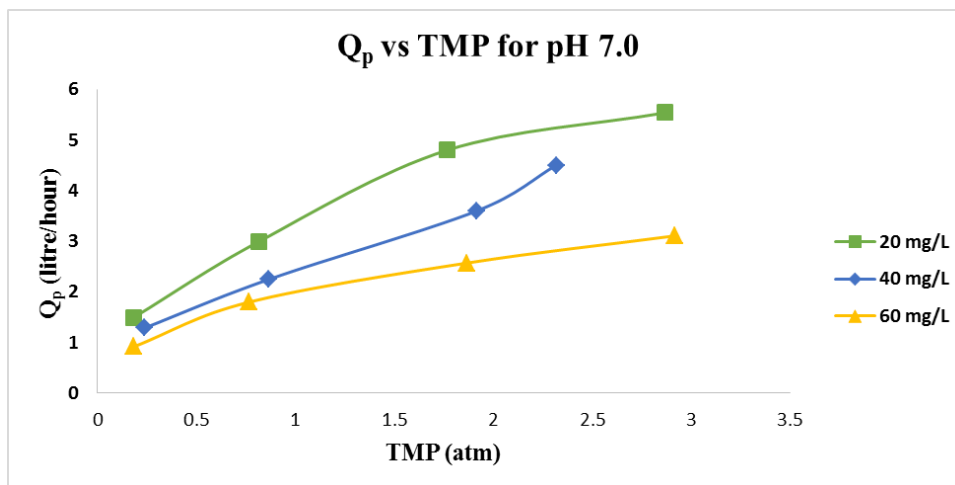


Figure 24: Effect of Transmembrane Pressure with respect to Permeate flow for pH 7.0

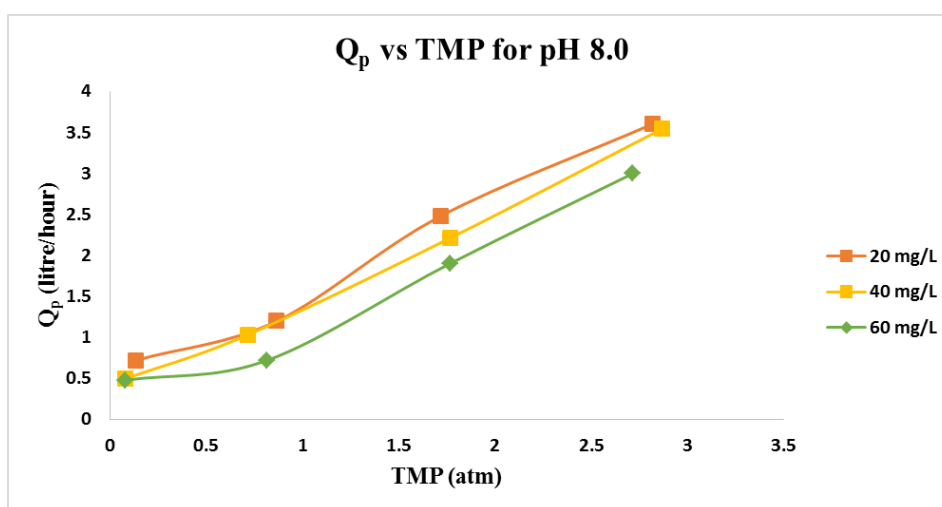


Figure 25: Effect of Transmembrane Pressure with respect to Permeate flow for pH 8.0

S K Gupta et al (2005) concluded that a good membrane always keeps the solute permeability constant with respect to other parameters like feed pressure, flow rate and concentration [25]. From the calculated values by using SK model, the solute permeability values were constant.

4. CONCLUSIONS

HPA 600 nanofiltration membranes showed high rejection for MB. Suitable pH values are neutral, acids and base. According to the experimental datas, pH 4 is showing good results compare to other pH levels. MB is a basic dye. So maintaining the pH level in acidic region will lead to its efficient removal. The results showed linear relation between transmembrane pressure and permeate flow. Separation performance of a MB (flux and rejection) was investigated.

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