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# Application of Box Wilson experimental design method for removal of acid red 95 using ultrafiltration membrane

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**Abstract.** The applicability of the ultrafiltration process for color removal from dye-containing water has been examined in this study. The optimization of major process variables, such as dye concentration, chitosan concentration and transmembrane pressure on permeate flux and color removal efficiency was investigated. To find the most appropriate results for the experiment, the Box-Wilson experimental design method was employed. The results were correlated by a response function and the coefficients were determined by regression analysis. Permeate flux variation and color removal efficiency determined from the response functions were in good agreement with the experimental results. The optimum conditions of chitosan concentration, dye concentration and pressure were 50 mg/l, 50 mg/l and 3 bars, respectively for the highest permeate flux. On the other hand, optimum conditions for color removal efficiency were determined as 50 mg/l of dye concentration, 50 mg/l of chitosan concentration and 1 bar of pressure.

**Keywords:** Box-Wilson experimental design; chitosan; decolorization; dye; ultrafiltration

## 1. Introduction

Color is an important part of the human world. A few decades earlier, the selection, application and use of dyes were not seriously considered regarding their environmental impacts, even the chemical compositions of dyes were unknown. The textile industry is the largest consumer of dyestuffs (Mughal *et al*. 2013). So, the disposal of textile wastewater is currently a major problem in Turkey. Textile industries produce a lot of wastewater, which contains a number of contaminants, including acidic or caustic dissolved solids, toxic compounds and also dyes (Sakkayawong 2005).

In general, dyes are difficult to remove because they are stable to light and oxidizing agents and with low biodegradability (Buscio *et al*. 2016). The most used technologies to treat wastewater containing dyes are based on physical-chemical or/and biological processes. Coagulation and sedimentation processes are known to be effective in eliminating the colors of insoluble dyes such as disperse ones. However, these are not conditions for soluble dyes including reactive dyes (Robinson *et al*. 2001). The well-known conventional coagulants such as alum, polyaluminum chloride, iron (II) sulfate and lime are widely used in the textile wastewater treatment. More than 90% of color removal from acid dyes could be achieved by adding activated carbon. However, it known to be insignificant for base and direct dyes (Shin *et al*. 2012). Chitosan has been used in the dye complexation using adsorption processes or coagulation. It is recommended for the treatment of azo-dyes because of its excellent properties as an environmentally friendly coagulant that can be obtained from renewable resources (Akdemir 2012). Although dyes in wastewater could be effectively destroyed by advanced chemical oxidation (Kang *et al*. 2002), the treatment cost is high.

Biological treatment processes are frequently used to treat textile effluents. These processes are generally efficient for biochemical oxygen demand ($BOD_5$) and suspended solids (SS) removal. However, they are largely ineffective for removing color which was visible even at low concentrations (Lazaridis *et al*. 2003).

Membrane technology has shown great potential to be applied to treat different types of dyes from textile wastewater. It also permits the reuse of both auxiliary chemicals and some concentrated dyes and produce a high quality water that can be reused in new textile processes (Buscio *et al*. 2015). In general, reverse osmosis and nanofiltration membranes are the most studied materials in the treatment of wastewater containing dyes. The main limitation of membrane processes such as reverse osmosis and nanofiltration is the reduction of permeate flux, which is caused by the accumulation of particles on the membrane surface (Buscio *et al*. 2016). Ultrafiltration membranes exhibit low fouling and high efficiency to separate chemicals with high molecular weight or insoluble dyes such as disperse (Kaykioğlu *et al*. 2017).

It should be noted that a significant portion of the reported work on dye removal has been subjected to the traditional test method, which allows one of the independent parameters to change, while the others remain constant. This classical or traditional method of experimentation requires a lot of experimental work that takes time, does not consider the working effect between

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experimental parameters and leads to low fertility in optimization. These limitations of the classical method can be avoided by applying a response surface methodology that includes statistical design of experiments in which all factors are diversified together during a series of experimental studies (Khayet *et al*. 2011). The Box-Wilson experimental design is a response surface methodology used for evaluation of a dependent variable as functions of independent variables (Bali 2004).

Box Wilson experimental design method has been used in some studies for the treatment of textile wastewater. Bali (2004) used this method for investigation of the ability of the oxidative UV/$H_2O_2$ process to decolorize aqueous solutions of three azo dyes. Dyestuff, $H_2O_2$ concentrations and reaction time were considered as independent variables and color and total organic carbon removal efficiency was considered as dependent variable in the Box-Wilson statistical design method in his study. In another study, Box-Wilson method was also used for the solar photocatalytic degradation of textile dyestuff with Fe(III)/$H_2O_2$/solar UV process to optimize the wastewater flowrate, oxidant and catalyst concentrations as significant factors for maximum decolorization and organic matter removal (Parıltı and Akten 2010).

In the content of this study, the treatability of dye-containing wastewaters using an ultrafiltration membrane was investigated. The dyestuff (Acid Red 95) was used in the experimental studies since it is a widely used textile dyestuff in Turkish textile industry. Chitosan, a biological cationic polymer, has also been used to increase color removal efficiency. Experiments carried out within the scope of the study were designed according to the Box-Wilson experimental design method. Optimal dye concentration, chitosan concentration and transmembrane pressure parameters maximizing permeate flux and color removal efficiency were determined.

## 2. Materials and methods

### *2.1 Dye and chitosan*

The dyestuff was obtained from the EKOTEN Textile Industry in Izmir, Turkey. Characteristics of the Acid Red 95 are presented in Table 1.

Chitosan was taken from Sigma-Aldrich (product number of 419419) with high molecular weight and the chemical structure of chitosan is depicted in Figure 1.

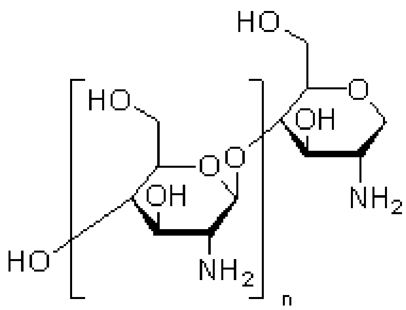
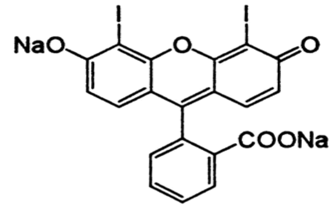


Fig. 1 The chemical structure of chitosan (García *et al*. 2014)

Table 1 Characteristics of the Acid Red 95 (AR95) (LGC Standards 2018)

| Acid Red 95 | |
|---|---|
| Commercial name | Superfix Red 195 |
| Classification | Single azo class |
| Molecular formula  | |
| λmax (nm) | 412 |
| Molecular weight (g/mol) | 628.1 |

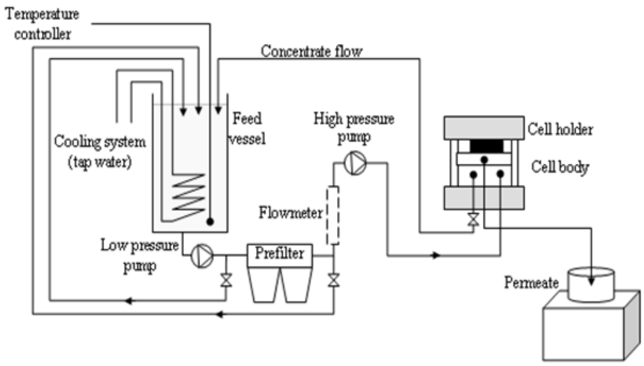



Fig. 2 Schematic flow diagram of the experimental set-up

### *2.2 Experimental system*

The membrane experiments were carried out in a laboratory-scale cross flow membrane system. The feed stream was pumped from the feed vessel to the feed inlet of cell body. A portion of the solution permeated through the membrane and flowed into the permeate carrier. The concentrate stream flowed back to the feed vessel. A cooling system with tap water in the feed vessel was used in all filtration experiments to keep the temperature at 22-24°C. 5 μm cartridge filter was used before ultrafiltration membrane as prefilter. Osmonics Sepa CF II membrane system described in detail in our previous works (Akdemir and Ozer 2008, Akdemir and Ozer 2009) has also been used in this study. At the beginning of the experiments, chitosan was added in the determined quantities to water containing different concentrations of dye and this sample was filled into feed vessel of experimental set-up. Permeate from membrane was collected in the permeate collection vessel. The pressure and the recycle flow rate were controlled by regulation valves. During the filtration experiments, weight of permeate in permeate carrier was continuously monitored.

Schematic flow diagram of experimental set-up is given in Figure 2. The ultrafilic MW membrane with a molecular weight cut-off of 100 kDa were used in this study. Membrane area was 0.0155 $m^2$ for all membrane experiments.

Table 2 Experimental conditions according to a Box-Wilson statistical design

| | Dye conc. (mg/l) | Chitosan conc.(mg/l) | Pressure (bar) |
|---|---|---|---|
| Axial point | | | |
| A1 | 250 | 100 | 2 |
| A2 | 50 | 100 | 2 |
| A3 | 150 | 150 | 2 |
| A4 | 150 | 50 | 2 |
| A5 | 150 | 100 | 3 |
| A6 | 150 | 100 | 1 |
| Factorial points | | | |
| F1 | 92 | 129 | 2.6 |
| F2 | 208 | 71 | 2.6 |
| F3 | 208 | 129 | 1.4 |
| F4 | 208 | 71 | 1.4 |
| F5 | 92 | 71 | 2.6 |
| F6 | 92 | 129 | 1.4 |
| F7 | 208 | 129 | 2.6 |
| F8 | 92 | 71 | 1.4 |
| Center point | | | |
| C | 150 | 100 | 2 |

### *2.3 Box-Wilson experimental design*

In the present study, a Box-Wilson experimental design was employed to evaluate the combined effect of three independent variables; dye concentration, chitosan concentration, pressure and designated as $X_1$, $X_2$ and $X_3$, respectively, on the ultrafiltration of dye wastewater as expressed by the permeate flux and color removal percentages. Dye concentration (DC, $X_1$) was varied between 50-250 mg/l, chitosan concentration (CC, $X_2$) was varied between 50-150 mg/l and pressure (P, $X_3$) was varied between 1 and 3 bar. The dye and chitosan concentrations were determined according to studies done with different dyes in the literature (Szygula *et al*. 2008, Akdemir 2012). For all experiments, flow rate was taken as 200 l/h and filtration time was taken as 120 minutes. In the preliminary experiments performed in the laboratory, the highest permeate flux and color removal efficiency value was obtained at 200 l/h flowrate and 120 minutes filtration. For this reason, all experiments were carried out at this flow rate and time. The minimum and maximum range of variables was investigated and experimental conditions determined by the Box-Wilson statistical design were presented in Table 2. The experiments consisted of six axial (A), eight factorial (F) and center points (C). The center point was repeated four times. Computation was carried out using multiple regression analysis using the least squares method (Parıltı and Akten 2010).

The following response function was used in correlating the permeate flux and color removal efficiency with independent parameters ($X_1$, $X_2$ and $X_3$).

Table 3 Observed and predicted permeate flux and color removal efficiency

| Experiment number | Permeate flux (l/m$^2$.h) | | Color removal efficiency | |
|---|---|---|---|---|
| | Observed | Predicted | Observed | Predicted |
| A1 | 14.40 | 16.10 | 0.93 | 0.94 |
| A2 | 20.13 | 18.79 | 0.85 | 0.84 |
| A3 | 14.63 | 15.23 | 0.85 | 0.86 |
| A4 | 15.87 | 15.62 | 0.85 | 0.84 |
| A5 | 20.67 | 22.22 | 0.79 | 0.81 |
| A6 | 19.74 | 18.55 | 0.83 | 0.81 |
| F1 | 19.82 | 19.64 | 0.84 | 0.85 |
| F2 | 19.43 | 18.30 | 0.91 | 0.93 |
| F3 | 15.87 | 15.56 | 0.88 | 0.90 |
| F4 | 16.26 | 16.18 | 0.91 | 0.90 |
| F5 | 19.43 | 19.48 | 0.85 | 0.83 |
| F6 | 16.65 | 17.51 | 0.90 | 0.88 |
| F7 | 19.59 | 17.96 | 0.83 | 0.79 |
| F8 | 16.26 | 17.62 | 0.67 | 0.71 |
| C | 14.32 | 14.36 | 0.88 | 0.89 |

$$Y = b_0 + b_1 X_1 + b_2 X_2 + b_3 X_3 + b_{12} X_1 X_2 + b_{13} X_1 X_3 + b_{23} X_2 X_3 + b_{11} x_1{}^2 + b_{22} x_2{}^2 + b_{33} x_3{}^2 \quad (1)$$

The Statistica 5.0 computer program was employed for the determination of the coefficients of Eq. (1) by regression analysis of the experimental data where Y is predicted yield; $b_o$ is constant; $b_1$, $b_2$ and $b_3$ are linear coefficients; $b_{12}$, $b_{13}$, $b_{23}$ are cross product coefficients; and $b_{11}$, $b_{22}$, $b_{33}$ are quadratic coefficients.

## 3. Results and discussion

### *3.1 Box-Wilson experimental design method results*

The permeate fluxes and color removal efficiencies obtained from the experiments are summarized in Table 3. The observed permeate fluxes varied between 14.32 and 20.67 L/m$^2$.h and color removal efficiencies varied between 67 % and 93 %. The observed permeate fluxes and color removal efficiencies were compared with the predicted ones obtained from the response function.

The results attained by the ultrafiltration experiments that were performed under 120 minutes of filtration time were used to determine the coefficients of the response functions and the coefficients were further used in calculating predicted values of permeate flux and color removal efficiencies (Eqs. 2 and 3).

The factors in front of the model terms indicate the intensity and direction of the influence of the independent variable. A positive effect of a factor means that the response is improved when the factor level increases and a negative effect of the factor reveal that the response is inhibited when the factor level increases. On the basis of the coefficients given in Eq. (2), the variable of pressure ($X_3$)

exhibited the highest positive influence on permeate flux. The negative effect of dye concentration ($X_1$) is also shown from this equation. The effect of chitosan concentration ($X_2$) on permeate flux is quite low. According to Eq.(3), it can be stated that the color removal efficiency increases with decreasing concentration of dye and chitosan and pressure.

The coefficients were used in calculating predicted values of permeate flux and color removal efficiencies. The correlation coefficients ($R^2$) between the observed and predicted values were 0.90 and 0.94 for permeate flux and color removal, respectively. These results indicated excellent agreements between the observed and predicted values. The effects of the operating variables on the permeate flux and color removal performance of the system were determined by obtaining the projections of the response functions on certain planes of the known parameter values.

$$Permeate\ flux = 77.76547 - 0.1047(DC) - 0.009071(CC) + 0.70089(P) - 0.000075(DC*CC) + 0.000409(DC*P) + 0.0000817(CC*P) + 0.000309(DC)^2 + 0.000429(CC)^2 + 0.002411(P)^2 \quad (2)$$

$$Color\ removal = -1.3289422 - 0.0047182(DC) - 0.00132597(CC) - 0.0152732(P) - 0.0000245(DC*CC) - 0.000126(DC*P) - 0.0000431(CC*P) + 0.0000004(DC)^2 - 0.0000142(CC)^2 - 0.0000302(P)^2 \quad (3)$$

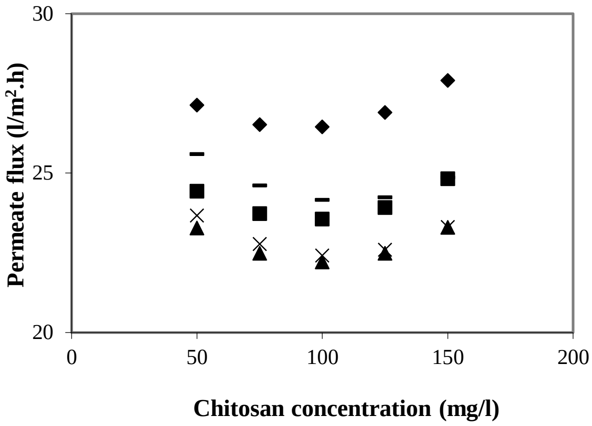



Fig. 3 Variation of permeate flux with chitosan concentration as a function of dye concentration at 2 bar pressure. Dye concentration: (♦) 50 mg/l, (−) 100 mg/l, (■) 150 mg/l, (x) 200 mg/l, (▲) 250 mg/l

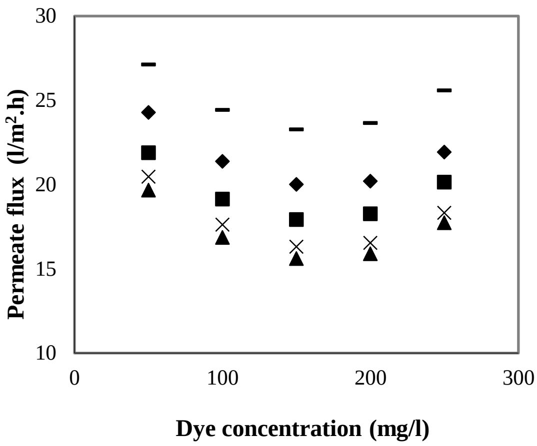



Fig. 4 Variation of permeate flux with dye concentration as a function of pressure at 150 mg/l chitosan concentration. Pressure: (−) 3 bar, (♦) 2.5 bar, (■) 2 bar, (x) 1.5 bar, (▲) 1 bar

### *3.2 Permeate flux*

In the first stage of the experimental studies carried out in the scope of the study, the effects of dye concentration, chitosan concentration and transmembrane pressure on permeate flux were investigated.

In the first experiment where the pressure was kept constant at 2 bar, the flux change at different chitosan and dye concentrations was examined and the results are shown graphically in Figure 3. The highest flux values were obtained for the dye concentration of 50 mg/l. At a concentration of 50 mg/l of chitosan, the flux value initially decreased to 26.4 l/m$^2$.h at a concentration of 100 mg/l of chitosan. It was observed that the value of flux increased with the increase of chitosan. The flux measured at 50 mg/l dye and 150 mg/l chitosan concentrations is 27.1 l/m$^2$.h.

In another experimental study, the effect of increasing dye concentration and pressure was observed by keeping the chitosan concentration constant. Chitosan concentration of 150 mg/l was kept constant, the variation in permeate flux with different dye concentration and pressure values shown in Figure 4. When the graph was examined, it was observed that the increase in dye concentration resulted in a decrease in the flux of the resultant but a slight increase in the amount of flux when the dye concentration exceeded 200 mg/l. The increase in pressure also increases the permeate flux. The lowest flux values were obtained for 1 bar pressure while the highest flux values were observed at pressure value of 3 bar. The operation conditions for maximum permeate flux (27.9 l/m$^2$.h) was 3 bar pressure, 50 mg/l dye concentration and 150 mg/l chitosan concentration.

In the final stage of experimental studies to observe the pemeate flux, the dye concentration was kept constant and the effect of the change in the chitosan concentration and pressure on the permeate flux was investigated. The results for the 50 mg/l dye concentration are plotted in Figure 5. It can be seen from figure that the amount of flux decreased with increasing chitosan concentration, which is valid for all pressure values. However, after 100 mg/l of chitosan, the

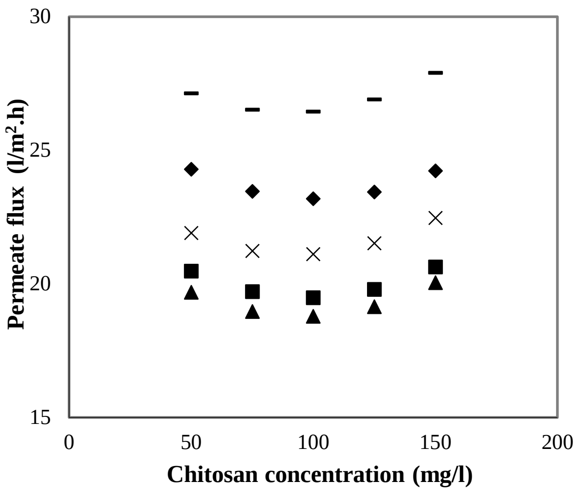



Fig. 5 Variation of permeate flux with chitosan concentration as a function of pressure at 50 mg/l dye concentration. Pressure: (−) 3 bar, (♦) 2.5 bar, (x) 2 bar, (■) 1.5 bar, (▲) 1 bar

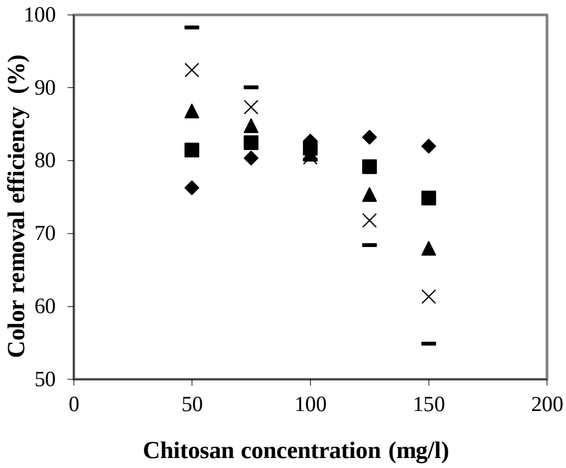



Fig. 6 Variation of color removal efficiency with chitosan concentration as a function of dye concentration at 2 bar pressure. Dye concentration: (♦) 50 mg/l, (■) 100 mg/l, (▲) 150 mg/l, (x) 200 mg/l, (−) 250 mg/l

amount of flux increased by 0.5 $l/m^2.h$, but this is negligible. As a result, increasing chitosan concentration does not affect the filtrate value to a large extent.

### *3.2 Color removal*

In the second stage of experimental studies, the effects of dye concentration, chitosan concentration and pressure on the color removal efficiencies have been investigated. First of all, the variation of the chitosan and dye concentrations on the color removal efficiency at constant pressure was investigated and the results are given in Figure 6. When the effect of increase in chitosan concentration on color removal efficeiny was examined, the color removal efficiency is found to be 98% at 50 mg/l of chitosan concentration for 250 mg/l of dye concentration. If the concentration of chitosan is increased by maintaining the same value of pressure (2 bar) at the highest dye concentration in operation (250 mg/l), the decolorization efficiency appears to decrease. From here it can be said that when the concentration of chitosan in the wastewater at constant pressure is increased, the color removal efficiency decreases proportionally for high dye concentration. There is no positive effect on the color removal efficiency of chitosan. At low dye concentrations (50 mg/l), the increase in chitosan concentration is not very effective in color removal efficiency. The removal efficiency for the 50 mg/l chitosan concentration was 76%, while the efficiency for the 150 mg/l chitosan increased to 80%. As a result there is no need to add chitosan to keep the color removal efficiency at a high level.

When the results obtained in the experiments are plotted, the relationship between the color removal efficiency and the dye concentration can be observed easily. The variation of the color removal efficiency for the chitosan concentration of 150 mg/l with the different dye concentrations and the different pressure values is given in Figure 7. If the dye concentration is increased by keeping

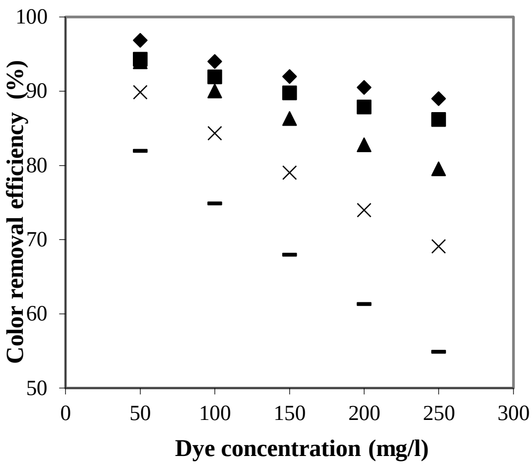



Fig. 7 Variation of color removal efficiency with dye concentration as a function of pressure at 150 mg/l chitosan concentration. Pressure: (♦) 1 bar, (■) 1.5 bar, (▲) 2 bar, (x) 2.5 bar, (−) 3 bar

the chitosan concentration constant at 150 mg/l, the color removal efficiency is reduced for all pressure. It can be said that increasing the pollution load from the membrane reduces the color removal efficiency in the same way. The increase in pressure also affects the color removal efficiency in the negative. Because, deposited organic matter in the fouling layer are scoured and carried at higher pressures. So, captured material is carried into permeate. Therefore color concentration of permeate increases. For a concentration of 50 mg/l of chitosan, the color removal efficency obtained at 1 bar pressure is 96%, but when the pressure is increased to 3 bar, this value decreases to 81%.

In the last study for the purpose of color removal with dye wastewater using ultrafiltration membrane, the effect of the change in the chitosan concentration and pressure on the color removal efficiency was examined by keeping the dye

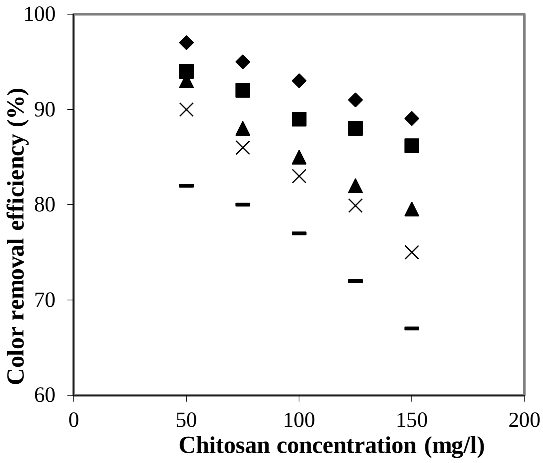



Fig. 8 Variation of color removal efficiency with chitosan concentration as a function of pressure at 250 mg/l dye concentration. Pressure: (♦)1 bar, (■) 1.5 bar, (▲) 2 bar, (x) 2.5 bar, (−) 3 bar

concentration constant at 250 mg/l. The variation of dye removal efficiency with chitosan concentration at different pressure values is given in Figure 8. When the graph was examined, it was observed that the increase in the chitosan concentration decreased the color removal efficiency. The addition of chitosan did not contribute positively to the color removal efficiency. Decrease in color removal efficiency has been accompanied by increase in pressure. Therefore, the optimum operating conditions for color removal are those where the chitosan concentration and the pressure are minimum.

As a result of all experimental studies, optimal conditions for ultrafiltration of dye wastewater with chitosan were 50 mg/l dye concentration, 50 mg/l chitosan concentration and 1 bar pressure.

## 4. Conclusion

Color removal from wastewaters containing dye by using ultrafiltration membrane and chitosan was investigated in this study. The Box-Wilson statistical experiment design were used to generate statistically reliable results.

- Predictions obtained from the response functions were in good agreement with the experimental results indicating the reliability of the method used.
- When the optimum conditions for the permeate flux were examined, the dye concentration was found to be 50 mg/l, the chitosan concentration was 50 mg/l and the pressure was 3 bar for the highest permeate flux (27.1 $l/m^2.h$).
- Optimum conditions for color removal efficiency were determined as 50 mg/l of dye concentration, 50 mg/l of chitosan concentration and 1 bar of pressure.
- Usage of high doses of chitosan did not have much effect on the permeate flux and color removal efficiency.
- Since higher color removal efficiencies are obtained when working at low pressures, experiments using low pressure and low chitosan dosages will be more economical.

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