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# Pilot scale evaluation of feasibility of reuse of wine industry wastewater using reverse osmosis system: modeling and optimization

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ABSTRACT

In this experiment, reverse osmosis (RO) treatment process was investigated under different operating conditions such as pH, flux, transmembrane pressure, and temperature for the wine industry wastewater treatment using response surface methodology. Box–Behnken response surface design with four variables and three levels was used to optimize and study the effect of process parameters. Pareto analysis of variance was done to analyze the result and second-order polynomial model was created to predict the responses. Optimum process parameters were found to be as follows: pH of 10, flux of 70 L/h, transmembrane pressure of 20 bar, and temperature of 30˚C. Under these conditions, predicted value of responses was as follows: Color removal of 91%, COD removal of 93%, TDS removal of 97%, permeate flux of 24 L/$m^2$ h, operating cost of 14 rupees with water recovery of 69%. The properties of RO-treated permeate water were compared with Indian standards and found to be good fit for reuse.

*Keywords:* Reverse osmosis; Wine wastewater; Reuse; Box–Behnken design; Operating cost; Optimization

## 1. Introduction

It is well known that nowadays, industrial water use is a major factor of global water crisis due to drastic increase in population and industries [1]. The water usage trends in India in the year 2012 show that 13% of the natural water sources are used for industries. An overview by the World Commission on Water revealed that fresh water usage will be increased globally by around 60% in the year 2050 [2]. Hence, it is very important that effective water management activities to protect the water environment. In India, various industries consume large amount of fresh water for their process as well as industries discharge highly polluted wastewater to the nearby ecological system. Particularly, wine industry is considered as one of the top water consumers and discharge highly polluted wastewaters. The wastewater produced in

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wine industry contains salts, suspended solids, and other auxiliary chemicals, which cause an organic and inorganic pollution [3]. In addition, wastewater discharged from these industries is notorious for its recalcitrant characteristics. The quality of this wastewater depends on the treatment methods, auxiliaries, and raw materials used. Approximately, wine industry produces 4,500 ton of wastewater annually and its discharge into the rivers and canals affects the aquatic life and creates a negative impact on water sustainability. So, a new approach to zero liquid discharge (ZLD) has a hopeful role in protecting the ecosystem from wine industry wastewater [4].

Nowadays, wastewater treatment and reuse become more attractive method in the wine industry, due to the scarcity of fresh water and increasingly strict rule concerning its release [5]. In wine industries, commonly used conventional water treatment methods include physical, chemical, and biological treatment methods that are not allowed to reuse the water since they do not have sufficient water to achieve the desired percent of removal of the contaminant. In contrast, RO treatment has become a top promising water treatment method for industries [6]. The RO wastewater treatment techniques are of top interest because they decrease the number of unit operations, reuse processed water, and recover useful byproducts. In addition, they have several advantages when compared to conventional wastewater treatment techniques which include continuous and automatic operation, selective separation, easy scale up, easy and well-arranged process conduction, low-space requirement, and purification without chemical [7]. Furthermore, the process factors in RO treatment process such pH, flux, transmembrane pressure, and temperature mainly affect the treatment efficacy and the optimization of these variables will rise the process efficiency with reasonable operating cost [8]. But, the one factor at a time optimization studies required large number of trials and there is chance for poor or misleading results as well as it also fails to study the interactive effect of process factors on treatment efficiency. Last few decades, response surface methodology (RSM) plays an effective method for investigating the interactive effects of more variables influencing the responses by changing them simultaneously and performing a minimum number of trials [9]. RSM is a group of statistical methods generally used to study the performance of complex systems and modeling any kind of complex process [10]. However, to the best of our knowledge, RO treatment process to evaluate the reusability of wine industry wastewater using RSM has not yet explained in literature. Hence, in this research work, an attempt has been made to find out the efficiency of RO process to treat wine industry wastewater using RSM. Finally, the quality of produced water was compared with Indian standard in order to examine its market viability.

## 2. Materials and methods

### 2.1. Raw materials and chemicals

In this study, wastewater was obtained from a local wine industry, Tamil Nadu, India and its characteristics were as follows: pH 4; Color: 265 Pt-Co; COD: 4,432 mg/L; TDS: 3,985 mg/L. Analytical reagent grade chemicals such as Hydrochloric acid (HCl) and Sodium hydroxide (NaOH) were used.

### 2.2. RO experimental setup

Picture of the pilot scale RO process experimental setup is shown in Fig. 1. The bench scale filtration system is fitted with spiral wound module (Membrane area: 2 $m^2$; Salt rejection: 98%; MWCO: 200 Da; Water permeability: 4 $L/m^2$ h bar). The wine industry wastewater was stored in a double wall tank (5 L), without stirring, and then continuously pumped through membrane modules by means of a diaphragm


Fig. 1. Picture of pilot-scale RO unit.

pump (Hydra-Cell model D-07, positive displacement pump, maximum pressure 70 bar, maximum Flux 100 L/h, nominal power 1.8 kW). Tests were run in module dimensions of 3.9 cm width × 33.5 cm length. The main process parameters (pH, flux, transmembrane pressure, and temperature) were measured. Operating pressure is adjusted finely with a spring-loaded pressure-regulating valve (SS-R4512MM-SP, India) on the concentrate outlet and monitored by a digital pressure gauge (Ceraphant T PTC31). This is helpful for independent control of transmembrane pressure and flux. Temperature is monitored and adjusted by temperature controller tank and 0.1 N HCl or NaOH adjusted pH of the wastewater. After RO process, permeate was analyzed for color, COD, and TDS removal.

### 2.3. Analytical methods

Physicochemical properties of the wine industry wastewater are studied according to the APHA standard techniques explained elsewhere [11]. The removal efficiency is calculated as follows [12]:

$$\text{RE} = \left(\frac{c_0 - c_e}{c_0}\right) \times 100 \tag{1}$$

where $c_0$ and $c_e$ is the initial and final concentrations of color, COD, and TDS, respectively. Operating cost of RO process was calculated using unit price (5.14 Rupee per KWh), according to Indian industrial power supply price.

### 2.4. Statistical experimental design

In this present study, Box–Behnken response surface design (BBD) with four factors at three levels was employed to optimize and find out the effect of process parameters such as pH ($A$), flux ($B$), transmembrane pressure ($C$), and temperature ($D$) on RO treatment process to treat wine industry wastewater. Process factors and their ranges (Table 1) were found out based on the preliminary studies. Experimental runs were carried out based on a BBD and the whole design consisting of 29 runs (Table 2) with five center points were designed. Then, the Design-Expert 8.0.7.1 (State-Ease Inc., Minneapolis, MN, USA) statistical package was used for the statistical calculations. The relationship between the responses and five independent variables was evaluated by developing the second-order polynomial mathematical models and the generalized form of equation is given below [13]:

$$Y = \beta_0 + \sum_{j=1}^{k} \beta_j X_j + \sum_{j=1}^{k} \beta_{jj} X_j^2 + \sum_{i} \sum_{<j=2}^{k} \beta_{ij} X_i X_j + e_i \tag{2}$$

Table 1
Process variables and their ranges

| Process variables | Level −1 | 0 | 1 |
|---|---|---|---|
| $A$ | 2 | 6 | 10 |
| $B$ (L/h) | 20 | 60 | 100 |
| $C$ (bar) | 15 | 30 | 45 |
| $D$ (℃) | 30 | 40 | 50 |

Notes: $A$, pH: $B$, Flux: $C$, transmembrane pressure: $D$, temperature.

Table 2
BBD experimental design with results

| Run | $A$ | $B$ | $C$ | $D$ | $Y_1$ | $Y_2$ | $Y_3$ | $Y_4$ | $Y_5$ |
|---|---|---|---|---|---|---|---|---|---|
| 1 | 10 | 60 | 15 | 40 | 82.76 | 88.65 | 95.24 | 24 | 25.70 |
| 2 | 6 | 60 | 30 | 40 | 86.78 | 90.96 | 96.28 | 23 | 23.28 |
| 3 | 10 | 100 | 30 | 40 | 86.72 | 92.54 | 96.22 | 23 | 12.08 |
| 4 | 10 | 60 | 30 | 30 | 83.54 | 85.96 | 91.28 | 21 | 18.20 |
| 5 | 2 | 60 | 15 | 40 | 56.78 | 65.32 | 71.58 | 16 | 11.05 |
| 6 | 10 | 60 | 30 | 50 | 87.72 | 92.58 | 97.22 | 23 | 13.06 |
| 7 | 6 | 20 | 15 | 40 | 39.48 | 43.66 | 53.54 | 12 | 13.31 |
| 8 | 6 | 20 | 30 | 30 | 31.92 | 40.58 | 43.57 | 10 | 4.32 |
| 9 | 6 | 60 | 30 | 40 | 86.78 | 90.96 | 96.28 | 24 | 18.09 |
| 10 | 2 | 60 | 30 | 30 | 47.08 | 51.26 | 56.58 | 13 | 2.36 |
| 11 | 6 | 60 | 45 | 30 | 57.02 | 61.2 | 66.52 | 15 | 23.44 |
| 12 | 6 | 20 | 45 | 40 | 46.1 | 50.28 | 56.28 | 13 | 18.40 |
| 13 | 6 | 60 | 15 | 50 | 70.39 | 74.57 | 79.89 | 19 | 14.91 |
| 14 | 6 | 60 | 45 | 50 | 93.78 | 97.96 | 98.54 | 24 | 20.56 |
| 15 | 6 | 100 | 45 | 40 | 74.78 | 78.96 | 84.28 | 20 | 26.78 |
| 16 | 2 | 20 | 30 | 40 | 33.54 | 33.48 | 38.8 | 7 | 5.29 |
| 17 | 6 | 60 | 30 | 40 | 86.78 | 90.96 | 96.28 | 23 | 19.43 |
| 18 | 6 | 100 | 30 | 50 | 70.72 | 74.9 | 82.54 | 19 | 11.82 |
| 19 | 6 | 60 | 30 | 40 | 86.78 | 90.96 | 96.28 | 23 | 8.94 |
| 20 | 6 | 60 | 30 | 40 | 86.78 | 90.96 | 96.28 | 23 | 11.51 |
| 21 | 2 | 100 | 30 | 40 | 61.28 | 65.46 | 70.78 | 16 | 11.05 |
| 22 | 6 | 20 | 30 | 50 | 61.82 | 68.54 | 71.32 | 17 | 14.96 |
| 23 | 6 | 100 | 15 | 40 | 77.45 | 79.26 | 84.58 | 20 | 11.05 |
| 24 | 6 | 60 | 15 | 30 | 66.08 | 70.26 | 75.58 | 20 | 11.05 |
| 25 | 2 | 60 | 30 | 50 | 71.78 | 75.96 | 81.28 | 19 | 5.55 |
| 26 | 10 | 60 | 45 | 40 | 74.78 | 80.54 | 85.24 | 20 | 27.76 |
| 27 | 10 | 20 | 30 | 40 | 56.66 | 65.54 | 66.16 | 16 | 11.05 |
| 28 | 6 | 100 | 30 | 30 | 72.78 | 76.96 | 82.28 | 19 | 9.25 |
| 29 | 2 | 60 | 45 | 40 | 61.78 | 65.96 | 75.48 | 17 | 17.17 |

Notes: $A$, pH: $B$, flux: $C$, transmembrane pressure: $D$, temperature: $Y_1$, color removal: $Y_2$, COD removal: $Y_3$, TDS removal: $Y_4$, permeate flux: $Y_5$, operating cost.

Adequacy of the developed mathematical model is investigated by the analysis of variance (ANOVA). Regression coefficients of second-order polynomial model and its effects were analyzed with the help of the *F*-values at probability levels ($p \leq 0.05$). The experimental result is analyzed with different statistical parameters such as coefficient of determination ($R^2$), adequate precision (AP), and coefficient of variation (CV%) to emulate the statistical significance of the developed polynomial equation. Then, the interactive and individual effects of parameters on response are analyzed by constructing three-dimensional (3D) response surface plots from developed mathematical models [14]. Optimization of parameters for the highest RO treatment efficiency is performed by Derringer's desired function method [15].

## 3. Results and discussions

In this study, performance evaluation of reverse osmosis (RO) system to treat wine industry wastewater was studied in various processing conditions and four factors with three levels of BBD response surface design were used to examine and optimize the process variables. BBD experimental design consisting of 29 trials was performed and the results are presented in Table 2.

### 3.1. Mathematical modeling

To select the suitable mathematical model to explain the RO process, different models such as interactive, linear, cubic, and quadratic are analyzed by sequential model sum of squares (Table 3). The results indicate that linear and interactive (2FI) models exhibit lower *F*-value and high *p*-value; whereas quadratic model having high *F*-values and lower *p*-values are exhibited. Cubic model is found to be aliased [16,17]. So the quadratic model is chosen to describe the effects of parameters on the RO treatment of wine industry wastewater. Meanwhile, second-order polynomial equations have been created using coefficient of process factors (individual and combine) to the generalized quadratic model (Eq. (2)), which can use to predict the efficiency of RO treatment for various sets of combinations of four factors. The final models obtained in terms of coded variables are specified below:

$$Y_1 = 86.78 + 11.66A + 14.52B + 1.28C + 8.15D + 0.58AB - 3.25AC - 5.13AD - 2.32BC - 7.99BD + 8.11CD - 8.12A^2 - 19.51B^2 - 8.52C^2 - 6.84D^2 \quad (3)$$

$$Y_2 = 90.96 + 12.36A + 13.83B + 1.10C + 8.19D - 1.25AB - 2.19AC - 4.52AD - 1.73BC - 7.50BD + 8.11CD - 7.59A^2 - 19.23B^2 - 8.42C^2 - 6.65D^2 \quad (4)$$

$$Y_3 = 96.28 + 11.41A + 14.25B + 0.49C + 7.92D - 0.48AB - 3.48AC - 4.69AD - 0.76BC - 6.87BD + 6.93CD - 7.61A^2 - 19.55B^2 - 7.50C^2 - 7.51D^2 \quad (5)$$

$$Y_4 = 22.99 + 3.32A + 3.48B - 0.13C + 1.84D - 0.65AB - 1.13AC - 1.13AD - 0.16BC - 1.88BD + 2.64CD - 2.09A^2 - 5.16B^2 - 1.68C^2 - 1.67D^2 \quad (6)$$

$$Y_5 = 16.25 + 4.61A + 1.23B + 3.92C + 1.02D - 1.18AB - 1.02AC - 2.08AD + 2.66BC - 2.02BD - 1.68CD - 2.26A^2 - 3.63B^2 + 5.35C^2 - 3.62D^2 \quad (7)$$

where $Y_1$, $Y_2$, $Y_3$, $Y_4$, and $Y_5$ are color removal (%), COD removal (%), TDS removal (%); permeate flux (L/m$^2$ h), and operating cost (rupee), respectively. In common, developed response surface models may predict a pitiable or ambiguous results. So, the sufficiency of developed mathematical models were assessed by creating analytical plot (Fig. 2) such as predicted vs. actual plot, which helps to study the connection between experimental and predicted result [18,19]. The result revealed that residuals for the prediction of each response are minimum and it shows a good relation between experimental value and the value predicted by the developed mathematical models. Moreover, statistical significance of the developed mathematical models was examined using ANOVA, which is presented in Table 4. The higher *F*-value and lower *p*-values of individual and combined process variables confirmed that the developed models are very significant. The developed mathematical model was evaluated by the determination co-efficient ($R^2$), AP and co-efficient of variance (CV%), which clearly affirmed that the deviations between predicted and experimental data are low and confirms the reliability of the present study [20].

### 3.2. Effect of process variables on the RO process

Three-dimensional (3D) response surface plots were charted from the developed model (Eqs. (3)–(7)) to investigate the individual and interactive effect of process factors on the responses and also used to find

Table 3
Sequential model sum of squares and model summary statistics for RO process

| Source | Sum of squares | DF | Mean square | *F*-value | Prob. > *F* | Remarks |
|---|---|---|---|---|---|---|
| *Sequential model sum of squares for $Y_1$* | | | | | | |
| Mean | 138,019.3 | 1 | 138,019.3 | | | |
| Linear | 4,977.442 | 4 | 1,244.36 | 8.35807 | 0.0002 | |
| 2FI | 688.9204 | 6 | 114.8201 | 0.71657 | 0.6413 | |
| Quadratic | 2,744.398 | 4 | 686.0995 | 68.6918 | <0.0001 | Suggested |
| Cubic | 124.5809 | 8 | 15.57261 | 6.12598 | 0.0201 | Aliased |
| Residual | 15.25236 | 6 | 2.54206 | | | |
| Total | 146,569.9 | 29 | 5,054.135 | | | |
| *Sequential model sum of squares for $Y_2$* | | | | | | |
| Mean | 157,206.68 | 1 | 157,206.68 | | | |
| Linear | 4,950.36 | 4 | 1,237.59 | 8.57 | 0.0002 | |
| 2FI | 607.58 | 6 | 101.26 | 0.64 | 0.6987 | |
| Quadratic | 2,644.96 | 4 | 661.24 | 43.69 | <0.0001 | Suggested |
| Cubic | 210.97 | 8 | 26.37 | 171.04 | <0.0001 | Aliased |
| Residual | 0.93 | 6 | 0.15 | | | |
| Total | 165,621.48 | 29 | 5,711.09 | | | |
| *Sequential model sum of squares for $Y_3$* | | | | | | |
| Mean | 180,228.24 | 1 | 180,228.24 | | | |
| Linear | 4,752.62 | 4 | 1,188.16 | 8.50 | 0.0002 | |
| 2FI | 520.40 | 6 | 86.73 | 0.55 | 0.7633 | |
| Quadratic | 2,703.92 | 4 | 675.98 | 71.68 | <0.0001 | Suggested |
| Cubic | 116.18 | 8 | 14.52 | 5.50 | 0.0261 | Aliased |
| Residual | 15.84 | 6 | 2.64 | | | |
| Total | 188,337.21 | 29 | 6,494.39 | | | |
| *Sequential model sum of squares for $Y_4$* | | | | | | |
| Mean | 10,038.51 | 1 | 10,038.51 | | | |
| Linear | 318.48 | 4 | 79.62 | 7.63 | 0.0004 | |
| 2FI | 53.96 | 6 | 8.99 | 0.82 | 0.5662 | |
| Quadratic | 184.10 | 4 | 46.03 | 52.10 | <0.0001 | Suggested |
| Cubic | 10.80 | 8 | 1.35 | 5.17 | 0.0302 | Aliased |
| Residual | 1.57 | 6 | 0.26 | | | |
| Total | 10,607.41 | 29 | 365.77 | | | |
| *Sequential model sum of squares for $Y_5$* | | | | | | |
| Mean | 131,970.65 | 1 | 131,970.65 | | | |
| Linear | 5,125.84 | 4 | 1,281.46 | 8.41 | 0.0002 | |
| 2FI | 661.61 | 6 | 110.27 | 0.66 | 0.6808 | |
| Quadratic | 2,869.24 | 4 | 717.31 | 78.80 | <0.0001 | Suggested |
| Cubic | 120.02 | 8 | 15.00 | 12.12 | 0.0035 | Aliased |
| Residual | 7.43 | 6 | 1.24 | | | |
| Total | 140,754.78 | 29 | 4,853.61 | | | |

Notes: $Y_1$, color removal: $Y_2$, COD removal: $Y_3$, TDS removal: $Y_4$, permeate flux: $Y_5$, operating cost.

out the optimal condition of each process variable for maximum treatment efficiency [21]. In this experiment, the models have more than two variables. So, the 3D plots are drawn by keeping one variable at a constant level, whereas the other two variables were assorted in their range, which are shown in Figs. 3–4.

### 3.2.1. Effect of pH

pH is one of the crucial factors, which affects the efficiency of the RO treatment of wine industry wastewater. So, the experiments were performed to examine the effect of pH (2, 6, and 10) in RO treatment process and the results are shown in

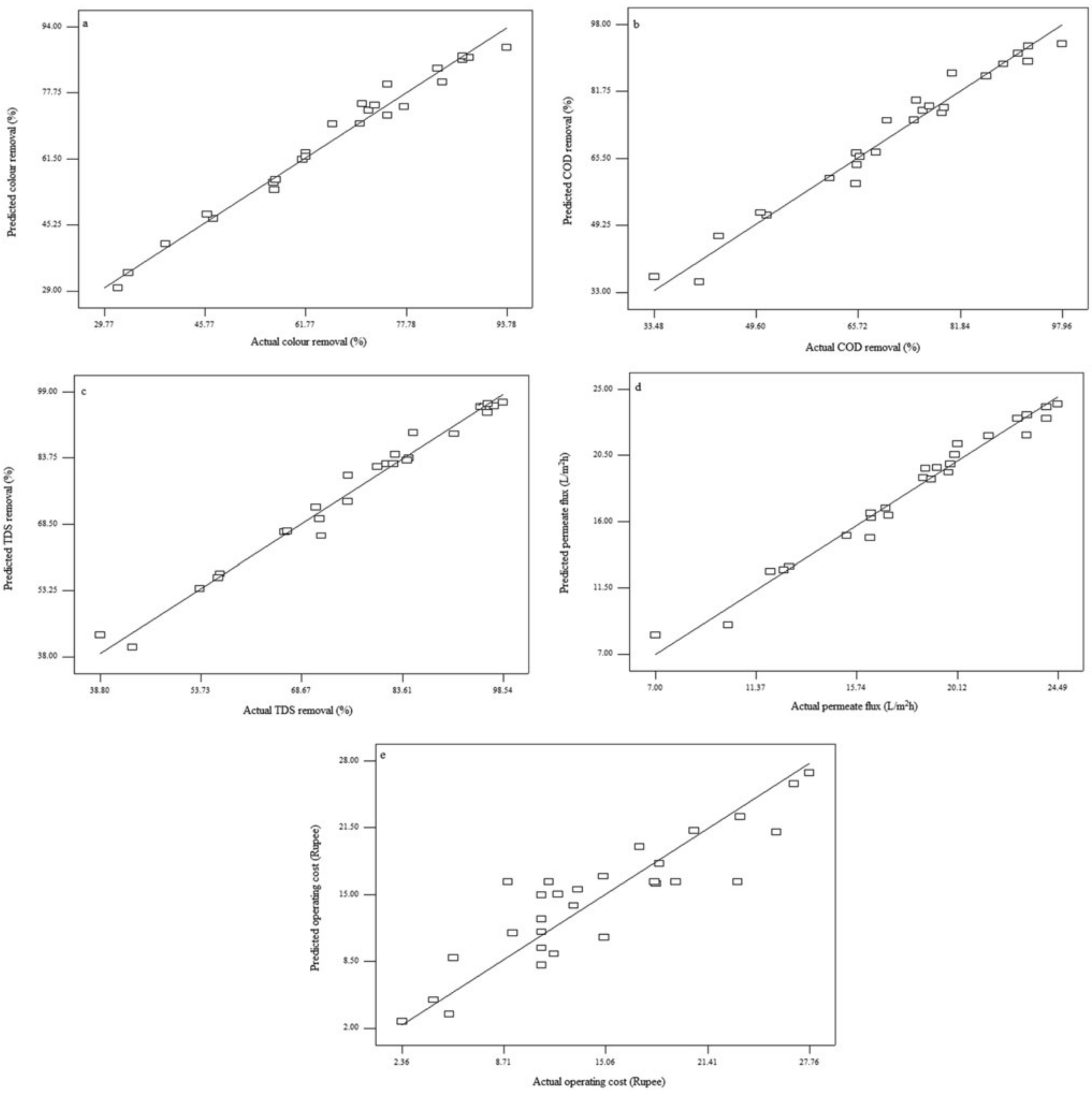


Fig. 2. Model adequacy plots: (a) colour removal, (b) COD removal, (c) TDS removal, (d) permeate flux, and (e) operating cost.

Fig. 3(a)–(e). From the results, it is observed that the color removal, COD removal, TDS removal, permeate flux, and operating cost are increased linearly with increasing pH from 2–10 i.e. throughout the experiments. The increase in pH would change the surface nature of membrane module for effective separation, thus treatment efficiency is increased. Similar kind of trend is reported for the heavy metals removal from industrial wastewater using membrane process [22].

### 3.2.2. *Effect of Flux*

Flux is one of the important factors for the wine industry wastewater treatment using RO process,

Table 4
ANOVA results for responses

| | $Y_1$ | | $Y_2$ | | $Y_3$ | | $Y_4$ | | $Y_5$ | |
|---|---|---|---|---|---|---|---|---|---|---|
| Source | *F*-value | *p*-value | *F*-value | *p*-value | *F*-value | *p*-value | *F*-value | *P* value | *F*-value | *p*-value |
| Model | 60.15 | <0.0001 | 38.71 | <0.0001 | 60.42 | <0.0001 | 45.00 | <0.0001 | 357.57 | <0.0001 |
| *A* | 163.39 | <0.0001 | 121.21 | <0.0001 | 165.52 | <0.0001 | 149.36 | <0.0001 | 2523.93 | <0.0001 |
| *B* | 253.21 | <0.0001 | 151.72 | <0.0001 | 258.43 | <0.0001 | 164.71 | <0.0001 | 152.55 | <0.0001 |
| *C* | 1.95 | 0.1840 | 0.96 | 0.3447 | 0.31 | 0.5860 | 0.23 | 0.6408 | 174.84 | <0.0001 |
| *D* | 79.79 | <0.0001 | 53.19 | <0.0001 | 79.72 | <0.0001 | 46.23 | <0.0001 | 1784.59 | <0.0001 |
| *AB* | 0.13 | 0.7191 | 0.41 | 0.5325 | 0.10 | 0.7592 | 1.94 | 0.1859 | 7.84 | 0.0142 |
| *AC* | 4.22 | 0.0592 | 1.26 | 0.2797 | 5.12 | 0.0400 | 5.82 | 0.0301 | 0.001 | 0.9673 |
| *AD* | 10.54 | 0.0059 | 5.40 | 0.0357 | 9.33 | 0.0086 | 5.78 | 0.0306 | 33.25 | <0.0001 |
| *BC* | 2.16 | 0.1637 | 0.79 | 0.3888 | 0.24 | 0.6283 | 0.12 | 0.7366 | 44.69 | <0.0001 |
| *BD* | 25.57 | 0.0002 | 14.89 | 0.0017 | 20.03 | 0.0005 | 15.94 | 0.0013 | 20.5 | 0.0005 |
| *CD* | 26.36 | 0.0002 | 17.39 | 0.0009 | 20.36 | 0.0005 | 31.49 | <0.0001 | 67.29 | <0.0001 |
| $A^2$ | 42.80 | <0.0001 | 24.69 | 0.0002 | 39.80 | <0.0001 | 32.03 | <0.0001 | 148.98 | <0.0001 |
| $B^2$ | 247.31 | <0.0001 | 158.41 | <0.0001 | 262.77 | <0.0001 | 195.17 | <0.0001 | 1.1 | 0.3115 |
| $C^2$ | 47.18 | <0.0001 | 30.37 | <0.0001 | 38.64 | <0.0001 | 20.84 | 0.0004 | 5.9 | 0.0293 |
| $D^2$ | 30.40 | <0.0001 | 18.98 | 0.0007 | 38.84 | <0.0001 | 20.54 | 0.0005 | 21.1 | 0.0004 |
| CV% | 4.58 | | 3.98 | | 4.05 | | 3.25 | | 4.99 | |
| $R^2$ | 0.9846 | | 0.9954 | | 0.9835 | | 0.9912 | | 0.9758 | |
| AP | 27.81 | | 25.64 | | 26.54 | | 31.24 | | 22.54 | |

Notes: $Y_1$, color removal: $Y_2$, COD removal: $Y_3$, TDS removal: $Y_4$, permeate flux: $Y_5$, operating cost.

which is associated with removal effectiveness and overhead. To study the effect of flux on RO process, experiments were performed with different flux (20, 60, and 100 L/h) and results are shown in Fig. 3(a)–(e). The results showed that color removal, COD removal, TDS removal, permeate flux, and operating cost are increased with increasing flux up to 80 L/h. This could be described by fact that, decreasing resistance to flux due to enhanced solute build-up at the membrane surface. Beyond the flux of 80 L/h shows the negligible effect on the process efficiency of RO treatment of wine industry wastewater. This is in agreement with results published by other author [23].

### 3.2.3. Effect of transmembrane pressure

Transmembrane pressure is also an important factor that influences the RO treatment of wine industry wastewater. Therefore, in this experiment, influence of transmembrane pressure on the RO treatment process is examined by varying its range (15, 30, and 45 bar) and the results are presented in Fig. 4(a)–(e). The result showed that color removal, COD removal, TDS removal, and permeate flux are increased with the increasing transmembrane pressure up to 40 bar, due to the increase in transmembrane pressure that creates the concentration difference and effective driving force to separate the pollutants from wine industry wastewater, which leads to maximum treatment efficiency. Beyond the transmembrane pressure of 40 bar shows the negligible effect on RO process. The increase in transmembrane pressure accumulates more pollutants in the membrane surface. As the transmembrane pressure rises, more pollutants accumulate on the membrane surface and form a gel layer. This layer results in rise in the osmotic pressure, causing increasing flux decline by the reduction of driving force [24]. Operating cost is linearly increased with increasing in transmembrane pressure, due to the consumption of higher power supply [25].

### 3.2.4. Effect of temperature

Temperature is a major factor which affects the efficiency of RO treatment of wine industry wastewater and its influence are investigated by varying temperature (20, 35, and 50˚C) and the results are depicted in Fig. 4(a)–(e). The result revealed that color removal, COD removal, TDS removal, and permeate flux are increased with the rising temperature up to 40˚C. It is identified that the fluxes rise with rising temperature. This is because of the reduction in solvent viscosity, the thermal expansion of the membrane materials, and the increasing of solvent diffusion coefficient in the membranes. Beyond the temperature of 40˚C seems the decreased treatment efficiency due to the negative impact of temperature on RO treatment. This is mostly because of the fact, rising amount of

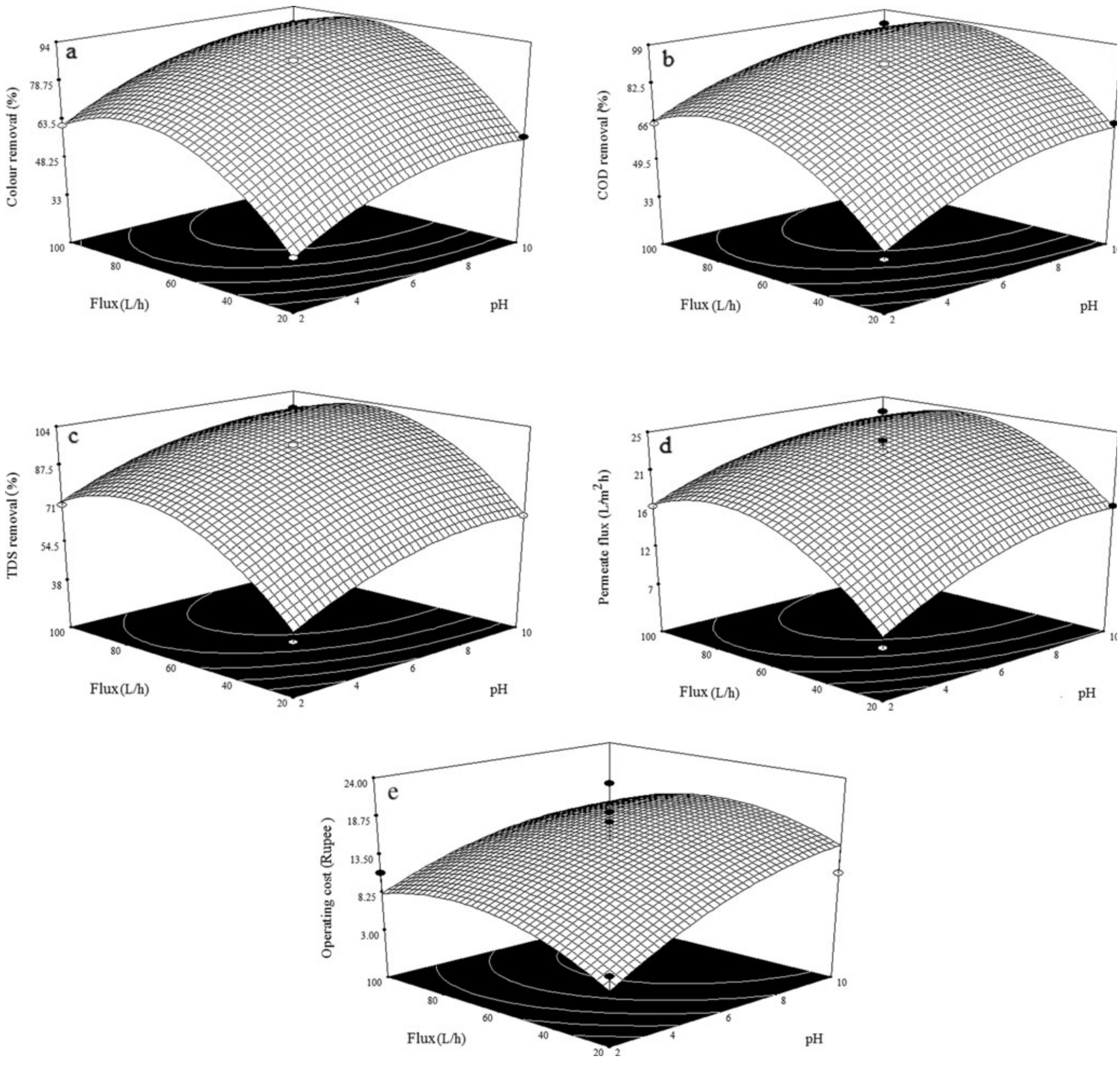


Fig. 3. Response surface plots representing the effect of process variables (*A* and *B*) on RO process: (a) colour removal, (b) COD removal, (c) TDS removal, (d) permeate flux, and (e) operating cost.

pollutants that can easily pass at higher temperatures resulted in plugging within the expanded pores and membrane surface [26]. Whereas, operating cost is linearly increased with increases in the temperature.

### 3.3. Optimization and validation

Simultaneous optimization of the multiple responses was carried out using Derringer's desired function methodology in order to investigate the optimum parameters for maximum treatment efficiency of RO treatment in wine industry wastewater. This will optimize any combination of one or more goals; the goals may apply either factors or responses. The possible goals are: maximize, minimize, target, within range, none (for responses only), and set to an exact value (factors only). In the present study, goals of the process variables were selected as in a range and the response goal was selected as maximize, except operating cost. This numerical optimization

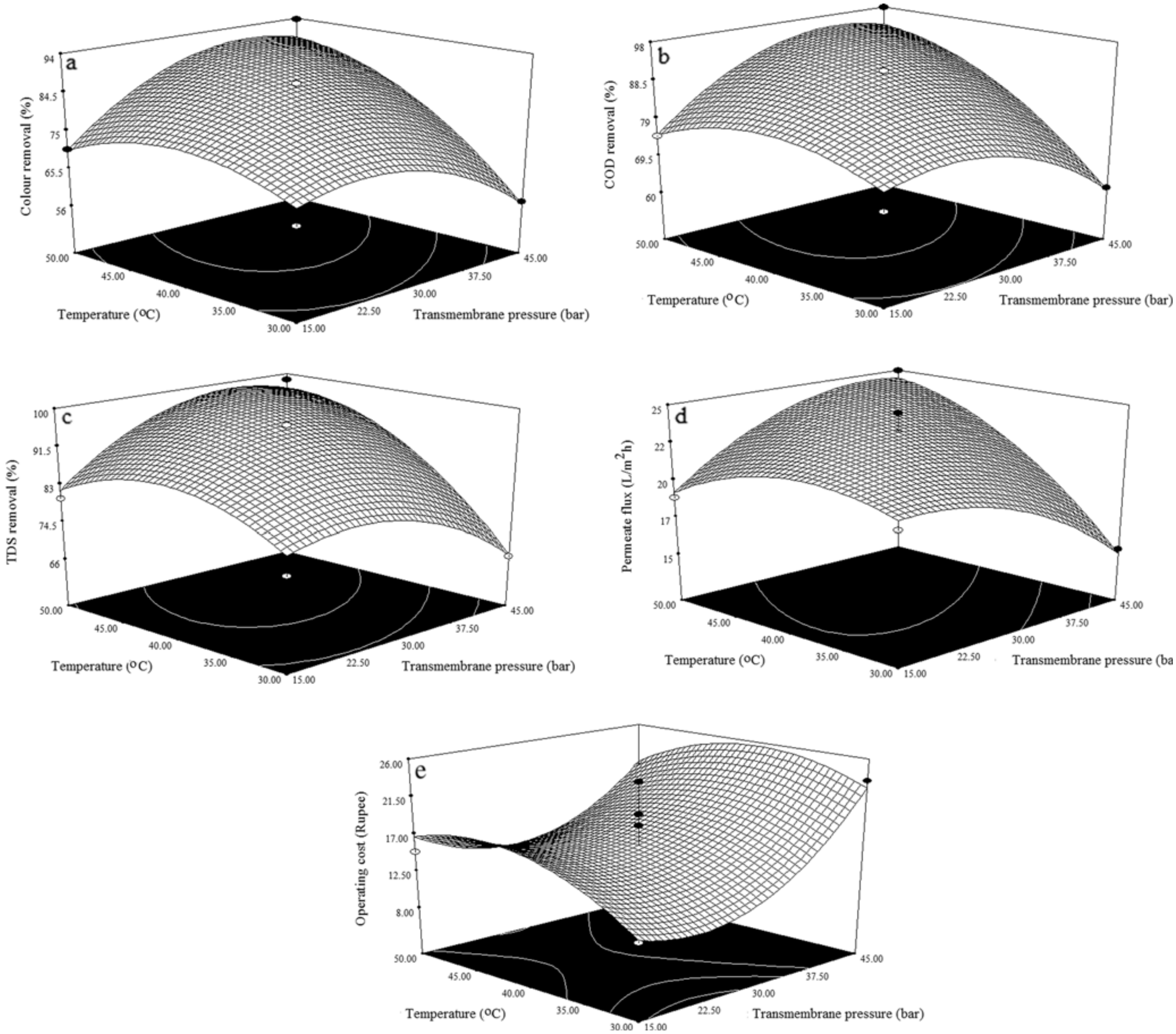


Fig. 4. Response surface plots representing the effect of process variables (*C* and *D*) on RO process: (a) colour removal, (b) COD removal, (c) TDS removal, (d) permeate flux, and (e) operating cost.

method evaluates a point that maximizes the desirability function and optimum conditions were found to be as follows: pH of 10, Flux of 70 L/h, transmembrane pressure of 20 bar, and temperature of 30˚C with a desirability value of 0.999. 91% of color, 93% of COD, 97% of TDS was removed at optimum operating conditions and also permeate flux of 24 $L/m^2 h$ was obtained. The operating cost for the recovery of 69% water from wine industry wastewater is found to be 1.8 rupees per liter. Then, the suitability of optimum conditions for the predicting optimum response values are tested based on above-mentioned conditions. Triplicate experiments were performed under the optimized conditions and the mean values (<2% error) obtained from real experiments, demonstrated the validation of the optimized conditions [27].

### 3.4. *Quality of treated wine industry wastewater*

To find out the reusability of RO-treated wine industry wastewater, the quality of RO-treated water was determined and compared with Indian standards. The obtained results are given in Table 5. It clearly suggested that all the compared properties of

Table 5
Comparison chart of RO-treated wine industry wastewater and Indian standards

| Characteristics | RO-treated wine industry wastewater | Indian standards | WHO standards |
|---|---|---|---|
| pH | 6.8 | 5.0–7.0 | 5 |
| Color (Pt-Co) | 24 | 15–25 | 13 |
| COD (mg/L) | 310 | 150–300 | 175 |
| TDS (mg/L) | 120 | 100–250 | 180 |

RO-treated wastewater are close to Indian standard, thus it may act as an alternate water source in local market and industries [28].

## 4. Conclusion

In this study, BBD response surface design (BBD) was used to study and optimize the RO process factors such as transmembrane pressure, pH, flux, and temperature on the treatment of wine industry wastewater for reuse. The results revealed that all the process factors have significant effects on the RO treatment process and quadratic models were developed for predicting the responses. Optimum set of the independent variables are obtained by derringer's desired function methodology and it is found to be: pH of 10, flux of 70 L/h, transmembrane pressure of 20 bar, and temperature of 30˚C. Under these conditions, 91% of color, 93% of COD, 97% of TDS were removed and also 24 L/m$^2$ h of permeate was obtained. Finally, 69% of water is recovered from the wine industry wastewater for reuse. Recovery cost of water is found to be 1.8 rupees per liter. These results indicate that the proposed pilot-scale RO process is an effective and economically viable method to recover the 69% of reusable wastewater from wine industry wastewater.

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