

# Removal of colour and chemical oxygen demand from textile effluent by Fenton oxidation method

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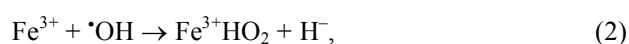
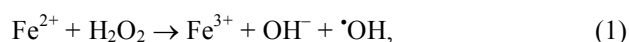
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**The main objective of this study is to focus on the removal of colour and chemical oxygen demand (COD) from the secondary treated effluent (SE) and reverse osmosis (RO) concentrate, using Fenton oxidation as an advanced oxidation process. In order to identify the feasibility and economics, the experiments were conducted in these two streams of the textile-based effluent treatment plant. In this study, COD and colour removal efficiencies were observed as 75% and 94% in the SE and 85% and 99% in the RO concentrate respectively. After comparing the operating cost between these two streams, treating of SE with Fenton oxidation was found to be an economical, sustainable option for removing the colour and COD from the SE. This option will improve the performance of membrane filtration systems in effluent treatment plants that are based on zero liquid discharge.**

**Keywords:** Chemical oxygen demand, colour, Fenton, textile effluent, ZLD.

TEXTILE companies play a crucial role in the Indian economy. Majority of the textile companies are small, medium and micro enterprises (SME) and are distributed throughout the country. These textile companies, especially dyeing companies, consume enormous amounts of water through various operations, such as scouring, bleaching, dyeing and washing. In addition to consuming water, these companies also use dyes, salts and other auxiliaries in the process, which generate highly coloured effluent along with other pollutants such as biological oxygen demand (BOD), chemical oxygen demand (COD), total suspended solids (TSS) and salts<sup>1-5</sup>. Therefore, colour and organic pollutant removal is an integral function of effluent treatment plants<sup>6</sup>. Various configurations of treatment methods such as physical, biological and chemical methods<sup>4,5</sup> are integrated to mitigate the colour and COD issues from the dye-contaminated effluent<sup>7,8</sup>. Biological treatment is widely used to treat effluent containing high organic load<sup>7</sup>. Even though it has certain advantages, biological treatment is not an appropriate

option. After biologically treating composite textile effluent, it still contains significant concentrations of organic load, which is highly non-biodegradable. The combined biological treatment with Fenton is expected to produce less sludge<sup>9</sup>, cost less, and be more effective than any other single treatment technology for the treatment of textile dye effluent<sup>10,11</sup>. Among advanced oxidation processes (AOPs), Fenton<sup>12-15</sup> is widely used for wastewater treatment and seems to be favourable because it breaks down the pollutant. The removal of organics by AOP is required in effluent treatment plants. Fenton oxidation is an effective post-treatment/polishing step that transforms the organic compound into by-products and further reduces the effluent's toxicity<sup>7</sup>. Most conventional treatment methods employed in the textile dyeing industry for removal of colour and COD include coagulation/flocculation by addition of chemicals, and activated carbon adsorption of treated effluent<sup>16-18</sup>. Among various conventional methods, chemical treatment methods generate huge amounts of sludge<sup>13,19</sup> along with an increase in total dissolved solids (TDS) in the treated effluent or secondary pollution due to the addition of chemicals<sup>20,21</sup>. The Fenton process not only oxidizes the organic compounds but also enhances the coagulation function by forming ferric-hydroxo complexes<sup>1,7,22</sup>. The chemistry of Fenton involves<sup>23</sup> the generation of  $\cdot\text{OH}$  radicals, as shown in the reaction below (1). Fenton's reagent cannot be used alone, but can be coupled with other treatment techniques<sup>24</sup>. However, production of iron sludge in the form of  $\text{Fe}(\text{OH})_3$  is considered a shortcoming of this process which necessitates further separation and disposal<sup>15</sup>. Further, when compared to other coagulants, the presence of residual oxidized iron would have a minimal impact or cause no loss in specific flux and salt rejection in RO membranes<sup>25</sup>. Therefore, Fenton oxidation is a suitable pre-treatment methodology for treating secondary textile effluent prior to RO.



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Among the AOPs, Fenton's reaction has a diminutive reaction time to oxidize the organics; thus, Fenton's reagent is used when the removal of high organic pollutants or COD is required<sup>26</sup>. The generated 'OH radical oxidizes the organics in the solution into carbon dioxide (CO<sub>2</sub>) and water (H<sub>2</sub>O) by cascade oxidation reaction<sup>27</sup>. These 'OH radicals possess a strong oxidizing potential of +2.8 V and can attack a wide variety of toxic contaminants in the solution<sup>28</sup>.

The main aims of this work are to determine the influences of various parameters on the degradation of colour and recalcitrant organics present in SE and RO concentrate, and to find the most cost-effective option between these streams in order to optimize operating cost. The effects of Fe<sup>2+</sup>, H<sub>2</sub>O<sub>2</sub>, quantity of sludge generation and operating cost were studied to discover the ideal operating conditions for removal of colour and recalcitrant COD from the SE and RO concentrate with a lower Opex.

## Materials and methods

Composite samples of biologically treated effluent and RO concentrate from a textile dyeing-based effluent treatment plant were collected at the outlet of the clarifier and RO reject. Samples were then filtered through a sand filter and homogenized separately before conducting the experiments. The pH of both effluents was adjusted to 5 by adding H<sub>2</sub>SO<sub>4</sub> (ref. 29) and measured using a pH meter. A separate jar test was conducted for both samples with varying concentrations of Fe<sup>2+</sup> and H<sub>2</sub>O<sub>2</sub> at a constant pH of 5. Five trials were conducted for each sample to find the maximum colour and COD reduction and optimal dose of H<sub>2</sub>O<sub>2</sub> and Fe<sup>2+</sup>. COD and colour oxidation were achieved by a Fenton reagent composed of FeSO<sub>4</sub>, 7H<sub>2</sub>O and H<sub>2</sub>O<sub>2</sub>. The necessary quantities of Fe<sup>2+</sup> were added to the samples and thoroughly stirred using a magnetic stirrer. Then H<sub>2</sub>O<sub>2</sub> solution was slowly added to all jars. The experimental setup constituted suitable mixing arrangements in which a 1000 ml sample was studied. After the addition of Fe<sup>2+</sup> and H<sub>2</sub>O<sub>2</sub>, all jars were allowed to settle for 5 hours for completion of the reaction. Then samples were collected from each jar and analysed for colour and COD as per the standard methods for examination of water and wastewater<sup>30</sup>. The colour of samples was measured by absorbance measurement using spectrophotometer at three different wavelengths (436, 525 and 620 nm), and absorbance results were converted to 'Indexes of Transparency', i.e. DFZ (Durchsichts-Farb-Zahl) number. DFZ number was calculated based on the eq. (4)<sup>31</sup>

$$DFZ = 100 \left( \frac{E\lambda}{D} \right), \quad (4)$$

$E\lambda$  is the extinction (at a known wave length) and  $D$  is the path length of cell (mm).

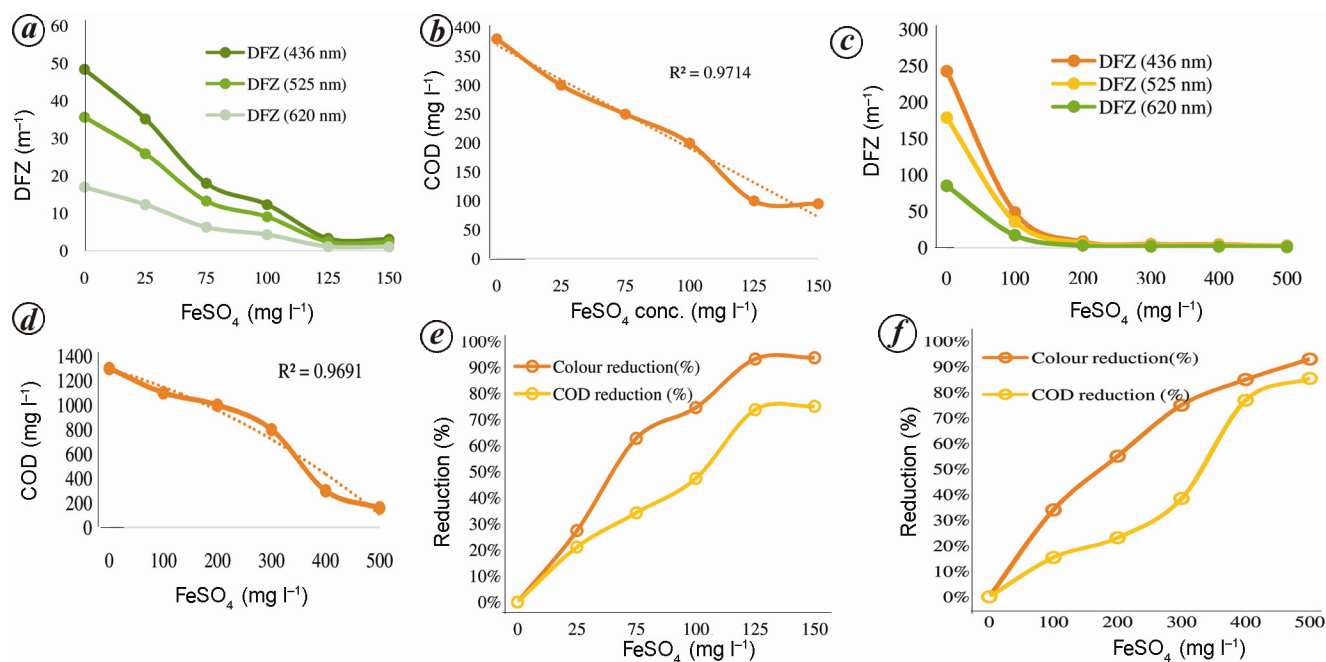
## Results and discussion

### *Effect of Fe<sup>2+</sup> dosage on removal of colour and COD*

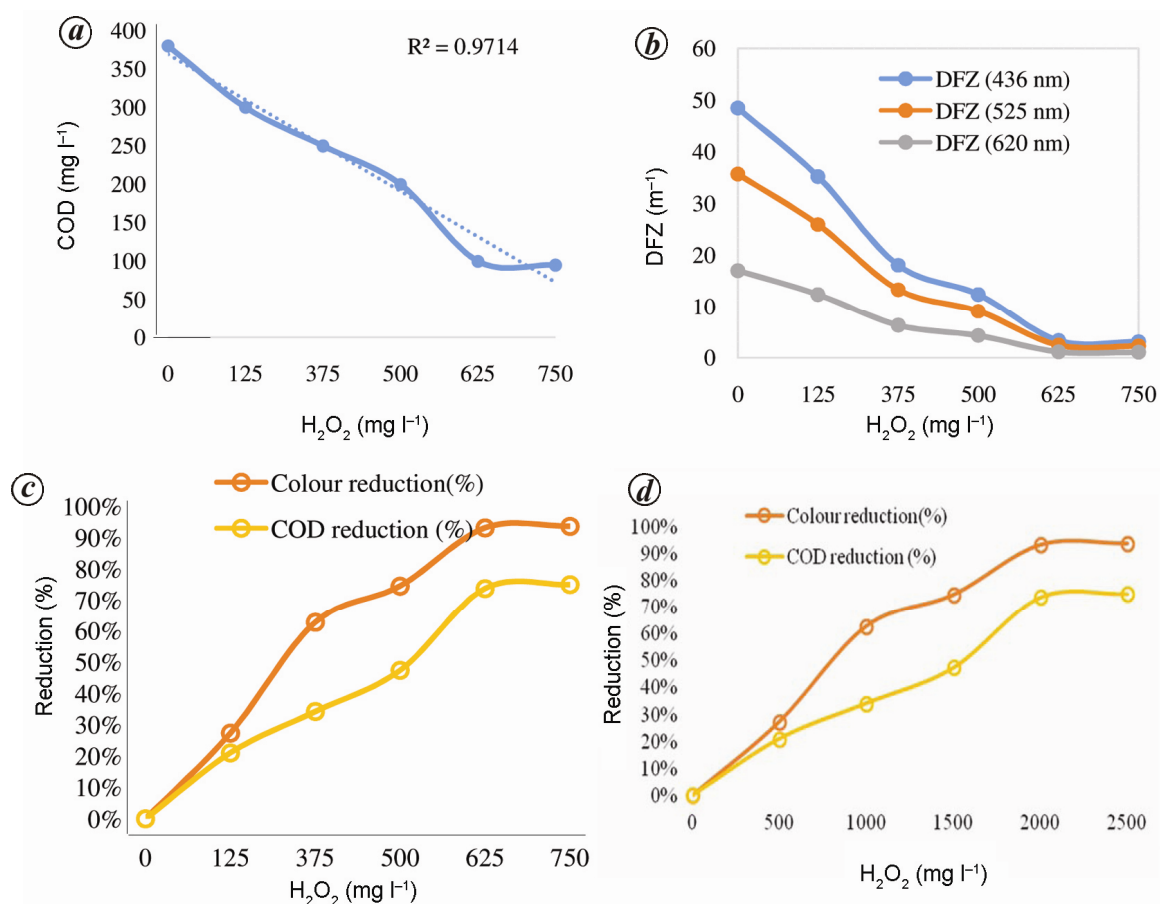
The effect of Fe<sup>2+</sup> dosage on the removal of colour and COD was studied at various concentrations in two samples of different salinity levels. Several studies have reported the use of various proportions of the two reactants – Fe<sup>2+</sup> and H<sub>2</sub>O<sub>2</sub> – for removal of organics<sup>32–34</sup>. To observe the effect of dosages in the oxidation of wastewater and to find the optimal concentration, a series of tests were conducted at different concentrations of Fe<sup>2+</sup> from 0 to 150 mg l<sup>-1</sup> for SE. As shown in Figure 1 a–f), when the concentration of iron increases, colour reduction in the sample proportionately increases<sup>35</sup>. Initially at 0 mg l<sup>-1</sup>, colour value (i.e. DFZ number) of the sample was 48 m<sup>-1</sup> (436 nm), 35 m<sup>-1</sup> (525 nm), and 18 m<sup>-1</sup> (620 nm) and maximum colour reduction was obtained at an Fe<sup>2+</sup> concentration of 150 mg l<sup>-1</sup>, i.e. reduction in DFZ values were 3.1 m<sup>-1</sup> (436 nm), 2.3 m<sup>-1</sup> (525 nm) and 1.1 m<sup>-1</sup> (620 nm). Therefore, optimal dose of Fe<sup>2+</sup> concentration is 125–150 mg l<sup>-1</sup>. From the results, it was found that as Fe<sup>2+</sup> concentration increases, the COD and colour removal efficiencies proportionately increased<sup>9</sup>. Further, consumption of Fenton reagent in the treating of SE was much lower<sup>36</sup> than the RO concentrate. Similarly, concentration of Fe<sup>2+</sup> in the colour reduction was also studied for the high salinity effluent (TDS 40,000 mg l<sup>-1</sup>), i.e. RO concentrate. As shown in Figures 1 c, d and 2 d, maximum colour reduction was obtained at 500 mg l<sup>-1</sup>. Fe<sup>2+</sup> dosage rate in the RO concentrate was 4-fold that required for SE, mainly due to high concentration of organics and higher salinity when compared to SE. COD reduction in both samples was also studied, and maximum COD reduction was observed in the SE at Fe<sup>2+</sup> dosage of 125–150 mg l<sup>-1</sup> (Figure 1 b and e), i.e. COD reduced from 380 to 95 mg l<sup>-1</sup>. Likewise, the maximum COD reduction in RO concentrate was observed at Fe<sup>2+</sup> dosage of 500 mg l<sup>-1</sup> (Figure 1 d and f). Colour and COD values were measured only after 5 hours of hydraulic retention.

### *Effect of H<sub>2</sub>O<sub>2</sub> dosage on removal of colour and COD*

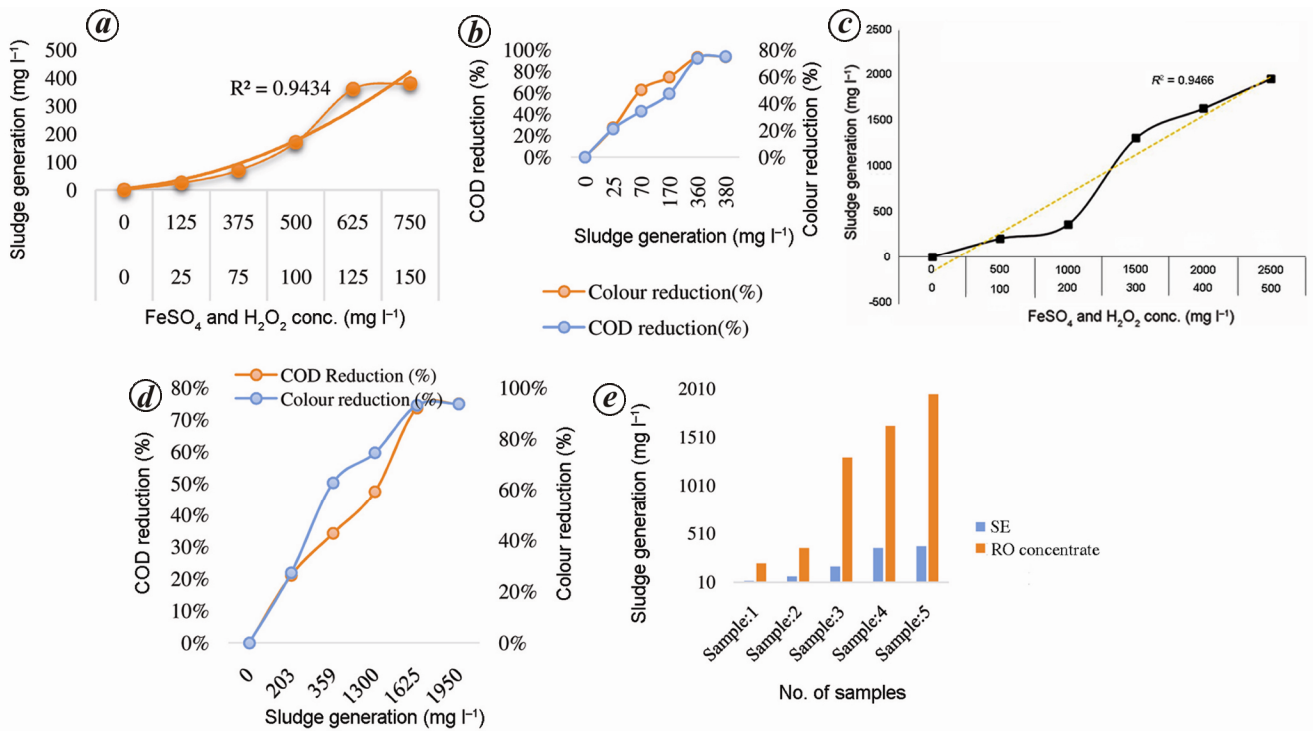
The effect of H<sub>2</sub>O<sub>2</sub> on colour and COD reduction were also studied in both streams and presented in Figure 2 a–d as well as Table 1. Figures show that the removal of colour and COD from the SE increased as the concentration of H<sub>2</sub>O<sub>2</sub> increased at pH 5.0. In the absence of iron, there is no evidence of hydroxyl radical formation in the wastewater<sup>35</sup>. The dosage of H<sub>2</sub>O<sub>2</sub> was 125, 375, 500, 625 and 750 mg l<sup>-1</sup>, and Fe<sup>2+</sup> was 25, 75, 100, 125, 150 mg l<sup>-1</sup>. In the presence of H<sub>2</sub>O<sub>2</sub>, the colour removal increased



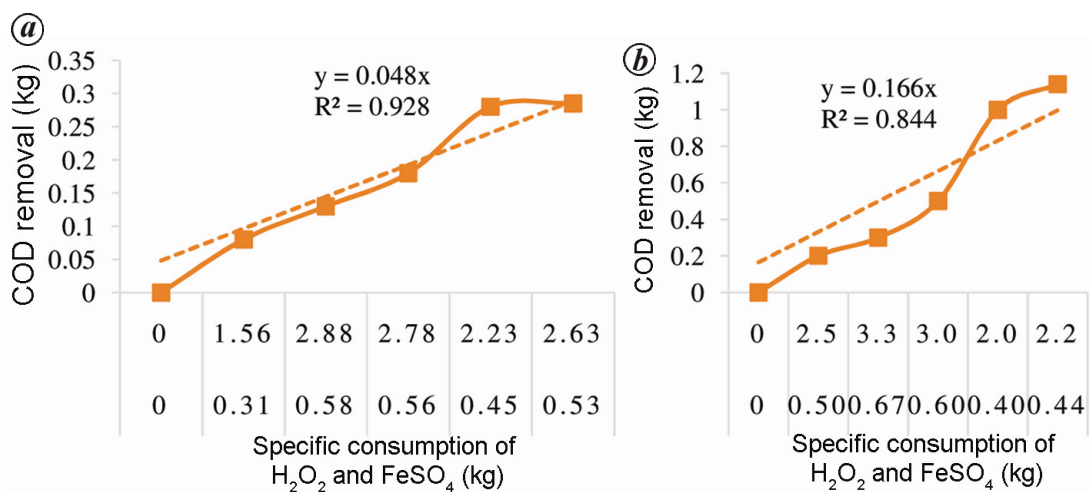
**Figure 1.** Effect of  $\text{Fe}^{2+}$  dosage on (a) colour reduction in the secondary treated effluent; (b) COD reduction in the secondary treated effluent; (c) colour reduction in the RO concentrate; (d) COD reduction in the RO concentrate; (e) colour and COD reduction in the secondary treated effluent; (f) colour and COD reduction in the RO concentrate.



**Figure 2.** Effect of  $\text{H}_2\text{O}_2$  dosage on reduction of (a) COD in the secondary treated effluent; (b) colour in the secondary treated effluent; (c) colour and COD in the secondary treated effluent and (d) colour and COD in the RO concentrate.



**Figure 3.** Effect of (a) concentration of Fe<sup>2+</sup> and H<sub>2</sub>O<sub>2</sub> on sludge generation in the SE; (b) percentage reduction of COD and colour on sludge generation in the SE; (c) concentration of Fe<sup>2+</sup> and H<sub>2</sub>O<sub>2</sub> on sludge generation in the RO concentrate; (d) percentage reduction of COD and colour on sludge generation in the RO concentrate. (e) Comparison of sludge generation between SE and RO concentrate.



**Figure 4.** Specific consumption of Fe<sup>2+</sup> and H<sub>2</sub>O<sub>2</sub> in reduction of (a) COD in secondary effluent and (b) COD in RO concentrate.

up to 94% and the COD removal increased up to 75%. Colour and COD reduction were calculated according to eqs (5) and (6)

$$\% \text{ Decolourization} = \frac{\text{Abs}_0 - \text{Abs}}{\text{Abs}_0} \times 100. \quad (5)$$

Here Abs<sub>0</sub> and Abs are absorbance values before and after oxidation of wastewater.

$$\% \text{ COD removal} = \frac{\text{COD}_i - \text{COD}_f}{\text{COD}_i} \times 100. \quad (6)$$

where COD<sub>i</sub> and COD<sub>f</sub> are initial and final (after oxidation) values of COD.

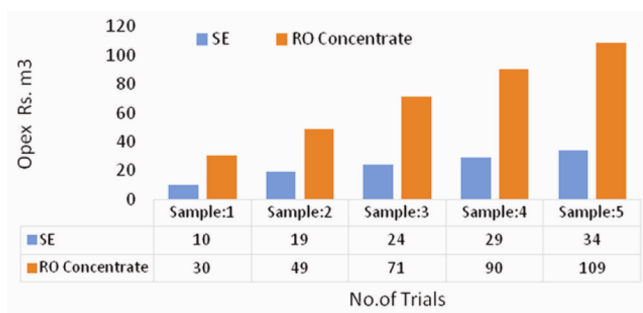
Since a high level of colour removal is much easier to achieve than a high level of organic matter oxidation/mineralization, the effect of process limitations on mineralization targeting was studied to find optimal

**Table 1.** Performance summary of Fenton process on removal of COD and colour from the SE and RO concentrate

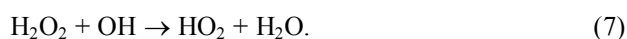
Reactor design parameters	Secondary treated effluent (SE)					RO concentrate						
	Control sample	Sample: 1	Sample: 2	Sample: 3	Sample: 4	Sample: 5	Control sample	Sample: 1	Sample: 2	Sample: 3	Sample: 4	Sample: 5
Sample volume (ml)	1000	1000	1000	1000	1000	1000	1000	1000	1000	1000	1000	1000
FeSO <sub>4</sub> dosing (mg l <sup>-1</sup> )	-	25	75	100	125	150	-	100	200	300	400	500
H <sub>2</sub> O <sub>2</sub> dosing (mg l <sup>-1</sup> )	-	125	375	500	625	750	-	500	1000	1500	2000	2500
Specific consumption of FeSO <sub>4</sub> kg <sup>-1</sup> of COD	-	0.31	0.58	0.56	0.45	0.53	-	0.50	0.67	0.60	0.40	0.44
Specific consumption of H <sub>2</sub> O <sub>2</sub> kg <sup>-1</sup> of COD	-	1.56	2.88	2.78	2.23	2.63	-	2.5	3.3	3.0	2.0	2.2
pH	8.1	5	5	5	5	5	8.1	5	5	5	5	5
TDS mg l <sup>-1</sup>	8000	8110	8140	8160	8180	8195	40,000	40,000	40,100	40,150	40,180	40,200
COD mg l <sup>-1</sup>	380	300	250	200	100	95	1300	1100	1000	800	300	160
Δ COD gm l <sup>-1</sup> (removal rate)	-	0.08	0.13	0.18	0.28	0.285	-	0.2	0.3	0.5	1.00	1.14
COD reduction (%)	-	21	34	47	74	75	-	15	23	38	77	85
Colour (absorption)	48	35	18	13	3.3	3.1	242	49	8.1	5	4.2	2.38
DFZ (436 nm), m <sup>-1</sup>	36	26	14	9	2.5	2.3	178	36	6.0	4	3.2	1.75
DFZ (525 nm), m <sup>-1</sup>	17	13	6.3	4.3	1.2	1.1	85	17	2.8	1.7	1.5	0.83
Colour reduction (%)	-	27%	63%	75%	93%	94%	-	80%	97%	98%	98%	99%
DFZ (436 nm), m <sup>-1</sup>	-	27%	63%	75%	93%	94%	-	80%	97%	98%	98%	99%
DFZ (525 nm), m <sup>-1</sup>	-	27%	63%	75%	93%	94%	-	80%	97%	98%	98%	99%
DFZ (620 nm), m <sup>-1</sup>	-	25	70	170	360	380	-	203	359	1300	1625	1950
Sludge generation mg l <sup>-1</sup>	-	25	70	170	360	380	-	203	359	1300	1625	1950

**Table 2.** Comparison of Capex and Opex between SE and RO concentrate

Sl. no.	Item description	SE (1 MLD)		RO concentrate (0.6 MLD)	
		INR/annum	INR kg <sup>-1</sup> of dyed fabric	INR/annum	INR kg <sup>-1</sup> of dyed fabric
<b>Capex</b>					
1.	Capital cost in INR	102,66,667	–	2,590,000	–
2.	Annuity @ 15% RoI for 15 years tenure	1,755,775	0.24	442,934	0.10
3.	Sub-total	1,755,775	0.24	442,934	0.10
<b>Opex</b>					
4.	Treatment chemicals	11,916,338	1.63	21,276,945	4.85
5.	Electrical energy	140,160	0.02	84,096	0.01
6.	Manpower	973,333	0.13	584,000	0.13
7.	Maintenance	205,333	0.02	31,080	0.01
8.	Sludge disposal cost	410,078	0.06	23,806,395	5.40
9.	Sub-total	13,645,242	1.86	45,782,516	10.45
10.	Total capex and opex	15,401,017		46,225,450	
	Capex + opex (Sl. no: 3 + 9) INR/m <sup>3</sup> of system inflow	42	–	211	–
11.	Capex + opex (Sl. no.: 3 + 9) INR kg <sup>-1</sup> dyed fabric	–	2.1	–	11.0

**Figure 5.** Comparison of Opex between SE and RO concentrate.

conditions<sup>26</sup>. Colour and COD removal efficiencies were increased when the H<sub>2</sub>O<sub>2</sub> dosage rate was higher than 125 mg l<sup>-1</sup>. Based on series of jar tests, greater reduction of COD was observed when the concentration of H<sub>2</sub>O<sub>2</sub> increased<sup>35</sup>. Similarly, the effect of H<sub>2</sub>O<sub>2</sub> on colour and COD reduction from RO concentrate was also studied. Figure 2d and Table 1 show the removal of colour and COD from the RO concentrate at different H<sub>2</sub>O<sub>2</sub> dosages (500, 1000, 1500, 2000, 2500 mg l<sup>-1</sup>). The pH was kept at 5.0 and Fe<sup>2+</sup> was at 100, 200, 300, 400, 500 mg l<sup>-1</sup>. After addition of H<sub>2</sub>O<sub>2</sub>, the colour and COD removal efficiency were increased to 99% and 85% respectively. According to eq. (7), the concentration of <sup>•</sup>OH is likely to increase cumulative to H<sub>2</sub>O<sub>2</sub> dosage, leading to amplified oxidation rates of organic matter present in the effluent. However, surplus H<sub>2</sub>O<sub>2</sub> interferes with the determination of COD. The free residual H<sub>2</sub>O<sub>2</sub> in the Fenton process consumes K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub>, leading to an increase of inorganic COD<sup>37</sup>. However, above the limiting point, <sup>•</sup>OH proficiently reacts with H<sub>2</sub>O<sub>2</sub> and yields HO<sub>2</sub> radicals, which are less reactive than <sup>•</sup>OH, as shown in eq. (7)



Therefore, increasing HO<sub>2</sub>, results in a negligible contribution<sup>10</sup>.

### Effect of Fe<sup>2+</sup> and H<sub>2</sub>O<sub>2</sub> concentration on sludge generation

Besides the efficiency of the Fenton process, a study on sludge generation was also conducted. As shown in Figure 3a and b and Table 1, while treating SE, the sludge generation gradually increased up to 380 mg l<sup>-1</sup>. Based on the regression analysis performed ( $R^2 = 0.94$ ), it is reported that the maximum reduction in COD and colour has a direct impact on the generation of sludge. Therefore, the quantity of sludge generation indicates the efficiency of the Fenton process in the reduction of organic pollutants in the effluent. Figure 3b shows that while the generation of sludge (i.e. 360 mg l<sup>-1</sup>) increases, the COD and colour reduction proportionally increase up to 75% and 94% respectively. This sludge would require additional expenditures for disposal in effluent treatment plants. Likewise, the same study was carried out for the RO concentrate (Figure 3c-e and Table 1). A high amount of sludge generation (1950 mg l<sup>-1</sup>) was observed at 500 mg l<sup>-1</sup> of Fe<sup>2+</sup> and 2500 mg l<sup>-1</sup> of H<sub>2</sub>O<sub>2</sub>. Based on the data obtained in the experiments, a regression analysis was carried out, and a positive correlation was found between the concentration of Fe<sup>2+</sup> and H<sub>2</sub>O<sub>2</sub> and the quantity of sludge generated, i.e.  $R^2 = 0.92$ . COD and colour reduction of about 85% and 99% respectively, were observed at a sludge concentration of 1950 mg l<sup>-1</sup>. Further, a comparative study for sludge generation was also conducted between SE and RO concentrates. As shown in Figure 3e, higher sludge generation was found in the RO concentrate than SE. This indicates the presence of higher concentration of organics in the

RO concentrate, which leads to higher consumption of Fenton reagent and generation of chemical sludge.

### *Comparison of specific consumption of $Fe^{2+}$ and $H_2O_2$ on the reduction of COD from SE and RO concentrate*

In order to estimate efficient utilization of the Fenton reagent by the organic substance present in the effluent, a specific consumption rate of  $Fe^{2+}$  and  $H_2O_2$  by COD was studied in all trials of both samples. In both cases, consumption of  $Fe^{2+}$  and  $H_2O_2$  varied from 0.3 to 0.5 kg and from 2.2 to 2.5 kg/kg of COD destruction, respectively (Figure 4 a and b).

Further, regression analysis was performed to find if a correlation exists between consumption of the Fenton reagent and COD reduction rate for both the cases, and it was found to be  $R^2 = 0.92$  and  $0.84$  respectively (Figure 4 a and b). This indicates that maximum COD reduction was obtained only when the concentration of Fenton reagent increased (Table 1).

### *Process economics*

After completing the bench scale experiments, the economic feasibility of the Fenton process was evaluated to assess the suitability of the system for treating of SE and RO concentrate. Capex (capital expenditure) and Opex (operational expenditure) were estimated for both streams for the typical capacity of 1 MLD (million litres/day) and 0.6 MLD of SE and RO concentrate respectively. At the capex of both systems, all civil, electromechanical and instrumentation items were considered and annuity on capital was worked out for 15-year tenure with a rate of interest (ROI) of 15%/annum. Annuity on the capital cost was calculated based on eq. (8)

$$\text{Annuity on capital cost: } \frac{P_i(1+i)^n}{(1+i)^n} \quad (8)$$

where  $P$  is the total capital cost,  $i$  the rate of interest and  $n$  is the life of components in years.

Similarly, in the opex head, all consumables such as chemicals, manpower, electrical energy, maintenance cost and sludge disposal cost were considered. As shown in Table 2, Capex and Opex are significantly lower in treating of SE, i.e. INR 42/m<sup>3</sup> of system inflow or INR 2.1/kg of dyed fabric produced, whereas in the RO concentrate stream, it is about INR 210/m<sup>3</sup> of system inflow or INR 11/kg of dyed fabric produced. Capex and Opex on dyed fabric were calculated based on effluent generation of 50 litres/kg of dyed fabric (i.e. 1 m<sup>3</sup> of effluent would be generated for each 20 kg of dyed fabric in soft

flow machines with an M : L (material to liquor) ratio of 1 : 5). From Table 2, it can be deduced that implementation of the Fenton process in SE is the most sustainable option when compared to the RO stream. In Figure 5, treatment costs for all trials of both samples are indicated through a bar chart. From this figure, it is seen that higher operating cost was in sample-5 of both streams, where maximum COD and colour reduction were obtained.

Capex and Opex for both streams were calculated based on the colour and COD reduction achieved in sample-5 of SE and RO concentrate.

### **Conclusion**

The treatment of SE and RO concentrate by the Fenton oxidation was considered in this study. The experimental result shows that reduction in COD and colour from both samples increased while the concentration of  $Fe^{2+}$  and  $H_2O_2$  increased. The ideal values of the functional factors during the oxidation of the colour and COD in the SE by the Fenton's process are  $Fe^{2+} = 125\text{--}150 \text{ mg l}^{-1}$ ,  $H_2O_2 = 750 \text{ mg l}^{-1}$ , pH = 5.0. Likewise, in RO concentrate, ideal values of the functional factors during oxidation of colour and COD by the Fenton process are  $Fe^{2+} = 500 \text{ mg l}^{-1}$ ,  $H_2O_2 = 2500 \text{ mg l}^{-1}$  and pH = 5.0. Complete decolourization of the colour does not mean that the organics are completely degraded. Hence the degradation of organics in terms of COD removal was also examined. From the figures and tables, it is seen that maximum colour and COD values were obtained when  $Fe^{2+}$  and  $H_2O_2$  were consumed at the rate 0.5 kg/kg COD (SE) and 2 kg/kg COD (RO concentrate) respectively.

Apart from the treatment efficiency point of view, cost wise comparison was also conducted to determine an economically sustainable option for treating the textile effluent. Finally, it was found that treating SE through the Fenton process is more viable than treating RO concentrate, because sludge generation (380 mg l<sup>-1</sup>) and operating cost (INR 42/m<sup>3</sup>) were lower than with RO concentrate. In addition, treating of secondary treated effluent prior to the RO system would be an added advantage, since most of the organics would be removed, resulting in minimal/no fouling of RO membranes, which in turn increases membrane life by reducing the need for chemical cleaning of RO membrane.

This process can be employed to treat and polish SE, especially in textile dyeing-based ZLD effluent treatment plants, where higher-grade water and salt are recovered for reuse in the dyeing process. The Fenton process is suitable for treating coloured effluents effectively. Moreover, this method is cost-effective and generates less sludge than conventional methods of chemical treatment.

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