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Study of Fenton Oxidation Process on Dyeing Wastewater and Improving its Catalysis

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Abstract— *The aim of this study was to find optimum dose of ferrous sulphate and hydrogen peroxide in Fenton oxidation process for treatment of dyeing wastewater. Fenton process is the advanced oxidation method used mainly for COD and colour removal. Experiments are done on synthetic wastewater prepared from three different dyes; acidic, disperse and reactive; and also effluent obtained from industry. The maximum removal of effluent COD was obtained more than 95% in Fenton treatment. In the second part of the study is find alterations to improve the Fenton treatment and the study the sludge generation and colour of wastewater after treatment. Experiments are done on synthetic wastewater prepared from four different dyes; acidic, disperse, reactive and azo dye; and also effluent obtained from industry. Ferric chloride and ferric nitrate showed better efficiency than conventional method of using ferrous sulphate as catalyst in treatment. The sludge generated was found to be in par with the COD removed during treatment. Fenton treatment was found to give its own colour to wastewater.*

Index Terms— — Advanced oxidation process, COD, Colour removal , Dyeing wastewater, Fenton treatment, Ferrous sulphate, Hydrogen peroxide

1 INTRODUCTION

Dyeing industry has become a major source of pollution due to increasing demand of textile products resulting proportional increase in production and application of synthetic dyes. More than 10,000 commercially available dyes are used in dyeing unit [1]. Textile industry can be classified into three categories cotton, woollen and synthetic fibres depending upon the raw materials used. The textile dyeing industry consumes large quantities of water and produces large volumes of wastewater from different steps in the dyeing and finishing processes. Wastewater from printing and dyeing units is often rich in colour, containing residues of reactive dyes and chemicals, such as complex components, many aerosols, high chroma, high COD and BOD concentration as well as much more hard-degradation materials. The toxic effects of dyestuffs and other organic compounds, as well as acidic and alkaline contaminants from dying industry on the general public are widely accepted. At present the dyes are mainly aromatic and heterocyclic compounds with colour display groups and polar groups. The structure is more complicated and stable resulting in greater difficulty to degrade the printing and dyeing wastewater [2].

Fenton oxidation process

The Advanced Oxidation Processes (AOPs) are alternative processes involving the generation of highly oxidizing radicals, specially the hydroxyl radical ($OH^{\bullet}$, E_0 =2.8 V versus NHE), capable of oxidizing high recalcitrant contaminants to CO_2 and H_2O. Among the different existing AOPs, the Fenton process arises as very promising and alternative wastewater technique that can be applied to the remediation of textile wastewaters, either alone or in combination with an aerobic biological treatment (pre or post biological treatment). The process is highly effective in the degradation of toxic and/or non-biodegradable compounds (e.g. aliphatic compounds, nitroaromatics, azo-dyes, phenols, chlorobencene, etc.) and has a special interest because it make possible the achievement of high reactions yields with a low cost treatment [3]. The generally accepted mechanism of the Fenton process proposes that hydroxyl radicals are produced in accordance with Equation (1), while the catalyst is regenerated through Equation (2), or from the reaction of Fe^{3+} with intermediate organic radicals (Equations (3)–(5)):

$$Fe^{2+} + H_2O_2 \rightarrow Fe^{3+} + HO^{\cdot} + HO^{-}$$
$$k = 76\ L\ mol^{-1}\ s^{-1} \qquad (1)$$
$$Fe^{3+} + H_2O_2 \rightarrow Fe^{2+} + HO^{2\cdot} + H^{+}$$
$$k = 0.01\ L\ mol^{-1}\ s^{-1} \qquad (2)$$
$$RH + HO^{\cdot} \rightarrow R^{\cdot} + H_2O \qquad (3)$$
$$R^{\cdot} + Fe^{3+} \rightarrow R^{+} + Fe^{2+} \qquad (4)$$
$$R^{+} + HO^{-} \rightarrow R\text{-}OH \qquad (5)$$

2 MATERIAL AND METHODOLOGY

Lab scale reactor

One litre batch reactor was used for both Fenton treatments. The reactor was cylindrical in shape with internal diameter 10.5cm and height 14cm with an effective volume of 1 litre. The reaction is done on jar test apparatus with similar reactors together. The experiments were done at room temperature around 30 to 35^0C. The wastewater was prepared, filled in reactor in required quantity (500 mL) and the reagents required were fed to the reactor. Then the reactor was kept in jar test for experiments.

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Synthetic dyeing wastewater

The composition and characteristics of synthetic wastewater is given in table 1. Four different dyes were used individually to prepare wastewater of each dye. The dyes used were: Colocid Rhodamine B Conc (acidic dye), Colocid Yellow FG (reactive dye), Coralene Yellow C4G (disperse dye) and Corafast Blue G (azo dye).

Table 1.
Characteristics of simulated wastewater

Materials used	Conc. (mg/L)	Functions of material
Dye	250	Dyeing
NH_4Cl	200	Helps in fixation
NaCl	1000	Fixing the dye
Na_2CO_3	1000	Fixing the dye
$NaHCO_3$	2000	pH buffer
Starch	750	Sizing in reactor
Glucose	1000	Substrate for bacterial growth
Acetic acid	500	Substrate in reactor
Sulphuric acid	357	pH neutralization
KH_2PO_4	250	Nutrient
$CaCl_2.2H_2O$	10	Nutrient

Each chemical were added in required concentration to make the required quantity of wastewater.
The average COD of wastewater prepared from different dyes is given in Table 2.

Table 2.
COD of wastewater from different dyes

Dye	Type of dye	COD (mg/L)
Coralene Yellow C4G	Disperse	1668 ± 156
Colocid Rhodamine B Conc	Acidic	1779 ± 84
Colocid Yellow FG	Reactive	2136 ± 216
Corafast Blue G	Azo	2080 ± 160

Experimental setup and procedure
se for all the three dyes where found out as described above. The required quantity of wastewater required for the experiment is predetermined. The same quantity of tap water is taken in a container. The chemicals required to make the wastewater is added to this water in required quantity as given in table 1. The wastewater is mixed well for uniform concentration. One litre of wastewater is filled in each reactor. The initial COD of wastewater is found out.
In Fenton treatment, the pH of wastewater is made to 3 using concentrated sulphuric acid. Then the catalyst, ferrous sulphate, $FeSO_4.7\ H_2O$ is added in required quantity and mixed well. Then hydrogen peroxide, H_2O_2 (35% solution), is added in required quantity and mixed. The reactors are placed in jar test apparatus and stirred at 150 rpm for 1 hour. The effluent is kept for settling of sludge in imhoff cone and supernatant is taken. The COD of supernatant of each reactor is measured and optimum dose if found out.
In activated carbon treatment, powdered activated carbon is added to each reactor of concentrations 0.6, 0.8, 1.0, 1.2 and 1.4 g/L. The reactors are placed in jar test apparatus and stirred at 150 rpm for one hour. The effluent is filtered using filter paper and the effluent COD is measured. The activated carbon concentration which gives the maximum removal is the optimum dose.

Analysis of effluent
The wastewater after Fenton reaction was taken from the jar test apparatus and its pH was made above 7 by adding NaOH solution. This will ensure that no further Fenton reaction takes place. The effluent was poured into an imhoff cone and was allowed to settle for 1 hour. The supernatant was taken and COD was found out.
In case of activated carbon treatment, after one hour reaction time the wastewater was filtered by using a filter paper and the COD of filtrate was found out.
COD was measured in closed COD apparatus. Sample is diluted 5 times. The COD was done as per standard method (APHA Standard).

3 RESULTS AND DISCUSSION

3.1 Optimum doses

Initially the optimum dose of hydrogen peroxide and ferrous sulphate for the 3 different dyes were found out. Then the optimum activated carbon dose for all the three dyes where found out as described above.

Disperse dye
The disperse dye used for experiment was Coralene Yellow C4G. The wastewater was prepared from this dye as per previously mentioned composition. The dye concentration was 250 mg/L.
Initially the hydrogen peroxide concentration was fixed at 6 g/L and the ferrous sulphate concentration was changed to get optimum value for maximum COD removal. The percentage reduction in COD for different catalyst concentration is given below in figure 1.

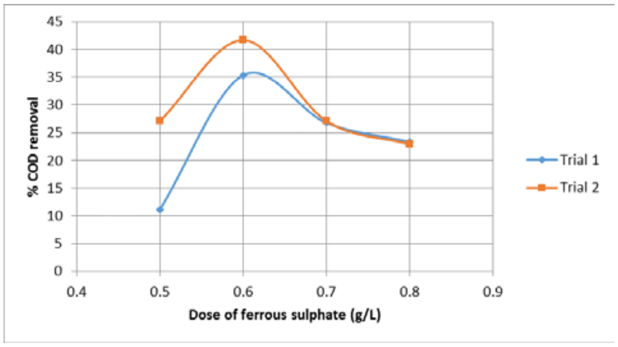

Fig1: Optimum ferrous sulphate concentration

The optimum dose of ferrous sulphate was found to be 0.6 g/L for disperse dye.

Now with this optimum dose of catalyst, the hydrogen peroxide dose was changed and its optimum dose was found out. The various dose and the percentage reduction in COD is given in figure 2.

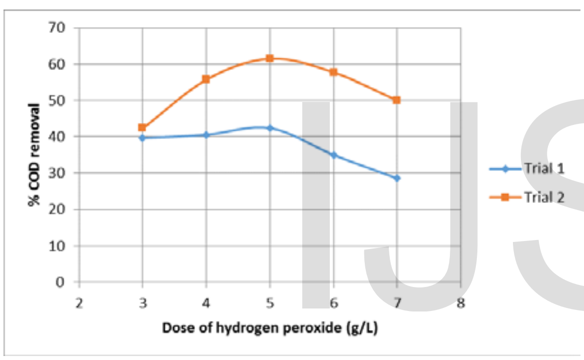

Fig 2: Optimum hydrogen peroxide concentration

The optimum dose of hydrogen peroxide was found to be 5 g/L. To get more precise value of optimum dose of hydrogen peroxide, doses near to 5 g/L is studied. Tests are done with hydrogen peroxide values lesser and greater than the optimum value obtained.

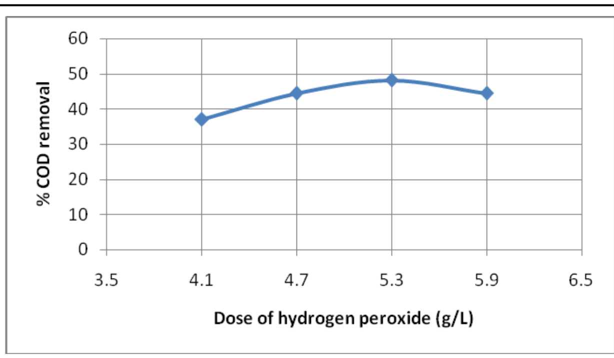

Fig 3: Optimum hydrogen peroxide concentration

Optimum dose of hydrogen peroxide can be found to be 5.3 g/L. Thus for Fenton reaction of disperse dye optimum dose of hydrogen peroxide is 5.3 g/L and that of ferrous sulphate is 0.6 g/L. At the optimum dose maximum removal of more than 61% was obtained by Fenton treatment.

Acidic dye

The acidic dye used was Colocid Rhodamine B conc. The wastewater prepared using this dye had dye concentration 250 mg/L. Initially the hydrogen peroxide concentration was fixed at 6 g/L and the ferrous sulphate concentration was changed to get optimum value for maximum COD removal. The optimum dose of ferrous sulphate was found to be 0.7 g/L for acidic dye.

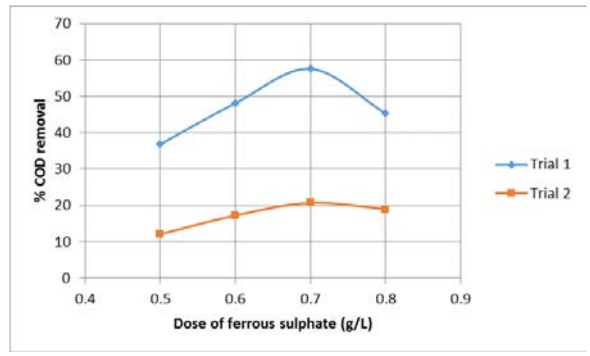

Fig 4: Optimum ferrous sulphate concentration

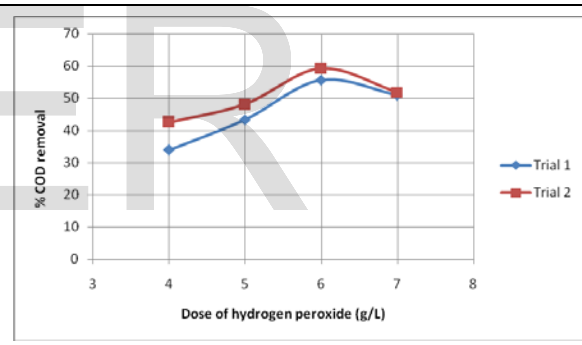

Fig 5: Optimum hydrogen peroxide concentration

Now with this optimum dose of catalyst, the hydrogen peroxide dose was changed and its optimum dose was found out. The optimum hydrogen peroxide dose for disperse dye was found to be 6 g/L.

To get more precise value of optimum dose of hydrogen peroxide, doses near to 6 g/L was studied.

From the figure 6, the optimum dose of hydrogen peroxide was found to be 6.3 g/L. Thus for Fenton reaction of acidic dye optimum dose of hydrogen peroxide is 6.3 g/L and ferrous sulphate is 0.7g/L.

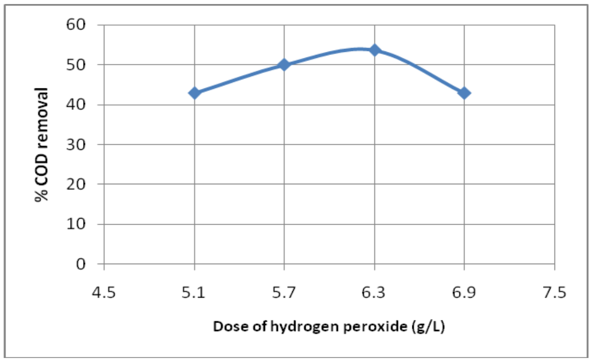

Fig 6: Optimum hydrogen peroxide concentration

Reactive dye

Reactive dyes have good fastness properties owing to the bonding that occurs during dyeing. Tests were done to find optimum value of ferrous sulphate required in Fenton process for maximum COD removal. The concentration of hydrogen peroxide was fixed at 6 g/L and concentration of ferrous sulphate was varied.

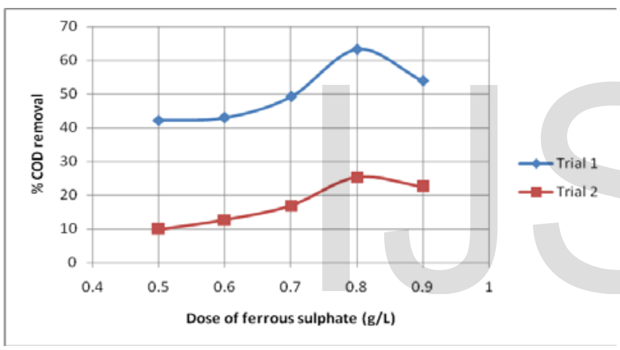

Fig 7: Optimum ferrous sulphate concentration

From the figure 7, the optimum dose of ferrous sulphate was obtained to be 0.8 g/L. With this optimum value of ferrous sulphate various concentrations of hydrogen peroxide was tested to find its optimum values.

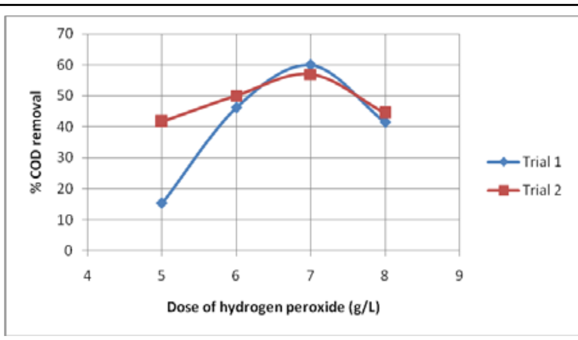

Fig 8: Optimum hydrogen peroxide concentration

From the figure 8, the optimum dose of hydrogen peroxide was obtained as 7 g/L for reactive dye in each trial. To get more accurate optimum value tests were conducted on concentrations closer to 7 g/L.

From figure 9, the maximum removal was obtained at hydrogen peroxide concentration of 6.7 g/L. Thus for reactive dyeing wastewater optimum dose is 0.8 g/L ferrous sulphate and 6.7 g/L hydrogen peroxide.

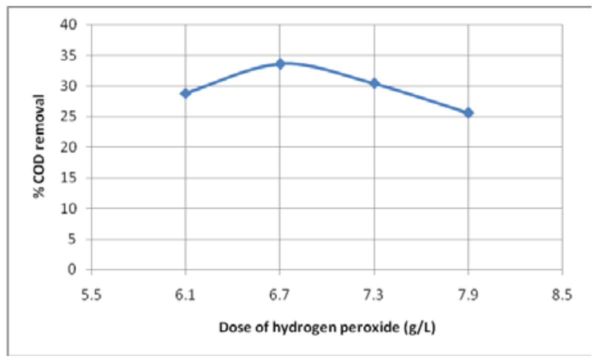

Fig 9: Optimum hydrogen peroxide concentration

Azo dye

Azo dyes contains nitrogen as azo group as part of their molecular structures; more than half the commercial dyes belong to this class. In this experiment, the wastewater produced from azo dye was treated using Fenton reagent. The concentration of hydrogen peroxide was fixed at 6 g/L.

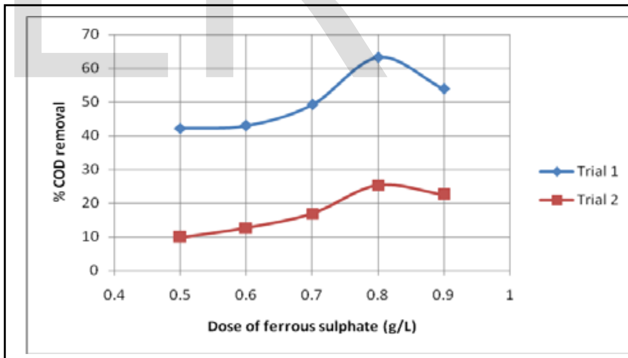

Fig 10: Optimum ferrous sulphate concentration

From the figure 10, the optimum dose of ferrous sulphate was obtained to be 0.8 g/L. With this optimum dose of ferrous sulphate, various concentrations of hydrogen peroxide were tested to get its optimum value.

From the figure 11 the optimum dose of hydrogen peroxide was obtained to be 7 g/L. Experiments were done to get more precise value.

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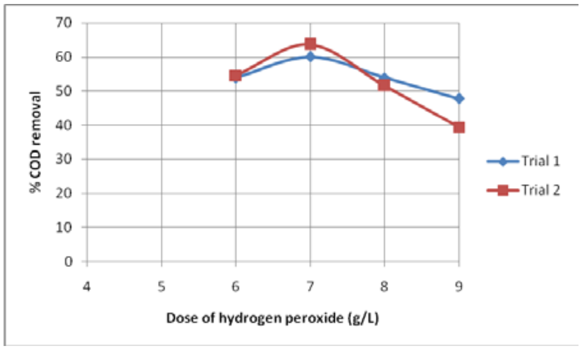

Fig 11: Optimum hydrogen peroxide concentration

The optimum dose of hydrogen peroxide was obtained to be 7 g/L. Experiments were done to get more precise value. For this doses above and below 7 g/L was selected in equal intervals and the same procedure was done. Thus for azo dye optimum ferrous sulphate dose is 0.8 g/L and optimum hydrogen peroxide dose obtained as 6.7 g/L.

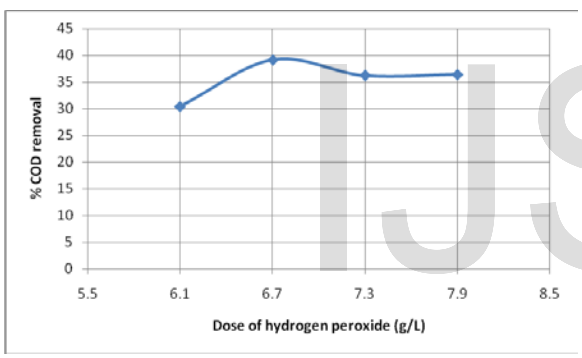

Fig 12: Optimum hydrogen peroxide concentration

3.2 Improved fenton process

In Fenton treatment, usually ferrous sulphate is used as catalyst. Study was done to find out other compounds that can be used as catalyst for Fenton reaction. For this, wastewater was prepared with a mixture of dyes and various salts were tested. The different salts used and their efficiency is given in table 3.

From the results, ferric chloride and ferric nitrate showed much better results than ferrous sulphate in removing COD from wastewater.

Table 3

Testing efficiency of various salts for Fenton treatment

Compounds tested	Percentage reduction in COD	Result
$FeSO_4.7H_2O$	61.38	Positive result
$FeCl_3$	95.04	Positive result
$CuSO_4.5H_2O$	24.74	Less efficient
$MnSO_4.H_2O$	0	No reaction
$ZnSO_4.7H_2O$	3.8	Less efficient
$Fe(NO_3)_3$	87.63	Positive result

Different dyes were compared with ferric chloride and ferric nitrate along with ferrous sulphate and their relative efficiencies were compared.

Disperse dye

Table 4

Comparing various catalysts

Catalyst	% reduction in COD	% increase in efficiency
$FeSO_4.7H_2O$	46.6	
$FeCl_3$	60	22.33
$Fe(NO_3)_3$	53	12.08

Acidic dye

Table 5

Comparing various catalysts

Catalyst	% reduction in COD	% increase in efficiency
$FeSO_4.7H_2O$	52.63	
$FeCl_3$	57.89	9.09
$Fe(NO_3)_3$	55.26	4.76

Reactive dye

Table 6

Comparing various catalysts

Catalyst	% reduction in COD	% increase in efficiency
$FeSO_4.7H_2O$	58.33	
$FeCl_3$	61.88	5.73
$Fe(NO_3)_3$	59.22	1.50

Azo dye

Table 7
Comparing various catalysts

Catalyst	% reduction in COD	% increase in efficiency
$FeSO_4.7H_2O$	53.33	
$FeCl_3$	73.33	27.27
$Fe(NO_3)_3$	66.67	20.0

From these results it is obvious that ferric chloride and ferric nitrate prove to be a better catalyst that ferrous sulphate for the four types of wastewater. Thus they can be used as a better catalyst than the conventional Fenton treatment using ferrous sulphate as catalyst. From the observations made, the sludge produced by ferrous sulphate takes more time to settle completely.

3.3 Dyeing industry wastewater

Dyeing wastewater was obtained from one dyeing industry near Surat, Gujarat. The experimental procedure was similar to that of synthetic wastewater prepared earlier. The wastewater was having a dark navy blue colour. The initial properties of wastewater were studied.
Initial COD = 960 mg/L
Initial pH = 11.52

Fenton treatment

The dyeing wastewater was taken and its pH was made to 3. The initial COD was found to be 960 mg/L. Initially the hydrogen peroxide concentration was fixed at 6 g/L and the concentration of ferrous sulphate was varied to find its optimum value.

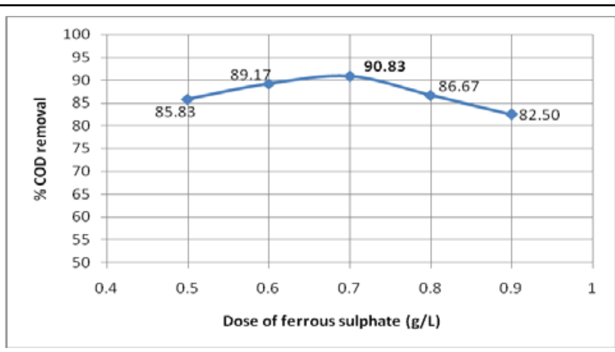

Fig 13: Optimum ferrous sulphate concentration

From the results obtained the optimum dose of ferrous sulphate was found to be 0.7 g/L. With this optimum dose the concentration of hydrogen peroxide was varied to find the optimum value. The initial COD of sample was 912 mg/L.

From the results obtained the optimum hydrogen peroxide dose was 7 g/L. Thus for the dyeing wastewater, the optimum

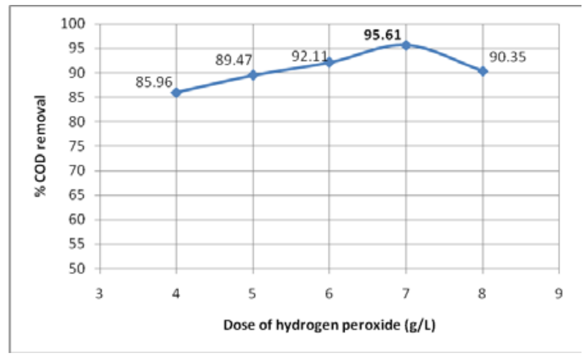

Fig 14: Optimum hydrogen peroxide concentration

ferrous sulphate dose was 0.7 g/L and that of hydrogen peroxide is 7 g/L.

Improvements in Fenton process

After the treatment of Fenton process with ferrous sulphate as catalyst, experiments were done with ferric chloride and ferric nitrate as catalyst.

Table 8
Improved Fenton process

Catalyst	*Final COD (mg/L)*	*% reduction*
$FeSO_4.7H_2O$	42	95.39
$FeCl_3$	34.67	96.20
$Fe(NO_3)_3$	133.37	85.38

From the results ferric chloride was the best catalyst in treatment of the dyeing wastewater. It had more than 96% removal efficiency and the treated wastewater had COD less than permissible discharge standards. It was observed that treatment using ferric nitrate produce lesser sludge than by using conventional ferrous sulphate as catalyst. The ferrous sulphate produced 0.408 g/L of sludge while ferric chloride produced 0.322 g/L of sludge. More over sludge produced after ferric chloride treatment settles faster than other catalyst produced sludge. Thus for effective treatment of dyeing wastewater it is better to use ferric chloride as catalyst.

3.4 Sludge generation

One of the main disadvantages of Fenton process is the production of sludge after treatment. The COD and colour removed from the wastewater settles as concentrated sludge. The quantity of sludge produced is directly proportional to the COD removed in treatment. Usually for 1 litre of wastewater treated the volume of sludge produced will be around 35 to 45 ml after complete settlement. Thus the major issue involved is the treatment and disposal of sludge. Various studies have been going on in reuse of sludge. But most of these methods are either inefficient or too costly.

3.5 Quantity of sludge produced

As mentioned earlier, the quantity of sludge produced depends upon the COD removed.

Table 9
Quantity of sludge produced

Dye	*Type*	*Fenton reaction* Optimum hydrogen peroxide conc. (g/L)	*Fenton reaction* Optimum ferrous sulphate conc. (g/L)	*Sludge produced (mL)*
Coralene Yellow C4G	Disperse	5.3	0.6	40
Colocid Rhodamine B Conc	Acidic	6.3	0.6	37
Colocid Yellow FG	Reactive	6.7	0.8	38
Corafast Blue G	Azo	6.7	0.8	41
Industrial WW	Mixture of dyes	7.0	0.7	30

The important observations made was that even after settling majority of sludge consists of water itself. When the 30mL/L of sludge produced from industrial wastewater was dried in hot air oven at 105°C, the weight of sludge produced was 0.408 g/L

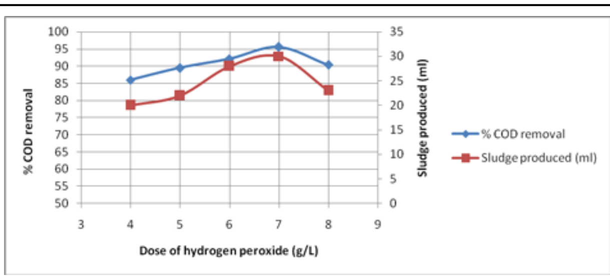

Fig 14: Quantity of sludge generated

3.6 Settling of sludge

After Fenton treatment the wastewater was poured into imhoff cone to allow sludge to settle. Usually it takes 4 to 5 hours retention time for complete settlement of sludge. After this period, there will be clear difference between sludge and supernatant. The supernatant will have low COD and colour.

In order to increase the speed of settling, alum can be used. It was observed that when alum solution was added to the treated wastewater, complete settlement takes place within one hour.

3.7 Colour of supernatant after Fenton treatment

It was observed that after Fenton treatment, irrespective of colour of dye the supernatant after settling has slight yellow colour. So absorbance test was done to find the absorbance value of each treated wastewater. The wave length of yellow was selected at 575nm.

From the table it is clear that disperse and acidic dye have more colour than reactive and azodye. Thus the Fenton is more efficient in removing colour of reactive and azo dye. Experiment was done to see if Fenton reaction itself is giving any colour to the treated wastewater. For this wastewater was prepared without adding any dye and Fenton treatment was carried out with 0.7 g/L ferrous sulphate and 7 g/L hydrogen peroxide. It was observed that the resultant wastewater was having slight yellow colour as for dyeing wastewater. At 575nm, it had an average absorbance of 0.0007. Thus Fenton itself is giving colour to the wastewater. It can be concluded that the colour present in treated wastewater can be due to incomplete reaction of Fenton reagents. In order to remove this colour further treatment is required as Fenton treatment proved that it cannot remove colour by 100%, moreover it produces its own colour.

Table 10
Absorbance of treated wastewater

Dye	*Type*	*Average absorbance*
Coralene Yellow C4G	Disperse	0.0170
Colocid Rhodamine B Conc	Acidic	0.139
Colocid Yellow FG	Reactive	0.036
Corafast Blue G	Azo	0.0068

4 Conclusion

Treatment of dyeing wastewater is a challenge due to its varying effluent characteristics, high COD and colour. The stringent rules make it important to have more efficient treatment technologies. The optimal doses of hydrogen peroxide and ferrous sulphate for four different types of dyes are studied and study on sludge produced and colour of treated wastewater is also done. Different types of dyes showed different affinity for the Fenton reaction and COD removal. For disperse dye, the optimum dose of ferrous sulphate and hydrogen peroxide is found to be 0.6 g/L and 5.3 g/L respectively, which can be effective for 61.5 % COD removal. At the same time 40 mL/L of sludge is generated. For acidic dye the optimum doses of ferrous sulphate and hydrogen peroxide are 0.6 g/L and 6.3 g/L respectively with 59.26 % COD removal and 37 mL/L of sludge. For reactive dye, the optimum dose of ferrous sulphate and hydrogen peroxide doses are 0.8 g/L and 6.7 g/L which is effective for 63.18 % COD removal with 38 mL/L of sludge generated after treatment. For Fenton treatment of azo dye the optimum ferrous sulphate and hydrogen peroxide doses are 0.8 g/L and 6.7 g/L respectively with 63.64 % COD removal generating 41 mL/L of sludge. For dyeing wastewater, optimum ferrous sulphate dose is 0.7 g/L and hydrogen peroxide dose is 7 g/L. The removal efficiency is more than 95% and the treated wastewater was having lower COD below permissible limits. The sludge produced was 30 mL/L of wastewater treated at optimum doses. The removal, treatment and disposal of sludge is a major concern in case of Fenton treatment The sludge produced is rich in colour and COD. So special treatment methods are required before disposal. The Fenton treatment also gives residual colour after treatment of wastewater. Thus using Fenton as a pre-treatment method and followed by using another treatment option will give better results. This can reduce the quantity of sludge produced also, as the quantity of sludge produced depends upon the catalyst and reagents added.

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