

# Heterogeneous Fenton and Microfiltration for the Treatment of Textile Waste Water

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**Abstract** - The textile industry produces wastewater that contributes to the pollution of water since it uses a lot of chemicals. This study investigates the use of integrated processes of separation of the heterogeneous Fenton and microfiltration membrane to handle the wastewater. The efficacy of flyash is tested in heterogeneous Fenton pretreatment analysis. Study will research the effects of some primary operating parameters such as catalyst dose, pH, hydrogen peroxide concentration, reaction time on the efficiency of the procedure. It will figure out the optimal pH levels, catalyst dosage, and treatment period. Chemical oxygen demand and efficiencies for removing color would also be calculated. The post-treatment was performed with microfiltration membrane cellulose acetate. The pH and flow rate effect for 0.12 m/s, 20 m/s, 50 m/s was systematically analyzed. It discusses the impact of pH and flow rate on filtration cycle. Analysis will be found for the removal efficiency of the combined heterogeneous Fenton and microfiltration system if the process is appropriate for the treatment of textile effluent. The cod removal and colour removal was 80.4% and 88.6% respectively

**Key Words:** Heterogeneous Fenton, Flyash, Filtration, Microfiltration, Hydrogen peroxide.

## 1. INTRODUCTION

The textile business is one of the biggest assembling ventures. In each phase of textile industry different sorts of colors are utilized to shading their items. The color containing wastewater is typically discharged straightforwardly into the close by channels, waterways, stale, lakes or tidal ponds. Such wastewater removal may make harm the nature of the getting water bodies, the seagoing eco-framework and the biodiversity of condition. The coloring business effluents contain high BOD and COD worth, suspended solids, harmful mixes and the shading that is seen by natural eyes at low focus. In handling of materials, the industry utilizes various colors, synthetic compounds, helper synthetic compounds and measuring materials. Accordingly, debased waste water is produced which can cause natural issues except if appropriately treated before its removal. Pollutants released by the global textile industry are uninterruptedly doing unimaginable harm to the environment. It pollutes land and makes them useless and barren in the long run. Textile manufacturing units also release hazardous waste into the nearby land.

Conventional wastewater treatment that incorporates dyes requires biological oxidation, chemical oxidation, and adsorption. Biological methods are usually inexpensive and easy to implement, and are currently being used to extract organics and color from dyeing and textile wastes. However, modern biological processes such as activated sludge processes cannot easily remove this wastewater, since the structure of most industrial dye compounds is typically complex and many dyes are non-biodegradable due to their chemical existence and molecular scale. At present, several methods have been developed to treat textile wastewater but they cannot be used individually because this wastewater has high salinity, color and non-biodegradable organics. In coagulation process, large amount of sludge is created which may become a pollutant itself and increase the treatment cost. Oxidation process such as ozonation effectively decolorizes almost all dyes except disperse dyes, but does not remove COD effectively. Electrochemical oxidation produce pollutants which increases the treatment cost. Within AOPs, the Fenton process is an interesting solution, because it allows high rates of degradation at room temperature and pressure conditions using cheap chemicals and simple reactants to handle. [1] Oxidation of Fenton is a homogeneous catalytic oxidation process which uses a mixture of hydrogen peroxide and ferrous ions. Production of highly oxidizing species can be enhanced by applying solar light to Fenton reactants, promoting sustained chemical reactions, and increasing performance relative to other AOP-based methods. This cycle is known as solar photo-Fenton oxidation. There is no single process capable of adequate treatment mainly due to the complex nature of these effluents. The use of combined processes has been suggested recently to overcome the disadvantage of individual unit processes. Most of the existing processes include an initial step of activated sludge treatment to remove the organic matter followed by oxidation, UV radiation, membrane separation, or adsorption. [2]

### 1.1 Objective of study

- (1) To study the effectiveness of fly ash in Heterogeneous Fenton pretreatment
- (2) To find out the efficiency of combination of Heterogeneous Fenton and Microfiltration in treatment of textile waste water.

## 2. METHODOLOGY

### 2.1 Sample Collection

The 20L of textile waste water was collected from Handloom Development Centre, Vatakara. Industrial water demand is about 40 to 50 m<sup>3</sup>/day, and the dyeing process produces 20 to 30 m<sup>3</sup> of colored wastewater every day while processing cotton. Methylene blue, Remazol Red F 3B, Olive R, and Ramazol blue CG are the dyes commonly used by the industry. The wastewater Samples were collected in an air tight container. The samples are Closed and sealed tight to avoid any oxidation and contamination. Samples were stored in refrigerator to avoid further biodegradation at 4°C and analyzed.

### 2.2 Analytical methods

The parameters such as pH, COD, BOD<sub>5</sub>, TSS, TDS, Alkalinity, Turbidity, Sulphides, Chlorides, and Color were analyzed as the characteristics were determined by standard method procedures.

**Table -1:** Instruments and methods for analysis

Parameters	Instrument	Method
pH	Water quality analyzer	Electrometric method
Alkalinity		Double end point titration method
turbidity	turbidimeter	Nephelo turbidity method
chloride		Mohr method
BOD	BOD incubator	Winkler method
COD	Reflux apparatus	Potassium dichromate reflux method
TDS	Water quality analyzer	Electrometric method
sulphide	Iodometric method	
color	Colorimetric Method	Double beam U V Spectrophotometer

### 2.3 Catalyst preparation

Treatment with heterogeneous Fenton is called a pretreatment process. Pretreatment was achieved using catalyst fly ash.

Fly ash were obtained from NAATH ready mix materials, Thamarasery for the heterogeneous catalyst preparation. The samples obtained were made clear of all impurities and then they were sieved using a 200 micron sieve. Catalyst was

prepared by the incipient process of impregnation. In 20 mL of distilled water 6 g of FeSO<sub>4</sub>.7H<sub>2</sub>O dissolved. After that, the solution was applied with 10 g of fly ash. The mixture was agitated in a water bath and heated at 100 °C before the water finished evaporating. The sample was overnight dried in an oven at 100 °C. [3].

### 2.4 Chemicals

The chemicals used in the experiment include 1N H<sub>2</sub>SO<sub>4</sub> or NaOH to change the pH to produce peroxide radicals by 30 percent H<sub>2</sub>O<sub>2</sub>. Additionally, 0.1N Sodium bisulphite (NaHSO<sub>3</sub>) was applied to avoid the H<sub>2</sub>O<sub>2</sub> reaction.

### 2.5 Experimental procedure

The experiment with the Heterogeneous Fenton was performed in a glass beaker with ability 1L. The sample volume used had been 250 mL. Before startup, the desired pH value was changed using either 1N H<sub>2</sub>SO<sub>4</sub> or NaOH. Then a given weight of prepared fly ash catalyst was added and very well mixed with wastewater for 10 min. and added given volume of H<sub>2</sub>O<sub>2</sub>. The experimental setup was placed in shaker or jar test apparatus. The reaction time begins with the addition of H<sub>2</sub>O<sub>2</sub> and thorough mixing of the reagents is necessary. Upon reaching the necessary contact time 0.1N Sodium bisulphite (NaHSO<sub>3</sub>) was added to stop the H<sub>2</sub>O<sub>2</sub> reaction. The determining factors for the Fenton reaction are pH, H<sub>2</sub>O<sub>2</sub> catalyst concentration and reaction time. Several trials have been performed based on these factors.

The membrane filtration experiments took place at varying pH and flow levels. Adding the dilute 0.1 M NaOH or H<sub>2</sub>SO<sub>4</sub> changed the pH solution to the correct pH. Many separation systems use pressure gradient as the main driving force, but here gravitational force has been taken as the principal driving force. And during the study a 6 cm head was held in feed chamber. These pretreated samples were passed through the jar containing the membrane, and spectrophotometer was used to determine the concentrations of the effluent at different levels. Permeate was collected every ten minutes, and measured its COD and absorption. Membranes are then regenerated using trypsin and sodium bicarbonate solution

The configuration of the membrane filtration is shown in Figure 1. The system consists mainly of two components- a feed tank and filtration unit. The total chamber feed capacity for the process was 8L. The filtration device was a hollow spiral wound membrane. The membrane module was kept within a container with an inlet and a 1.5 L capacity outlet.

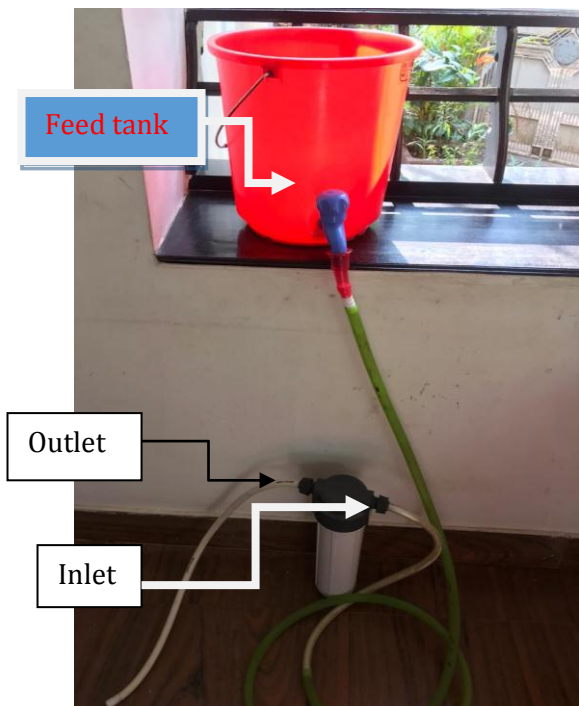


Fig -1: Experimental setup

Separation experiment was conducted using the above mentioned filtration set-up. Feed solution was allowed to flow under gravity and the entire module was sealed perfectly to prevent leakage. Liquid feed has been filtered at specific pH and flow rate for the experiments. Heterogeneous Fenton effluent that uses fly ash is used as the solution for feed. COD and color of the effluent is determined. [4]

### 3. RESULTS AND DISCUSSION

This section deals with the results and discussions that show promising results of textile wastewater treatment using Fenton and microfiltration techniques.

Table -2: shows the characteristics of untreated textile wastewater.

Analytical Parameters	Value
pH	10.92
Turbidity (NTU)	60
Chloride (mg/l)	195
Hardness (mg/l)	165
Sulphide (mg/l)	56
Total Suspended Solids (mg/l)	66

Total Dissolved Solids (mg/l)	993
BOD (mg/l)	440
COD (mg/l)	1210

### 3.1 Results of the effect of catalyst concentration, contact time, pH, and concentration of H<sub>2</sub>O<sub>2</sub>

Table -3: Effect of catalyst concentration (fly ash) and time on Colour and COD removal

Reaction time (min)	Catalyst concentration (g/L)	% COD removal	% Colour Removal
30 min	0.3	15.00	26.51
	0.4	16.30	27.10
	0.5	18.40	29.50
	0.6	15.00	27.27
60 min	0.3	40.00	51.20
	0.4	40.30	53.01
	0.5	41.45	53.89
	0.6	36.30	50.45
90 min	0.3	55.20	67.32
	0.4	55.30	68.49
	0.5	58.40	72.44
	0.6	51.00	67.9
120 min	0.3	56.40	67.02
	0.4	58.10	68.13
	0.5	58.40	72.42
	0.6	51.76	68.39

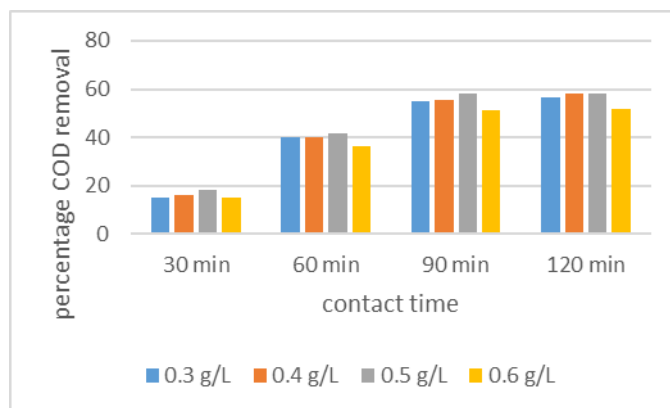


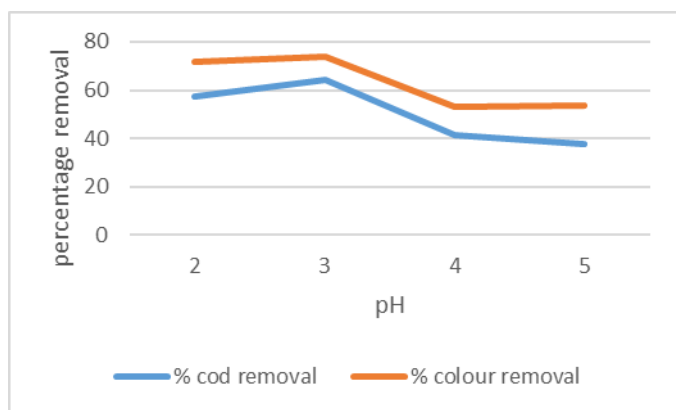
Chart -1: Effect of catalyst concentration and time on Colour removal (At 8.8 mM H<sub>2</sub>O<sub>2</sub>, pH 3)

The concentration of catalysts is a factor that significantly affected the efficiency of treatments. The effect was studied in values of 0.3, 0.4, 0.5 and 0.6 g / L, the concentration of H<sub>2</sub>O<sub>2</sub> was 8.8 mM (1 ml / L) and the pH was 3 throughout 120 minutes. Catalyst effect of 0.2 g / L shall be omitted. It can be seen from table 1 that the decomposition of dye and COD removal continues to increase up to 72.44% and 58.4 % respectively as the catalyst concentration increases

**Table -4:** Effect of pH on COD and Colour removal

pH	% COD removal	% Colour removal
2	57.43	71.92
3	64.12	73.94
4	41.60	52.97
5	37.43	53.65

The results of table 4 shows that acidic surroundings are conducive to the formation of hydroxyl radicals, proven when the treatment affect appears to decrease while increasing pH. Here, the higher removal efficiency of dye 73.94 % and COD 64.12 % occurs at pH 3.



**Chart -2:** Effect of pH on COD and Colour removal (At 8.8 mM H<sub>2</sub>O<sub>2</sub>, reaction time 90 min)

**Table -5:** Effect of H<sub>2</sub>O<sub>2</sub> concentration on Colour and COD removal

H <sub>2</sub> O <sub>2</sub> conc (ml) (30 % wt)	H <sub>2</sub> O <sub>2</sub> conc (mM)	% COD removal	% Colour removal
0.5	4.4	63.8	59.6
0.75	6.6	54	63.12
1	8.8	60.98	72.65
1.25	11.02	64.3	75.6
1.5	13.23	64	71.68

Table 5 demonstrate the effect of hydrogen peroxide concentration in study conditions pH 3, catalyst dose 0.5 g / L and 90 minute reaction time. The effect of dosing H<sub>2</sub>O<sub>2</sub> was investigated by varying initial H<sub>2</sub>O<sub>2</sub> concentration. It achieved the best initial concentration of H<sub>2</sub>O<sub>2</sub> at 11.02 mM. Removal of COD was 64.3% and removal of color was 75.6%.

**Table -6:** Optimized condition for Heterogeneous Fenton-Fly ash

Parameter	Value
Catalyst Concentration (g/L)	0.5
Reaction	90 min
pH	3
H <sub>2</sub> O <sub>2</sub> Concentration (mM)	11.02

Table 6 shows the optimized condition for the treatment obtained from the above trails. These conditions are used to treat the untreated textile water. Table 6 shows the characteristics of textile waste after the primary treatment with heterogeneous Fenton. It can be seen that COD is reduced by 64.3% and color removal was seen to be 75.6%. Initial COD was reduced to 431.97 mg/L and BOD was reduced to 153.48 mg/L. The obtained result is much greater than permissible limits. Further reduction is possible through microfiltration.

**Table -7:** Characteristics of wastewater after Heterogeneous Fenton treatment

catalyst Concentration (g/L)	% COD removal	COD (mg/L)	% Colour removal
0.5	64.3	431.97	75.6

Analytical Parameters	Value	Percent removed
COD (mg/l)	431.97	64.3
BOD (mg/l)	153.48	65.11
pH	6.86	37.15
Turbidity (NTU)	24.4	59.32
Sulphide (mg/l)	18.32	67.27
Chloride (mg/l)	119.05	38.94
Hardness (mg/l)	105.18	36.25
Total Suspended Solids (mg/l)	38.58	41.53
Total Dissolved Solids (mg/l)	601.81	39.39

**Table -8:** Effect of flow rate on Colour and COD removal by microfiltration

	Collecting time (min)	% Colour removal	% COD removal
0.12	10	80.8	70.01
	20	83.9	70.75
	30	84.1	75.67
	40	88.4	77.9
	50	88.6	80
	60	88.6	80.4
20	10	81.47	67
	20	82.37	68.1
	30	82.35	70.59
	40	83.9	75
	50	83.9	75.8
	60	84.56	76
50	10	76.7	64.5
	20	76.7	65.3
	30	77.8	70.02
	40	77.86	70.67
	50	78.75	75
	60	80	77.6

Table 8 shows the result of colour and COD removal by microfiltration. The feed solution is passed through the experimental setup at a pH7 and reaction time is kept 60 mins. The flow rate was kept at 0.12m/s, 20m/s, 50m/s based on the inlet pipe opening condition. pH 7 and 0.12 m/s flow rate are the Optimum parameters obtained . At pH 7, 88.6 percent average color removal and 80.4 percent COD removal.

Table 8 shows the result of the treatment of pretreated waste water with microfiltration.

**Table -9:** characteristics of treated waste water

Analytical Parameters	Value	Percent removed
COD (mg/l)	237.16	80.4
BOD (mg/l)	88	80
pH	7.14	34.54
Turbidity (NTU)	24.4	75.6
Sulphide (mg/l)	nil	
Chloride (mg/l)	40.34	79.31
Hardness (mg/l)	41.25	36.25

Total Suspended Solids (mg/l)	19.8	70
Total Dissolved Solids (mg/l)	248.25	75

From table 9 it can be seen that at pH 7 and 0.12 m/s flow rate, 88.6 percent average color removal and 80.4 percent COD removal is obtained.

#### 4. CONCLUSIONS

Treatment of wastewater is one of the big problems facing textile producers. Actually, several methods for treating textile wastewater have been developed but they cannot be used individually because this wastewater has high salinity, color and organics that are not biodegradable. Coagulation is mostly used nowadays .In coagulation process, large amount of sludge is created which may become a pollutant itself and increase the treatment cost. Potential capacity of fly ash as a catalyst was examined in Heterogeneous Fenton process in this report. The COD and color removal was observed to be 64.3% and 75.6% at catalyst concentration of 0.5 g/L at pH 3 and a reaction time of 90 min. After the microfiltration COD removal increases to 80.4% and color removal observed was 88.6% at a pH 7the flow rate then observed was 0.12 ml/s.

As a preliminary treatment the results shows a removal of COD and color by more than 75% hence further studies are to be carried out in the field of textile waste water treatment incorporating Fenton process with secondary process. The possibility of other efficient catalyst for Fenton process has to be further researched. Future studies can be carried out by using composite cellulose membrane such as cellulose iron, cellulose nitrate etc. ,studies can be conducted using solar photo Fenton incorporated with microfiltration.

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