

Physiochemical Treatment of Textile Industry Effluents

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Summary: The study mainly focuses on the application of chemical Coagulants (Lime, Alum and Ferrous Sulfate) and Advanced Oxidation Processes (AOPs) (Ozone Treatment and Fenton Process, alone and in combination) to treat textile industry effluents, optimization of coagulation process for various Coagulants in terms of process conditions, including coagulant dose, pH and settling time. The results revealed that Alum was most effective. The efficiency of coagulation process was dose dependent and 400 mg/L dose of Alum alone showed maximum color removal of 47%, 57% and 54% of yellow, red and blue dyes, respectively in addition to the COD removal of 44%. The combined applications of Alum and Lime (300:75 mg/L) and Lime and Alum (300:75 mg/L) showed slightly better COD removal of 51%. However, color removal efficiency of all coagulants was at par. The Ozonation process appeared the most promising for the treatment of waste water and color/COD removal, the efficiency of which increased with increasing the treatment time at constant Ozone dose. For less polluted effluents, 97% color removal was obtained after 1 minute and after 15 minutes for highly polluted effluents; The COD removal efficiency of the process for less polluted effluents was around 89% after 5 minutes Ozonation and for highly polluted effluents 88% COD removal after 40 minutes. The performance of Fenton process was extremely low as compared to Ozonation process. Increase in pH, significantly decreased the color removal efficiency of the process. COD removal efficiency of Fenton process increased with an increase in settling time.

Key words: Textile industrial effluents, Chemical Coagulants, Advanced Oxidation Processes, Ozone Treatment, Fenton Processes, Colour removal efficiency, Chemical Oxygen Demand (COD).

Introduction

Textile processing sector is one of the most important and largest industrial sectors of Pakistan, and accounts for 65% of country's export, 46% of industrial production, 38% of employed industrial manpower, 9% of Gross National Product. The textile industry is water intensive and 20-350m³ of waste water is produced for each ton of fabric [1]. Textile effluent is highly colored and has higher contents of Chemical Oxygen Demand (COD), Biological Oxygen Demand (BOD) and dissolved solids in addition to the presence of toxic chemicals, detergents and oil and grease [2, 3]. The pollutants concentration however, largely depends upon the fabric quality, dye stuff and shade variation [4]. COD contents and color values in textile industry effluents were higher than permissible limits and require higher chemical consumption during treatment [3, 5] and esthetically displeasing.

A wide range of techniques are being used worldwide to treat textile wastewater [6]. These methods include chemical coagulation [7, 8], biological processes [9], membrane techniques (REF) and advanced oxidation processes [5]. The Advanced Oxidation Processes (AOPs) are advantageous over traditional treatment technologies due to generation

of hydroxyl radicals, which can oxidize a wide range of organic compounds and can remove color through oxidation cleavage of chromospheres [10-13]. The main objective of the study are: Treatment of Textile Dying industry effluents with Cotton fiber to optimize the coagulation process for various coagulants i.e., Ferrous Sulphate (FeSO₄.7H₂O), Lime (CaO) and Alum (KAl(SO₄)₂.12H₂O) in terms of process conditions, including coagulant dose, pH and settling time, to apply the advanced oxidation process including Ozonation process, and Fenton alone and in combination to treat the effluent, and to determine the effects of process conditions such as Ozone dose, initial dye contents, iron catalyst concentration, Hydrogen Peroxide (H₂O₂) concentration, pH treatment time and settling time on the overall efficiency of these processes.

Experimental

Textile waste water was characterized in two groups (Table-1) on the basis of pH, TDS, COD and ammonia nitrogen contents. These Textile industrial effluents subjected to different coagulation process (for various coagulants in terms of process conditions, including coagulant dose, pH and settling

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time), advanced oxidation process (Ozonation process, and Fenton processes alone and in combination to treat the effluent) and to determine the effects of process conditions such as Ozone (O_3) dose, initial dye contents, iron catalyst concentration, H_2O_2 concentration, pH treatment time and settling time on the overall efficiency of these processes.

Table-1: Effect of pH on the color and COD removal efficiency of Fenton process at optimized dose of $FeSO_4$ (100mg/L) at different pH levels

Parameter	Color and COD removal (%) at various pH values						
	3	4	5	6	7	8	9
COD	24	4.3	-5	-32	-45	-180	-218
pH	2.97	4.39	5.89	6.99	7.92	7.7	8.42
Yellow (Abs _{424nm})	30	23	23	12	12	-36	-40
Red (Abs _{516nm})	36	33	33	24	12	-45	-45
Blue (Abs _{620nm})	34	31	25	22	9	2	2

Coagulation process

The pH of the waste water was varied from 3 to 9 and it was dosed with coagulants i.e., Lime (CaO), Alum ($KAl(SO_4)_2 \cdot 12H_2O$) and Ferrous Sulphate ($FeSO_4 \cdot 7H_2O$) as shown in Table-2. The coagulant was rapidly mixed for two minutes initially, followed by slow mixing for 30 minutes. Newly formed flocs were allowed to settle down for 30 minutes, and the supernatant was decanted and analyzed following standard methods [33]. Settled sludge volume was also measured after each experiment.

Table-2: Effect of settling time on the COD and Color removal efficiency removal efficiency of Fenton process at various H_2O_2 concentrations at constant dose of Fe^{2+} 100mg/L

H ₂ O ₂ dose ml/L	COD removal (%) at various doses of H ₂ O ₂ and settling time		Color removal (%) at various doses of H ₂ O ₂ and settling time								
			Yellow (Abs _{424nm})			Red (Abs _{516nm})			Blue (Abs _{620nm})		
	15 min	60 min	15	30	60	15	30	60	15	30	60
0.5	40	57	13	13	16	58	59	59	27	43	45
1	34	54	43	55	56	70	81	81	59	65	76
1.5	24	39	40	43	46	77	79	79	74	76	76
2	-2	24	53	55	56	83	84	84	80	82	82

Fenton process

500ml waste water was treated in each experiment. An optimal dose of Hydrogen Peroxide (H_2O_2) was determined by keeping optimal dose of Ferrous Sulphate ($FeSO_4 \cdot 7H_2O$) constant. H_2O_2 dose varied in the range of 0.5-3ml/L at constant pH (3) of the waste water. H_2O_2 was quickly mixed with waste water for starting 2 minutes and followed by 30 minutes of slow mixing; waste water was allowed to settle for 15-60 minutes and was then determined the overall removal efficiency of the Fenton process.

Ozonation process

Ozone was generated by using Ozone generators (ENALY, HGOZ/1000, USA) with Ozone

(O_3) production rate 1g/hr at air flow rate of 1.1 L/minute. Ozone was bubbled through 200ml of real textile effluent via diffused fixer at the bottom of the reactor, in batch mode. The Ozone exposure time varied between 1-40 minutes and color and COD removal was determined after an interval of every 5 minutes.

Color removal efficiency

The dye concentration in reaction mixture at different reaction times was determined by measuring the absorption intensity at 424, 516 and 620 nm by using UV- Visible Spectrophotometer since actual waste water had different kinds of dyes. Decolorization (%) was calculated using following relationship

$$D(\%) = \frac{C^0 - C_t}{C^0} \times 100$$

where:

D% = Decolorization percentage

C^0 = Initial concentration of dye

C_t = Concentration of dye at certain treatment time

COD removal efficiency

Standard method 5220 B (APHA, 1998) i.e., Reflux method is used for COD removal efficiency [33]. COD reduction (%) was determined by using following formula

$$COD(\%) = \frac{COD^0 - COD_t}{COD^0} \times 100$$

where

COD% = COD reduction percentage

COD^0 = initial COD of the effluent

COD_t = COD of the effluent after treatment

Results and Discussion

Effect of different doses of different coagulants on COD and Color Removal Efficiency

The performance of different doses of Coagulants (Ferrous Sulphate, Lime and Alum) is shown in Table-3. It appears from the results that 100 mg/L dose of Ferrous Sulphate demonstrates maximum color and COD removal. Beyond the optimal dose (100 mg/L), COD removal efficiency

was significantly reduced. Ferrous Sulphate dose above 200 mg/L substantially added pollution load to the effluent which is due to an increase in Total Dissolved Salts/ Total Soluble Salts contents. The data regarding the effect of different doses of lime showed that COD removal efficiency was good at dose 100 mg/L. With the increase in lime dose, COD removal efficiency decreases dramatically because of an increase in Total Soluble Salts contents of effluents. In case of color removal, lime appeared to be in effective [14, 15]. It was evident that removal efficiency of Alum for all parameters was increased substantially with increasing Alum dose, and 400 mg/L dose appeared to be optimal thereby reducing Chemical Oxygen Demand up to 44% and color (yellow, red and blue dyes) level to 47, 57 and 54%, respectively.

Table-3: Basic Physio-chemical Characteristics of Textile industrial effluents

Parameter	Value	
	Effluent 1	Effluent 2
pH	9.5	9.5
COD (mg/L)	464	1154
NH ₃ (mg/L)	5.72	8.96
Yellow (A _{424nm})	1.83	2.72
Red (A _{516nm})	2.24	3.29
Blue (A _{620nm})	2.16	3.19

Table-2 showed the efficiency of combined application of alum and lime. In case of constant Lime dose (300mg/L), an Alum dose of 25mg/L showed maximum Chemical Oxygen Demand (COD) and color removal. Color removal efficiency of the system was better than Lime alone [16]. In case of constant Alum dose (300mg/L), a Lime dose of 75mg/L showed maximum COD removal [17, 18]. However, color removal efficiency of the system was not better than the lime alone (Table-3). Results also revealed that combined applications of Alum/Lime (300:75) and Lime/Alum (300:75) showed COD removal around 51% in both cases. However, a combination of Lime/Alum (100:300) proved

effective, and more than 50% color removal efficiency was obtained for all wavelengths [19]. Similarly, the application of Alum alone showed better color removal [20,21] as compared to Ferrous Sulphate and Lime alone (Table-4).

Table-4: Coagulants doses used in the jar test.

Type of coagulant	Dose range (mg/L)					
Lime	100	200	300	400	500	600
Alum	100	200	300	400	500	600
Ferrous sulfate	50	100	150	200	250	300
Alum+ lime (300mg/L)	25	50	75	100	-	-
Lime+ alum (300mg/L)	25	50	75	100	-	-

Effect of Ozonation process on Color Removal Efficiency

The effects of Ozone exposure time, Ozone dose and initial dye concentration on the color and COD removal efficiency of the process is shown in Table 5 and 6. The color removal efficiency of the process increased with increasing the Ozone treatment time, which was a function of Ozone dose (Table-7). The treatment time decreased dramatically with increasing ozone dose [22]. At constant Ozone dose, the color removal (%) was however a function of Ozonation time [23]. The performance of the process significantly declined as the dye concentration of the effluent was increased (table 5). Highly polluted Textile Effluents (effluent 2) required much longer time for the color and COD removal as compared to Less Polluted Effluents (effluent 1). Undoubtedly, an increase in Ozonation time enhances the ozone contents in liquid phase, which overall boosts the degradation rate constant [17, 24]. However removal efficiency is increased trivially when Ozone concentration in the liquid phase approaches its maximum value [25]. This may be due to the fact that the process is controlled by the rate of chemical reaction and any further improvement in Ozone mass transfer would have a diminishing effect on the observed reaction rate [25].

Table-5: COD and Color Removal efficiency (%) of different doses of Ferrous Sulphate, Lime and Alum.

Parameter	Different doses of Ferrous Sulfate (mg/L)				Different doses of Lime (mg/L)				Different doses of Alum (mg/L)							
	50	100	200	300	100	200	300	400	500	600	100	200	300	400	500	600
COD	11	16	16	-31	42	43	44	28	25	25	30	34	41	44	41	41
pH	10.76	10.83	10.73	8.67	9.84	9.87	9.89	9.92	9.94	9.96	9.52	9.41	9.41	9.43	9.37	9.32
Yellow (Abs _{424nm})	8	28	13	-32	4	3	4	2	2	2	11	30	35	41	33	9
Red (Abs _{516nm})	6	35	22	-11	7	5	7	4	4	3	41	51	53	57	40	34
Blue (Abs _{620nm})	9	32	24	-8	8	8	8	5	5	5	39	51	53	54	38	34

Table-6: Efficiency of Different doses of Alum at a constant dose of lime (300mg/L) and Different doses of Lime at constant dose of Alum (300mg/L)

Parameter	Different doses of Alum (mg/L) at a constant dose of lime (300mg/L)				Different doses of Lime (mg/L) at a constant dose of Alum (300mg/L)			
	25	50	75	100	25	50	75	100
COD	48	48	51	47	48	48	51	41
pH	11.93	11.93	11.96	12.01	9.61	9.53	9.52	9.63
Yellow (Abs _{424nm})	38	36	37	33	16	35	40	52
Red (Abs _{516nm})	38	34	36	32	28	35	40	52
Blue (Abs _{620nm})	35	31	33	29	26	32	37	50

Table-7: Comparison of the efficiencies of doses of different coagulants alone and in combination.

Coagulant dose (mg/L)			Color removal (%)			COD removal (%)	pH
Lime	Ferrous sulfate	Alum	Yellow	Red	Blue		
100	0	0	4	7	6	42	9.8
0	100	0	28	35	32	23	10.8
0	0	400	41	57	54	44	9.4
300	0	75	37	36	33	51	11.9
75	0	300	40	40	37	51	9.5
100	0	300	52	52	50	41	9.6

The effect of treatment time on the color and COD removal efficiency of the process was shown at constant Ozone (O_3) dose of 5 g/hr (Table-8 and 9). Experiment was repeated seven times. Only 5 min Ozonation time was sufficient to effectively remove the dye contents of the effluents (Table-6) and Table-7 revealed that initial COD contents (1154 mg/L) were reduced to 470 mg/L after 5 min treatment time and COD removal efficiency of the process increased with an increase in Ozonation time. The characteristics of raw and treated effluent after Ozonation (Table-10) revealed that Ozonation process effectively removed the color and COD contents [26, 27].

Effect of Fenton process on Color Removal Efficiency

The efficacy of Ferrous Sulphate with respect to its dose showed that the dose of 100mg/L was appeared to be optimal for the color and COD removal (Table-11). Beyond that dose, the efficiency of the iron was not only dropped but also cause an increase in the pollution load due to the elevated level

of solids in the effluent [14, 28]. The effect of pH on the performance of Fenton process (Table-12) revealed that the pH varied between 3-9, with maximum COD and color removal at pH 3. But further increase in pH decreased the removal efficiency which was due to the formation of Ferric complex which caused the precipitation of both Ferrous and Ferric ions [18, 29].

The effectiveness of H_2O_2 for color and COD removal with respect to its dose (0.5-2mg/L) and settling time (Table-13) demonstrated that 0.5mg/L dose of H_2O_2 appeared to be optimal and showed maximum COD removal efficiency at 60 minutes settling time, above this dose, the efficiency was dramatically decreased [13]. In case of color removal, 1mg/L H_2O_2 dose appeared to be optimal and showed maximum color removal in the first 15 min, beyond that increase in removal efficiency was insignificant [20, 13, 31].

Table-8: Effects of ozone treatment time, initial dye concentration and ozone dose on the color removal efficiency of Ozonation process

Ozonation time (min)	Color removal (%) efficiency of Ozonation process for effluents of various dye concentrations (mg/L) at different ozone doses				
	Effluent 1			Effluent 2	
	1 g/hr	5 g/hr	1 g/hr	5 g/hr	
1	-	97	43	65	
3	-	98	59	73	
5	-	99	70	89	
10	94	-	83	96	
15	98	-	90	97	
20	100	-	94	99	
25	-	-	99	-	

Table-9: Effect of initial dye concentration and Ozonation time on the COD removal efficiency of Ozonation process at constant ozone dose (5 g/hr Ozone dose) for real textile effluents

Ozonation time (min)	COD removal (%) efficiency of the ozonation process for effluents of different COD contents	
	Effluent 1 (464 mg/L)	
		Effluent 2 (1154 mg/L)
1	55	37
3	82	48
5	89	59
10	96	67
15	-	72
20	-	75
25	-	79
30	-	84
35	-	85
40	-	88

Table-10: Statistical analysis of color removal efficiencies of the Ozonation process with respect to treatment.

Ozonation time	Color removal efficiency of the Ozonation process for effluent 2								
	Yellow (424nm)			Red (516nm)			Blue (620nm)		
	Mean removal (%)	Mean value (abs)	SD	Mean removal (%)	Mean value (abs)	SD	Mean removal (%)	Mean value (abs)	SD
1	59	1.1	0.057	73	0.89	0.022	76	0.756	0.023
3	72	0.68	0.008	90	0.34	0.021	85	0.461	0.011
5	83	0.404	0.056	95	0.149	0.005	96	0.107	0.005
10	90	0.257	0.045	96	0.127	0.009	97	0.089	0.009
15	92	0.192	0.021	97	0.123	0.009	97	0.089	0.009
20	94	0.156	0.018	97	0.123	0.012	98	0.045	0.012
25	94	0.147	0.028	97	0.123	0.017	98	0.045	0.017

Table-11: Statistical analysis of COD removal efficiencies of the Ozonation process with respect to treatment.

Ozonation time (min)	Mean COD value (mg/L)	SD	Removal % range
5	470	55	50-62
10	378	37	63-70
15	320	46	66-72
20	285	18	74-77
25	232	36	75-81
30	183	18	81-86
35	172	69	82-90
40	135	70	85-97

Table-12: Characteristics of effluent before and after treatment with ozonation process.

Parameters	Effluent		NEQS limits
	Raw	Treated	
COD (mg/L)	1154	135	150
pH value	9.48	9.49	6-10 Ph
Color (A _{424nm})	2.72	0.155 (94%)	-
Color (A _{516nm})	3.29	0.09 (97%)	-
Color (A _{620nm})	3.18	0.07 (98%)	-

Table-13: Color and COD removal efficiency of Ferrous Sulphate with respect to its different doses.

Parameter	Different doses of Ferrous Sulphate (mg/L)			
	50	100	200	300
COD	11	23	16	-31
pH	10.76	10.83	10.73	8.67
Yellow (Abs _{424nm})	8	27	13	-32
Red (Abs _{516nm})	6	34	22	-11
Blue (Abs _{620nm})	9	32	24	-8

In the study combine doses of Lime and Alum proved more effective. Similarly Ozone doses also show drastic reduction in Colour and COD removal efficiencies at different times. Ozonation, Hydrogen Peroxide treatment and Fenton processes are classified as Non Photochemical Oxidation processes [25]. Fenton process is cost effective, easy to apply and effective for a wide range of organic compounds with easy to handle chemicals [28]. One of the advantages of Fenton's reagent is that no energy input is required for the activation of Hydrogen Peroxide [32]. Fenton process consists of four stages, first of all pH is adjusted to low pH, then reaction takes place at pH 3-5, then pH is neutralized to 7-8 and finally precipitation occurs [30]. These Fenton processes require less reaction time. On the other hand, Advanced Oxidation Processes are defined as the processes which involve production and use of powerful but relatively non-selective Hydroxyl ions in sufficient quantities [24], as these Hydroxyl radicals has highest oxidation potential to oxidize the complex compounds present in textile effluents [23]. Theses Advanced Oxidation Processes convert the dissolved organic pollutants in CO₂ and

H₂O. Ozonation has high efficiency at even high pH (>11) levels, because it reacts with almost all organic and inorganic chemicals present in the effluents indiscriminately [27]. Ozone reacts with reacting medium in two different ways; direct molecular and radical type chain reactions simultaneously depending upon the pH and concentration and composition of effluents [28]. The combination of Ozone and Hydrogen Peroxide are used for the treatment of highly polluted effluents, the oxidation of whome is difficult and consume large amount of oxidant [19, 32]. Ozonation is low cost treatment method (with high initial cost but very low running cost) resulting in sludge destruction as compared to Lime, Ferrous Sulphate and Alum treatments which are sludge collection processes [27]. In Ozonation process, a photolysis of Ozone in water with ultra violet radiation in the range of 200-280nm can lead to yield of Hydrogen Peroxide producing Hydroxyl radical [22]. Different kinds of Ultra Violet radiations are use as source, among which Mercury vapor lamp is most common [25]. So it is recommended that Ozonation process should be used to treat these types of waste waters.

Conclusion

Alum coagulant was proved more effective as compared to other coagulants. Efficiency of coagulation process was dose dependent. The combined application of lime and alum showed better COD removal efficiency as compared to alone application of Lime and Alum. Ozonation process was proved more effective and its removal efficiency increases with increasing ozone treatment time at constant Ozone dose. The effluent of higher pollution level (effluent 2) required much longer treatment time as compared to less polluted effluent (effluent 1). Fenton process proved to be very low in efficacy. At pH 3, process showed COD and color removal but above this pH, color removal efficiency was significantly decreased. COD removal efficiency of Fenton process increased with increase in settling time. Ozonation is low cost treatment method (with high initial cost but very low running cost) resulting in sludge destruction as compared to Lime, Ferrous Sulphate and Alum treatments (27) which are sludge collection processes. So it is recommended that Ozonation process should be used to treat these type of waste waters.

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