

Ozonation application in activated sludge systems for a textile mill effluent

D. Orhon, H. Dulkadiroğlu, S. Doğruel, I. Kabdaşlı, S. Sozen and F. Germirli Babuna

Environmental Engineering Department, Istanbul Technical University, I.T.Ü. İnşaat Fakültesi, 80626 Maslak, Istanbul, Turkey

Abstract The study investigates the effect of partial ozonation of textile wastewater, both at the inlet (pre-ozonation) and the outlet (post-ozonation) of biological treatment, for the optimization of COD and color removals, both typical polluting parameters associated with the textile industry. Pre-ozonation provides at optimum contact time of 15 minutes 85% color removal, but only 19% COD reduction. Removal of the soluble inert COD fraction remains at 7%, indicating selective preference of ozone for simpler compounds. Post-ozonation is much more effective on the breakdown of refractory organic compounds and on color removal efficiency. Ozonation after biological treatment results in almost complete color removal and a 14% soluble inert COD reduction. The polishing effect of post-ozonation also proves quite attractive from an economical standpoint, involving approximately 50% of the ozone utilization at the same ozone flux rate and contact time, yet providing a lower soluble residual COD level.

Keywords Biological treatment; knit fabric processing; COD fractionation; COD removal; color removal; ozonation; soluble inert COD; textile wastewater

Introduction

Textile industry wastewater is typically associated with high COD and color. A significant fraction of the COD imparted by different dyeing and finishing processes is usually non-biodegradable, which bypasses the treatment step and becomes a major problem in meeting the effluent limitations. Color is equally objectionable both from a regulatory and esthetic aspects. From a scientific and technical perspective, selection of the appropriate treatment step for the effective removal of both color and residual COD has always been a major concern.

The investigated plant involves knit fabric-finishing processes mostly using reactive dyes. Reactive dyes are much more soluble compared to others which generate wastewater having strong color (Jedele, 1982; Grau, 1991) and non-biodegradable organic residues, difficult to remove in conventional chemical and biological treatment systems (Sewekow, 1993). Although chemical precipitation is an effective treatment method for color removal from wastewater containing dyes with limited solubility, such as disperse dyes, it gives unsatisfactory results for soluble dyes (Kuo, 1992). Apart from the removal efficiency potential provided, sludge generation is a major disadvantage for chemical treatment since it requires additional and costly handling and disposal, (Kuo, 1992; Gaehr *et al.*, 1994). Biological treatment is a common treatment alternative applied on textile wastewaters, but it increases the non-biodegradable organic matter, initially present in the wastewater, through generation of soluble residual microbial products (Orhon and Artan, 1994). Chemical oxidation with ozone, however, is the preferred process for the dyes used in textile operations in order to achieve high color removal efficiencies and to improve the biodegradability without sludge production (Gaehr *et al.*, 1994; Schönberger and Kaps, 1994).

Ozone can be applied either before or after the biological treatment process. *Pre-ozonation* may be expected to ease biological treatment by converting the more slowly

biodegradable COD into simpler compounds or by reducing the amount of inert organic matter. *Post-ozonation*, on the other hand, may have a polishing effect on the effluent quality. It is therefore important to set the basis for the selection of the appropriate location for ozone application. The right choice between pre- or post-ozonation alternatives is significant for the optimum use of the chemical oxidation potential provided, both for the overall COD and color removal, and for the reduction of soluble non-biodegradable COD fraction, often a major problem in meeting the stringent effluent quality restrictions.

In this framework, the objective of the study was to investigate the effect of partial ozonation of textile wastewater, both at the inlet and the outlet of biological treatment, for the optimization COD and color removal, both typical polluting parameters associated with the textile industry. Changes induced by ozonation on soluble COD components and specifically, on the fate of soluble inert COD fraction, were also evaluated under different operating conditions. For this purpose, the biological treatment effluent was selected, as it is likely to contain only non-biodegradable COD. Ozonation of textile wastewater, as a technically effective process in conjunction with biological treatment, was also evaluated in terms of COD fractionation, a new concept for treatability-oriented wastewater characterization.

Materials and methods

Description of the textile plant

The study generated and evaluated experimental data from a textile plant located at Ayazağa/Istanbul, processing cotton and synthetic knit fabrics and their blends. The plant operates 6 days a week, 250 days a year, employing 165 people in 3 shifts a day. The process requires an average of 751 m³ of water per day, supplied partly from local wells and the remaining portion by tankers, with a water balance fully justifying the need for wastewater recovery and reuse. The textile mill has a treatment plant that consists of an equalization tank, a neutralization unit and a conventional activated sludge system operated at a sludge age of 7 days, providing full treatment before discharge into the an adjacent creek.

The plant used in the survey reflects a typical example of knit fabric finishing subcategory, involving batch-wise operations such as dyeing, bleaching, kiering etc. applied to cotton, viscose rayon, polyester, polyamide knit fabrics and cotton/polyester, polyester/viscose rayon blend knit fabrics. The investigated plant has a total volumetric wastewater load of 72 m³ ton⁻¹ fabric that is consistent with the literature (Germirli Babuna *et al.*, 1998a, 1999).

Experimental and analytical methods

The experimental survey is conducted on equalization tank samples and biologically treated wastewater. Biologically treated wastewater is taken after 20 h hydraulic retention time from the equalization tank in order to represent the same wastewater quality of equalization tank sample.

Ozone was produced by a laboratory ozone generator PCI GL1. Ozonation experiments were conducted at 15 psi (103.45 kPa) pressure by a feed gas flowrate of 1.42 l min⁻¹ using a sample of 1 litre in a 1.5 litre semi-batch bubbled gas washing bottle reactor with an effective depth of 23 cm. Ozone gas was supplied at the bottom of the reactor through a sintered glass plate diffuser. All pieces in contact with ozone were stainless steel, glass or Teflon® and stable in the presence of ozone. Two gas washing bottles containing 2% potassium iodide solution were connected to the reactor in series in order to determine the output ozone gas passing through the reactor.

Experimental studies related to biological treatability involved soluble COD fractionation. Influent soluble inert COD fraction, S_p, was measured according to the method

proposed by Orhon *et al.* (1999). The method consists of running two aerated batch reactors of the same volume, one fed with the filtered wastewater and the other with glucose. Both reactors were started with the same initial COD concentration and seeded to secure an initial biomass concentration of around 40 mg VSS per litre. The seed was obtained from a lab-scale fill and draw aerobic reactor operated at steady state with a feed of 50% glucose and 50% wastewater mixture. Aliquots removed periodically from the mixed liquor were analyzed for soluble COD. Experiments were continued until the observation of a stable threshold COD level coupled with no measurable biomass activity.

Influent readily biodegradable COD fraction, S_s , was obtained respirometrically as defined in the method proposed by Ekama *et al.* (1986). OUR measurements were conducted in samples periodically taken from 2 litre aerobic batch reactors where the initial F/M ratio was adjusted to 0.2 g COD/g VSS. The reactors were seeded with the biomass obtained from fill and draw reactors acclimatized to wastewater, operated at sludge ages of 10 days. The heterotrophic yield coefficient, Y_H , was assumed as 0.67 g COD/g cell COD in accordance with the data reported in the literature (Germirli Babuna *et al.*, 1998a; Orhon *et al.*, 2001). OUR measurements were performed in Manotherm RA-1000 continuous respirometer connected to PC. pH of the biological reactors was kept in the range of 7.0–8.0 and continuous aeration was supplied to maintain a dissolved oxygen concentration of above 6 mg l⁻¹. The rapidly hydrolysable COD component, S_H , as the remaining soluble COD fraction, was calculated by mass balance.

COD measurements were performed as defined in ISO 6060 (1986). All other analyses for conventional characterization were conducted in accordance with *Standard Methods* (1998). The color was measured in platinum-cobalt unit and absorbance values in three different wavelengths (436, 525 and 620 nm) after filtering the samples from 0.45 µm membrane filter papers. Soluble fractions were defined as filtrates of the samples subjected to vacuum filtration using also 0.45 µm membrane filters. Whatman GF/C glass-fiber filter papers having a pore size of approximately 1.2 µm were used for total suspended solids (TSS) and volatile suspended solids (VSS) measurements.

Results and discussion

Wastewater characterization

The two wastewater samples investigated in the study, the one taken from the equalization tank to characterize the biological treatment influent, and the other a composite from the aeration tank effluent, were analyzed in this part of the study, for conventional pollutant parameters. The results of conventional wastewater characterization both for biological process influent and effluent are summarized in Table 1.

As shown in this table, the influent reflects the typical properties of a textile wastewater containing high COD, color and total dissolved solids and relatively low particulate pollutants and nutrients (Germirli Babuna *et al.*, 1998a, 1999). The biological treatment unit operates well with a good settling activated sludge and a total COD removal efficiency of around 80%.

Ozonation experiments

After conventional characterization the samples were subjected to ozonation experiments in order to notice the impact of the ozone flux rate on COD and color removal. In the first step, the feeding time was set as 5 minutes for a wide range of ozone flux levels changing from 18 to 97 mg min⁻¹. The results obtained are outlined in Table 2. For the biological treatment influent, a steadily increasing improvement in color removal was observed, starting from 22% at 18 mg O₃ min⁻¹, reaching 68% for 97 mg O₃ min⁻¹. The corresponding COD reduction was almost negligible for the majority of ozonation experiments. It

Table 1 Conventional wastewater characterization

Parameter	Biological treatment influent	Biological treatment effluent
Total COD (mg l ⁻¹)	925	215
Soluble COD* (mg l ⁻¹)	530	185
Soluble COD** (mg l ⁻¹)	660	190
TSS** (mg l ⁻¹)	175	15
VSS** (mg l ⁻¹)	155	15
NH ₃ -N (mg l ⁻¹)	35	0
Ortho-PO ₄ * (mg l ⁻¹)	2	5
Color* (Pt-Co Unit)	1,840	1,380
Absorbance* 436 nm	0.621	0.463
525 nm	0.629	0.461
620 nm	0.346	0.263
pH	9.17	8.50
Alkalinity (mg CaCO ₃ l ⁻¹)	610	800
Conductivity (μMhos cm ⁻¹)	9,700	12,900
Chloride (mg l ⁻¹)	2,240	2,575

* filtered from 0.45 μm membrane filter paper

** filtered from Whatman GF/C glass-fiber filter paper

remained limited to 5% at 97 mg O₃ min⁻¹. Experiments on the biological treatment effluent were also conducted for an ozone flux range of 19–99 mg O₃ min⁻¹. The results obtained could be characterized, as shown in Table 2, with a higher color removal range of 53–92%, coupled with an equally higher COD removal rate of 5–14%, but still quite low from a practical standpoint.

Results outlined in Table 2 indicate that the optimum ozone flux should be chosen in relation to color removal rates, in view of limited COD reductions. No significant color removal could be achieved beyond an ozone flux of 4 l O₃ min⁻¹. Therefore a value of around 40 mg O₃ min⁻¹ was selected as the optimum ozone flux for the biological treatment influent. In order to set a basis for comparative evaluation, the same ozone flux was also selected as the optimum level for the ozonation studies of biologically treated wastewater.

In the second step, both samples were ozonated for four different feeding periods of 5, 10, 15 and 30 minutes at the selected optimum ozone flux rate of 40 mg min⁻¹ (Table 3).

According to the results given in Table 3, the COD reduction efficiency improved from 4% to 24% for the equalization basin sample, with a parallel increase in color reduction, from 48% to 96%. The ozonation studies of biologically treated wastewater at the optimum ozone flux rate of 40 mg min⁻¹ resulted in an almost total color removal and a somewhat limited COD reduction of 14%. The difference between COD and color removal efficiencies for both samples can be attributed to the selective character of ozone reactions with different COD components present in these samples. It should also be argued that the observed difference in COD removal efficiencies between the two textile wastewater samples, or any other of samples, cannot be explained using total COD alone, but it requires a further in-depth look at their COD composition.

Effect of ozonation on COD fractionation

Differentiation of various COD components in terms of their biodegradation properties will provide a better insight into how ozonation reactions proceed with different COD fractions within the textile wastewater. COD fractionation, introduced and experimentally defined as part of the new activated sludge modeling concepts (Henze *et al.*, 1987; Orhon and Artan, 1994) is used in this part of the study to determine the change of the wastewater composition after ozonation. Aside from separating the total COD into soluble (S_T) and

Table 2 Results of preliminary ozonation experiments

Sample	Ozone flux (mg min ⁻¹)	Ozone feeding time (min)	Utilized ozone (mg)	pH	Total COD (mg l ⁻¹)	Total COD removal (%)	Soluble COD (mg l ⁻¹)	TSS (mg l ⁻¹)	VSS (mg l ⁻¹)	Color (Pt-Co Unit)	Color removal (%)	436 nm Absorbance	525 nm Absorbance	620 nm Absorbance
Biological Treatment Influent	18	5	21	8.94	925	1	530	175	155	1,840	–	0.621	0.629	0.346
	41	5	30	8.85	890	4	495	170	155	1,440	22	0.397	0.307	0.095
	62	5	53	8.85	890	4	500	170	155	960	48	0.343	0.227	0.082
	82	5	74	8.79	885	4	480	165	155	760	59	0.203	0.094	0.002
	97	5	84	8.76	880	5	480	165	150	620	66	0.131	0.043	0.000
Biological Treatment Effluent	19	5	21	8.50	215	–	185	15	15	580	68	0.109	0.025	0.000
	40	5	24	8.48	205	5	185	15	15	1,380	–	0.463	0.461	0.263
	60	5	28	8.47	190	12	180	10	10	645	53	0.215	0.150	0.046
	82	5	31	8.46	190	12	180	<10	<10	310	78	0.119	0.061	0.036
	99	5	34	8.43	185	14	175	<10	<10	200	86	0.074	0.025	0.000
										155	89	0.023	0.000	0.000
										110	92	0.010	0.000	0.000

Table 3 Results of ozonation experiments at optimum ozone flux

Sample	Ozone flux (mg min ⁻¹)	Ozone feeding time (min)	Utilized ozone (mg)	pH	Total COD (mg l ⁻¹)	Total COD removal (%)	Soluble COD (mg l ⁻¹)	TSS (mg l ⁻¹)	VSS (mg l ⁻¹)	Color (Pt-Co Unit)	Color removal (%)	436 nm Absorbance	525 nm Absorbance	620 nm Absorbance
Biological Treatment Influent	40	5	30	8.85	925	–	530	175	155	1,840	–	0.621	0.629	0.346
	40	10	93	8.56	805	13	590	105	100	960	48	0.343	0.227	0.082
	40	15	125	8.54	750	19	600	75	75	420	77	0.167	0.098	0.065
	40	30	710	8.52	700	24	445	65	65	280	85	0.157	0.063	0.020
Biological Treatment Effluent	40	5	24	8.48	215	–	185	15	15	70	96	0.037	0.018	0.010
	40	10	39	8.38	200	7	170	10	10	310	–	0.463	0.461	0.263
	40	15	64	8.35	185	14	170	<10	<10	70	78	0.119	0.061	0.036
	40	30	244	7.96	185	14	170	<10	<10	40	95	0.065	0.042	0.026
										20	97	0.011	0.000	0.000
										20	99	0.000	0.000	0.000

Table 4 COD fractionation after ozonation experiments

Sample	Ozone feeding time (min)	Utilized ozone (mg)	C_T (mg l^{-1})		X_T (mg l^{-1})		S_T (mg l^{-1})		S_S (mg l^{-1})		S_H (mg l^{-1})		S_I (mg l^{-1})	
			Conc.	Removal	Conc.	Removal	Conc.	Removal	Conc.	Removal	Conc.	Removal	Conc.	Removal
Biological Treatment Influent	-	-	925	-	395	-	530	-	175	-	215	-	140	-
	5	30	890	35	395	0	495	35	90	85	245	30*	160	20*
	10	93	805	120	215	180	590	60*	85	90	370	155*	135	5
Biological Treatment Effluent	15	125	750	175	150	245	600	70*	10	165	460	245*	130	10
	-	-	215	-	30	-	185	0	0	-	0	-	185	-
	5	24	200	15	15	15	185	0	0	0	0	0	185	0
Biological Treatment Effluent	10	39	190	25	20	10	170	15	0	0	5	5*	165	20
	15	64	185	30	15	15	170	15	0	0	15	15*	155	30

* increase

Table 5 Effect of ozonation on COD ratios

Sample	Ozone feeding time (min)	Utilized ozone (mg)	C_T (mg l^{-1})	X_T (mg l^{-1})	$\frac{X_T}{C_T}$	S_T (mg l^{-1})	$\frac{S_T}{C_T}$	S_S (mg l^{-1})	$\frac{S_S}{C_T}$	S_H (mg l^{-1})	$\frac{S_H}{C_T}$	S_I (mg l^{-1})	$\frac{S_I}{C_T}$
Biological Treatment Effluent	5	30	890	400	45	495	55	90	10	245	27	160	18
	10	93	805	215	27	590	73	85	10	370	46	135	17
	15	125	750	150	20	600	80	10	1	460	62	130	17
Biological Treatment Effluent	-	-	215	30	14	185	86	0	0	0	0	185*	86
	5	24	200	200	15	8	185	92	0	0	0	0	185*
	10	39	190	20	10	170	90	0	0	5	3	165*	87
Biological Treatment Effluent	15	64	185	15	8	170	92	0	0	15	8	155*	84

* also including microbial products

particulate COD, (X_T), the fractionation experiments in this study were carried out to identify mainly the soluble COD fractions, namely, readily biodegradable (S_S), rapidly hydrolysable (S_H) and inert (S_I) COD, as the particulate components constitute only a minor part of the total COD. In this context, the effect of ozonation on COD fractionation, especially on the soluble inert COD fraction, was determined for ozone contact times of 5, 10 and 15 minutes. A contact time of 30 minutes, an extreme level in practice, was discarded in this part. The results of COD fractionation are given in Table 4, and the relative evaluation of each fraction as percent of the total COD is presented in Table 5.

According to Tables 4 and 5, an increase in both the concentration and percentage of soluble COD component is observed at higher ozone contact times for the equalization tank sample. An ozone contact time of 5 minutes resulted in a decrease of readily biodegradable COD content from 175 mg l^{-1} to 90 mg l^{-1} . Prolonged ozone application for 15 minutes resulted with almost total S_S oxidation. S_I in the raw equalization basin sample was 140 mg l^{-1} , corresponding to 26% of the total soluble COD (S_T), and to 15% of the total COD (C_T), a level in accordance with the soluble inert COD content experimentally ascertained for similar textile effluents (Orhon *et al.*, 1992; Orhon and Ubay Çokgör, 1997; Germirli Babuna *et al.*, 1991, 1998a, 1998b, 1999). Ozonation induced, as illustrated in Figure 1, a S_I increase of 14% with a contact time of 5 minutes, possibly due to generation of less biodegradable organic by-products, as reported in the literature (ATV-Handbuch, 1997). When the contact time was extended to 15 minutes, S_I was brought down to 130 mg l^{-1} , corresponding to a reduction efficiency of 7%, together with a color removal of 85%. As presented in Table 4, S_I content of biological treatment effluent was decreased from 185 mg l^{-1} to 155 mg l^{-1} , indicating to a removal rate of 16%. The total soluble COD level, however, remained practically unchanged around 170–185 mg l^{-1} and the color removal rate between 78 and 97%, as shown in Table 3.

The equalization tank sample initially contained 43% particulate COD and 57% soluble COD. At ozone feeding time of 15 minutes this ratio changed to 20/80% in favor of the soluble COD fraction because of hydrolysis mechanism. The impact of ozonation on soluble COD fractions; namely readily biodegradable COD (S_S), rapidly hydrolysable COD (S_H) and inert COD (S_I) was also investigated. For contact times utilized in ozonation experiments, ozone preferentially attacked S_S . In summary, pre-ozonation resulted in a significant decrease of readily biodegradable COD, generated a significant level of rapidly hydrolysable COD and did not appreciably affect the initial level of soluble inert COD.

Respirometric evaluation of the biological treatment effluent indicates from the OUR profile shown in Figure 2a, that it contains only non-biodegradable organic matter. In the

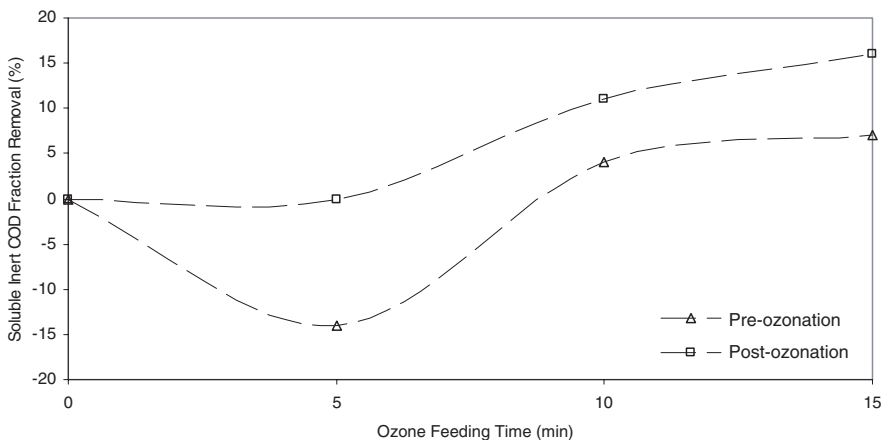


Figure 1 Relationship between soluble inert COD removal and ozone contact time

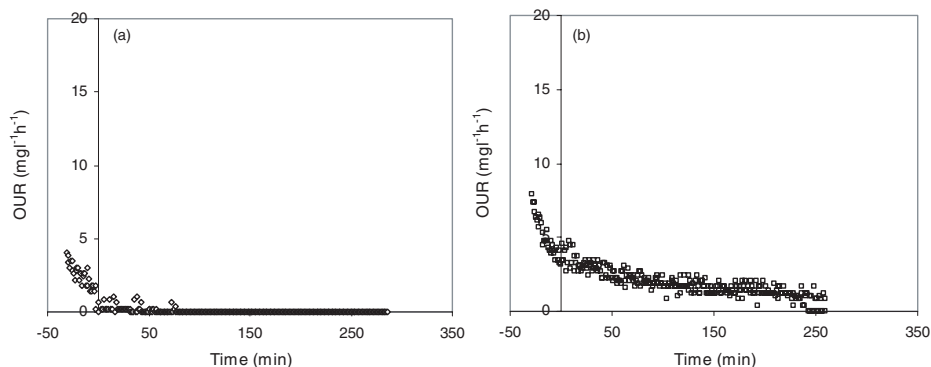


Figure 2 OUR profiles of biologically treated wastewater. (a) Non-ozonated biologically treated wastewater. (b) Ozone contact time of 15 minutes

effluent, the ratio between particulate and soluble COD components seemed to be rather constant, maintaining the level of around 10/90%. Ozone was found to be only effective after 10 minutes of contact time with post-ozonation. Ozonation for 5 minutes maintained all soluble COD fractions at the same level. A minimal increase in S_H and a slight decrease in S_I were observed at higher contact times. For all ozone feeding times applied no S_S generation was obtained. Post-ozonation, aside from achieving higher color and inert COD removal efficiencies compared to the pre-ozonation alternative, proved quite attractive from an economical standpoint, involving approximately 50% less ozone utilization at the same ozone flux rate and contact time.

Conclusions

Pre-ozonation of the equalization tank sample results in a higher color (from 48% to 85%) and a limited COD removal (from 4% to 19%) indicating the existence of a selective reaction. By evaluating the impact of ozone on soluble and particulate COD components, S_T generation through particulate COD breakdown was observed. The effect of ozonation on soluble COD fractions has shown that the oxidation of readily biodegradable COD was the major mechanism. In this respect, pre-ozonation induced a competing mechanism with biological treatment. It was observed to secure a significant shift from particulate slowly hydrolysable COD to rapidly hydrolysable COD. Soluble inert COD concentration was increased at lower doses, due to generation of less biodegradable organic by products. It slightly decreased at higher contact times.

Post-ozonation on the other hand, provided an almost complete color removal and a higher removal for the soluble inert COD, the only soluble COD component in the effluent. It generated no readily biodegradable COD and a very little rapidly hydrolysable COD. The soluble inert COD removal rate was relatively higher compared to pre-ozonation. It also proved to be more efficient, as the utilized ozone with post-ozonation was approximately 50% of the ozone applied to the equalization tank sample.

Consequently, chemical oxidation with ozone after biological treatment proved more effective in terms of color and soluble inert COD removal efficiencies. The selective preference of ozone for simpler organic compounds could also be avoided with post-ozonation application. In conclusion, for the textile wastewater containing high levels of color and soluble inert COD, chemical oxidation with ozone after biological treatment may be recommended as the appropriate additional treatment step in meeting stringent effluent limitations.

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