

In-plant control applications and their effect on treatability of a textile mill wastewater

H. Dulkadiroglu, G. Eremektar, S. Dogruel, H. Uner, F. Germirli-Babuna and D. Orhon

Istanbul Technical University, Civil Engineering Faculty, Environmental Engineering Department, Ayazaga Kampüsü, 80626, Maslak, Istanbul, Turkey

Abstract Water minimization and exploration of the potential for wastewater recovery and reuse are priority issues of industrial wastewater management. They are extremely significant for the textile industry commonly characterized with a high water demand. The study presents a detailed in-plant control survey for a wool finishing plant. A comprehensive process profile and wastewater characterization indicate that process water consumption can be reduced by 34%, and 23% of the wastewater volume can be recovered for reuse. Treatability of reusable wastewater fraction and the effect of in-plant control applications on effluent treatability were also investigated.

Keywords Industrial pollution control; in-plant control; textile industry; treatability; wastewater reuse; water conservation; wool finishing

Introduction

Implementation of in-plant control techniques for textile industries may be tailored for the main purpose of achieving significant reductions in water use, raw material and energy consumption, wastewater production and in some cases even wastewater load (UNEP IE, 1994). In-plant control applications can be grouped under four headings: (i) water minimization (water conservation); (ii) wastewater recovery and reuse; (iii) chemical substitution and (iv) recovery of valuable substances (material reclamation) (Van Veldhuisen, 1991). Significant reductions in water use can be achieved by preventing the unnecessary water consumption practices in textile mills. On the other hand, one of the major in-plant control techniques is the recovery of wastewaters originated from one operation for reuse in a second operation, directly or after being treated. The usage of inefficient washing equipment, poor housekeeping practices, feeding freshwater at all operations requiring water and the application of excessively long washing cycles leading to consumption of unnecessarily high amounts of water are among the reasons of obtaining elevated wastewater volumes (UNEP IE, 1994).

Segregating the relatively less polluted wastewater fraction for reuse (Orhon *et al.*, 2000, 2001a) and preventing unnecessary water consumption are observed to generate a stronger wastewater to be treated. Thus, the feasibility of such in-plant control applications must be evaluated by comparing the savings obtained on fresh water demand versus elevated end of pipe wastewater treatment costs together with cost of treating reusable streams, where applicable.

In this context, this study involves a comprehensive plant survey including wastewater generation and characterization together with the identification of recoverable streams and the assessment of unnecessary water consumption for a selected wool finishing plant. The systematic approach presented can be adopted for other industrial applications dealing with textile processing. The study also outlines the results of a detailed treatability survey. Biological treatability studies covering the assessment of COD fractionation are conducted on raw wastewater samples obtained before and after the application of proposed appropriate in-plant control measures. In this context, the effect of reuse and water conservation

practices on the biological treatability of the plant effluent is evaluated. Treatability of reusable wastewater fraction is also investigated.

Characteristics of the investigated plant

The wool finishing plant investigated in the study handles previously *dyed wool*, *wool-lycra*, *wool-polyester* and *wool-polyester-lycra blends* fabric-finishing operations. The plant operates nine different processes and all of them are batch-wise operations. Acetic acid together with different types of detergents, crease-proofing agents and softeners are added to either fill and draw or shower baths in order to obtain the required finishing on fabrics. As can be seen from the production data summarized in Table 1, three types of previously dyed fabric namely; *A type*, *B type* and *C type*; are subjected to finishing operations in the plant. When a dyeing process is applied to fabric, such fabrics are defined as A type of fabrics. The fabrics manufactured from dyed yarns are named as B type of fabrics. Lastly, C type fabrics are the fabrics produced from dyed tops or dyed fibers.

Five different processes namely, *B type-100% wool and 96% wool + 4% lycra fabric*; *C type-100% wool and 96% wool + 4% lycra fabric*; *A type-50% wool + 50% polyester and 48% wool + 48% polyester + 4% lycra fabric*; *B type-50% wool + 50% polyester and 48% wool + 48% polyester + 4% lycra fabric*; and *C type-50% wool + 50% polyester and 48% wool + 48% polyester + 4% lycra fabric* finishing processes are selected for the investigation in terms of identifying their recoverable wastewater streams and water conservation practices. The selected processes constitute approximately 82% of the total production. A *type-100% wool and 96% wool + 4% lycra* fabric finishing operations are not included in the survey as the used wide washing equipment is not suitable for wastewater recovery and water conservation applications.

Materials and methods

Source-based wastewater samples were collected from the selected processes. Wastewater samples were obtained from each discharge of fill and draw rinsing baths. A sampling frequency of every 2 or 3 minutes for short shower rinsings was applied, while sample collection every 10 minutes was used for long shower rinsings. Temperature, pH, conductivity, total and soluble COD, and color values for all collected samples were detected in order to identify the unnecessary water consumption points and reusable streams. Apart from COD, all analyses for conventional characterization were performed as defined in *Standard Methods* (1998). COD measurements were accomplished by ISO 6060 (1986) method.

Table 1 Production data

Process	Processed product (kg day ⁻¹)	Type of equipment	Production (%)
<i>100% wool and 96% wool + 4% lycra</i>			
A Type dyed fabric finishing	907	Wide Washing	13.6
→ B Type dyed fabric finishing	483	Turbo	7.2
→ C Type dyed fabric finishing	1,256	Turbo	18.8
<i>50% wool + 50% PES and 48% wool + 48% PES + 4% lycra</i>			
→ A Type dyed fabric finishing	1,135	Spiral	17.0
→ B Type dyed fabric finishing	1,263	Turbo	18.9
→ C Type dyed fabric finishing	1,138	Turbo	20.1
<i>Others</i>			
A Type dyed fabric finishing	52	Various	0.8
B Type dyed fabric finishing	196	Various	2.9
C Type dyed fabric finishing	39	Various	0.6

→ Investigated processes

Filtrates of samples subjected to vacuum filtration by means of Millipore membrane filters with a pore size of 0.45 μm were defined as soluble fractions. The Millipore AP40 glass fiber filters were used for suspended solids (SS) and volatile suspended solids (VSS) measurements. Absorbance measurements were conducted on samples filtered from 0.45 μm membrane filters, at 3 different wavelengths, namely 436, 525 and 620 nm. Pharmacia LKB Novaspec II model spectrophotometer was used for this purpose. All experiments were conducted at room temperature. pH adjustments were made by NaOH or H_2SO_4 solutions. Each data point in the study was calculated as the mean of three replicate measurements.

Three composite wastewaters representing; *raw* wastewater, *reusable* wastewater and *remaining* wastewater after separation of the streams suitable for recovery and reuse; were prepared in proportion to their relative flow rates in the processing scheme.

Chemical treatability experiments consisting ozonation and physico-chemical precipitation, were conducted on the reusable wastewater. Ozone used for the ozonation experiments was produced by means of a laboratory *ozone generator PCI GL1*. The experiments were conducted at 15 psi (103.45 kPa), using a sample of 1 litre in a 1.5 litres 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. Two gas washing bottles, connected in series, containing 2% KI solution were connected to the reactor for the determination of ozone output. Lab-scale jar-test apparatus adjusted to provide 5 minutes flash mixing, 30 minutes flocculation and 30 minutes settling was used for coagulation flocculation with $\text{FeCl}_3 \cdot \text{H}_2\text{O}$ and alum [$\text{Al}_2(\text{SO}_4)_3 \cdot 18\text{H}_2\text{O}$]. Anionic polyelectrolyte, UCE AP273 was also added when needed. Whereas, three subsequent 3 minutes flash mixing and 3 minutes settling jar-test cycle was adopted for sodium bentonite applications. Biological treatability studies for the determination of COD fractions were conducted on *raw* and *remaining* composite wastewater samples. The particulate and soluble inert COD components, X_{I1} and S_{I1} of the wastewater were determined according to an experimental procedure proposed by Germirli *et al.*, 1993. Respirometric measurements conducted as batch tests with the seed biomass taken from a fill and draw reactor operated at a sludge age of 10 days, were adopted for the assessment of the readily biodegradable COD, S_{S1} (Ekama *et al.*, 1986). The reactors were started with an initial F/M ratio of 0.6 g VSS (g COD) $^{-1}$. They were constantly aerated to maintain a dissolved oxygen concentration of 6–8 mg l^{-1} and pH of reactors was kept at 7–7.5 by adding phosphate buffer. For the determination of S_{S1} , the heterotrophic yield coefficient, Y_{H} , was accepted as 0.67 g COD (g COD) $^{-1}$ as given in literature for a similar wastewater (Orhon *et al.*, 2000). OUR measurements were conducted with a *WTW OXI DIGI 2000* oxygen meter. The following mass balance equations on soluble COD, S_{T1} and total COD, C_{T1} of the wastewater, were applied to calculate the remaining COD fractions namely, influent rapidly hydrolysable COD, S_{H1} and slowly hydrolysable COD, X_{S1} :

$$S_{\text{H1}} = S_{\text{T1}} - S_{\text{S1}} - S_{\text{I1}} \quad (1)$$

$$X_{\text{S1}} = C_{\text{T1}} - S_{\text{T1}} - X_{\text{I1}} \quad (2)$$

Methodology for in-plant control

Characterization of segregated wastewater streams is the first step in an in-plant control methodology covering water conservation and reuse applications. The obtained results must then be evaluated in a way to assess the unnecessary water consumption points and identify reusable wastewater streams throughout the operations. Although it is possible to

Table 2 Reuse criteria for textile dyeing wastewaters

Parameters	Li and Zhao (1999)	Hoehn (1998)
pH	6.5–8.0	6.5–7.5
Total COD (mg l ⁻¹)	0–160	<50
TSS (mg l ⁻¹)	0–50	<500
TDS (mg l ⁻¹)	100–1,000	
Total hardness (mg CaCO ₃ l ⁻¹)	0–100	90
Chloride (mg l ⁻¹)	100–300	<150
Total chromium (mg l ⁻¹)	0.1	
Iron (mg l ⁻¹)	0–0.3	0.1
Manganese (mg l ⁻¹)	<0.05	0.05
Conductivity (μS cm ⁻¹)	800–2,200	
Alkalinity (mg CaCO ₃ l ⁻¹)	50–200	

find a few reuse criteria for textile dyeing wastewaters in literature, there is no consensus on the given figures as shown in Table 2 (Hoehn, 1998; Li and Zhao, 1999).

Therefore, the specific demands of the manufacturer, together with the mentioned criteria must be considered, while defining the reusable wastewater streams. It must be kept in mind that the product quality is the key term in such in-plant control applications. The required product quality must be maintained after water conservation and reuse applications.

B type 50% wool + 50% polyester and 48% wool + 48% polyester + 4% lycra dyed fabric finishing operation is selected to illustrate the adopted methodology. As can be seen from the process flow-chart given in Figure 1, this finishing process consists of 6 rinsings of which 3 are fill and draw type rinsing and the rest is shower rinsing; and a subsequent fill and draw softening step. Acetic acid, detergent and softening agents are the used auxiliaries added to different baths.

An example of the results of characterization study performed on each discharge of fill and draw rinsing baths and samples collected from shower rinsings with various frequencies is tabulated in Table 3.

According to the results obtained from the investigation of selected processes, the following points are proposed for water conservation and reuse:

1. As all the wastewater samples contain fibres originating from the fabric, total COD values differ considerably for each replicate analysis (the figures given in the table for total COD correspond to mean values). Soluble COD values are more dependable for evaluation in this respect.
2. Shower rinsings can be ended after a point where the soluble COD values of the wastewaters reach 50 mg l⁻¹.

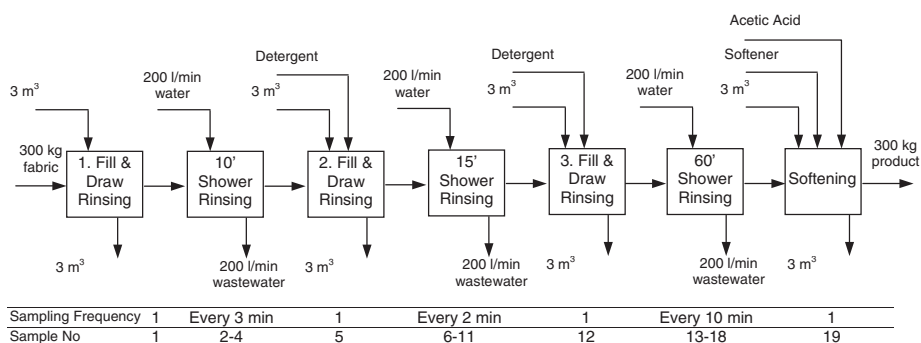


Figure 1 Flow-chart of the selected process

Table 3 Results of the detailed characterization study conducted on the selected process

Sample No (Sampling frequency)	pH	Conductivity ($\mu\text{S cm}^{-1}$)	Total COD (mg l^{-1})	Soluble COD (mg l^{-1})	Absorbance		
					436 nm	525 nm	620 nm
1	6.23	440	950	305	0.072	0.042	0.030
2 (3')	6.47	410	760	220	0.053	0.033	0.024
3 (6')	6.59	400	260	75	0.015	0.009	0.006
4 (9')	6.54	400	250	70	0.013	0.008	0.005
5	5.85	540	2,460	1430	0.126	0.078	0.057
6 (2')	6.62	400	500	350	0.054	0.036	0.027
7 (4')	6.47	400	440	190	0.039	0.023	0.016
8 (6')	6.65	380	280	150	0.022	0.013	0.009
9 (9')	6.37	390	260	110	0.014	0.007	0.005
10 (12')	6.50	390	150	110	0.014	0.006	0.004
11 (15')	6.55	390	140	75	0.013	0.005	0.003
12	6.33	410	940	700	0.058	0.031	0.022
13 (10')	6.79	400	110	65	0.009	0.004	0.004
14 (20')	6.91	400	75	55	0.008	0.004	0.003
15 (30')	9.96	400	50	40	0.007	0.004	0.003
16 (40')	6.96	390	50	35	0.006	0.003	0.002
17 (50')	6.98	390	50	30	0.006	0.003	0.001
18 (60')	7.14	400	30	20	0.005	0.001	0.000
19	4.91	625	1,020	700	0.024	0.017	0.015

3. Streams having soluble COD values lower than 650 mg l^{-1} can be segregated for reuse. This value seems high at a first glance, however when dilutions are considered, a final reusable wastewater fraction with a soluble COD value of approximately 200 mg l^{-1} is obtained. In order to be on the safe side, discharges of first fill and draw rinsings and fill and draw rinsings with auxiliary addition are not summed up with reusable streams even if they have soluble COD values lower than the above mentioned level. Wastewaters originating from first fill and draw rinsings are not considered within the reusable stream as the effect of previous dyeing operations on the wastewater quality can not be predicted soundly. The same uncertainty is also valid in fill and draw rinsings where brands of auxiliaries added differ.

The next step of the adopted methodology is investigating the possible changes in the product quality with applying the proposed water conservation and reuse practices. The result of this survey showed that shower rinsings must not be reduced under 5 minutes for water conservation purposes otherwise the product quality deteriorates.

Table 4 summarizes the proposed in-plant control applications for B type 50% wool + 50% polyester and 48% wool + 48% polyester + 4% lycra dyed fabric finishing operation. According to the results, 60 minutes shower rinsing can be stopped after 20 minutes. By

Table 4 In-plant control applications of the selected process

Sample No	Type of operation	Wastewater generation ($\text{m}^3 (300 \text{ kg fabric})^{-1}$)	Reusable streams ($\text{m}^3 (300 \text{ kg fabric})^{-1}$)	Water conservation streams ($\text{m}^3 (300 \text{ kg fabric})^{-1}$)
1	1st fill and draw rinsing	3	–	–
2–4	10 min. shower rinsing	2	2	–
5	2nd fill and draw rinsing	3	–	–
6–11	15 min. shower rinsing	3	3	–
12	3rd fill and draw rinsing	3	–	–
13–18	60 min. shower rinsing	12	4 (first 20 min)	6 (from 20 to 60 min)
19	Softening	3	–	–
TOTAL		29	9	6

doing so, a water conservation of 6 m³ per 300 kg finished fabric is obtained. All the wastewater streams originating from 10 and 60 minutes shower rinsings together with the rest of 60 minutes shower rinsing (first 20 minutes) can be added to the reusable fraction. As a result 9 m³ out of total 29 m³ per 300 kg finished fabric, can be directed towards reusable wastewaters collection tank.

The results of applying the methodology presented for the example dyed fabric finishing operation indicate that a 34% reduction in water consumption is possible and 23% of the generated wastewaters can be recovered and reused after being subjected to a pretreatment as given in Table 5.

Treatability studies

Treatability of reusable streams

As mentioned earlier some reuse practices require previous treatment. Ozonation and chemical treatment are among the most commonly used pollutant removal methods applied to reusable wastewater streams (Sewekow, 1995). Therefore an experimental study by the use of ozone and various coagulant-flocculants is performed in order to investigate the treatability of reusable streams. Raw wastewater characterization of two samples collected from the reusable streams equalization tank is presented in Table 6. Total chromium, iron and manganese contents of raw reusable wastewater are within the required quality limits given by the manufacturer. TSS, TDS, total hardness, chloride, conductivity and alkalinity levels of the raw reusable wastewater on the other hand are below the limits given in the reuse criteria (Table 2). Therefore only COD concentration must be reduced prior to a reuse application.

Table 5 Results of the proposed in-plant control applications

Process	Processed product [kg fabric (day) ⁻¹]	Water usage			Water conservation		Reusable streams	
		wastewater generation [m ³ (300 kg fabric) ⁻¹]	(m ³ day ⁻¹)	(%)	(m ³ day ⁻¹)	(%)		
<i>100% wool and 96% wool + 4% lycra</i>								
B Type dyed fabric finishing	483	22	35.4	11.3	32	4.8	14	
C Type dyed fabric finishing	1,256	23	96.3	33.5	35	12.6	13	
<i>50% wool + 50% PES and 48% wool + 48% PES + 4% lycra</i>								
A Type dyed fabric finishing	1,135	15	56.8	26.5	47	18.9	33	
B Type dyed fabric finishing	1,263	29	122.1	25.3	21	37.9	31	
C Type dyed fabric finishing	1,338	30	133.8	53.5	40	26.8	20	
TOTAL	5,475	-	444.4	150.1	34	101	23	

Table 6 Characterization of raw reusable wastewater

Parameter	Sample I	Sample II
Total COD (mg l ⁻¹)	180	235
Soluble COD (mg l ⁻¹)	120	175
TSS (mg l ⁻¹)	15	15
TDS (mg l ⁻¹)	340	345
Total hardness (mgCaCO ₃ l ⁻¹)	0	0
Chloride (mg l ⁻¹)	<100	<100
Total chromium (mg l ⁻¹)	<0.5	<0.5
Iron (mg l ⁻¹)	<1	<1
Manganese (mg l ⁻¹)	<0.3	<0.3
Conductivity (μS cm ⁻¹)	550	625
Alkalinity (mgCaCO ₃ l ⁻¹)	135	120
Color (Pt-Co Unit)	20	30
pH	7.10	7.19

The wastewaters of Sample I are subjected to treatability tests with alum, FeCl_3 and sodium bentonite; whereas ozonation experiments are conducted on filtered wastewaters of Sample II from Millipore AP40 glass fiber filters. The results of ozonation experiments tabulated in Table 7 show that, even with extreme ozone dosages, the soluble COD reduction efficiency can only be improved to 40%, giving an effluent soluble COD concentration of above 100 mg l^{-1} .

The results of chemical treatability are given in Table 8. Although manufacturers do not prefer the usage of iron salts as coagulants, since such applications may cause staining problems in the final product, experimental data on the treatability of FeCl_3 is also generated. Good sludge characteristics and sufficient COD removal efficiencies are achieved with sodium bentonite applications, while also adequate removal of total COD is observed to be possible with alum, which presents a much cheaper application.

Effect of in-plant control applications on effluent treatability

As chemical treatment alone gives unreliable and insufficient results in meeting the stringent effluent limitations, biological treatment is the most commonly used technology applied to wastewaters originating from textile finishing industry (Germirli Babuna *et al.*, 1998, 1999; Orhon *et al.*, 2001b). Recent developments in the concept of biological treatability emphasize the importance of biodegradability or in other words COD fractions of wastewaters (Orhon and Artan, 1994). Elimination of unnecessary water consumption and segregation of reusable streams on the other hand, generate a stronger wastewater likely to contain elevated levels of residues to be handled (Orhon *et al.*, 2001a). The main issue to be investigated in this context is assessing the biodegradability of both raw and remaining wastewaters and evaluating the obtained results in terms of feasibility of in-plant control practices. Table 9 shows the conventional characterization of *raw* and *remaining* wastewaters. The *remaining* wastewaters reflect a stronger nature as expected.

Table 7 Results of ozonation tests

Parameter	Ozone feeding time				
	-	5 minutes	10 minutes	15 minutes	30 minutes
Ozone flux (mg min^{-1})	-	58	58	58	58
Utilized ozone (mg)	-	25	150	405	1255
Soluble COD (mg l^{-1})	175	155	140	130	105
Soluble COD removal (%)	-	11	20	26	40
Conductivity ($\mu\text{S cm}^{-1}$)	625	640	620	620	600
Alkalinity ($\text{mgCaCO}_3 \text{ l}^{-1}$)	120	110	90	60	60
Color (Pt-Co Unit)	30	10	0	0	0
Color Removal (%)	-	67	100	100	100
pH	7.19	7.68	7.52	7.38	7.31

Table 8 Results of chemical treatability

Parameter	Alum			FeCl_3			Sodium bentonite		
	50	75	100	50	75	100	500	1,000	1,500
Dosage (mg l^{-1})	50	75	100	50	75	100	500	1,000	1,500
Total COD (mg l^{-1})	55	65	30	70	65	60	75	85	55
Total COD removal (%)	69	64	83	61	64	67	58	53	69
Conductivity ($\mu\text{S cm}^{-1}$)	600	600	600	570	580	590	580	660	900
Alkalinity ($\text{mgCaCO}_3 \text{ l}^{-1}$)	50	90	65	125	105	85	155	180	240
Color (Pt-Co Unit)	0	0	0	0	0	0	0	0	0
TDS (mg l^{-1})	405	405	400	385	350	355	350	455	615
SVI (ml g^{-1})	140	105	105	95	115	155	30	25	15
pH	6.03	6.26	6.26	6.50	6.41	6.59	7.63	7.07	7.59

Table 9 Conventional characterization of raw and remaining wastewaters

Parameter	Raw wastewater	Remaining wastewater
Total COD (mg l ⁻¹)	687	1,460
Soluble COD (mg l ⁻¹)	455	970
TSS (mg l ⁻¹)	85	190
VSS (mg l ⁻¹)	80	180
TDS (mg l ⁻¹)	380	640
TKN (mg l ⁻¹)	19.5	30
NH ₃ -N (mg l ⁻¹)	8	18
TP (mg l ⁻¹)	0.8	1.2
Conductivity (μS cm ⁻¹)	620	655
Alkalinity (mgCaCO ₃ l ⁻¹)	108	106
Color (Pt-Co)	220	440
pH	7.11	6.16

Table 10 tabulates the COD fractions of raw and remaining wastewaters. Soluble inert COD, S_{I1} , and particulate inert COD, X_{I1} , both by-pass the treatment system without being affected by biochemical reactions. The readily biodegradable COD, S_{S1} , readily hydrolysable COD, S_{H1} and slowly hydrolysable COD, X_{S1} are the components of biodegradable part.

According to the figures given in the table, the biodegradable COD accounts for 85% of both raw and remaining wastewaters. Raw and remaining wastewaters have the same ratio of initial soluble inert COD and initial particulate inert COD, 5% and 10%, respectively. Both wastewaters contain hydrolysable fractions that are similar to each other. Application of in-plant control practices practically has no effect on the COD fractionation, although it increases the initial soluble inert COD level over 100%.

Conclusions

In-plant control applications covering water conservation and reuse alternatives, treatability of reusable wastewater fraction and the effect of in-plant control on the biological treatability of effluents have been investigated for a textile plant dealing with wool finishing operations in this study. The following issues can be emphasized to highlight results obtained.

Reusable streams and unnecessary water consumption points throughout the operations are identified by means of a comprehensive process profile and wastewater characterization. The product quality is checked after the application of water conservation practices. The experimental results indicate that a 34% reduction in water consumption is possible and 23% of the generated wastewaters can be recovered and reused after being subjected to a suitable pretreatment. Ozonation of reusable streams does not give satisfactory results, while adequate removal efficiencies can be achieved with alum and sodium bentonite.

The application of in-plant control has no distinct effect on the COD fractionation of wastewater streams as both raw and remaining wastewaters are observed to contain similar

Table 10 COD fractionation of wastewaters

Influent COD Fractions	Raw wastewater		Remaining wastewater	
	[mg l ⁻¹]	[%]	[mg l ⁻¹]	[%]
Readily biodegradable COD, S_{S1}	220	32	485	33
Initial soluble inert COD, S_{I1}	32	5	67	5
Rapidly hydrolysable COD, S_{H1}	203	30	418	29
Slowly hydrolysable COD, X_{S1}	160	23	341	23
Initial particulate inert COD, X_{I1}	72	10	149	10
Total COD	687		1,460	

readily biodegradable, rapidly and slowly hydrolysable fractions and initial inert COD ratios. Such an application imparts, however, a higher soluble inert COD level due to dealing with a more concentrated remaining wastewater. An increase over 100% is determined in initial soluble inert COD level when in-plant control measures are implemented.

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