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Research article

Analysis of the best available techniques for wastewaters from a denim manufacturing textile mill

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ABSTRACT

The present study was undertaken as the first plant scale application and evaluation of Best Available Techniques (BAT) within the context of the Integrated Pollution Prevention and Control/Industrial Emissions Directive to a textile mill in Turkey. A "best practice example" was developed for the textile sector; and within this context, BAT requirements for one of the World's leading denim manufacturing textile mills were determined. In order to achieve a sustainable wastewater management; firstly, a detailed wastewater characterization study was conducted and the possible candidate wastewaters to be reused within the mill were identified. A wastewater management strategy was adopted to investigate the possible reuse opportunities for the dyeing and finishing process wastewaters along with the composite mill effluent. In line with this strategy, production processes were analysed in depth in accordance with the BAT Reference Document not only to treat the generated wastewaters for their possible reuse, but also to reduce the amount of water consumed and wastewater generated. As a result, several applicable BAT options and strategies were determined such as reuse of dyeing wastewaters after treatment, recovery of caustic from alkaline finishing wastewaters, reuse of biologically treated composite mill effluent after membrane processes, minimization of wash water consumption in the water softening plant, reuse of concentrate stream from reverse osmosis plant, reducing water consumption by adoption of counter-current washing in the dyeing and finishing processes. The adoption of the selected in-process BAT options for the minimization of water use provided a 30% reduction in the total specific water consumption of the mill. The treatability studies adopted for both segregated and composite wastewaters indicated that nanofiltration is satisfactory in meeting the reuse criteria for all the wastewater streams considered.

1. Introduction

The textile industry is a water intensive sector with a great variety of process steps requiring use of large amounts of water and chemicals (GilPavas et al., 2017; Yurtsever et al., 2016b). This fact put efforts on the minimization of use of, and where applicable reuse of, raw materials and water within the production steps. Being a water intensive sector (typically 200–400 L per kg of fabric) (Dasgupta et al., 2015); in textile industry, wastewater discharges are of primary concern rather than gaseous emissions and solid wastes. Given the great variety of fibres, chemicals and other auxiliaries in use, textile manufacturing processes generate wastewaters which contain residuals of a wide variety of chemicals and auxiliaries of different nature and therefore are not adequately treated in conventional wastewater treatment plants (Dasgupta et al., 2015). The presence of dyes, metals, phenols, toxic compounds and/or phosphates of which are mostly resistant to conventional biological treatment, can pass untreated through the conventional wastewater treatment systems, and end up in the receiving streams where they may cause adverse effects. It is therefore important to minimize the use of these chemicals which result in difficult-to-treat wastewaters via chemical substitution and also excess use of water in textile plants (EU, 2003). The

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stringent environmental regulations for discharge today are forcing the dyers and finishers in the textile industry to examine the potential for recycling the water from the waste stream by new technologies (Van der Bruggen et al., 2004).

Ozturk et al. (2015a,b) indicated the importance of the implementation of in-plant control techniques for textile industries for the purposes of achieving significant reductions in water use, raw material and energy consumption, wastewater production and in some cases even reductions in wastewater load. They conducted a comprehensive plant survey including wastewater generation and characterization together with the identification of recoverable streams and the assessment of unnecessary water consumption in a textile mill employing wool and acrylic fibre production and subsequent dyeing, and indicated that significant levels of reductions in consumptions and discharges/emissions could be achieved by the application of suggested best available techniques (BAT) options. Van Veldhuisen (1991) grouped in-plant control applications under four headings: (i) water minimization (water conservation); (ii) wastewater recovery and reuse; (iii) chemical substitution, and (iv) recovery of valuable substances (material reclamation). 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 for reuse, directly or after being treated which might result in a stronger wastewater as dictated by the water conservation hierarchy improving environmental and economic performance of industrial facilities (Orhon et al., 2000, 2001).

The European Integrated Pollution Prevention and Control (IPPC) Bureau produces sectoral BAT reference documents, so-called BREFs to describe applied techniques, present emissions and consumption levels, techniques considered for the determination of BAT, giving special consideration to the criteria listed in Annex III of the Industrial Emission Directive (IED) (2010/75/EU) that repealed and replaced Directive 2008/1/EC on IPPC. The BREFs are the main reference documents used by the European Union (EU) Member States when issuing operating permits/licences for the activities specified in the IED. The BREFs are considered as the tools that will provide better consistency of implementation across the EU Member States. It is also likely that requirements for setting emission limits will be more consistently applied across Europe with the availability of up to date BREF documents. Thus, in assessing BATs for wastewater emissions from any IED activity, BREFs can be taken as the major reference along with other references available in the literature.

Ibáñez-Forés et al. (2013) proposed a methodology for identifying sustainable and most appropriate BAT for industrial facilities. The methodology adopted involves the use of life cycle assessment (LCA) approach to guide the selection of candidate BAT options for the control of hot spots identified in industry, and an environmental, economic, technical and social assessment. The application of the proposed approach was illustrated by a case study on ceramic tiles production. Chung et al. (2013) suggested a similar methodology addressing the environmental and economic concerns and competitiveness that are associated with the BAT for wastewater facilities in the leather tanning and finishing industry. The techniques that are applied in the leather tanning and finishing industry were compared with regard to regulatory compliance, economic feasibility, and environmental and technical aspects. Bréchet and Tulkens (2009) applied a multi-dimensional methodology to identify best combinations of these available techniques at the plant level, applied it to a plant in the lime industry; and concluded that there is in general not a single BAT, but well a best combination of BAT to be used. The tool developed consists of a decision tool based on linear programming modelling of the operations applied during production and on internalization of the external costs generated by these operations.

In a recent article, Ozturk et al. (2015b) employed multi-criteria decision-making methods to determine the most suitable BAT for a textile mill. A total of 14 BAT including good management practices, water minimization and chemical minimization/substitution were considered and a feasibility study was carried out to determine potential benefits and savings for each candidate BAT. The potential savings achievable after the implementation of BAT were indicated as 43.51% in water consumption, 16.39% in chemical consumption, and 45.52% in combined wastewater flowrate. In another recent study (Ozturk et al., 2016), it was indicated that in textile wastewater management, various wastewater streams can be segregated and directly reused without treatment in the production processes. After segregation of relatively clean wastewater streams, the remaining combined wastewater could be reused after employing advanced treatment technologies.

Due to highly polluted nature of textile wastewaters, water reuse may only be possible after proper treatment, which depends on the concentrations of pollutants and the reuse criteria. The possible treatment options include both physiochemical (coagulation-flocculation (Verma et al., 2012), adsorption (Jorfi et al., 2017), membrane filtration (Dasgupta et al., 2015), advanced oxidation (Soares et al., 2017), etc.) and biological (activated sludge (Kumar et al., 2014), anaerobic treatment (Yurtsever et al., 2016c), membrane bioreactor (Jegatheesan et al., 2016), etc.) processes. Verma et al. (2012) reported that more effective color removal is possible with novel pre-hyrolysed coagulants such as Polyaluminium chloride (PACl), Polyaluminium ferric chloride (PAFCl), Polyferrous sulfate (PFS) and Polyferric chloride (PFCl). The color removal efficiency of the process (58–100%) highly depends on the nature of wastewater, used coagulants and the aids, dosage, and pH. Although activated sludge treatment performance may be satisfactory to discharge the wastewater to sewerage, it may require additional physicochemical treatment to meet reuse criteria (Sahinkaya et al., 2008). In order to improve the biological process performance, membrane bioreactors (MBRs) are considered as a possible alternative. Yurtsever et al. (2016a, 2016c, 2015) reported that anaerobic MBR can also be effectively used in decolorization of textile wastewater and the process has high tolerance to increased salinity and sulfate concentration. In another study, Malpei et al. (2003) evaluated the feasibility of upgrading a full scale activated sludge process by a MBR and compared the performance of the processes. In the activated sludge process, although high COD removal efficiency (~90%) was observed, the effluent COD and TSS showed high fluctuations, i.e. 90–490 mg/L and 40–60 mg/L, respectively. In the MBR, the fluctuation was only 2.3% with the average effluent COD concentration of 137 ± 39 mg/L with free of TSS. Similarly, color removal efficiency of MBR was 96.5%. Hence, water may be reused after MBR treatment if high conductivity of the treated water is not a concern. Generally, reuse criteria may not be met after biological and/or chemical treatment due to remaining COD, color and especially dissolved inorganics (high conductivity) in the treated effluent. Hence, nanofiltration (Dasgupta et al., 2015) or reverse osmosis processes (Zheng et al., 2015) may be required for the remaining pollutant and conductivity removal.

This study aims to evaluate BAT for the treatment of the wastewater from a denim manufacturing mill in Turkey, within the framework of the EU's IED/IPPC philosophy. The plant produces denim textile starting from raw cotton. Textile production starts with fibre manufacturing, continues with sizing, dyeing, weaving and ends up with finishing. Among these; sizing, dyeing and finishing are the wet processes. The dyeing process of the factory is composed of pre-treatment, dyeing, rinsing and softening operations; and uses indigo, indanthrene and sulphur dyes either individually or sequentially. The finishing process has chemical treatment and rinsing stages requiring use of large quantities of chemicals and water, producing hot and alkaline wastewaters. The plant extracts water from wells; softens through ion exchange and reverse osmosis processes and discharges its biologically treated wastewaters into the sewer line that ends up with a municipal wastewater treatment plant. The mill produces 20,000 tons of cotton fibre; 45 million meter of denim fabric per year with a daily water consumption of about 7500 tons, which corresponds to water consumption before the implementation of selected in-plant BAT measures (Kocabas et al., 2009). This is the first plant scale implementation of the Integrated Pollution Prevention and Control/Industrial Emissions Directive and the respective BREF document to a textile mill in Turkey.

2. Methods

In order to develop water minimization strategies, a detailed study program was undertaken. Some of the identified strategies were implemented at the textile mill to evaluate the efficiencies of the selected strategies in reality. The approach used for the identification of the strategies are provided below.

2.1. Approach

In the present study, firstly a detailed plant-wide survey was conducted and data on different wastewater streams were collected. The findings have indicated that dyeing and finishing processes are responsible for about 80% of the total wastewater generation. Thus, water reuse in dyeing and finishing processes appeared as essential in terms of water minimization. To this end, a wastewater sampling program was carried out for a period of one and a half month time for the segregated wastewater streams of dyeing and finishing processes and also for the composite effluent stream to identify the pollution profiles and to evaluate the possible reuse alternatives. Secondly, the BAT options for the management of wastewaters and also for the minimization of water consumption including reuse options were identified based on a comparison with the BREF Textile Document. The applicability of these options at the plant scale was discussed and evaluated with the factory technical management. Also, additional water minimization strategies that were not covered by the BREF Textile Document were developed by the mill staff considering their technical and economic viability. Amongst the identified BAT options, some were “in-plant” measures and not “end-of-pipe” type, and some of these were implemented straightaway by the plant and provided considerable reductions in overall specific water and energy consumptions (Kocabas et al., 2009).

Initial investigations carried out in the mill have indicated that, none of the wastewater streams could be directly reused in the plant and some streams were identified as reusable after proper treatment. In line with these findings, a wastewater management strategy was adopted which includes treatability of the candidate wastewater streams for their possible reuse within the mill through physicochemical/biological treatment process alternatives considering the reduction achieved/to be achieved in water consumption via employing BAT measures.

The wastewater in the dyeing plant is mainly composed of spent dye bath wastes, rinsing wastewaters, and machinery cleaning wastes. Rinsing wastewater samples were taken from pre-rinsing tanks which are located prior to the dye baths, and from post-rinsing tanks following dyeing. Spent dye baths and machinery cleaning wastes were not sampled separately; instead samples were taken from the overall effluent channel of the dyeing line. The wastewater from the finishing line was sampled from the rinsing tanks and the overall effluent channel. The parameters to be analysed in the collected samples were identified in accordance with the BREF Textile Document and the water reuse criteria for textile wastewaters (see Table 1). Major parameters selected for the characterization were Chemical Oxygen Demand (COD), color, conductivity, and turbidity along with some others as presented in Table 1.

Table 1
Water reuse criteria for textile wastewaters (Capar et al., 2006).

Parameter	Reuse criteria
COD (mg/L)	80
Color (Pt-Co)	20
Turbidity (NTU)	1
Suspended solids (mg/L)	5
Dissolved solids (mg/L)	500
Total hardness (mg/L as $CaCO_3$)	60
Conductivity (μS/cm)	1000
pH	6–8

After the detailed wastewater characterization study, a wastewater management strategy was developed for the mill. For this purpose, wastewater streams that may be segregated and/or that can be treated for possible reuse within the plant were identified. Alternative treatment processes which might be applicable and which are worth to investigate for the wastewater streams in the plant were selected. As indicated before, not only wastewater treatability studies, but also some in-plant BAT measures for the minimization of water consumption and therefore minimization of wastewater generation were considered. Table 2 summarizes the treatment technologies applied to different wastewaters for their possible reuse within the mill. The wastewater treatability options were initially screened taking into account their COD and color removal efficiencies, water recovery performances and sludge generation potentials to determine the most appropriate technically applicable alternative/s.

2.2. Analytical methods

During wastewater characterization studies, COD and color measurements were performed using HACH DR-2000 Model spectrophotometer following USEPA approved HACH Method No.8000. Conductivity and pH measurements were conducted using Hach Sension 378 pH-Conductivity meter. Turbidity, Total Suspended Solids (TSS) and alkalinity measurements were performed according to the Standard Methods (APHA, 2012).

3. Results and discussion

The results of the present study have been grouped and discussed in two main sections. Firstly, the characterization studies of the wastewater streams are presented and secondly the wastewater management strategy including both the reduction of water

Table 2
Wastewater treatment studies conducted.

Wastewater	Treatment technology
Indigo dyeing wastewaters	Physicochemical pre-treatment + Membrane filtration
Finishing wastewaters	Membrane filtration
Wastewater treatment plant influent	Biological treatment + Membrane filtration
Wastewater treatment plant influent	Membrane bioreactor

Table 3
Characteristics of the dyeing wastewaters.

Parameter	Value
pH	8.9–12.7
Conductivity (mS/cm)	2.5–35
COD (mg/L)	520-3250
Color (Pt-Co)	650-10,000
Alkalinity (mg/L $CaCO_3$)	240-8500
TSS (mg/L)	90–350
Turbidity (NTU)	25–320

consumption and management and treatment of different wastewater streams are given.

3.1. Wastewater characterization study

The wastewater characterization study was conducted for the dyeing, finishing and composite wastewater streams and the results are presented in Table 3, Table 4 and Table 5, respectively. As expected, none of the wastewater streams were appropriate for direct reuse in the mill. They were all characterized by very high COD and color levels and with extreme pH values.

During the wastewater characterization study, the production scheme in the factory was quite variable and there was denim production with 12 different dyeing recipes. An evaluation of the recipes applied during the sampling period and the past four months prior to the sampling period indicated that the analysed dyeing wastewater samples are representative of the typical production in the denim dyeing line (85% of total production of the factory). Table 3 presents the dyeing wastewater characteristics. Since the production may vary widely not only during a year (because of seasonal changes and fashion) but even over a single day (according to the production programme), the resulting wastewater characteristics are even more difficult to standardise and compare.

The waste mercerization wash water containing caustic and fibres came out to be the major stream to be reused within the finishing process. This stream contains high amount of sodium hydroxide which can be recovered and reused. The plant places special emphasis on this waste stream since it is of great economic value and also demands acid addition for neutralization during the treatment. The characteristics of the finishing wastewater are given in Table 4.

Currently the mill has a wastewater treatment plant employing activated sludge process operated at a quite high food to microorganisms ratio (>0.15 kg BOD_5/kg MLSS.day), which seems contrary to the BAT Textile Document. However, the treatment plant still meets the discharge standards to the sewage system. Nevertheless, the wastewater generated was also subject to an extensive characterization as it was considered part of the overall BAT assessment study.

Table 5
Characteristics of the composite wastewater generated at the mill.

Parameter	Value
COD (mg/L)	2300–2500
Color (Pt-Co)	2000–4000
Conductivity (mS/cm)	5–6

Table 6
Average wastewater quantities (3-year average values).

Process	ton/year	%
Sizing	55,900	5.7
Dyeing	415,500	42.2
Finishing	512,500	52.1
TOTAL	983,900	100.0

Accordingly, characterization studies of the composite wastewater treated in the wastewater treatment plant were performed and the results shown in Table 5 were obtained. As can be seen, the composite wastewater flowing into the wastewater treatment plant was characterized with a very high COD, color and conductivity.

During the wastewater characterization studies, flow rate of each wastewater stream was also considered. To this end, wastewater flow rate data for the last three years were obtained from the plant management and the average values were evaluated. As seen in Table 6, finishing process appears to be the process that generates the highest amount of wastewater; whereas, sizing process is responsible for only 5.7% of the whole wastewater from the mill. From this table, it is clear that the primary attention should be given to dyeing and finishing process wastewaters in the mill to achieve a sustainable water management.

3.2. Wastewater management studies

In developing a wastewater management strategy for the mill, firstly, possible water conservation opportunities were considered. The results from this part of the study are presented below under “Minimization of Water Consumption”. Secondly, for dyeing and finishing wastewaters, optional reclamation techniques were evaluated. The results obtained from these treatability studies are also presented below under the relevant sections, namely, “Management of Dyeing Wastewaters”, “Management of Finishing Wastewaters”, “Management of Composite Mill Wastewater”, “Pilot Scale Membrane Bioreactor (MBR) Application”. In this respect, membrane filtration preceded by physicochemical treatment was proposed for the reclamation of dyeing wastewater. For the finishing wastewater; only membrane filtration was deemed sufficient as this wastewater was lower in TSS. The treatment alternatives considered for the combined wastewater were the

Table 4
Characteristics of the finishing wastewaters (Varol et al., 2015).

Parameter	Wastewater from Post-Rinsing Tank #							Mixed wastewater
	1	2	3	4	5	6	7	
pH	12.3	12.1	11.8	11.7	11.4	11.1	8.9	11.3
Conductivity, mS/cm	150	60	20	13	9	5	1	18
TSS, mg/L	1632	793	248	172	144	131	40	132
TDS, mg/L	61,947	23,043	8348	5186	4279	3154	1151	6236
Alkalinity, mg $CaCO_3$/L	41,163	15,002	4920	3204	2246	1216	382	4196
Color, Pt-Co	10,113	7059	3689	3651	3568	3078	844	2994
Turbidity, NTU	879	584	534	561	406	379	138	289
COD, mg/L	8524	5202	2296	2080	2330	2007	542	1863
NaOH, g/L	67.5	34.7	6.3	2.8	1.9	1.4	–	–

Table 7
Total water consumption in the mill (Kocabas et al., 2009).

Year	Total Water Consumption (ton/day)	Total Water Consumption (kg/meter fabric)
0	7419	55
1[a]	6064	47
2	4813	43
3	4600	40

[a] Start of the project.

application of membrane filtration for the effluent from biological treatment and the application of MBR (Table 2).

3.2.1. Minimization of water consumption

Along with the wastewater characterization studies, a detailed water mass balance analysis was conducted to identify consumption patterns in each process. This is to optimize the use of water and to develop possible minimization options. It was found that water consumption is highly variable in the plant due to variations in the production scheme and should be closely monitored. More than 50% of the total extracted water is used for dyeing and finishing processes; and the rest being used for other purposes such as sizing, steam generation, good housekeeping, etc. Moreover, dyeing and finishing processes are responsible for 80% of the total wastewater generated.

The possible BAT options for the minimization of water consumption were identified based on comparison with the BREF Textile Document (EC, 2003). The applicability of these options at the plant scale was discussed and evaluated with the factory technical management. Also, additional water minimization strategies that were not covered by the BREF Textile Document (EC, 2003) were developed by the mill staff considering their technical and economic viability. The identified BAT options were (Kocabas et al., 2009):

1. *Installation of flow control valves in dyeing and finishing processes*: Installation of flow control valves on the individual machines utilized in different processes enables flow control in processes. Furthermore, flow control valves are utilized in order to document water use and evaluate improvements and hence help in monitoring the water consumption.
2. *Counter-current washing in dyeing and finishing processes*: In this washing method, the cleanest wash water enters at the final wash stage, and the drained water from this stage is then sent backwards through each of the previous stages counter to the fabric. Thus, the more contaminated wash water contacts the more contaminated fabric as it enters the washing process, and the least contaminated wash water contacts the cleanest fabric. This method of washing provides water savings as compared to the traditional washing method of supplying clean water at every stage of the washing.
3. *Reuse of dyeing and finishing wash waters after a treatment scheme*: Wash waters with chemicals and dyestuffs are treated in order to remove the impurities; and, if reuse criteria for textile industry are met, these waters are reused in the processes and hence minimize total water consumed by eliminating the extraction of water from the wells.
4. *Recovery and reuse of sodium hydroxide from mercerization wash waters in the finishing process*: After sodium hydroxide recovery from wash waters of mercerization, water is recirculated into the process to be reused and hence washing efficiency is improved along with reduced chemical cost.
5. *Reuse of concentrate stream of reverse osmosis plant*: the use of concentrate stream for cleaning purposes (e.g. toilets) in the mill improves the total water consumption performance of the mill by eliminating the extraction of water for cleaning purposes.
6. *Minimization of wash water consumption in the water softening plant (reuse of wash waters from regeneration of ion exchangers)*: elimination of excess water consumption in regeneration of ion exchangers improves water consumption performance of the mill, reducing the amount of total extracted water.
7. *Reuse of compressor cooling waters in production processes*: reuse of softened cooling waters of compressors in production processes reduces the amount of water extracted from the wells and thus increase total water consumption performance of the mill.

Among these BAT measures listed above, 1, 2, 5, 6 and 7 were implemented during the project period, 3 and 4 were considered as possible wastewater treatment options and laboratory scale treatability studies were conducted. In the proceeding sections, the results obtained from these studies are presented.

The adaptation of the suggested BAT options to the mill provided a 30% reduction in total water consumption. The total water consumption pattern of the mill is given in Table 7 for a period of four years. A gradual decrease is observed for the project period. Water minimization has been mainly achieved by installing flow control valves to dyeing and finishing processes, use of semi-counter current rinsing in both lines, minimization of wash water consumption in the water softening plant, reuse of concentrate stream from reverse osmosis plant and use of compressor cooling waters in production processes.

3.2.2. Management of dyeing wastewaters

Wastewaters from dyeing process appeared to require high color and COD removals for possible reuse. Coagulation-flocculation and filtration processes were selected as the pretreatment alternatives for the reuse of dyeing wastewaters. The three most commonly applied dyeing recipes were identified and wastewater generated from each recipe was mixed in equal volumes so as to represent the general trend of the dyeing line wastewater. Coagulation-flocculation with aluminum sulfate

Table 8
Results of treatability studies for dyeing wastewaters (Unlu et al., 2009).

Parameter	Mixed dyeing wastewater	Coagulation-flocculation[a] Removal (%)	MF[b] Removal (%)	MF + UF[c] Removal (%)	NF[d] Removal (%)
COD (mg/L)	1000	40–50	25–30	80–85	97
Color (Pt-Co)	6000	40–50	70–75	90–95	99
Conductivity (mS/cm)	6	–[e]	~0	~0	60

[a] Optimum dose was 1000 mg/L for both coagulants.

[b] Millipore MF membranes (Pore sizes 0.45 μm, 5 μm, 8 μm, 10 μm, 20 μm; operating pressure 3 bars).

[c] Millipore PES UF membranes (100, 50, 10, 5 kDa; operating pressure 4 bars).

[d] NF270, Dow-Filmtec, USA.

[e] There occurs an increase in conductivity with coagulation.

Table 9
Results of caustic recovery studies (Varol et al., 2015).

Parameter	Mercerizing wastewater	UF[a] Removal (%)	NF[b] Removal (%)	NF[c] Removal (%)
COD (mg/L)	8500–19,000	83	91	92
Color (Pt-Co)	7600–10,200	93	99	97
NaOH (g/L)	25–68	100[d]	100[d]	100[d]

[a] Polyethersulfone GR95PP, Alfalaval, Denmark.
[b] Polyethersulfone NP010, Microdyn Nadir, Denmark.
[c] Polyethersulfone NP030, Microdyn Nadir, Denmark.
[d] The values presented are caustic recovery values.

($Al_2(SO_4)_3\cdot 18H_2O$) and ferric chloride ($FeCl_3\cdot 6H_2O$), dead-end microfiltration (MF) and MF coupled with ultrafiltration (UF) and cross-flow nanofiltration (NF) were tested. Table 8 shows the characteristics of the mixed dyeing wastewater and the results of its treatability studies.

As presented in Table 8, coagulation-flocculation method is not an efficient pre-treatment alternative for the dyeing wastewaters not only due to low COD and color removals but also due to requirement of high coagulant doses. High coagulant dose leads to elevated treatment costs along with large volumes of chemical sludge to be handled. GilPavas et al. (2017) studied pre-treatment of textile wastewater with coagulation-flocculation process and the COD removal efficiency was 48% under optimum operational conditions, i.e. 700 mg/L $Al_2(SO_4)_3$ at pH 9.96. Compared to coagulation and flocculation process, higher color removal was observed in the MF process (70–75%). The color removal efficiency increased (90–95%) with UF process. Similarly, Harrelkas et al. (2009) reported that COD and color removal efficiencies of MF and UF processes following coagulation and process were 37%, 42% and 65%, 74%, respectively. The membrane filtration technique was found to achieve high color and COD removals. While the sequential application of microfiltration (MF) and ultrafiltration (UF) as pre-treatment was found to be promising in color and COD removals; the effluent required to be further treated by nanofiltration (NF) for an improvement in effluent conductivity. Application of NF was found to remove 60% conductivity and provide reusable water. Similarly, Alcaina-Miranda et al. (2009) reported 41–69% conductivity rejection in the pre-treated textile wastewater depending on the type of nanofilter used (NF270 or Duraslick NF) and pH (5–11). In another study, Bes-pi (2005) tested nanofiltration process with Desal DK2540 NF to remove conductivity from chemically pre-treated textile wastewater. The conductivity reduced from 2.8 mS/cm to around 1 mS/cm with the corresponding efficiency of around 70%.

3.2.3. Management of finishing wastewaters

Preliminary studies as regards to caustic recovery from the highly alkaline hot mercerizing wastewater coming from finishing line have yielded very satisfactory results and a high quality caustic with a concentration of 270–300 g NaOH/L (over 90% of the original spent caustic waste solution volume) that can be recycled back to the mercerization process after an evaporation step.

Although BREF Textile Document (EC, 2003) suggests three-stage evaporation and purification for the recovery of caustic, this process was not applicable in the pilot plant due to foaming problems encountered during operation. Thus, cross-flow membrane filtration was tested at lab scale with polyethersulfone (PES) UF and NF membranes which are resistant to high pH and high temperatures. All membranes tested provided complete caustic recovery at a *trans*-membrane pressure of 4.0 bar. However, NF membranes were found to be superior to UF membrane in the removal of color and COD. Table 9 summarizes the results of the preliminary investigations, which indicate a very high removal of COD and color along with complete recovery of caustic.

Table 10
Results of biological treatment coupled with membrane filtration (Sahinkaya et al., 2008).

	COD (mg/L)	Color (Pt-Co)	Conductivity (mS/cm)
Raw wastewater	2300 –2500	2000 –4000	5–6
Effluent from activated sludge process	200–300	300–600	7–7.5
NF effluent[a]	5–40	0–8	2–2.5
Reuse criteria	80	20	2.5

[a] Membrane Used: MF270, Dow-Filmtec, USA.

3.2.4. Management of composite mill wastewater

As regards the composite mill wastewater, two different treatability studies were conducted. First, the possible reuse of the total mill wastewater was investigated as part of the wastewater management strategy adopted. Second, MBR application was studied as suggested by the BREF Textile Document for combined mill effluents.

The reuse of wastewater after a biological treatment process requires a tertiary treatment to be applied. In the present study, coupling of activated sludge treatment with NF to improve denim textile wastewater quality to reuse criteria was considered (Sahinkaya et al., 2008). A lab scale 10 L completely mixed fed-batch biological reactor was operated by feeding composite wastewater obtained from the mill. No sludge recycling was applied, thus, the hydraulic retention time was kept equal to the solids retention time with a value of 8 days. The reactor temperature was constant at 25 °C, dissolved oxygen concentration was in the range of 2–3 mg/L and the reactor pH changed from 6.5 to 8. The COD removal efficiency observed was 91 ± 2% and 84 ± 4% on the basis of total and soluble COD, respectively. The color removal efficiency was 75 ± 10. The high conductivity of the wastewater, did not adversely affect the system performance. Although biological treatment was quite effective in removing color and COD, it was not satisfactory in meeting the reuse criteria. Hence, further treatment to reach target reuse quality was investigated using NF. In another study, Yurtsever et al. (2016a) reported that salinity in simulated textile industry wastewater did not adversely affect the biological removal performances of color and COD, which should be due to adaptation of biomass to high salinity environment.

The effluent of the biological reactor was diverted to NF process to investigate its possible reuse within the mill. To remove coarse particles, dead-end microfiltration (MF) with 5 μm pore size was applied before NF. The color removal by NF was almost complete and permeate color was as low as 10 Pt–Co (Table 10). Similarly, quite high COD rejections (80–100%) were observed. Permeate conductivity was in the range of 1.98–2.67 mS/cm corresponding to 65% conductivity rejection. As can be seen from Table 10, the final effluent's COD, color and conductivity were below the reuse criteria indicating that the process chain of activated sludge followed by NF is reusable. In parallel, wastewater fluxes were also good; between 31 and 37 L/m^2/h (LMH) at 5.07 bars corresponding to around 45% flux declines compared to clean water fluxes. In conclusion, NF after biological treatment appeared as a possible alternative for denim textile wastewaters to meet reuse criteria. Bes-pi (2005) studied nanofiltration performance of chemically pre-treated textile wastewater. The permeate COD concentration of NF was between 59 and 80 mg/L depending on the volume concentration factor at feed COD concentration of 700 mg/L, corresponding to 89–92% rejection efficiency. Flux was around 60 LMH at 20 bar pressure.

Table 11
Performance of MBR system (Yigit et al., 2009).

Parameter	Feed Average (Min-Max)	Permeate Average (Min-Max)
COD (mg/L)	1411 (686–2278)	37 (13–60)
TSS (mg/L)	137 (60–212)	0.6 (0.4–0.8)
Turbidity (NTU)	292 (194–419)	0.31 (0.18–0.65)
Conductivity (μS/cm)	5125 (1578–9440)	4910 (2820–8150)
Color (Pt Co)	2447 (286–8100)	53 (32–105)

Avlonitis et al. (2008) reported similar performance with TRISEP (4040-XN45-TSF) NF membrane as total decolorization of cotton dye effluents together with 72% salinity rejection were reported.

3.2.5. Pilot scale MBR application

The objective of this part of the study was to investigate the performance of an aerobic MBR system for the treatment of the mixed wastewater from wet processes of the textile industry and the impacts of MBR operation parameters on treatment performances. A pilot-scale MBR system (ZW®-10 unit, GE Water&Process Technologies, Zenon Membrane Solutions) containing a submerged hollow fiber membrane module in the aeration tank was operated aerobically for about three months on-site at a continuous flow mode. The system was operated at two different operational stages: (i) no sludge wastage (infinite solids retention time) with a typical permeate flux of 20 L/m^2-h, and (ii) a solids retention time of 25 days with the same flux.

During the whole operation period, despite the high dissolved solids content in the influent, very high treatment efficiencies were achieved. The performance of the system was not adversely affected by the variations in the influent characteristics, food/microorganism ratio, organic loading rate, specific substrate utilization rate and dissolved oxygen level and provided continuous and consistent COD removal and nitrification. At the solids retention time of 25 day, effluent quality was slightly better than at infinite solids retention time condition. The results presented in Table 11 clearly show that complex and highly polluted denim textile wastewaters could be treated very effectively with MBR systems. The average values of some effluent quality parameters found in the entire operation period were turbidity, 0.31 NTU; TSS, 0.6 mg/L; BOD_5, 15 mg/L; COD, 37 mg/L; NH_3-N, 1.0 mg/L; NO_3-N, 9.6 mg/L; and TN, 10.5 mg/L. Color values of as high as 8100 Pt-Co were significantly reduced to about 50 Pt-Co levels, indicating that MBR effluent could be reused in the production process. However, since the dissolved solids could not be removed by UF as employed in MBR processes, further treatment (i.e. NF or reverse osmosis) is required prior to reuse of MBR effluent, especially in dyeing and finishing processes. On the other hand, MBR effluent could be directly reused for washing and rinsing of the tanks in the production line, on-site irrigation, and for other minor usages. Furthermore, the MBR effluent could be mixed with other process waters to decrease fresh water consumption. The results of this part of the study indicated that complex and highly polluted textile wastewaters can be treated much more effectively by MBR processes than conventional activated sludge systems, and the treated wastewaters by MBR have high potential for reuse in the textile industry. Similarly, Malpei et al. (2003) reported high COD (~94%) and color (~97%) removal efficiencies in MBR equipped with UF hollow fibre membrane treating textile industry wastewater. In another study, Niren and Jigisha (2011) reported over 90% COD and color removals in a UF hollow-fibre submerged MBR treating textile industry wastewater.

4. Conclusions

This study addressed the selection/application of the most appropriate and sustainable in-process and end-of-pipe BAT options for the management of wastewaters from a denim textile manufacturing facility. The results indicate that dyeing and finishing wastewaters are with the highest pollution load and therefore with the most environmental impacts due to high COD and color loads. They were found to be responsible for about 80% of the total wastewater generation. Thus, water reuse in these processes appeared as essential in terms of water minimization. In terms of water consumption, these two processes were found to be responsible for more than 50% of the total extracted water. The rest of water was found to be used for other purposes such as sizing, steam generation, good housekeeping, etc.

After a wastewater sampling program that is carried out to identify the pollution profiles of the segregated wastewater streams of dyeing, finishing and also for the composite effluent stream from the industry, the possible end-of pipe BAT options for the management of segregated wastewaters and also in-process BAT options for the minimization of water consumption including direct reuse options were identified, based on comparison with the BAT indicated in the literature. In-process BAT measures taken, resulted in a 30% reduction in the total water consumption of the facility.

The BAT options suggested for the reclamation of segregated wastewater streams were NF of dyeing wastewaters with/without coagulation/MF + UF pretreatment and recovery and reuse of caustic from mercerization wash waters. For the composite plant effluent, NF of biologically treated composites textile mill effluent and MBR treatment of the composites wastewater were adopted. The laboratory-scale tests conducted for all these treatment alternatives were satisfactory in meeting the water reuse criteria. NF was effective in reclaiming caustic from finishing effluent.

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Nomenclature

BAT	Best Available Techniques
BOD_5	5-day Biochemical Oxygen Demand
BREF	BAT Reference
COD	Chemical Oxygen Demand
IED	Industrial Emissions Directive
IPPC	Integrated Pollution Prevention and Control Directive
MBR	Membrane Bioreactor
MF	Microfiltration
MLSS	Mixed Liquor Suspended Solids
NF	Nanofiltration
UF	Ultrafiltration
TDS	Total Dissolved Solids
TN	Total Nitrogen
TSS	Total Suspended Solids

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