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Molecular weight distributions in cotton-dyeing textile wastewaters

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ABSTRACT

Many different chemicals are used in the textile industry, with its effluent being a major pollution source if not treated properly. High-strength textile wastewater requires an environmentally friendly, cost-effective and highly efficient treatment before being discharged into the environment. The goal of this study is to determine the appropriate treatment alternatives of high-strength textile wastewater based on the pollution strength and molecular weight distribution (MWD) analyses of pollutants from a cotton-dyeing textile mill. Sequential filtrations through microfiltration and ultrafiltration membranes were conducted separately on wastewater samples from dying, bleaching and mixed processes. According to MWD analyses, half of the total organic carbon, chemical oxygen demand (COD) and biochemical oxygen demand (BOD_5) found in the wastewater which came from the bleaching and mixed processes were less than 1 kDa, while 56% of the COD and BOD_5 found in the wastewater from dyeing was between 100 and 1 kDa. Moreover, the total kjeldahl nitrogen content of the bleached and dyed wastewaters was mostly higher than 1 kDa. Furthermore, UV_{254} absorbance in the mixed, bleaching and dyeing treatments was <1 kDa with ratios of 61, 49 and 25%, respectively. According to the Specific Ultraviolet Absorption values, the organic contents of all of these processes were hydrophilic. Experimental results indicated that biological and physical methods are more appropriate than chemical methods for treating the effluents of cotton-dyeing.

Keywords: Cotton-dyeing effluents; Molecular weight distribution; Organic matter; Treatment alternatives

1. Introduction

Textile industries generate a huge quantity of wastewater with a high concentration of pollutant constituents. Effluents from textile industries are mainly characterised as having an intense colour and high concentrations of organic and inorganic constituents [1]. Conventional treatment processes have commonly been applied to the treatment of textile wastewater. Applications of individual treatment processes are insufficient for meeting discharge limits, while coagulation–flocculation, along with gravity settling, is one of the most effective methods for partially removing soluble colloids and colours from textile wastewater. Therefore, various combined treatment processes have been recently used for treating that

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kind of wastewater [2,3]. On the other hand, the sole monitoring of changes made to pollutant concentrations is inadequate for providing a deep analysis of the fate of pollutants during the treatment process. Furthermore, a molecular weight distribution (MWD) analysis, as well as an analysis of pollutant concentrations, is useful for evaluating treatment alternatives and monitoring the removal of pollutants [4,5].

The MWD of organic matters constantly changes in all treatment processes. Comprehensive research studies have been done to determine the relationship between MWD and treatment performance. The results from biological treatment studies have indicated that low fractionated organics can be effectively removed by microorganisms [6,7]. Zhao et al. [8] and Campagna et al. [9] reported higher organic removal when the materials had a molecular weight less than 1 kDa, while Dulekgurgen et al. [10] obtained the highest treatment efficiency with less than 2 nm. On the other hand, high-molecular organics could be effectively removed by chemical and physical processes. It has been proven that flocculation plus sedimentation is an effective option for the treatment of pollutants with molecular weights higher than 50 μm. Chian [11], for instance, applied chemical precipitation and obtained elevated removal efficiencies for high-molecular-weight particles (>5 kDa). Similarly, Uner et al. [12] studied the chemical treatability of textile finishing wastewater and reported a 87% COD removal efficiency for particles with high-molecular weight (0.45 μm-100 kDa) compared to a 57% removal efficiency for particles with lower MWs (<1 kDa). Furthermore, conventional filtration methods provided the removal of pollutants with MWs higher than 3 μm [13].

In addition to organics, MWD analyses have been used for the evaluation of other pollution parameters. Ged and Boyer (2013) evaluated the MWD of phosphorus fractions of aquatic dissolved organic matter [14]. Forty-four percentage of dissolved organic phosphorus was less than 1 kDa, with 40% being higher than 10 kDa. Their experimental results indicated that the coagulation process was insufficient for the removal of dissolved organic phosphorous with a molecular weight of less than 1 kDa. The MWD of organic nitrogen was also investigated by Zhao et al. [8]. The majority of nitrogen with an MW with <500 Da was removed by means of a sequencing batch reactor (SBR), while the application of an SBR plus a nanofiltration (NF) membrane increased the treatment efficiency up to 80%. The study conducted by Qian et al. revealed that colour is formed by organic matters with MWs higher than 1 kDa [15]. Researchers reported that the colour removal and treatability of organic matters were increased by the granular activated carbon adsorption/O_3 process (with a range of 1–10 kDa) [15]. Using MWD analysis, Zhao et al. [8] applied NF and obtained 85% removal efficiency of UV_{254} by >500 Da [8].

Effluents from cotton-dyeing processes contain intensive dyeing chemicals and are a high-strength wastewater with elevated concentrations of pollutants. It may pose hazardous effects on the environment if it is directly discharged into receiving bodies without proper treatment. Turkey is a developing country which is rich in all forms of textile industry as well as cotton-dyeing mills. The development of appropriate treatment alternatives is quite important for maintaining a highly environmentally friendly and sustainable textile industry in a developing country. In this study, the MWD of pollutants in three different wastewater sources from a cotton-dyeing mill was investigated by applying sequential ultrafiltration (UF) to them. Additionally, concentration and MWD values were also used in order to evaluate the alternative treatment options.

2. Materials and methods

2.1. Wastewater characterisation

Wastewater samples were taken from a full-scale cotton-dyeing mill in Istanbul, Turkey. In the mill, the majority of its wastewater is generated from bleaching and dyeing processes. Effluents from each process are finally mixed into an equalisation tank. Two wastewater samples were obtained from the effluents of these processes. A mixed-wastewater sample was also taken from the equalisation tank prior to pH adjustment. All samples were kept at 4˚C in the laboratory before the experiments were conducted.

The organic content of wastewater samples were characterised by the analysis of chemical oxygen demand (COD), total organic carbon (TOC) and biochemical oxygen demand (BOD_5). All analyses were performed according to procedures in the standard methods [16]. The COD of the wastewater was determined using the closed reflux colourimetric method (5220D), while its TOC was measured by means of a TOC analyzer (Hach Lange, IL 550). Five-day biochemical oxygen demand tests were performed by following the SM-5210B method. Moreover, the total kjeldahl nitrogen (TKN) was analysed using a thermal digester (Velp Scientifica). Dissolved organic content was determined by the filtration of the samples through a 0.05 μm pore-sized filter, while the remaining organics above the filter were defined as particular matters. UV_{254} was measured using a double-beam UV–vis spectrophotometer (Shimadzu UV-1800) with 1 nm of resolution. Specific Ultraviolet Absorption (SUVA) values were calculated by dividing UV_{254} into dissolved organic content so as to determine the hydrophobicity of organics. Colour was spectrophotometrically determined by Hach lange (5000 DR), while pH and conductivity were measured using a multimeter (Thermo Orion 5 star).

2.2. MWD analysis

The membrane separation method was used for the MWD analysis. Separation experiments were performed with a stirred membrane cell (Amicon, Model 8400) with the diameter of 76 mm and an effective membrane area of 41.8 cm^2. The membrane separation cell consisted of a methyl-methacrylate glass holder with a total volume of 350 mL. Wastewater samples were sequentially filtered through MF and UF. The pore size of the MF membrane (Microdyn Nadir) was 0.05 μm while the nominal molecular weight limits (NMWL) of the UF membranes were between 0.1 and 100 kDa (PALL and Spectrum Inc.). All membranes were soaked with deionised water in order to remove glycerine before the filtration experiments. The sequential filtration experiments were performed until a final permeate volume of 150 mL was reached. During the filtration process, the cell was pressurised with N_2 gas. MF was conducted under 1 bar of pressure while the operating pressures of the UF analyses were 2 bars for the UF membranes between 100 and 1 kDa and 4 bars for the UF membranes between 0.5 and 0.1 kDa.

3. Results and discussion

3.1. The characteristics of the wastewaters

The main characteristics of the wastewater from the different processes are shown in Table 1. During all of these processes, pH values were higher than eight, with the highest pH value being at 9.1 during the mixed-filtration process. Conductivity values increased proportionally with pH values. The COD values were around 1,200 mg/L in the mixed and dyeing samples, while the bleached wastewater attained the highest value of 4,075 mg COD/L. The ratio of organic carbon was at the lowest in the dyed wastewater due to the intensive use of synthetic dye chemicals, whereas the contribution of domestic wastewater in the equalisation tank significantly improved the ratios of TOC/COD and BOD_5/COD. The BOD_5/COD ratio in wastewater is commonly used for determining the biodegradability of organic matters and whether or not biological treatment is more favourable when this ratio is over 0.3 [17]. The BOD_5/COD ratio of the mixed wastewater was higher than the bleached and dyed wastewaters since it also comprised of effluents from the rinsing and washing of the textiles with domestic wastewater. The biodegradability ratio is close to that of aerobic treatment. Nevertheless, anaerobic treatments could be considered as being a more appropriate treatment option with lower sludge and energy production, not to mention nutrient requirements. On the other hand, while high conductivity and colour may inhibit biological treatment, reverse osmosis and membrane distillation are more appropriate for removing conductivity. Furthermore, colour can be effectively removed by ozonation and ultrafiltration. Consequently, a single evaluation based simply on wastewater characteristics is insufficient for deciding whether or not a particular treatment process and MWD analysis can provide a comprehensive assessment.

3.2. The MWD of organics

Concentrations of COD, BOD_5 and TOC at varying molecular weight fractions are shown in Fig. 1. The particulate COD fractions (>0.05 μm) of mixed, bleached and dyed wastewaters were 1, 12 and 4%, respectively (Fig. 1(a)). These ratios indicate that the COD of all textile wastewaters are mainly comprised of dissolved organics. A fraction analysis of the mixed-wastewater sample indicated that 5% of its COD was in the range of 1–0.1 kDa, while the percentage of organics found in that same sample at over 1 kDa and under 0.1 kDa was found to be at around 47%. On the other hand, the corresponding dissolved COD fractions for 100–1 kDa, 1–0.1 kDa and <0.1 kDa were 23, 14 and 48% in bleached wastewater and 55, 8 and 27% in dyed wastewater, respectively.

The total BOD_5 values of the bleached wastewater were 2.5 times higher than that of the mixed wastewater and 4.37 times higher than that of the dyed wastewater (Fig. 1(b)). Similar to the COD, almost all BOD_5 samples were in soluble form. The soluble BOD_5 ratios of the mixed wastewater were 15, 2 and 83% from 100–1 kDa, 1–0.1 kDa and <0.1 kDa fractions, while the ratios were 40, 0 and 59% for the bleached wastewater and 56, 1 and 37% for the dyed wastewater. These figures indicate that each process has a different BOD_5 fraction, while the wastewater itself is mainly less than 0.1 kDa.

The TOC fractionations of wastewaters were as shown in Fig. 1(c). Similar to the COD and BOD_5, bleached wastewater had considerably higher TOC values than that of the dyed and mixed wastewaters.

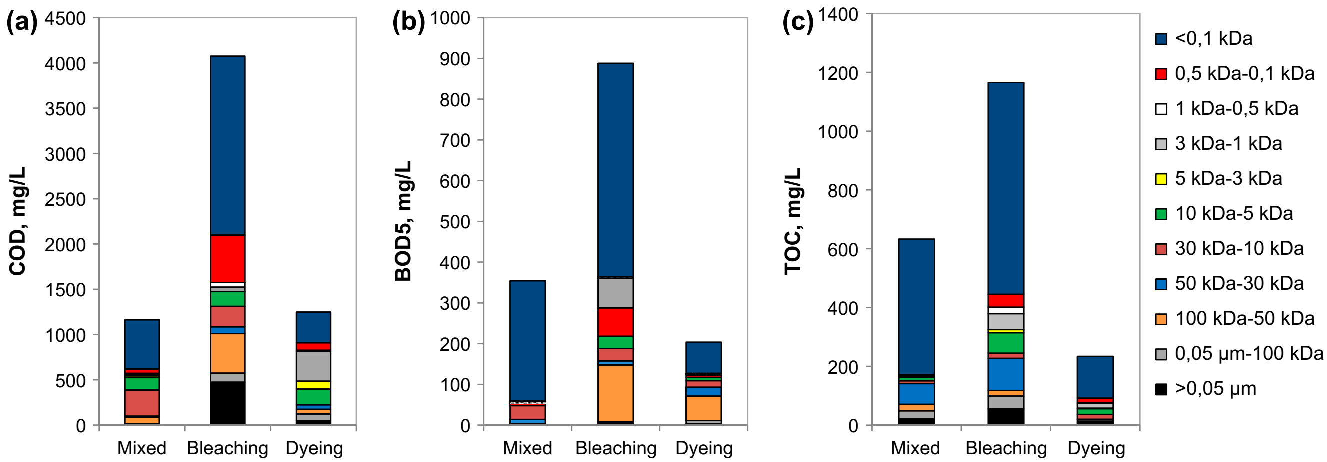

Fig. 1. MW distributions of organic matter (a) COD, (b) BOD_5, and (c) TOC.

The particulate TOC ratio in the dyed wastewater was 1.67%, with all of the TOC in the mixed and bleached wastewater being in a soluble form. TOC fractions from 100–1 kDa, 1–0.1 kDa and <0.1 kDa were 19, 1 and 73% for the mixed, 24, 6 and 62% for the bleached, and 24, 8 and 61% for the dyed wastewaters, respectively.

After each filtration step, the BOD_5/COD ratio of the mixed wastewater steadily increased with the decrease of fraction size, with the highest ratio being 54% from <0.1 kDa (Fig. 2). On the other hand, the BOD_5/COD ratios in both the bleached and dyed wastewaters changed very little during the sequencing filtration. Changes in the TOC/COD ratios of the samples are shown in Fig. 2(b). For the mixed wastewater, the TOC/COD ratio increased from 54 to 62% and 85% at the 10 kDa and 0.1 kDa fractions, respectively. The ratio also increased from 29 and 19% to 37 and 42% after 0.1 kDa fractioning of bleached and dyed wastewater.

These values indicate that low MW organic matters (<0.1 kDa) of the mixed wastewater is feasible for biological treatment. All fractions of the bleached and dyed wastewaters had relatively low biodegradability, with a BOD_5/COD ratio of less than 0.30. Almost 50% of the organic matters in the bleached wastewaters passed through the 0.1 kDa MW membrane. On the one hand, the bleached and mixed wastewaters are not feasible for being treated in RO or NF seeing as they have over 0.1 kDa MW, along with flocculation and precipitation [11,12]. Researchers reported that biological treatment is effective for low-molecular-weight organic matters [8,18] and that the anaerobic treatment has been applied to the bleached and mixed wastewaters in order to treat them seeing as they have a low biodegradability [16]. RO or NF membranes can easily be used for removing organic matters remaining after biological treatments. Moreover, pre-treatment with biological processes could effectively remove low-molecular organics and prevent the fouling of the RO and NF membranes, thereby improving the operation duration of the membrane systems. One should consider the fact, however, that the fractions of intermediate and large organics may increase after

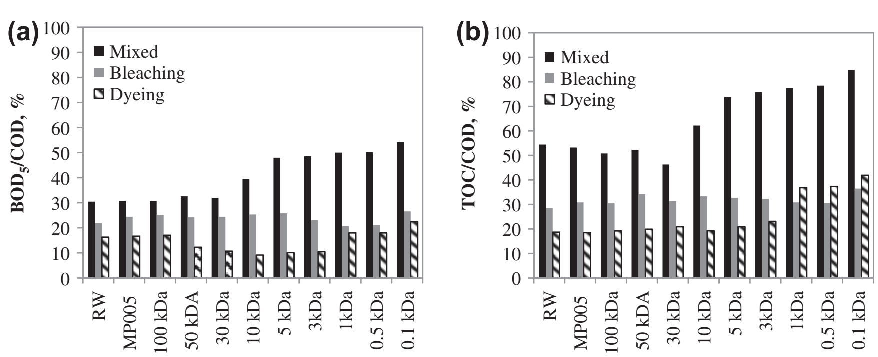

Fig. 2. The ratio of (a) BOD_5/COD and (b) TOC/COD.

such biological treatment [8]. In this case, intermediate and large organics can be efficiently removed by means of sedimentation [10,11]. In order to prevent the fouling of the NF or RO membranes, effluents from the sedimentation process need to be filtered through MF or UF membranes.

3.3. Analysis of UV–vis and SUVA

Ultraviolet and visible absorption spectroscopy (UV–vis) analyses are useful in determining the total amount of organic compounds in wastewater by measuring their absorbance at certain wavelengths. Organic matters are commonly identified at wavelengths of 220 and 280 nm [19]. In this study, UV_{254} measurements were made at a wavelength of 254 nm for the purpose of determining the aromatic compounds.

The UV_{254} and SUVA values which were obtained from the analysis results are shown in Figs. 3 and 4. After 0.05 μm of filtration, UV_{254} values decreased by 32, 44 and 5% for the bleached, dyed and mixed wastewaters, respectively. For the same wastewaters, UV_{254} absorbance values decreased by 38, 55 and 76% after the 0.1 kDa fraction. It has been reported that biological treatment is effective in removing aromatic organics and that, therefore, effluents can be safely filtered through the membrane [8]. Experimental results indicated that filtration using an NF with a MWCO of 1 kDa can reduce UV_{254} by 39, 51 and 75% for mixed, bleached and dyed wastewaters, respectively. Thus, by biologically processing wastewaters by means of MF or UF, NF filtration can then be used in order to remove its aromatic organics. The SUVA value of dyed wastewater was 1.79, while being less than 0.7 for mixed and bleached wastewaters. After MF/UF filtration, the SUVA of dyed wastewater decreased below one. It has been reported that organic matters with SUVA values less than two have hydrophilic properties [20]. In the present study, hydrophilic degrees of organic materials increased at lower MWs. Conventional coagulation–flocculation is not a proper method for removing highly hydrophilic organics [20]. Nevertheless, SUVA values can be decreased with the use of activated carbon processes [21,22].

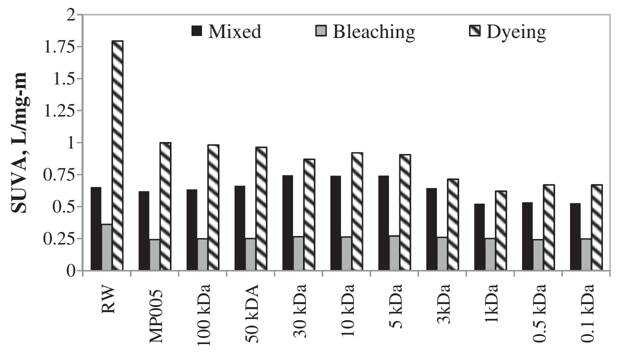

Fig. 4. SUVA values of mixed, bleaching and dyeing wastewaters.

3.4. The MWD of TKN

The TKN values of the wastewater samples at various molecular weights are presented in Fig. 5. Bleached wastewater had the highest TKN, while the dyed had the lowest. Very little change was observed in TKN values during the fractioning of the dyed wastewaters. On the other hand, the TKN of mixed wastewater was decreased by 49 and 53% after the 1 and 0.1 kDa fractions, respectively. The TKN of the bleached wastewater was reduced by 25 and 29% after the 1 and 0.1 kDa fractions, respectively. The removal of the TKN from the dyed wastewater was very low, with almost 20% for 10 kDa. After using the 10 kDa MWCO membrane, no change was examined in the lower MWCO membranes. These values show that when precipitation is added to flocculation, MF,

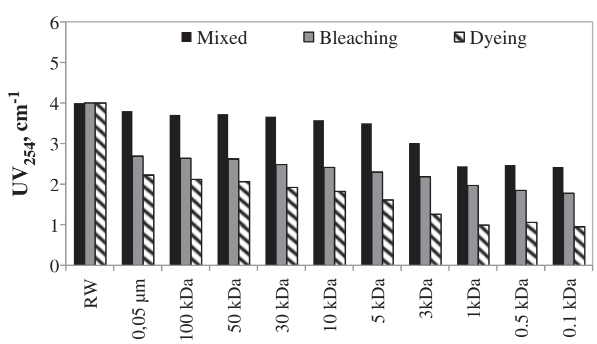

Fig. 3. UV_{254} absorption of mixed, bleaching and dyeing wastewaters.

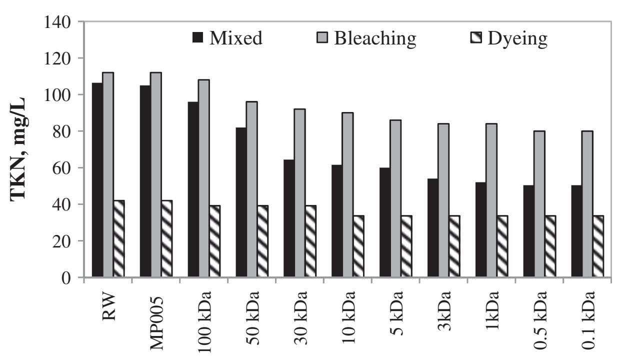

Fig. 5. MW distributions of TKN.

Table 1
Characterisation of textile wastewater

Parameters	Units	Mixed wastewater	Bleaching wastewater	Dyeing wastewater
pH	–	9.1	8.4	8.7
Colour	PtCo	465	na	460
Conductivity	mS/cm	22.9	8.8	10.4
COD	mg/L	1,163	4,075	1,250
TKN	mg/L	106	112	42
TOC	mg/L	633	1,165	234
BOD_5	mg/L	354	888	204
UV_{254} Abs.	cm^{-1}	4	4	4

UF and NF membranes are not sufficient for removing TKN. In their study, Zaho et al. [8] found that lower-molecular-weight organic nitrogen was effectively removed by means of biological processes. In the present study, TKN is mostly less than 1 kDa MW; this can also be reduced by biological treatment processes. In the case of anaerobic treatment, TKN could be efficiently removed along with organic matters. Moreover, UF and NF membranes can reduce the intermediate parts of the TKN (>1 and <100 kDa) from anaerobic treatment effluents. On the other hand, researchers reported that the anaerobic treatment results in 50–200 mg/L of ammonia [23]. As seen in Table 1, the amount of ammonia contained in dyed wastewater is insufficient for anaerobic treatment, while it might be sufficient for mixed- and bleached-wastewater treatments. In the case of there being a deficient amount of ammonia for the purpose of treating the processes separately, external ammonia chemicals can be added. However, one should bear in mind that the supplementation of external chemicals increases operation costs.

3.5. Colour removal

While colour was not measured in the transparent bleached wastewater, colour changes in the mixed and dyed wastewaters are as given in Fig. 6. Dyed wastewater had higher colour intensity than that of mixed wastewater. Filtration by means of an MF membrane (with a pore size of 0.05 μm) provided 50 and 55% colour removals in the mixed and dyed wastewaters, respectively. Following the 3 kDa MW fraction, the colour of the mixed wastewater was reduced to 95%, with no colour being observed with fractions less than 1 kDa. Moreover, a 0.1 kDa fraction resulted in decreasing the colour of the dyed wastewater by 92%. Those results indicate that particular and colloid materials produce almost 50% of colour, while the remaining 50% of the colour is only apparent colour.

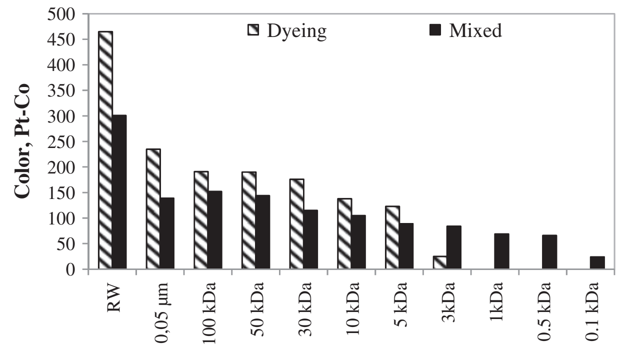

Fig. 6. MW distributions of colour.

Although the biological treatment process can remove coloured materials, the remaining colour has been reported in the effluents of such processes in most cases [10]. Hence, after biological treatment, additional processes are required to meet discharge limits. As seen in Fig. 6, almost half of the colour is removed from the wastewaters by utilising the MF and UF membranes. When an NF with an MWCO of 1 kDa is applied, the colour can be decreased by 100 and 77% from the mixed and dyed wastewaters, respectively. In addition to membrane filtration, ozonation can also be applied in order to remove colour. Ozonation effluents, however, may contain low-molecular-weight matters, thereby increasing the biodegradability of the organic matters. After ozonation, biological treatment or carbon adsorption should be applied in order to remove low-molecular-weight organics before being filtered through the NF or RO membranes. Furthermore, low-molecular-weight organics with high biodegradability are easily biodegraded by anaerobic microorganisms [24].

4. Conclusions

In this study, treatment alternatives for processing different wastewaters from cotton-dyeing mills were evaluated based on pollution characteristics and a MWD analysis. The following conclusions can be drawn:

(1) According to the BOD_5, COD and TOC values, aerobic biological treatment is not feasible for all of the processes' effluents, whereas anaerobic treatment can be applied along with sedimentation or MF/UF membranes before their being polished with NF or RO membranes.
(2) SUVA values show that all wastewaters are hydrophilic in character and that the conventional coagulation–flocculation should not be utilised seeing as it is not the proper method for removing highly hydrophilic organics.
(3) Most parts of the TKN <1 kDa can be removed by means of anaerobic treatment. UF and NF membranes may reduce the intermediate parts of the TKN following this anaerobic treatment.
(4) Particulate and colloidal materials produced almost half of the colour in the mixed and dyeing processes, while the bleaching process did not yield any colour.
(5) Almost half of the colour was removed by MFs with pore sizes of 0.05 μm, with the remaining being treated by the NF membrane.
(6) While treating the wastewaters anaerobically, MF or UF and NF membranes can be used for the removal of organic matters, UV_{254}, TKN and colour from the effluents of the mixed, bleaching and dyeing processes.
(7) This study presents alternative treatment options related to pollutant properties; however, better decisions can be made based on pilot-scaled treatment performances coupled with economic analyses.

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