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Investigation of ceramic membranes performance for tannery wastewater treatment

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ABSTRACT

The objective of this study was to investigate the treatment of highly polluted tannery wastewater using ceramic microfiltration (MF) and ultrafiltration (UF) membranes. The impact of membrane pore size and pressure on permeate flux, chemical oxygen demand (COD), and color reduction was examined. All experiments were performed at a lab-scale, using cross-flow ceramic membrane test unit. Three different single-channel tubular ceramic membrane modules (γ-Al_2O_3, Media, and Process Technology, Inc., USA) with average pore sizes of 10, 50, and 200 nm were used. Wastewater sample was obtained from the effluent of tannery at organized industrial district of the city of Isparta, Turkey. Clean water flux tests were conducted before and after wastewater treatment. Permeate flux was reduced due to membrane fouling after all operation and fouling was removed effectively using chemical cleaning procedures. More than 95% color removal was consistently achieved with both UF membranes (10 and 50 nm). COD reductions ranged between 58 and 90% at all pressures for UF membranes tested in the wastewater. As the test pressure of the UF ceramic membranes increased, COD and color reduction also increased. It was concluded that ceramic UF membranes with 10 nm average pore size can be used in removing COD and color from highly polluted tannery wastewater.

Keywords: Ceramic membrane; Color removal; Tannery; Microfiltration; Ultrafiltration

1. Introduction

Leather tanning industry is one of the most important and leading economic sectors in developing countries as in the cases of Turkey, China, India, Pakistan, and Brazil [1–3]. The main concern regarding the tanning industry is the highly polluted effluent with high organic matter and salt concentrations and other pollutants. High concentrations of pollutants with low biodegradability in tannery wastewater represent a serious technological and environmental challenge [4,5]. The tannery industry is extremely water-intensive, discharging into the environment an average of 30–35 m^3 of wastewater per ton of raw hide [3]. The estimated total wastewater discharge from tanneries is about 400 million m^3/year. Acids, alkalis, chromium salts, tannins, solvents, sulfides, dyes, auxiliaries, and many others compounds, which are used in the transformation of raw or semi-pickled skins into commercial goods, do not completely stick to skins and remain in the effluent [3]. Tannery effluent is a strong wastewater with complex characteristics of high

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chemical oxygen demand (COD), color, and conductivity values. It is associated with a high level of organic pollutants commonly characterized with COD concentrations of above 3,000 mg/L [3]. COD of total tannery wastewater is approximately 100–120 kg per ton of raw hide [6], i.e. approximately 55% of the COD of the global effluent. Tannery wastewaters have COD and SS values approximately five times higher than the municipal wastewaters, and high salt concentrations similar to brackish waters [7].

The Water Pollution Control Regulation (SKKY) issued in 2004 contains discharge criteria for tannery industries including COD, total nitrogen, Cr^{+6}, and pH levels. In Turkey, tannery wastewater treatment plants have to meet the discharge limit of 300 mg/L COD in terms of organic pollutants .The Turkish Ministry of Environment and Forestry made a revision regarding the color parameter in the Water Pollution Control Regulation in 2011, and created a new color discharge standard of 280 Pt–Co for tannery industry [8].

Treatment of industrial wastewaters is a major concern in terms of both pollution control and minimizing fresh water consumption. Tannery wastewaters have refractory groups of organic chemicals [4]. Thus, clean, economically as well as environmentally sustainable wastewater treatment technologies have been explored for the leather industry [9,10]. Membrane processes gained importance because of providing higher quality water, easier operational control, and less maintenance. The use of membrane technologies applied to the leather industry represents an economic advantage, especially in the recovery of chromium from residual waters of leather tanning. Also, several studies showed that crossflow microfiltration (MF) and ultrafiltration (UF) led to reduction in the polluting load of tannery wastewater [11–13]. Wang et al. [13] showed that a rejection above 90% of COD can be obtained at 25℃ under a trans-membrane pressure of 0.085 MPa by ultrafiltration with polyimide membrane at pH 9 for the purification and recycling of tannery degreasing wastewater. Similarly, up to 93% COD removal in soaking and liming wastewater of the tanning process were obtained with polymeric membranes [12]. Ceramic membranes are used in a wide range of filtration processes than polymeric membranes because of advantages such as mechanical strength, resistance to chemicals, extreme pH levels and temperatures, and ease of cleaning [14–17]. Ceramic membranes are commonly constructed from inorganic oxides such as alumina, silica, zirconia, and stannic oxide [18,19].

In order to minimize the pollution of tannery wastewaters, some authors proposed the adoption of ceramic membrane technologies. For water reuse, Bhattacharya et al. [20] have studied the tannery wastewater recovery by ceramic microfiltration and reverse osmosis. In their study, about 91% reduction in COD and BOD_5, 62% reduction in total organic carbon, and complete removal of sulfide were achieved in the direct microfiltration process, while turbidity reduced to below 1 NTU. The use of low-cost ceramic microfiltration membranes, made of Moroccan Perlite, was evaluated by Majouli et al. [21] to treat the effluents coming from beamhouse section of tannery. Perlite membrane led to significant reduction in COD (50–54%) and Total Kjeldahl Nitrogen (TKN) (56%) in the beamhouse effluent. Majouli et al. [22] also studied the treatment of beamhouse effluent of tannery by microfiltration through Cordierite/Zirconia and Alumina tubular ceramic membranes. Operation of this technique at a lower pressure (1 bar) resulted in a significant reduction in COD (60–65%) and TKN (57–59%).

The objective of this work is to investigate the performance of ceramic UF and MF membranes in the removal of color and COD from tannery wastewater. The use of MF/UF to treat tannery effluent has been almost exclusively focusing on polymeric membranes, while only a little research has been performed with ceramic membranes. The impact of membrane pore size and processing pressure on permeate flux and the removal of COD and color was determined. A total of three different pore sizes and pressures were tested with a purpose of investigating the COD and color removal performances of the ceramic UF and MF membranes.

2. Materials and methods

2.1. Water source

Membrane tests were conducted on tannery wastewater. Wastewater sample was obtained from the effluent of tannery at organized industrial district of the city of Isparta, Turkey. The raw wastewater sample was filtered with a 5-μm cartridge filter before membrane tests. Physicochemical characteristics of the filtered water are shown in Table 1. Filtered samples were collected in high-density polyethylene bottles and stored at 4℃ in dark until use. Tannery wastewaters are characterized by their conductivity and pH values. COD is 3,770 mg/L, which shows the amount of oxygen required for the oxidation of organic matter present in the effluent. Color is 9,140 Pt–Co due to the considerable amounts of suspended solids.

2.2. Membrane test unit

The ceramic membrane was operated at a cross-flow filtration mode. All the experiments were

Table 1
Physicochemical characteristics of the filtered raw wastewater (average values of triplicate measurements)

Parameters	Raw water
COD (mg/L)	3,770
Color (Pt–Co)	9,140
Color (ADMI)	6,000
pH	9.92
Conductivity (μS/cm)	26,200
Sulfate (mg/L)	1,120
Turbidity (NTU)	2.44
Cr^{+6} (mg/L)	0.89

conducted in the total recycle mode, whereby both concentrate and permeate were returned to the feed tank. Fig. 1 shows the cross-flow ceramic membrane test unit used in the experiments. The unit included housing for a single tubular ceramic membrane module. All tests were performed at a feed water temperature of 20 ± 2˚C by circulating water through the jacket around the feed tank using a water bath circulator (PolyScience, 9602). Permeate volumes collected during the tests were calculated by measuring the permeate weight using an electronic digital balance (accuracy of ±10 mg). The test system included a high-pressure pump (Hydra-Cell, D/G-03-B), a variable frequency drive (ABB, ACS150), and a 2.2 kW motor to adjust the pump speed and the feed flowrate. It also included a stainless steel feed tank (45 L max. solution volume), a ceramic membrane module and its housing, a concentrate control valve, pressure gauges, and stainless steel and/or Teflon tubings resistant to high pressure. The membrane operating pressures were adjusted by the concentrate control valve.

2.3. Ceramic membrane tests

Three different single-channel tubular ceramic membrane modules (γ-Al_2O_3, Media, and Process Technology, Inc., USA) were tested with average pore sizes of 10, 50, and 200 nm. Membrane characteristics are given in Table 2. The tested membrane pressures were 1, 2, and 4 bar. The duration of each membrane test was 10 h. Samples from feed tank and permeate were taken every hour for COD, color, conductivity, and pH measurements. Buffering for pH was not carried out in raw water tests. The differences between permeate and feed water pH values of the raw water were generally less than ±0.3. The test unit was operated at least for 2 h with the same feed water for conditioning the new membranes prior to the 10 h membrane tests. This pre-operation was also conducted for all flux measurement tests. Flow rates of concentrate and permeate streams, membrane unit, and pump outlet pressures were also recorded every hour. Chemical cleaning procedure was performed after each separation process. Fouled ceramic membrane was cleaned using NaOH solution (1 g/L) for 30 min at 85˚C. Subsequently, membranes were soaked in DDI water for 2 h. The test unit was operated at least for 4 h with the DDI for the rinsing step.

2.4. Analytic measurements

The treatment performance of the system was determined through measurements of color, COD, turbidity, total dissolved solids, conductivity, and pH according to the Standard Methods [23]. COD analysis was performed using Hach-Lange DR5000 spectrophotometer according to Standard Method 5220-D (closed

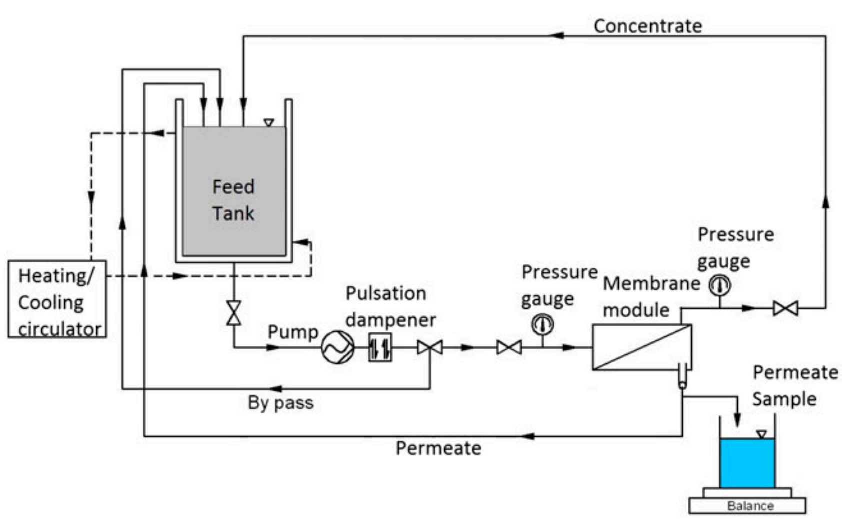

Fig. 1. The schematic diagram of the lab-scale, cross-flow ceramic membrane test unit.

Table 2
Characteristics of ceramic MF and UF membranes

γ-Al_2O Ceramic Membranes (nm)	Outer diameter (mm)	Inner diameter (mm)	Active length (cm)	Total filtration area (cm^2)
10	4	2	23.5	29.5
50	4	2	23.5	29.5
200	4	2	23.5	29.5

reflux colorimetric method). Color measurement was performed using DR5000 spectrophotometer according to the Standard Methods. Besides standard color measurements, ADMI (American Dye Manufacturers Institute) method was also used to monitor the color of wastewater effluent as an indicator of water quality. The ADMI measurement method is more suitable for industrial wastewater which contains intense color. The ADMI method can give more accurate results because of the ability of scanning a wide spectral band. ADMI value was measured according to EPA Method 110.1. This method is an extension of the Tristimulus Filter Method. Tristimulus values are converted to an ADMI single number color difference of the same magnitude assigned to platinum-cobalt standards, using the Adams-Nickerson Color Difference (DE). This method measures the sample's true color, independent of hue. Hach DR5000 spectrophotometer was used for measuring ADMI values because standard curves and complex equations have been installed in this instrument [24,25]. Conductivity was measured using a conductivity meter (WTW Inolab Cond. Level 1). pH was measured using WTW 340i pH meter. Turbidity was measured using WTW Turb 550 turbidimeter. All analytical measurements were conducted in triplicates.

3. Results and discussion

Before wastewater tests, clean water fluxes (CWF) were measured at different membrane pressures for ceramic UF and MF membranes. CWF and wastewater flux (WWF) values are shown in Fig. 2 for UF modules with 10 nm average pore size. As expected, CWF values increased with increasing membrane pressure for all membranes. Average CWF values were 91, 143, and 262 L/m^2 h (LMH) at pressures of 1, 2, and 4 bar, respectively, for 10 nm. Similar results were found for 50 and 200 nm membranes, as well. At constant pressure, maximum flux (1,075 LMH) was obtained by the 200 nm pore size membrane (data not shown). Similarly, it was found that permeability tends to rise as the pressure applied increased for the treatment of effluents coming from tannery beamhouse by microfiltration through Cordierite/Zirconia and Alumina tubular ceramic membranes [22].

For all membranes, WWF values generally decreased during 10 h of operation. Using the 10-nm pore size membrane, permeate flux values were 22, 28, and 41 LMH at pressures of 1, 2, and 4 bar after 10 h operation, respectively. WWF values found were about 80% lower than CWF values at 4 bar pressure with 10 nm pore size UF membrane. This finding indicates the extent of fouling due to raw water filtration. It is consistent with the literature, and Majouli et al. [22] explained this finding with contaminants changing the dynamic proprieties of water such as viscosity and density [22]. Due to high concentration of organic matter in the effluents, studies that use membrane technology in the leather industry reported major fouling problems [6]. Some authors proposed to recover the permeate flux by implementing cleaning methods, whether chemical or high pressure, which obtained good results [6,7]. Caustic soda is typically used to clean the membranes fouled by organic and microbial foulants. The function of caustic soda is mainly the hydrolysis of organics and the solubilization of inorganics [6].

After 10 h of operation, CWF test were conducted again to determine the fouling levels. CWF values after the operation were similar to the WWF values. Due to 80% permeate flow reduction, chemical cleaning was performed with NaOH solution. Table 3 shows the impact of cleaning on flux recoveries for 10-nm UF membranes. After chemical cleaning, CWF values were similar to those of raw membrane. This result shows that fouling was removed effectively by applying chemical cleaning procedures. Mendoza -Roca et al. [6] studied cleaning of membranes fouled by tannery wastewater with NaOH, sodium dodecyl sulfates, and two different enzymes, and their results showed that NaOH (1 g/L) have a better cleaning efficiency than the other alternates they used.

UF ceramic membrane separation significantly reduced the color in permeates compared to the feed water. More than 95% of color reduction was consistently achieved with 50 and 10 nm membranes. Table 4 shows the Pt–Co values for all membranes at 1 and

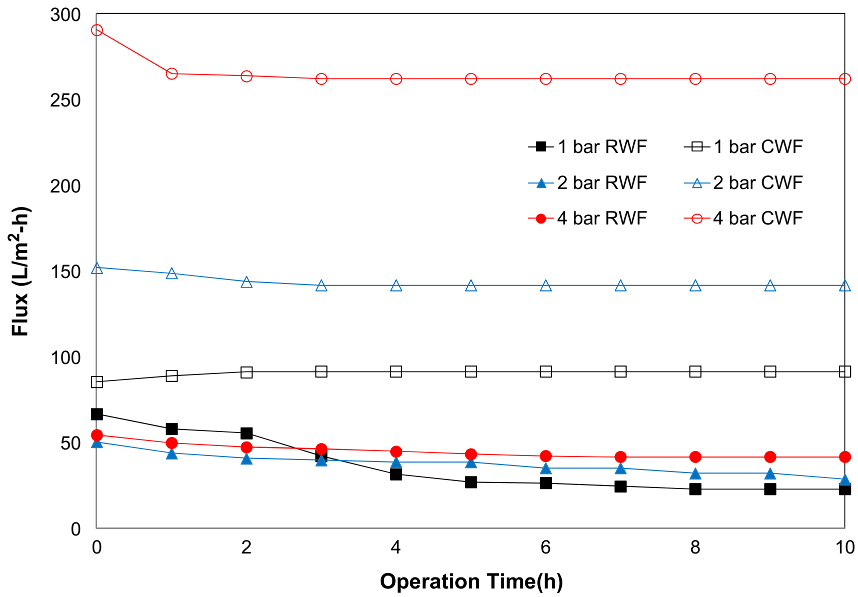

Fig. 2. Impact of membrane pressure on WWF and CFW (membrane average pore size: 10 nm, feed temp: 20 ± 2˚C, total recycle mode).

Table 3
Impact of cleaning on flux recovery for 10 nm ceramic UF membranes

Parameters (LMH)	1 bar	2 bar	4 bar
CWF	91	141	262
WWF	36	38	46
CWF_After Wastewater	31	28	26
CWF_After Chemical Cleaning	93	146	265

2 bar. The Turkish Ministry of Environment and Forestry has made a revision regarding the color parameter in the Water Pollution Control Regulation in 2011, and created a new color discharge standard as 280 Pt–Co for tannery industry. The color achieved in permeates of UF membranes with 10 and 50 nm average pore size was lower than 280 Pt–Co. Color levels as high as 9,140 Pt–Co were significantly reduced to about 10 Pt–Co levels for ceramic UF membranes.

Fig. 3 shows the color removals for all membranes at 1 and 2 bar as ADMI unit. These low color levels suggest that UF ceramic membranes even with 50-nm pore size are highly effective for removing color and meeting regulations. pH value and conductivity remained constant during all the filtration tests.

Fig. 4 shows the raw water COD removal achieved by all MF and UF membranes at 1 and 2 bar. The lowest removal was for 200-nm MF membrane, and COD removal was about 10% at 200-nm pore size membrane. Maximum COD removal was by the 10-nm pore size membrane at 2 bar. COD removal ranged between 58 and 78% at all pressures for all UF membranes tested. UF membrane with 10-nm pore size provided higher COD removal than those of 50 nm pore size membrane. As a general trend, COD removal increased with increasing membrane pressure from 1 to 2 bar for all membranes. Majouli et al. [22] reported 60 and 65% COD removal for Cordierite/ZrO2 and commercial Alumina membranes, respectively, at 1 bar.

Fig. 5 shows COD removal of UF membrane with 10-nm pore size at different pressures. In general,

Table 4
Color levels after 10 h operation as Pt–Co unit for all tested membranes at 1 and 2 bar

Tested membranes	Color value as Pt–Co at 1 bar	Color value as Pt–Co at 2 bar
10 nm	5	5
50 nm	57	57
200 nm	8,600	9,060
Feed Water	9,140	9,140

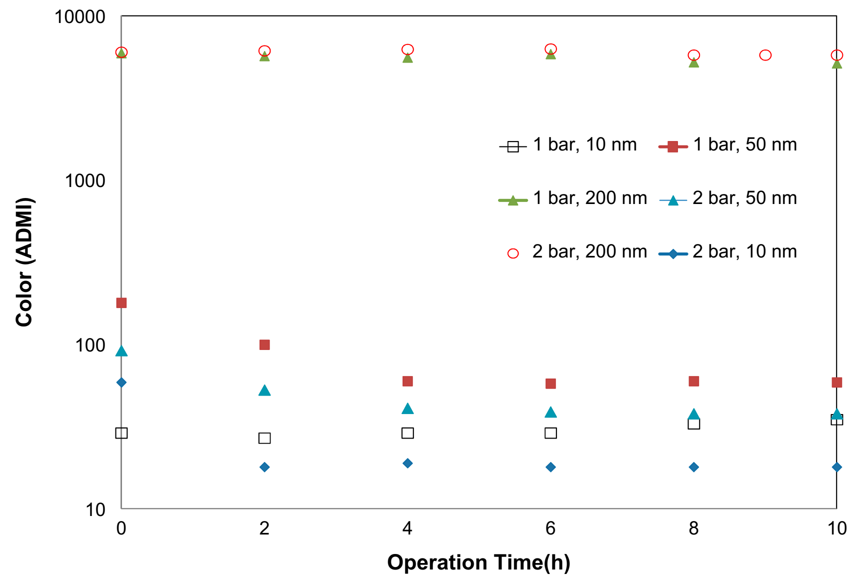

Fig. 3. Impact of membrane pressure and pore size on color removal in the raw water (feed temp: 20 ± 2˚C, total recycle mode).

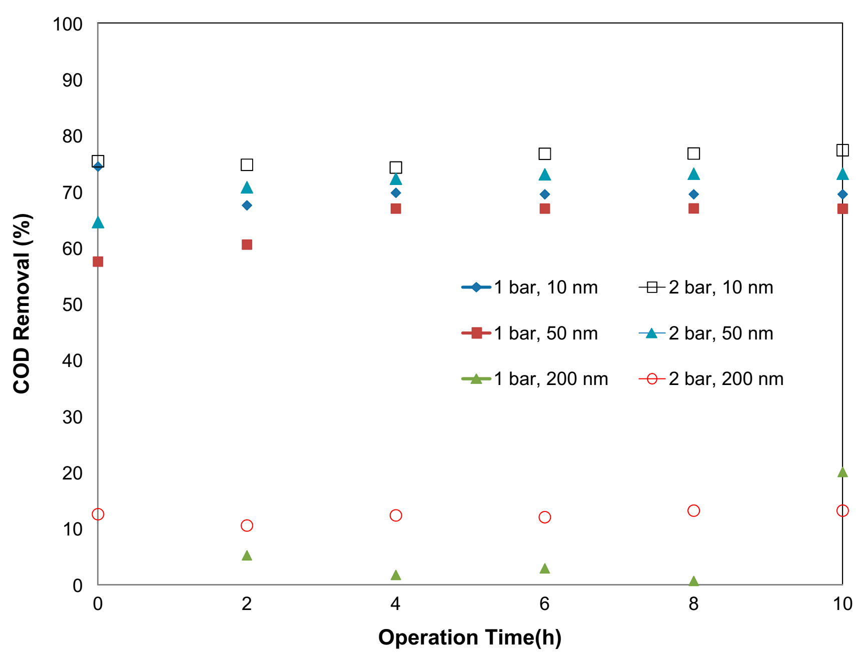

Fig. 4. Impact of membrane pressure and pore size on COD removal in the raw water (feed flowrate: 2.5 L/min, CFV: 3.3 m/s, feed temp: 20 ± 2˚C, total recycle mode).

COD removal increased with increasing membrane pressure from 1 to 4 bar. While a similar trend was obtained for all membranes, the maximum COD removal (90%) was achieved in raw water at 4 bar pressure with the membrane having 10 nm pore size.

Tannery wastewater can discharge into sewer systems (indirect discharge), where it should undergo full-scale treatment or direct discharge (streams and rivers). Discharge into receptive environment, as stated in the Regulations for the Control of Water Pollution, mentions the discharge limit of COD, color, and pH is 300 mg/L, 280 Pt–Co, and 6–9, respectively. Color values from as high as 91,400 Pt–Co levels were significantly reduced to about 5 Pt–Co level, which are

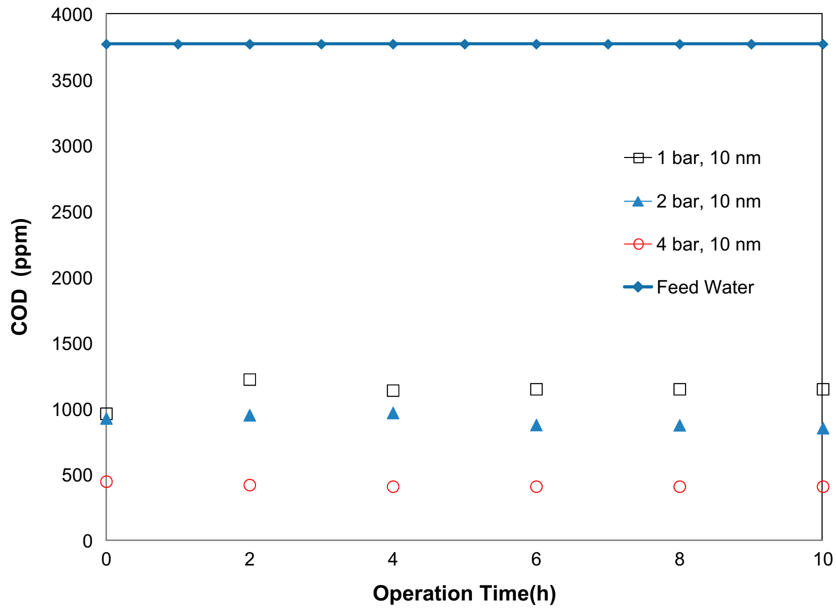

Fig. 5. Impact of membrane pressure on COD removal (pore size: 10 nm, feed flowrate: 2.5 L/min, CFV: 3.3 m/s, feed temp: 20 ± 2˚C, total recycle mode).

below discharge limits. However, since COD and pH values are still above discharge limit, further treatment processes (i.e. nanofiltration or reverse osmosis) are required prior to discharge into streams and rivers. There are local discharge limits into sewer systems to protect municipal wastewater treatment's operations and to ensure that its discharges comply with Regulations for the Control of Water Pollution requirements. Local discharge limit of COD into sewer systems is 1,000 mg/L and pH levels should be in the range of 6–12. Our results overall indicated that complex and highly polluted tannery wastewaters can be pre-treated effectively by ceramic membrane processes prior to sewer systems.

4. Conclusions

Ceramic UF and MF membranes were selected because of their potential advantages including chemical and thermal stability, physical strength, and longer operational lives. Initial tests indicated that flux obtained by the ceramic membrane increased with increasing average pore size at constant pressure. Color removal was higher than 95% and color levels were significantly reduced from as high as 9,140 Pt–Co to about 10 Pt–Co levels. COD removal ranged between 58 and 90% at all pressures for UF membranes tested. In general, COD removal increased with increasing membrane pressure (from 1 to 4 bar). A maximum of 90% COD removal was achieved in raw water at 4 bar pressure with the 10-nm pore size membrane. Permeate flux is reduced due to membrane fouling after wastewater treatment during all operations. Fouling was removed effectively by applying chemical cleaning procedures. The results indicated that highly polluted tannery wastewaters can be treated very effectively by ceramic ultrafiltration systems. Ceramic membranes can offer a more robust and long-term alternative to polymeric membranes in the treatment of tannery wastewater. Their superior chemical, thermal, and mechanical properties mean that not only can they be operated under harsh conditions, they can also be backwashed and cleaned with strong cleaning agents as well as sterilized at high temperatures, offering reliable performance over long periods of time.

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