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TREATMENT OF WASTE METALWORKING FLUIDS BY COAGULATION AND CROSS-FLOW MEMBRANE FILTRATION

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Abstract

The primary objective of this study was to treat waste metalworking fluids (MWFs) originating from the metalworking industry. To achieve this goal, a series of treatment methods were employed in sequence, and various parameters were analyzed, including chemical oxygen demand (COD), pH, total suspended solids (TSS), turbidity, oil/grease, total organic carbon (TOC), and biochemical oxygen demand (BOD). In the initial stage of wastewater pre-treatment, cross-flow microfiltration (MF) and chemical coagulation using $FeCl_2 \bullet 4H_2O$ as the coagulant were implemented. The results revealed that the MF membrane (TM10) exhibited removal efficiencies of 67.2% for COD, 93.2% for suspended solids (SS), 99.3% for turbidity, and 98.6% for oil/grease. On the other hand, coagulation with ferric chloride achieved removal rates of 24.9% for COD, 66.8% for SS, 50.2% for turbidity, and 91.6% for oil/grease. Clearly, the TM10 MF membrane was more effective in the pre-treatment stage. Following the pre-treatment, the wastewater underwent further treatment using three different ultrafiltration (UF) membranes with molecular weight cut-offs (MWCO) ranging from 1 to 10 kDa in a cross-flow system. The GE (1 kDa) membrane demonstrated the highest removal efficiency, which was comparable to the GH (2 kDa) membrane. However, both the GH and GE membranes achieved fluxes of 25.58 and 20.43 $L/m^2/h$ at 6 bar pressure, respectively. For the final stage of treatment, the most efficient nanofiltration (NF) membrane (TS80) achieved removal efficiencies of 96.2%, 99.9%, 100%, and 70.7% for COD, SS, oil/grease, and electrical conductivity (EC), respectively. It was concluded that the NF filtrate could be reused as process water. This study successfully implemented a series of treatment methods for waste metalworking fluids (MWFs), effectively removing various pollutants and providing potential options for reusing the treated wastewater in the metalworking industry.

Key words: coagulation, membrane process, metalworking fluid, oil removal, wastewater

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1. Introduction

Metalworking fluids (MWFs) play a central role in the metalworking industries, serving to cool and lubricate the interface between tools and products. The primary objectives are to extend tool life, minimize friction, and reduce heat generation (Brinksmeier et al., 2009; MacAdam et al., 2012). Moreover, they contribute to improving product quality, removing metal chips, and safeguarding tools against corrosion (Hilal et al., 2004; Misra and Sköld, 1999).

However, the widespread use of MWFs results in a staggering annual consumption of 2,000,000 m^3 globally, generating ten times more wastewater due to pre-use dilution (MacAdam et al., 2012).

MWFs can be categorized into two main types: water-based and oil-based. Water-based MWFs are further classified as semi-synthetic and synthetic, while oil-based MWFs encompass soluble oils and straight oils. Water-based MWFs currently dominate industrial applications where efficient cooling is imperative, but their usage leads to higher concentrations of organic chemical pollutants in wastewater. Basic constituents of water-based MWFs include insoluble lubricants, emulsifiers, pH buffers, defoamers, and biocides (Bensadok et al., 2007; Cheng et al., 2005; Misra and Sköld, 1999).

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Due to factors like thermal degradation, particulate contamination, and biological contamination, MWFs require periodic replacement during the manufacturing process. Consequently, a substantial amount of toxic liquid waste is generated, necessitating proper treatment. The treatment of waste MWFs holds significant importance across numerous industries, particularly in the metal processing sector and its associated machine shops (Hilal et al., 2004).

There are various methods for separating oil and other pollutants from MWFs wastewaters like membrane filtration (Behroozi and Ataabadi, 2021), coagulation (Ríos et al., 1998), electrocoagulation (Aytac, 2022; Bensadok et al., 2008), extraction (Talbi et al., 2009), oxidation (Portela et al., 2001; Zakar et al., 2021), biochemical (Chachou et al., 2015 Sabina et al., 2014) processes. Among them, membrane filtration stands out as a suitable method. It can offer high removal efficiency, especially when applied together with a pre-treatment. In addition, the membrane processes feature high degreasing efficiency, low energy cost and compact design, as well as stable effluent quality and easy installation and operation (Masoudnia et al., 2013).

In the first part of this study, coagulation and laboratory-scale cross-flow microfiltration (MF) were applied for the pre-treatment of MWF wastewater. Then, ultrafiltration (UF) and nanofiltration (NF) were used for further treatment. Experiments were carried out to determine the effects of membrane type process such as microfiltration, ultrafiltration and nanofiltration, membrane materials and pore sizes, as well as the effects of operating conditions such as transmembrane pressure (TMP) on the removal efficiency of relevant parameters and permeate flux.

2. Material and methods

Wastewater samples were collected from the camshaft plant in Sivas, Turkey. The samples were collected at intervals for one month and stored at 4°C in a refrigerator before the experimental analysis. Characterizations of the wastewater for the metalworking plant were given in Table 1. The destabilization of the emulsions was carried out with emulation agents: ferric chloride ($FeCl_2 \cdot 4H_2O$) purchased from Merck (Darmstadt, Germany). NaOH and HCl were purchased from Merck (Darmstadt, Germany). Jar test was carried out by using Velp Scientifica FC65 at a volume of 250 mL samples mixed at 120 rpm for 1 minute and 30 rpm for 30 minutes. After that, the sample was allowed to settle for 30 minutes. Samples were collected in to measure pH, COD, TSS, turbidity, TOC, electrical conductivity (EC) and oil/grease.

COD, turbidity, and EC were measured by Hach Lange DR3900, WTW Turb 355 IR and WTW Cond 330i/SET. Chemical oxygen demand (COD, SM 5220 B), pH, total organic carbon (TOC, SM 5310 B), and oil/grease (SM 5520 B) were analysed according to APHA Standard Methods 20th edition (APHA, 1999). Total suspended solids (TSS) were analysed according to DIN EN 872, turbidity was analysed according to DIN EN 7027 and biochemical oxygen demand (BOD) was analysed according to DIN EN 1899). pH measured by Adwa AD8000. The pH of wastewater was adjusted by using solutions of NaOH and HCl. Membrane experiments were carried out in SEPA CF Cell (Sterlitech Corporation), a laboratory scale crossflow membrane system. The schematic diagram of the experimental filtration setup is shown in Fig. 1. The effective membrane area is 0.014 m^2.

Membranes used in this study, JX (MF, PVDF, 0.3μm, 130 L/m^2h), GE (UF, PVDF, 1 kDa, 18 $L/m^2.h$), GH (UF, 2 kDa, 20 $L/m^2.h$) were obtained from GE Osmosis; TM10 (MF, 0.2 μm, 50 $L/m^2.h$), UE10 (UF, 10 kDa, 50 $L/m^2.h$), TS80 (NF, 150 Da, 20 $L/m^2.h$), XN45 (NF, 500 Da, 35 $L/m^2.h$) were obtained from TriSep and NF245 (NF, Polyamide, 200-400 Da, 52-72 $L/m^2.h$) was obtained from DOW.

Membrane experiments for each membrane type were examined under different pressures (0.2-2 bar in MF membranes, 1-6 bar in UF membranes, 5-10 bar in NF membranes) at a constant flow rate (3.5 m/s) and temperature (25°C). The amount of feed wastewater for each study was 5 L. Filtration was performed in total recycling mode for 5 to 10 hours. Flux was regularly recorded by a connected computer. Samples were taken for the measurement of COD, TSS, TOC, turbidity, oil/grease and EC parameters.

The fluxes were calculated by Eq. (1) (Baharuddin et al., 2014).

$$Flux, F : J = \frac{\Delta V}{(A \cdot \Delta t)} \tag{1}$$

where ΔV represent the permeate volume, A was the effective membrane area, and Δt was operation time. The result was found in $L/h/m^2$.

Removal efficiencies were calculated by Eq. (2) (Baharuddin et al., 2014; Behroozi et al., 2019):

$$Removal\ efficiency,\ R = 1 - \frac{Cp}{Cf} \tag{2}$$

where Cp and Cf represent pollutant concentration in the filtrate and pollutant concentration in the feed, respectively.

In order to check whether the membrane was still usable, relative permeate flux were calculated by the Eq. (3) (Park et al., 2020):

$$Relative\ permeate\ flux\ = Jr = \frac{J}{J_{H_2O}} \tag{3}$$

where JH_2O is the flux of ultrapure water passed through the membrane at the beginning of the study.

After filtration the membrane was cleaned for 10 min at a pressure of 1.5 bar and 3.5 m/s of flow velocity using distilled water. If necessary, chemical cleaning is done by rinsing with 0.1 M NaOH and 100 mg/L of NaOCl. Membranes were stored with 0.5% formaldehyde solution.

Table 1. Characterization of the wastewater for metalworking plant

Parameter	*pH*	*COD (mg/L)*	*TSS (mg/L)*	*Turbidity (NTU)*	*Oil/grease (mg/L)*	*TOC (mg/L)*	*BOD (mg/L)*
Wastewater characterization	9.02	74.791	3.150	530	710	850	4.438

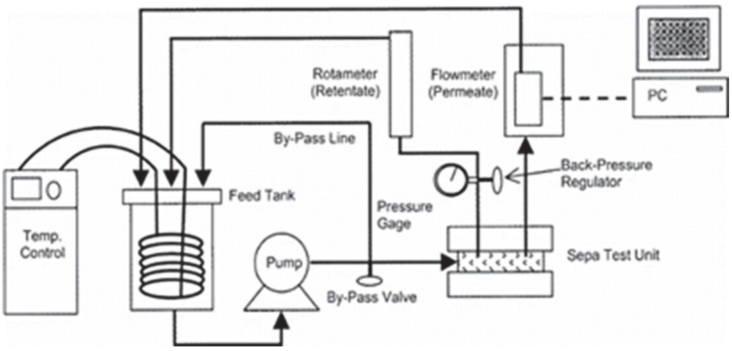

Fig. 1. The schematic diagram of laboratory scale membrane system (Matin et al., 2014)

3. Results

3.1. Coagulation

Three-stage coagulation experiments were carried out by using $FeCl_2 \cdot 4H_2O$ as the coagulant for a pre-treatment. The optimum coagulant dose (0.25 to 4 g/l), pH (6 to 9) and sedimentation time (10 to 60 minutes) was determined and experimental results were given in Table 2. For 2 g/L of $FeCl_2 \cdot 4H_2O$ at pH 9 and 60 min sedimentation time, COD, SS, oil/grease, TOC and turbidity values were found to be 24.87%, 66.78%, 91.56%, 97.95% and 50.19% respectively. It was observed that SS increased with increasing coagulant dose but decreased at 4 g/L. Sedimentation is not sufficient at 4 g/L coagulant dose. A similar situation is observed in the COD removal efficiency. The pollutants carried by the flocks that do not settle cause low COD removal efficiency. In addition, overdose and underdose of them negatively affect the coagulation process and the cost of chemical usage increases for an overdose of them (Sibiya et al., 2021). The concentrations are suitable for obtaining remarkable organic matter degradation values considering TOC removal of around 97% at 60 min sedimentation time. The high percentage of TOC removal even at a low concentration can be explained by adsorption and/or sweep coagulation in the form of precipitated hydroxide under alkaline pH conditions (Khouni et al., 2020; Ni et al., 2020).

However, it has been observed that the use of coagulation as a pre-treatment has damaged the membrane equipment and shortened the life span of the membrane. Also, Chesters et al. (2009) investigated the effects of cationic coagulants on membranes and suggested removing iron by filtration or using an antiscalant after the pre-treatment with a coagulant. Because of serious fouling problems and excessive contamination on the membrane surface, it was determined that coagulation is not suitable as a pre-treatment for membrane filtrations of MWFs.

3.2. Membrane treatment of MWFs

3.2.1. Use of MF membranes as a pre-treatment of MWFs

In the pre-treatment of wastewater, the use of MF membranes of JX (PVDF, 0.3 μm) and TM10 (PVDF, 0.2 μm) as an alternative to coagulation has been investigated for pre-treatment.

Different pressure (0.2, 0.5, 0.8, 2 bar) to microfiltration membranes were applied. The flux values obtained in pre-treatment studies with MF membranes were given in Fig. 2(a) for the JX membrane and Fig. 2(b) for the TM10 membrane. As shown in Fig. 2, the JX membrane was fouled with in a short time, while the TM membrane retained its flux values without fouling. Fouling is a common problem for hydrophobic membrane materials such as PVDF with hydrophobic wastewater materials such as oil/grease. Although both membranes are PVDF, different manufacturers produce membrane surfaces with different components and functional groups (Park et al., 2020).

The results of MF membranes for pre-treatment were evaluated in terms of COD, SS, TOC, turbidity and oil/grease parameters. The results were given in Table 3, as a comparison between the two membranes.

The percentage removal values were close to each other for the parameters studied in different pressures. At 0.8 bar, JX membrane was better for COD removal than other while TM10 membrane was obtained much better for COD and turbidity removal at 2 bar pressure. For both MF membranes, similar results were achieved in the parameters of COD, SS and TOC. As can be seen from Table 3, both membranes can provide 98.6% of oil/grease removal at any studied pressure. However, it was observed that oil droplets were formed in the filtrate of the JX membrane after a period of time. No oil droplets were observed in the filtrate of the TM10 membrane.

Alther et al. (2008) explained that oil and grease can be found in many different forms in wastewater. Oil and grease; free oil and grease can be found in water as mechanically emulsified oils, chemically emulsified oils, and dissolved oils. MWF contains chemically emulsified oil with size of droplets less than 20 microns. Both membranes used can hold these oil droplets due to their pore size. However, permeate obtained from the JX (0.3 µm) membrane has oil particles due to the presence of dissolved oils in the wastewater or the non-perfect surfaces of the membranes. Misra and Sköld (1999) also found that complete oil removal was attained if membrane pore sizes did not exceed 0.1 µm. Quicker fouling of the JX membrane, which has larger pores, may occur because oil particles can penetrate the pores of the membrane more easily (Kwon et al., 2000). Also, membrane fouling by oil droplets is a limiting factor because they reduce permeate flow by forming a cake layer on the membrane surface (Gutiérrez et al., 2008). It is seen that the time dependent flux drop in the JX membrane is much higher. Therefore, the use of TM10 (0.2 µm) membrane for pre-treatment was more suitable than the use of the JX membrane. The TM10 membrane was determined as a membrane suitable for pre -treatment because it provides higher flux at 2 bar pressure and provides relatively better efficiency.

To determine the use of coagulation or MF membranes as pre-treatment for the treatment of MWFs, comparison was done. The percentage removal efficiency for the TM10 membrane was found to be 67.2%, 93.2%, 99.3% and 98.6% for COD, SS, turbidity and oil/grease, respectively. On the other side, coagulation with $FeCl_2{\cdot}4H_2O$ were found to be 24.9%, 66.8%, 50.2% and 91.9% for COD, SS, turbidity and oil/grease, respectively. $FeCl_2{\cdot}4H_2O$ forms deposits in the pipes of the membrane system and prevents the pump in the system from operating smoothly. In addition, iron oxides accumulate on the membrane visibly. Due to all these, the TM10 membrane has been designated as the pre-treatment method in the treatment of the MWFs with the membrane system.

Table 2. Removal efficiencies of coagulation

Coagulant	*Coagulant dosage (g/L)*	*Removal Efficiency (%)*				
		COD	*SS*	*Oil/grease*	*TOC*	*Turbidity*
$FeCl_2{\cdot}4H_2O$ (At pH 9 and 60 min sed. time)	0.25	18.38	4.90	96.46	97.98	34.09
	0.5	20.54	64.34	91.39	97.54	65.22
	1	22.70	63.99	82.77	97.60	79.22
	1.5	22.70	70.98	92.67	97.82	40.27
	2	24.87	66.78	91.56	97.95	50.19
	3	28.11	63.29	87.63	97.94	44.21
	4	15.68	40.21	88.95	97.90	70.23

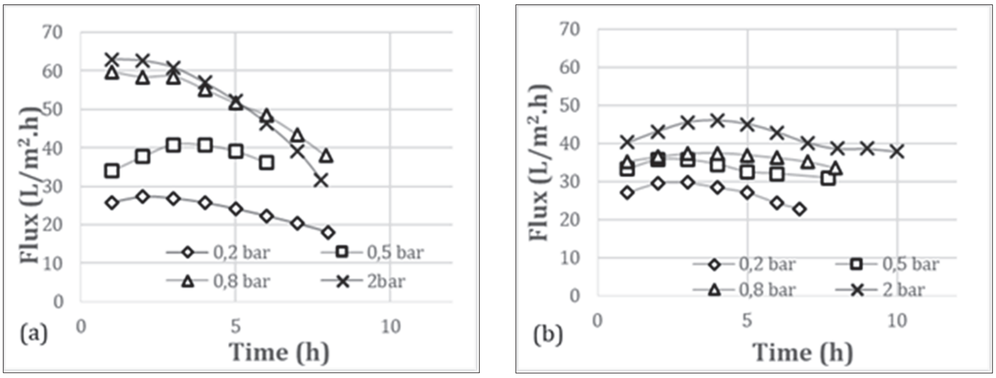

Fig. 2. Pressure-dependent flux values obtained on JX (0.3 µm) (a) and TM10 (0.2 µm), (b) membrane

Table 3. Removal efficiencies of MF membranes

	Pressure (bar)	*Removal Efficiency %*				
		COD	*SS*	*TOC*	*Turbidity*	*Oil/Grease*
JX	0.2	26.9	94	59.3	99.6	98.6
	0.5	55.4	97.3	52.7	87.1	98.6
	0.8	68.6	96	42.4	79.9	98.6
	2	63.5	92	50.3	99.2	98.6
TM10	0.2	37.3	94.3	52.4	99.7	98.6
	0.5	56.6	96.4	62.6	96.8	98.6
	0.8	57.1	99	62.4	96.8	98.6
	2	67.2	93.2	53.9	99.3	98.6

3.2.2. Ultrafiltration

The effluent of MF membrane was given to UF membranes to provide further removal of all studied parameters. For this purpose, UE10 (10 kDa), GH (2 kDa) and GE (1 kDa) UF membranes were used. Six different operating pressures between 1-6 bar were studied with UF membranes. The flux values obtained in pre-treatment studies with UF membranes were given in Fig. 3(a) for the UE10 membrane, Fig. 3(b) for the GH membrane and Fig. 3(c) for the GE membrane.

It may be seen from Fig. 3, UE10 (10 kDa) membranes have much higher flux values due to their higher MWCO. A flux of 70.61 L/m^2/h was obtained with the UE10 membrane at 6 bar. GE (1 kDa) and GH (2 kDa) membranes provided fluxes of 25.58 and 20.43 L/m^2/h, respectively at 6 bar. A similar result was also obtained in the study of Hilal et al. (2004), in which synthetic metalworking fluid wastewater was treated with UF and NF, and a flux of 49 L/m^2/h was obtained at 2 bar pressure in the 200 kDa UF membrane.

It may be seen from Table 4 that the GE membrane has higher removal efficiencies than the other UF membranes. UE10 showed low performance for COD and EC parameters due to the fact that its pore sizes were not as tight as other UF membranes. GH membrane gave similar results to the GE membrane. Considering that a higher flux was obtained, the GH membrane was preferred as optimal UF membrane for the treatment of MWFs wastewaters.

The results of treatment with UF membranes were evaluated in terms of COD, SS, EC, and turbidity parameters. The results were shown in Table 4, as a comparison between the three membranes.

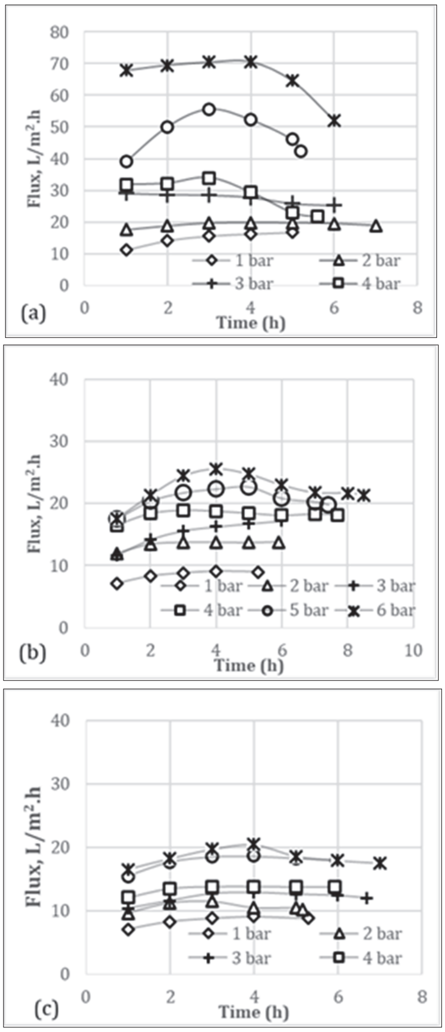

Fig. 3. Flux values obtained on UE10 (a), GH (b) and GE (c) membrane

Table 4. Removal efficiencies of UF membranes

	Pressure	***Removal Efficiency %***			
	(bar)	***COD***	***SS***	***EC***	***Turbidity***
UE10 (10 kDa)	1	42.8	86.1	11.5	98.4
	2	47.1	88.9	8.2	98.7
	3	42.5	83.3	9.2	98.5
	4	46	80.6	3	99.7
	5	45	83.3	3.9	98.2
	6	32	83.3	7.9	99.8
GH (2 kDa)	1	41.8	87.8	32.6	99.6
	2	50.9	87.8	28.6	99.7
	3	53	87.8	20.3	99.4
	4	65.6	87.8	18.8	99.7
	5	54.3	90.9	16.3	99.6
	6	55.8	97	18.1	99.7
GE (1 kDa)	1	55.1	84.3	28	99.3
	2	53.7	89.5	31.6	99.2
	3	60.7	94.8	23.9	98.5
	4	64.2	92.1	21	99.4
	5	68.4	92.1	23.2	98
	6	59.3	94.8	25	98.2

3.2.3. Nanofiltration

NF membranes were used to increase the COD removal efficiency, which could not be brought to a sufficient level after UF. NF membranes used for this purpose were TS80, XN45 and NF245 and have MWCO of 100-200, 500 and 200-400 Da, respectively. The working pressure was kept constant at 10 bar in NF studies. The flux values obtained in studies with NF membranes are given in Fig. 4. It can be seen from Fig. 4, 48.1, 22.21 and 13.5 L/m^2.h fluxes were obtained with XN45, TS80 and NF245 membranes at 10 bar pressure, respectively.

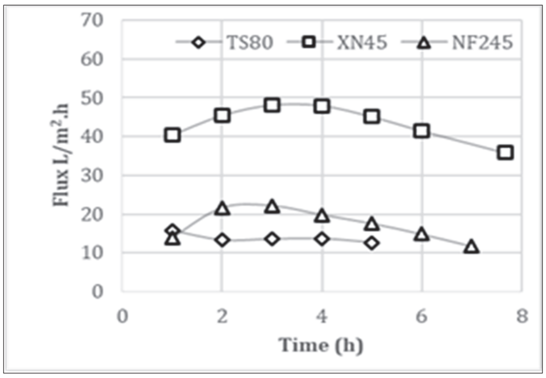

Fig. 4. Flux values obtained on TS80, XN45 and NF245 membranes at 10 bar

The results of the studies performed on the UF membranes were evaluated in terms of COD, EC and turbidity parameters. The results were given in Table 5, as a comparison of the three membranes. The highest removal rate among selected NF membranes was achieved with the TS80 membrane. However, this membrane showed the lowest flux value. Conversely, the XN45 membrane had the highest flow and lowest removal efficiencies. This was due to molecular weight limitations. The choice of high flux or high removal efficiencies is achieved in accordance with the needs.

Table 5. Removal efficiencies of NF membranes

	Removal Efficiency %			
	COD	**SS**	***Oil/grease***	***EC***
TS80 (100-200 Da)	96.2	99.9	100	70.7
XN45 (500 Da)	41.9	99.9	100	49.8
NF245 (200-400 Da)	67.0	99.9	100	59.5

4. Conclusions

Oil/grease removal was performed at high efficiency using $FeCl_2{\cdot}4H_2O$ coagulant in the coagulation process, but failure was determined by some parameters such as turbidity and SS. $FeCl_2{\cdot}4H_2O$ coagulant was not suitable as a pre-treatment before membrane processes due to iron oxide deposits in the system. It has been found that both MF membranes as pre-treatment can give lower the oil/grease concentration below 10 mg/L. However, TM10 (0.2 µm) membrane has better removal efficiency in the remaining parameters. For these reasons, membrane filtration using TM10 membranes was chosen as the appropriate method among coagulation and membrane filtration methods applied as pre-treatment.

It was found that the UE10 (10 kDa) UF membrane provides a much better flow rate than GE (1 kDa) and GH (2 kDa) but the removal efficiencies were less than others as expected. There was no significant difference between GE and GH membranes in terms of removal efficiency. Since higher flux is obtained with the GH membrane, it may be appropriate to use this membrane.

Among the NF membranes that were tested, the TS80 membrane provides the best removal efficiency. COD, SS, total oil/grease and EC removal after NF were found to be 96.2%, 99.9%, 100% and 70.7% respectively.

In this study, the following suggestions were made for the treatment of MWFs wastewater:

- As a pre-treatment we can advise using MF instead of coagulation such as $FeCl_2$ because of preventing the pump in the system from operating smoothly and iron oxides on the membrane accumulate visibly.
- It was determined that after using MF as pre-treatment, there is no need to use UF membrane and NF membrane can be used directly.
- After the treatment with the NF membrane, a high level of removal is achieved compared to the input values. This ensures that the treated wastewater can be reused as process water.

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