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Pre-concentration of Municipal Wastewater Using Flocculation-Assisted Direct Ceramic Microfiltration Process: Optimization of Operational Conditions

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Abstract Direct ceramic microfiltration (DCMF) is an effective technology to pre-concentrate organic matter (OM) for the subsequent anaerobic energy-recovering processes and a fast, cost-effective, easy treatment process for municipal wastewater. The major problem in DCMF is the rapid fouling of the membrane. In this study, to maximize OM recovery rates and prevent membrane fouling, the DCMF process was alternately paired with dosing of a cationic polyacrylamide (PAM) flocculant and chemically enhanced primary sedimentation (CEPS). The DCMF process tested in three stages: (i) optimization of flocculant concentration (0.5, 1, 1.5, and 2 mg/L PAM) and dosing point, (ii) optimization of operational conditions (pH, filtration/backwash duration, flux, and recovery rate) to control membrane fouling, and (iii) long-term operation of the DCMF process. The influence of PAM dosage points on DCMF fouling behavior was explored, and system operating parameters in terms of OM recovery and TMP change were optimized. The CEPS + DCMF setup was discovered to be a potential option for overcoming fouling. The highest chemical oxygen demand (COD) was 520 ± 20 mg/L in the concentrated wastewater using CEPS + DCMF experiments for 0.5 mg/L PAM. The highest OM pre-concentration was achieved at 90% recovery rate. After the optimization, COD concentration in the concentrate of the DCMF process reached 822 mg/L for the long-term (20 days) operation. The net potential energy production was calculated as 0.28 kWh/m^3 considering the theoretical COD of 1432 mg/L in the concentrate stream. As a novel approach, the CEPS + DCMF process can be used in place of conventional municipal wastewater treatment processes due to its acceptable OM removal performance, simple operation, small footprint, and potential energy generation.

Keywords Direct membrane filtration · Ceramic membrane · Pre-concentration · Chemically enhanced primary sedimentation · Municipal wastewater · Organic matter recovery

1 Introduction

Due to the scarcity and depletion of natural resources, wastewater should be considered as a renewable resource, from which water, OM, and nutrients can

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be recovered (Guven et al., 2019). In conventional wastewater treatment processes, large amounts of energy are consumed for aeration to promote biological oxidation for OM degradation and nutrient removal (Panepinto et al., 2016). OM in municipal wastewater can be regarded as a resource for energy production via anaerobic digestion (Sills et al., 2016) to generate methane, hydrogen, or volatile fatty acids (VFAs) as the concentration of OM is generally in the range of 250–1000 mg-COD L^{-1} (Aiyuk et al., 2004; Bielefeldt, 2009). Theoretically, the energy content in 1 g COD is around 3.5 kWh, which means average municipal wastewater may contain 1.66 kWh/m^3 energy. Hence, the energy contained in municipal wastewater maybe around four times higher than the energy required for a conventional activated sludge treatment process (Guven et al., 2019). Conversion of OM in wastewater to methane, hydrogen, and VFAs under anaerobic conditions is an alternative process for energy recovery. However, it is well known that the hydrolysis of suspended solids in municipal wastewater is the rate-limiting step at low temperatures (Xu et al., 2018) and may require a mesophilic temperature range of 25–37 °C. However, increasing the reactor temperature to the proper values may lead to the anaerobic process as energy-consuming rather than producing as around 1.2 kWh/m^3 energy is needed to increase water temperature for 1 °C (Xu et al., 2018). Hence, OM in the wastewater should be pre-concentrated to make the anaerobic treatment a more efficient and energy-positive process. Transitioning from aerobic towards anaerobic-based treatment processes offers the potential to considerably minimize the energy consumption of wastewater treatment by avoiding aeration and achieving energy-neutral wastewater treatment through biogas production (Dai et al., 2015; McCarty et al., 2011; Seib et al., 2016; Sills et al., 2016). For the pre-concentration of municipal wastewater, direct membrane filtration (DMF), dynamic sand filtration, centrifugation, CEPS, dissolved air flotation, and biological adsorption processes, high rate activated sludge process are applied individually or in combination (Guven et al., 2019; Jin et al., 2016; Wan et al., 2016). DMF of municipal wastewater has a significant potential owing to the simplicity of operation, low net energy requirement, high quality of treated water, high-water recovery ratio, and generation of the concentrated stream to be further used for energy generation (Gong et al., 2015, Huang et al., 2017, Kimura et al., 2017, Nascimento et al., 2017; Kimura et al., 2021; Nascimento & Miranda, 2021). Ceramic membranes (CMs) are more attractive than polymeric membranes due to their high filtration flux, low fouling behavior, and unique thermal and mechanical resistance (Liu et al., 2019; Meng et al., 2009; Oh et al., 2007; Zielińska & Galik, 2017). CMs have become more widely used in recent years for various water and wastewater treatments as a result of ongoing material development and lower prices (Li et al., 2020a). Although using CMs within the DMF process has become more attractive (Hafuka et al., 2020), limited studies have been reported on the use of CMs for DMF of municipal wastewaters (Zhao et al., 2019, Zhao et al., 2020).

The main problem in the application of the DMF process alone is fouling and the flux decrease like other pressure-driven processes (Arhin et al., 2016; Hube et al., 2020). To overcome the membrane fouling via removing or agglomerating the colloidal substances from wastewater, the CEPS process is an effective approach that applies chemical coagulants to greatly improve the removal of pollutants from wastewater by precipitation (Nair & Ahammed, 2015). CEPS has several advantages in wastewater treatment such as low investment cost (Aiyuk et al., 2004), low energy requirement (De Feo et al., 2008), ease to operate, and maintainance (Jordao & Volschan, 2004). For all these reasons, CEPS has been commonly utilized both in municipal wastewater treatment and also in industrial wastewater treatment for recent decades (Shewa & Dagnew, 2020). Moreover, CEPS has come into prominence in recent years, especially in OM/nutrient recovery from municipal wastewater and used as an alternative to conventional wastewater treatment processes (Czerwionka et al., 2020; Huang & Li, 2000; Lin et al., 2017; Taboada-Santos et al., 2020). Czerwionka et al. (2020) reported that biogas production was increased by 65–80% in anaerobic digesters after the CEPS process. Besides, with an increase in OM removal efficiency of primary settling tanks (Czerwionka et al., 2020). Zhao et al. (2019) also emphasized the advantages of the pre-coagulation process as a municipal wastewater pre-concentration method. They reported that the pre-coagulation process reduced the loading on membrane filtration, and high amounts of OM and phosphorus were recovered from the sewage (Zhao et al., 2019). However, the coagulation process, in which iron or aluminum salts are widely used (Hög et al., 2015; Bezirgiannidis et al., 2019; Li et al., 2020b; Alengebawy et al., 2021) as a fouling reducer for membrane processes, may cause metal contamination in both supernatant and sludge (Chakraborty et al., 2020). In addition, because the concentration of chemicals used in coagulation is higher than flocculation, it increases the treatment process cost. Metal salts used in the coagulation process may also negatively affect the anaerobic treatment performance of the concentrated stream (Mudhoo & Kumar, 2013). Hence, in recent years, many researches have been conducted on the recovery of OM from municipal wastewater, and still, the development of novel process alternatives is needed to develop energy-neutral processes. The DMF system is commonly implemented in previous studies for the pre-concentration of municipal wastewater (Lateef et al., 2013, Kimura et al., 2021). However, as few studies have been conducted on DMF applications using CMs, more research is required to optimize process performance and membrane cleaning strategies (Zhao et al., 2020). Furthermore, to the best of our knowledge, there is a gap in the literature for evaluation of the dosing point in the flocculation process using PAM, which would serve to increase process effectiveness. Particularly, in order to achieve long-term, sustainable membrane filtration, it is quite essential to consider the effect of PAM dosing point and operating parameters on permeate quality and membrane filtration performance. Hence, this study aims at evaluating the performance of PAM-assisted direct ceramic membrane filtration process for municipal wastewater treatment and OM recovery in the concentrated stream. The developed novel process was tested and optimized first in lab-scale before implementing it in pilot and then real-scale processes. In order to demonstrate the feasibility of the process on a real scale, the potential energy production potential was calculated based on the flow data of current municipal wastewater treatment plant in Kayseri, Turkey.

2 Materials and Methods

2.1 Membranes and Chemicals

Cationic polyacrylamide (PAM, Hydrofloc, Italy) of high molecular weight, medium–high cationic load, and bulk density of 0.7–0.8 g/cm^3 has been used as the flocculating agent. The stock solution of 0.1wt% PAM was used as polyelectrolyte in the flocculation experiments.

Sodium hydroxide (NaOH, Merck) and nitric acid (HNO_3, Merck) were used in cleaning the CM (Gruskevica & Mezule, 2021; Mei et al., 2017). All solutions were prepared using deionized water in the experiments.

The raw material of the operated CM was silicon carbide. The pore size of the membrane was 0.1 µm. The length × width × thickness of the membrane plate was 230 mm × 150 mm × 6 mm. The effective membrane area was 0.069 m^2.

2.2 Wastewater and Analysis

Raw municipal wastewater was collected from the primary settling tank effluent of the Kayseri municipal wastewater treatment plant (Turkey). The characteristics of samples are given in Table 1. Collected samples were stored at 4 °C, and samples were warmed up to room temperature (20 ± 5 °C) before use. The turbidity of wastewater samples was measured using a turbidimeter (TN100, Thermo Scientific, USA). Conductivity and pH were measured using 3620 IDS WTW multiparameter (WTW GMBH, Germany). Anion chromatography (AC) was performed on a Metrohm equipped with a Metrosep A Supp 5 (150 mm) analytical column and Metrosep C4 (4 mm) guard column to measure chloride (Cl^-), sulfate (SO_4^{2-}), phosphate-phosphorus (PO_4-P), and nitrite-nitrogen (NO_2-N). The chemical oxygen demand (COD), total nitrogen (TN), total suspended solid (TSS), and ammonia–nitrogen (NH_4-N) analyses were carried out following the "Standard Methods

Table 1 The characteristics of primary sedimentation tank effluent

Parameters	Raw wastewater
pH	7.3
Conductivity (µS/cm)	1476–1653
COD (mg/L)	324.4–468
NO_2-N (mg/L)	1.1–1.2
NH_4-N (mg/L)	41.9–41.7
PO_4-P (mg/L)	9.8–12.2

for Water and Wastewater" of the American Public Health Association (APHA, 2005).

2.3 Jar Tests

The flocculation performance of the cationic flocculant (PAM) was tested using a jar test with a six-paddled Jar Tester (Velp JLT6, Italy) on a bench of equal size. The procedure applied in jar test experiments is as follows: after the dosing of 0.5, 1, 1.5, and 2 mg/L PAM, 2 min rapid mixing (120 rpm), 15 min slow mixing (15 rpm), and 15 min sedimentation were performed. The supernatant liquid was carefully withdrawn from each beaker using a pipette from supernatant for COD, turbidity, TSS, and PO_4-P analysis. For the control sample, wastewater was settled for 15 min without dosing PAM. All tests were conducted at room temperature in the range of 20 – 25 °C. Before jar tests, the pH of the wastewater samples was adjusted to 7.0. Jar test was performed in duplicate, and the samples were characterized in each jar test. The average values are presented with standard deviations.

2.4 Experimental Set-up and Operational Conditions

After examining the effect of flocculant concentration on the removal of COD, turbidity, TSS, and PO_4-P in jar test experiments, three-step optimization experiments were performed in DCMF tests: (i) optimization of flocculant dosing point and concentration, (ii) optimization of operational conditions, and (iii) long-term operation. The methodology of DCMF filtration tests is summarized in Fig. 1.

In the first step of the DCMF experiments, polyelectrolyte dosing point and polyelectrolyte concentration were optimized. DCMF experiments were carried out for two different flocculant dosing points, including one-time PAM dosing in the CEPS process before DCMF (CEPS + DCMF) and continuous dosing of PAM during the DCMF process (PAM + DCMF). For the optimization of flocculant concentration and dosing point, the pH, flux, recovery ratio, and filtration/backwash duration were kept constant at pH 7.0, 20 LMH flux, 90% recovery rate, and 5/1 min filtration/backwash duration, respectively. The experiments were carried out by changing only the flocculant concentration and dosing point. NaOH and H_2SO_4 solutions were used to adjust the wastewater pH to 7.0. The filtration cycle included 5 min of filtration suction and 1 min of backwash. The flux was 20 LMH during the backwash. Ten percent of the feed flow was removed from the system as concentrate to keep the recovery rate constant at 90%. The HRT value was computed as 187 min based on the feed flow rate employed during the operation of the DCMF reactor, which is low enough to eliminate biological oxidation.

In the second step of DCMF tests, the operating parameters of the DCMF process were optimized using the optimal flocculant dosing point and concentration determined in the first stage of DCMF tests (Fig. 1). The operational parameters were optimized according to Table 2, by changing one parameter while keeping the others constant.

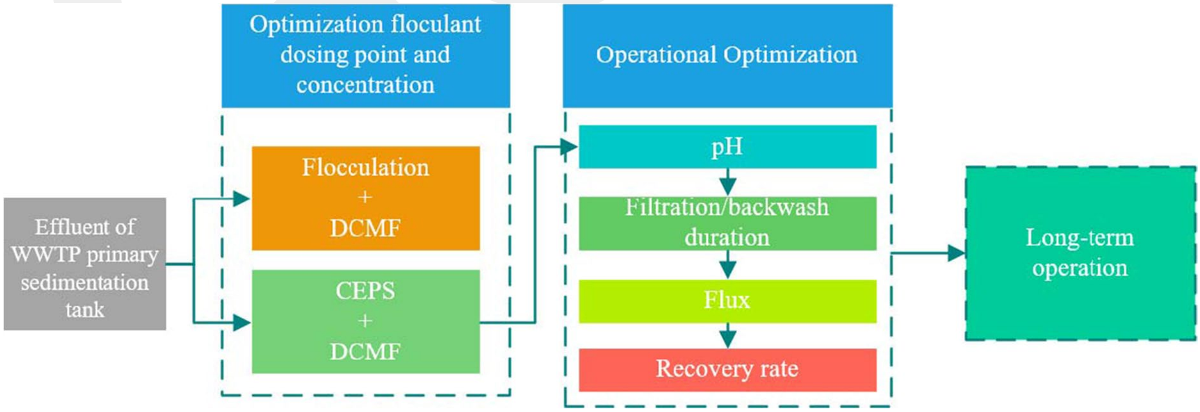


Fig. 1 Optimization of DCMF filtration

Table 2 The procedure followed in the optimization of the CEPS + DCMF process

Optimization parameters	Operational conditions				
	pH	Flux (LMH)	Water recovery rate (%)	Poly concentration (mg/L)	Filtration/backwash duration (min)
pH	6, 7, 8	20	90	0.5*	5/1
Filtration/backwash duration (min)	7	20	90	0.5*	5/1, 10/1, 10/2
Flux (LMH)	7	20, 30, 40	90	0.5*	10/2*
Recovery ratio (%)	7	20*	70, 80, 90	0.5*	10/2*

*These values were optimized in the dosing point optimization experiments

Schematic diagrams of PAM + DCMF and CEPS + DCMF processes are given in Fig. 2a and b, respectively. The dimensions of the reactor used in the system are 40 × 20 × 8.5 cm giving the total and effective volumes of 5.2 L and about 3.2 L, respectively. A flat-sheet membrane module was vertically fixed in the tank, and the filtrate was withdrawn through the membrane by a suction pump (Longer

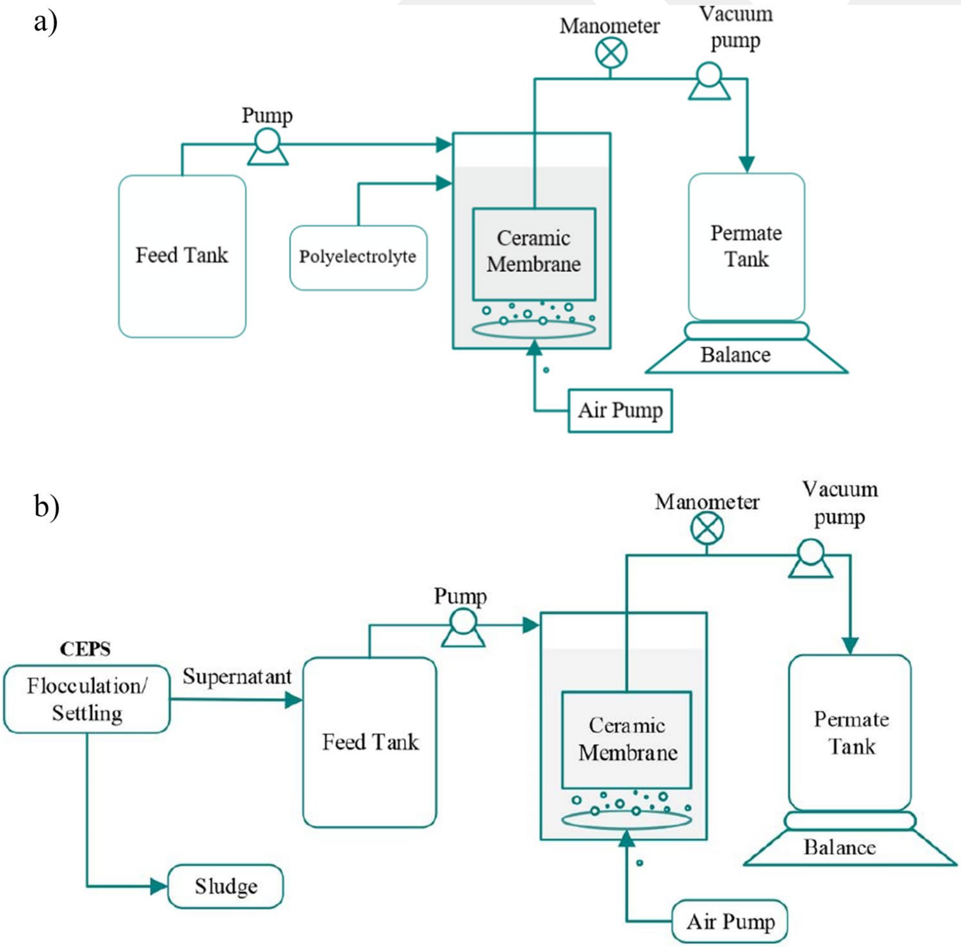


Fig. 2 Schematic diagram of the **a** PAM + DCMF and **b** CEPS + DCMF process

Pump, China). In addition, the formation of the cake layer was minimized by aeration of the membrane from the bottom of the reactor with a flow rate of 0.2 $m^3/(m^2h)$. The concentrated and feed wastewater samples were characterized in each DCMF test, which was performed in duplicate. The average values are presented with standard deviations.

In the third step, the long-term operation of the DCMF process was carried out after optimization of the dosing point and the operating conditions. CEPS + DCMF system was continuously operated for 20 days for treating primarily treated municipal wastewater. The system was operated for 6 h for every run each day. During the long-term operation, 3 virgin membranes, the properties of which are given in "Membranes and Chemicals", were used in a loop. The steps that take place in this loop are as follows; filtration was carried out in the DCMF reactor with one membrane, the second fouled membrane was physically cleaned after filtration, and the third membrane was chemically cleaned after physical cleaning. TMP behavior, as well as the characterization of concentrated wastewater and permeate, was investigated. During a typical operation, permeate was produced from 90% of the wastewater (23 mL/min) through the CM. Concentrated wastewater suspension was retained at 10% rates. Once the TMP reached 700 mbar, the membrane was cleaned by physical and chemical methods to recover its permeability. The membrane cleaning methods are given in detail in "Membrane Cleaning".

2.5 Membrane Cleaning

After each DCMF filtration experiment, the cake layer is physically cleaned with a sponge and backwashed with pure water. For the chemical cleaning of membranes, NaOH (0.5 M) and HNO_3 (0.5 M) solutions were used separately. According to the membrane cleaning procedure applied, the fouled membranes were taken out of the reactor and submerged in chemical solutions for offline cleaning. A three-stage cleaning procedure was applied to minimize flux loss in the CM used in DCMF experiments. Details of the cleaning procedure are as follows:

- Physical cleaning: After each DCMF filtration, the cake layer was cleaned with a sponge, and soft brushing was applied to the membrane and then 5 min backwash was applied with pure water for removing the remaining foulants from the membrane surface.
- Chemical cleaning with NaOH solution: Physically cleaned membrane was immersed in 0.5 M NaOH solution for 15 h. After NaOH cleaning, backwashing was applied with pure water for 5 min for removing the remaining pore-blocking foulants.
- Chemical cleaning with NaOH (0.5 M) and HNO_3 (0.5 M) solutions: This procedure was applied after each parameter optimization during the DCMF tests. The membrane was first immersed in 0.5 M NaOH and then 0.5 M HNO_3 solution for 15 h each. Following this chemical cleaning, the membrane was rinsed by backwashing with pure water for 5 min.

2.6 Specific Fouling Rate

The specific fouling rate (SFR, $g\,m^{-2}\,h^{-1}$) is defined as the increase in the mass of foulants per square meter per hour:

$$SFR = {}^{\Delta m}\!\big/_{(A\times\Delta t)}$$

where m is the foulant mass (g), A is the membrane area (m^2), and t is the filtration duration (h).

3 Results and Discussion

3.1 Flocculation Performance at different PAM Concentrations

Preliminary testing of polyelectrolytes with standard jar tests is essential for deciding the optimum polyelectrolyte dosage (Chong, 2012). Therefore, COD, TSS, and turbidity removals at different PAM concentrations were determined keeping pH at 7.0 in jar tests. Figure 3 shows the average COD, TSS, and turbidity removal rates and standard deviations for the duplicate jar test experiments. Even with the lowest concentration of 0.5 mg/L PAM, significant improvement in the sedimentation was observed, which in turn increased the removal efficiencies (Fig. 3). The COD removals without and with 0.5 mg/L PAM were 22% and 26%, respectively. Increasing PAM dosage

Fig. 3 COD, TSS, and turbidity removals at varying PAM concentrations in jar tests

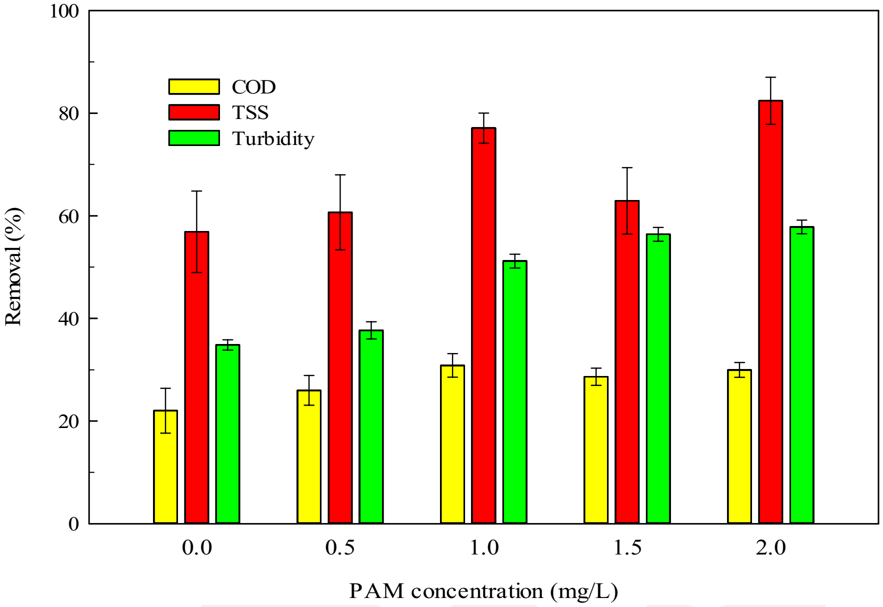


Table 3 The impacts of PAM concentration on pH, conductivity, and PO_4-P

Samples	pH	Conductivity (µS/cm)	PO_4-P (mg/L)
Raw wastewater	7.2 ± 0.1	2010 ± 25	5.6 ± 0.4
Without PAM	7.3 ± 0.2	2020 ± 14	5.5 ± 0.2
0.5 mg/L PAM	7.4 ± 0.0	1976 ± 8	5.6 ± 0.1
1 mg/L PAM	7.4 ± 0.1	1988 ± 9	5.7 ± 0.2
1.5 mg/L PAM	7.4 ± 0.1	1982 ± 9	5.0 ± 0.6
2 mg/L PAM	7.4 ± 0.0	1987 ± 10	2.4 ± 0.7

over 1 mg/L did not enhance COD removal efficiency, although 1, 1.5, and 2 mg/L PAM doses showed relatively similar values for COD removal efficiency (i.e., 31, 28, and 30%, respectively). Although the TSS removals were quite similar at 1 and 1.5 mg/L PAM dosages, the highest TSS removal (82%) was achieved at 2 mg/L PAM.

Besides COD, TSS, and turbidity measurements, the effluent of the jar test was also characterized in terms of pH, conductivity, and PO_4-P concentration (Table 3). After PAM dosage, the pH was slightly changed ranging between 7.2 and 7.4. The performance of flocculants is inherently not pH-dependent and does not cause any significant changes in pH (Ahmad et al., 2005, 2008; Amuda & Alade, 2006). In addition, conductivity decreased to 1976 ± 8 µS/cm in PAM dosed samples. The highest PO_4-P removal (57%) was obtained at 2 mg/L PAM.

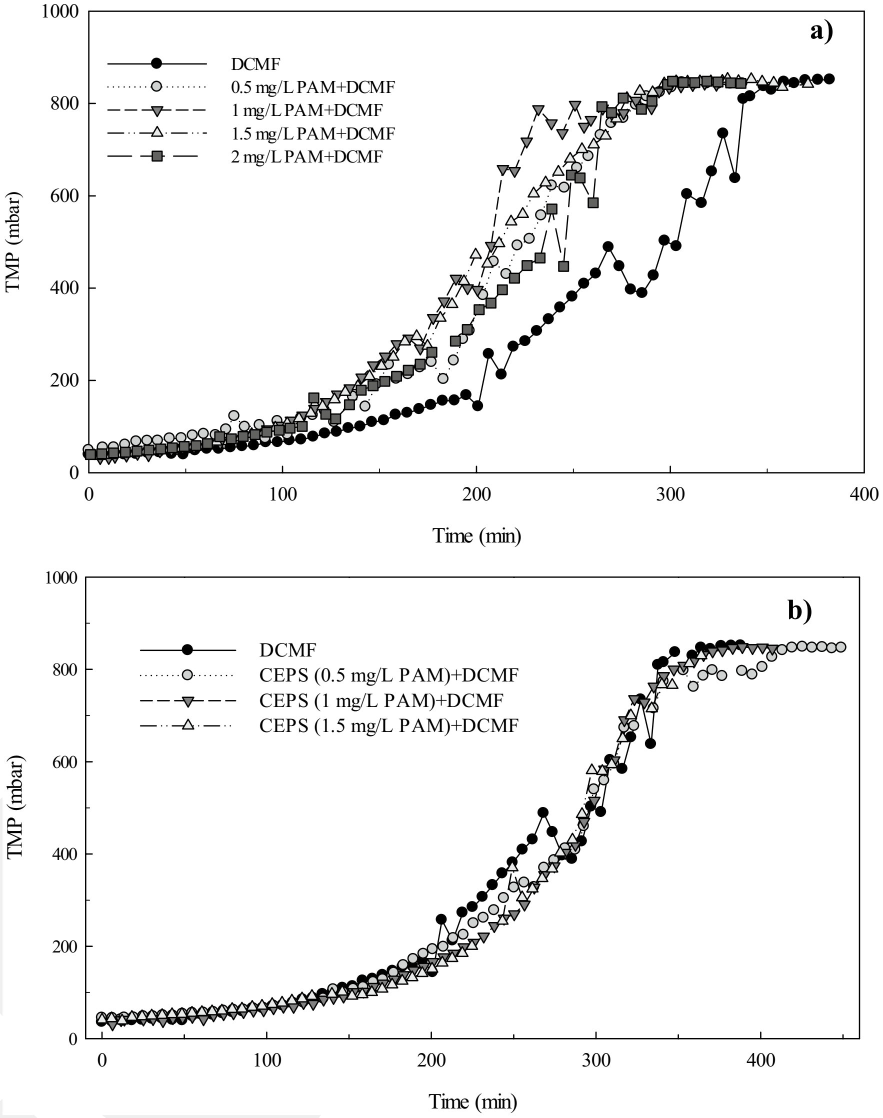
3.2 Determination of Optimum Configuration

To determine the optimum PAM dosing point and its concentration, DCMF experiments were conducted for two different configurations: PAM + DCMF and CEPS + DCMF as detailed in "Experimental Set-up and Operational Conditions."

The filtration performance of DCMF for two different configurations was determined in terms of the COD recovery and TMP elevation. The TMP changes for different configurations, i.e., PAM + DCMF and CEPS + DCMF, at varying PAM dosages, are provided in Fig. 4a and b. To evaluate the impact of PAM on filtration performance, the DCMF experiment was also conducted in the absence of PAM dosing.

In the DCMF tests without PAM addition, TMP increased above 350 mbar after 240 min. For the PAM + DCMF tests, using 0.5, 1, 1.5, and 2 mg/L PAM concentrations, TMP increased above 350 mbar after 205, 187, 184, and 200 min, respectively. On the other hand, TMP increased above 350 mbar after 268, 269, and 266 min at 0.5, 1, and 1.5 mg/L PAM concentrations, respectively, in CEPS + DCMF configuration. The positive effect of PAM dosing on TMP could not be obtained in PAM + DCMF experiments. Similarly, Malkoske et al. (2020) evaluated the effect

Fig. 4 TMP profile as a function of time for **a** PAM + DCMF and **b** CEPS + DCMF



of coagulation and flocculation on low-pressure MF and UF processes and obtained the greatest reduction in membrane fouling in CEPS + DMF processes (Malkoske et al., 2020). Additionally, the high molecular–weight polyelectrolytes may accumulate and form a gelatinous layer on the membrane surface, leading to a greater loss of flux and increasing the TMP (Dang et al., 2016). As seen in Fig. 4a, the PAM + DCMF cannot overcome the fouling problem due to this cake layer formation. Nevertheless, in the CEPS + DCMF process, gel layer formation on the membrane was prevented, which showed a positive effect in terms of TMP.

In order to investigate the effect of PAM dosing and CEPS process on cake layer formation, the cake layer was collected with water and softly brushed after each filtration experiment, and the SFR was calculated. Firstly, the concentrations of TSS were converted to mass for comparison, and the mass amounts of the cake layer were calculated. In the DCMF experiments without PAM, the mass of the cake layer was 270 ± 88 mg. In the PAM + DCMF experiments, the mass of the cake layer was 10.3 ± 1.3, 9.7 ± 1.1, 12.9 ± 1.4, and 13.3 ± 0.9 g/m^2, respectively, at 0.5, 1, 1.5, and 2 mg/L PAM dosages. Nevertheless, for CEPS + DCMF experiments, the TSSs of the cake layer were 3.3 ± 0.6, 3.1 ± 0.1, and 3.5 ± 0.7 g/m^2, respectively, at 0.5, 1, and 1.5 mg/L PAM dosages. According to the mass of the cake layer, the average SFR values and standard deviations for the duplicate filtration experiments are presented in Fig. 5. The increase in PAM dosage promoted the cake layer formation by supporting the adhesion of the solid matter on the membrane.

The characteristics of concentrated wastewater streams for two different DCMF configurations are given in Table 4. The PAM + DCMF experiments did not show better performance for the pre-concentration of the wastewater, like the performance of filtration performance based on TMP results. Since PAM + DCMF causes the polyelectrolyte to be attached to the reactor walls, membrane surface, reactor equipment such as pipes and connections, and COD content could not pass into the concentrate stream with the sludge pump (Du et al., 2019; Liu et al., 2017; Wang et al., 2011). The highest COD concentration of the concentrate (520 ± 20 mg/L) was achieved for 0.5 mg/L PAM dosing in CEPS + DCMF (Table 4). The lowest concentration of NH_4-N was obtained with 0.5 and 1.0 mg/L PAM CEPS + DCMF (37.9 and 35.7 mg NH_4-N/L) in concentrated wastewater samples. The low concentration of NH_4-N is an advantage in further anaerobic energy recovery studies, as it will be toxic to methanogens (Wang et al., 2015). Furthermore, PO_4-P was recovered in the CEPS + DCMF process at higher performance. PO_4-P concentration range in the concentrated streams of PAM + DCMF and CEPS + DCMF was between 8.0 and 9.9 mg PO_4-P /L and between 9.6 and 12.5 mg PO_4-P/L, respectively.

PAM is known as a common polymer used for flocculation while it is non-toxic to humans, animals, and plants. However, residual monomers in PAM can cause some environmental problems (Hennecke et al., 2018). Since the high concentration of the chemicals used will restrict the reuse of sludge in various applications with a circular economy approach, e.g., in agriculture, after appropriate treatment processes, small chemical dosages should be considered in the process (Aguilar et al., 2002; Kacprzak et al., 2017). In addition to the OM recovery and fouling tendency, the chemical costs must be taken into account in deciding the optimum chemical dosage (Ismail et al., 2012). Therefore, the optimum flocculant

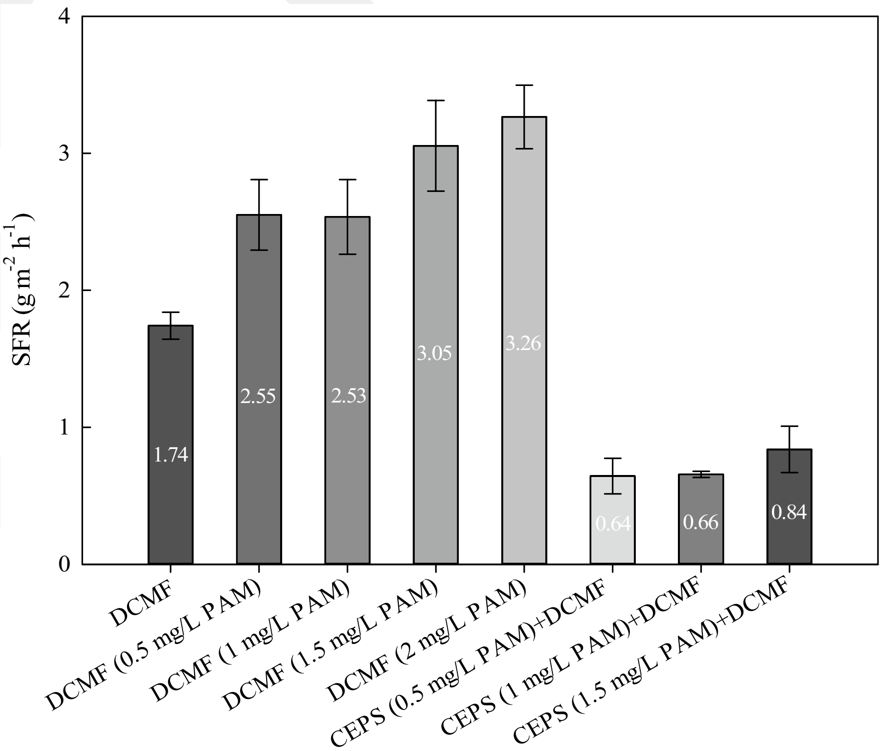


Fig. 5 Comparison of specific fouling rate (g m^{-2} h^{-1}) for DCMF, PAM + DCMF, and CEPS + DCMF processes

Table 4 Characteristics of the concentrated wastewater after DCMF process with different PAM concentration and configurations

Processes/parameters		pH	Conductivity (µS/cm)	COD (mg/L)	NO_2-N (mg/L)	NH_4-N (mg/L)	PO_4-P (mg/L)
DCMF		7.5 ± 0.3	1667 ± 105	495.7 ± 4.1	1.3 ± 1.0	47.8 ± 0.9	8.1 ± 0.6
DCMF (0.5 mg/L PAM)		7.5 ± 0.4	1642 ± 47	340.7 ± 42.4	1.5 ± 0.1	44.3 ± 2.2	8.1 ± 0.6
DCMF (1 mg/L PAM)		7.4 ± 0.2	1380 ± 404	352.7 ± 4.5	1.4 ± 0.2	44.1 ± 8.4	9.9 ± 3.2
DCMF (1.5 mg/L PAM)		7.5 ± 0.1	1628 ± 66	325.4 ± 18.2	1.2 ± 0.1	38.2 ± 1.1	8.0 ± 0.8
DCMF (2 mg/L PAM)		7.5 ± 0.1	1782 ± 19	331.6 ± 41.9	1.1 ± 0.1	49.4 ± 0.1	9.7 ± 2.7
CEPS (0.5 mg/L PAM) + DCMF	Feed*	7.4 ± 0.1	1586 ± 185	480 ± 181	1.4 ± 0.1	35.7 ± 9.1	11.5 ± 1.2
	Concentrated WW	7.5 ± 0.1	1474 ± 115	520 ± 20	0.9 ± 0.1	37.9 ± 6.6	11.3 ± 0.3
CEPS (1.0 mg/L PAM) + DCMF	Feed*	7.6 ± 0.5	1599 ± 62	352 ± 90	0.9 ± 0.1	36.8 ± 13.3	10.8 ± 2.2
	Concentrated WW	8.1 ± 0.7	1599 ± 75	341 ± 98	1.0 ± 0.1	35.7 ± 11.8	9.6 ± 2.3
CEPS (1.5 mg/L PAM) + DCMF	Feed*	7.5 ± 0.2	1664 ± 8	423 ± 75	1.1 ± 0.1	43.8 ± 0.2	12.3 ± 1.8
	Concentrated WW	7.8 ± 0.5	1663 ± 35	429 ± 79	1.0 ± 0.1	46.7 ± 3.1	12.5 ± 1.2

*Supernatant of CEPS process was used as feed

concentration considering OM recovery, filtration performance, and the chemical cost was determined as 0.5 mg/L PAM.

3.3 Optimization of Operational Conditions of DCMF Process

The effective membrane fouling control approaches by determining optimum operational conditions may extend the lifespan of the membranes and reduce operational costs (Hube et al., 2020; Jin et al., 2017). The optimization of operational conditions is crucial to achieve sustainable long-term operations of DMF processes (Jin et al., 2017; Lateef et al., 2013). After the determination of the optimum polyelectrolyte (PAM) concentration and process configuration, the operating conditions were optimized in terms of pH, flux, filtration/backwash duration, and water recovery ratio for the CEPS + DCMF mode of operation.

Inlet and outlet wastewater characteristics in the CEPS process are presented in Table 5. There are no significant removals of NO_2-N, NH_4-N, and PO_4-P as 2%, 4%, and no removal, respectively. However, the most significant removal was observed for TSS reduced from 184 ± 57 to 108 ± 55 after the CEPS process, which is crucial for controlling the cake layer developing on the membrane surface. Besides, COD was decreased from 496 ± 92 to 388 ± 86 mg/L in the CEPS process with 0.5 mg/L PAM dosing.

Table 5 The characteristics of raw wastewater and CEPS process effluent

Parameters	Raw WW	0.5 mg/L PAM dosing*
pH	7.4 ± 0.2	7.5 ± 0.2
Conductivity (µS/cm)	1624 ± 199	1678 ± 90
COD (mg/L)	496 ± 92	388 ± 86
TSS (mg/L)	184 ± 57	108 ± 55
NO_2-N (mg/L)	1.0 ± 0.4	1.1 ± 0.2
NH_4-N (mg/L)	47.0 ± 1.3	46.0 ± 2.2
PO_4-P (mg/L)	10.0 ± 0.9	9.6 ± 1.1

*The average values are presented here with ± indicating the standard deviations for 15 samples collected after every CEPS experiment

3.3.1 *Optimization of pH*

After determining the optimum polyelectrolyte concentration, pH was optimized with jar tests at 0.5 mg/L PAM dosing. The pH of the raw wastewater was 7.3. In determining the optimum pH in jar tests, the pH of the samples was adjusted to 6, 7, and 8 using H_2SO_4 or NaOH solutions. Each jar test was performed in duplicate for different pH values, and the conductivity and COD parameters were measured after each test, and the results are presented in Table 6. COD removal efficiencies at pH 6, 7,

Table 6 Effect of pH on COD removal performance of CEPS process conducted at 0.5 mg/L PAM

pH	Conductivity (µS/cm)	COD (mg/L)	COD removal (%)
Raw wastewater	1639	305 ± 5	–
7.3	1632 ± 3	210 ± 1	40.5 ± 0.3
6	1762 ± 8	180 ± 7	49.2 ± 2.0
7	1653 ± 16	190 ± 8	46.1 ± 2.3
8	1794 ± 7	180 ± 9	49.2 ± 2.5

and 8, were quite similar as 49.2, 46.1, and 49.2%, respectively. On the other hand, the lowest removal efficiency in terms of COD was obtained in the experiments conducted with the pH (7.3) of the raw wastewater. Filtration experiments were carried out at pH 7 and without pH adjustment due to the insignificant differences in COD removal obtained in the jar tests. It has also been stated that the flocculation is not a process that requires pH adjustment, unlike the coagulation process (Chong, 2012; Lee et al., 2014).

The effect of pH on TMP is presented in Fig. 6 for CEPS + DCMF experiments with 0.5 mg/L PAM. Although the pH had no noticeable impact on the TMP change, it was only revealed that the experiment that used the wastewater's natural pH performed better overall, particularly between the 280th and 380th minute of filtration.

Concentrated wastewater characteristics at the optimum pH are given in Table 7. COD concentrations for pH 7 and 7.3 were 520 ± 20 and 510 ± 10 mg/L, respectively. The COD concentration in the CEPS + DCMF process concentrate was unaffected by the pH of the wastewater.

At pHs 7.0 and 7.3, there was no discernible difference in filtration performance. Therefore, the experiments were carried out at the wastewater natural pH in the following optimization stages.

3.3.2 *Optimization of Flux and Filtration/Backwash Time*

Numerous studies have used intermittent filtration and/or backwash to mitigate membrane fouling (Hofs et al., 2011; Jin et al., 2010; Xue et al., 2016). In this study, the DCMF process was operated using aeration

Table 7 Characteristics of the concentrated wastewater after DCMF at different pHs

Parameters	pH 7	pH 7.3
pH	7.5 ± 0.1	8.2 ± 0.1
Conductivity (µS/cm)	1474 ± 115	1600 ± 8
COD (mg/L)	520 ± 20	510 ± 10
NO_2-N (mg/L)	0.9 ± 0.1	1.3 ± 0.0
NH_4-N (mg/L)	37.9 ± 6.6	44.4 ± 11.1
PO_4-P (mg/L)	11.3 ± 0.3	9.4 ± 0.5

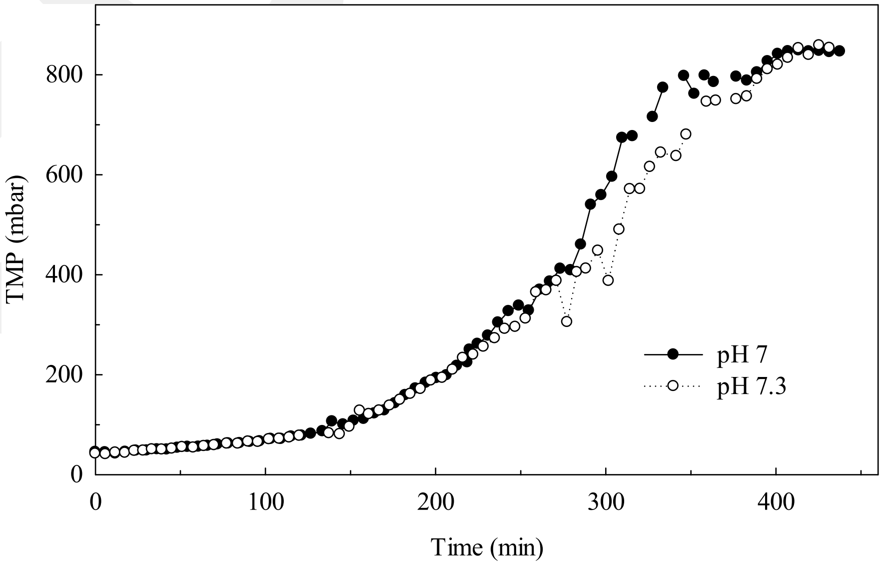


Fig. 6 TMP change in CEPS + DCMF process with different pH

and frequent backwashing together to reduce the membrane fouling. Thus, the formation of reversible fouling can be controlled with optimized backwash duration, resulting in less frequent chemical cleaning and reduced operating costs (Kalboussi et al., 2018). In these DCMF experiments, net flux was kept at 20 LMH, and the TMP change against time at different filtration/backwash durations (5/1, 10/1, 10/2 min) is given in Fig. 7. Major fouling of the membrane was detected when the system was operated with 10/1-min filtration/backwash time. As can be seen in Fig. 7, although the TMP increased in the system for 10/2 and 5/1 min of filtration/backwash times, the membrane operated with 10/2 filtration/backwash time had lower TMP after 250 min filtration time.

The concentrated wastewater characterizations in the DCMF tests carried out for the optimization of filtration/backwash durations are given in Table 8. It was determined that the wastewater was not concentrated at the rate of 10/1 min filtration/backwash, which was ended after 220 min of filtration because of the fouling of the membrane. The COD in the concentrated streams was 480 ± 10, 545 ± 34, and 362 ± 39 mg/L at 5/1, 10/2, 10/1 min filtration/backwash time, respectively.

The filtration/backwash duration of 10/2 min exhibited superior performance in terms of filtration performance. Then, filtration was carried out at 20, 30, and 40 LMH fluxes for the optimization of the process flux. The TMP behavior for the filtration is presented in Fig. 8. For the DCMF operated at 30 and 40 LMH fluxes, the TMP increased above 350 mbar after 68 and 40 min, respectively, and these filtration experiments were ended after 157 and 71 min, respectively. In the CEPS + DCMF experiment carried out at 20 LMH flux values, the TMP value increased over 350 mbar after the 220th min, and the experiment was ended at the 424th min. The higher fluxes required higher TMPs, often increasing the fouling rate and subsequently forcing more frequent cleaning, ultimately reducing the productivity (Campinas et al., 2021).

Characteristics of the concentrated wastewater after DCMF at 20, 30, and 40 LMH fluxes are given in Table 9. The highest COD concentration was

Table 8 Characteristics of the concentrated wastewater after DCMF with different filtration/backwash time

Parameters	Filtration/backwash duration (min)		
	5/1	10/2	10/1
pH	8.2 ± 0.1	7.8 ± 0.0	7.9 ± 0.4
Conductivity (μS/cm)	1600 ± 8	1695 ± 52	1663 ± 108
COD (mg/L)	480 ± 10	545 ± 34	362 ± 39
NO_3-N (mg/L)	1.3 ± 0.0	1.0 ± 0.1	0.9 ± 0.4
NH_4-N (mg/L)	44.4 ± 11.1	49.4 ± 3.4	49.6 ± 0.8
PO_4-P (mg/L)	9.4 ± 0.5	9.0 ± 1.4	9.1 ± 0.1

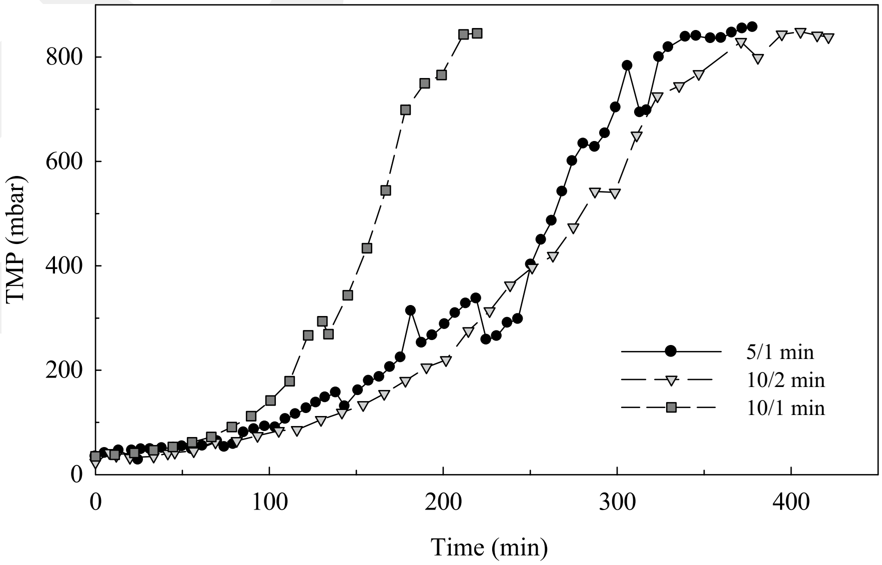


Fig. 7 Change in TMP in CEPS + DCMF process with filtration time with different filtration/backwash time

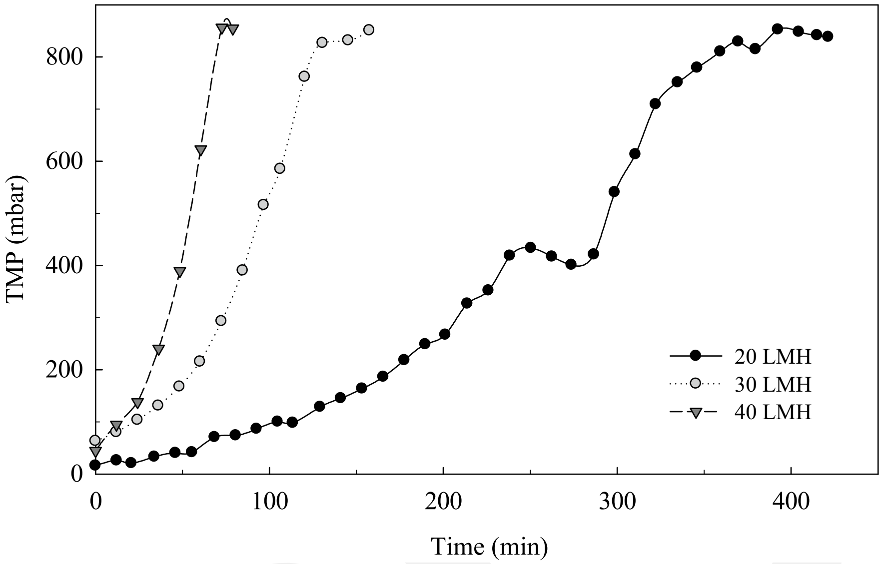


Fig. 8 TMP change in CEPS + DCMF process with different fluxes

Table 9 Characteristics of the concentrated wastewater after DCMF with different flux

Parameters	20	30	40
pH	7.8 ± 0.0	7.2 ± 0.1	7.4 ± 0.1
Conductivity (µS/cm)	1695 ± 52	1667 ± 30	1635 ± 7
COD (mg/L)	545 ± 34	420 ± 60	479 ± 27
NO_3-N (mg/L)	1.0 ± 0.1	ND	1.0 ± 0.7
PO_4-P (mg/L)	9.0 ± 1.4	9.2 ± 0.2	10.9 ± 0.4

reached at 20 LMH (545 ± 34 mg/L). These results were directly related to the duration of the experiment. Permeate production was approximately 7.8, 4.8, and 2.5 L for 20, 30, and 40 LMH fluxes, respectively. In the experiment conducted with a flux of 20 LMH, the duration of filtration extended up to 420 min (Fig. 8).

In the CEPS + DCMF process, the optimum flux was determined as 20 LMH in terms of TMP and COD recovery. In DMF studies conducted in the literature with the polymeric membrane with similar pore size (0.1 µ), the flux values vary between 4.2 and 20.8 LMH (Jin et al., 2016; Kimura et al., 2017; Lateef et al., 2013). However, CMs are usually operated at higher fluxes than the polymeric membranes, such as 20–40 LMH, but lower fluxes should be considered to reduce fouling, and associated energy demand for permeate withdrawing (Hofs et al., 2011).

3.3.3 *Optimization of Recovery Rate*

The effect of different water recovery rates of 70%, 80%, and 90% on DCMF system performance has been evaluated at 0.5 mg/L PAM, natural wastewater pH, the filtration/backwash time of 10/2 min, and at 20 LMH flux conditions. The TMP profile in the CEPS + DCMF experiments is given in Fig. 9 for 70%, 80%, and 90% membrane recovery rates. In DCMF experiments conducted at different recovery rates, similar TMP changes were observed until the 200th minute, while TMP was lower at 80% recovery rate in the rest of the filtration.

Characterization of concentrated wastewater obtained in CEPS + DCMF experiments conducted within the scope of optimization of the recovery rate is given in Table 10. Here, the best performance in terms of OM recovery was obtained for a 90% recovery rate and showed promising potential in energy recovery studies. In the literature, it is known that ceramic microfiltration membranes were used in recent years and studies with high recovery rates were carried out in DCMF systems similar to the experimental setup used in this study (Zhao et al., 2019, Zhao et al., 2020).

3.4 Long-Term Operation of CEPS + DCMF Process and Energy Production Potential

The CEPS + DCMF system was operated for 20 days for long-term treatment of primarily treated municipal

Fig. 9 TMP change in CEPS + DCMF process with the different recovery rates

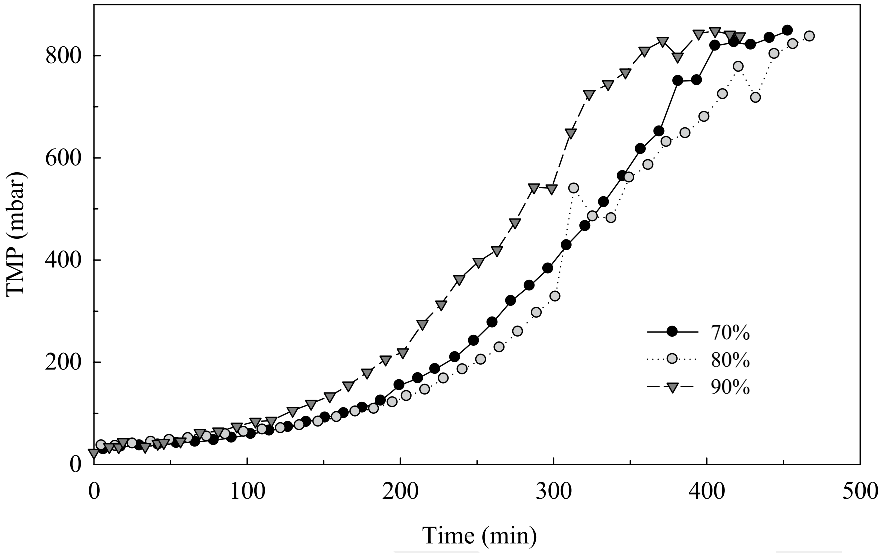
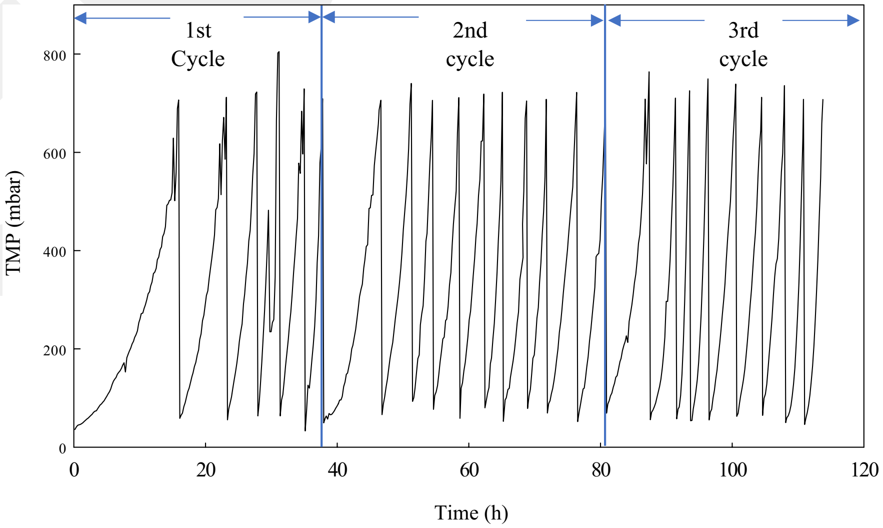


Table 10 Characteristics of the concentrated wastewater with different recovery rates

Parameters	70% recovery	80% recovery	90% recovery
pH	8.0 ± 0.1	7.7 ± 0.2	7.8 ± 0.0
Conductivity (µS/cm)	1689 ± 76	1678 ± 54	1695 ± 52
COD (mg/L)	444 ± 86	443 ± 73	546 ± 34
NO_3-N (mg/L)	1.1 ± 0.2	1.4 ± 0.2	1.0 ± 0.1
PO_4-P (mg/L)	10.4 ± 0.4	10.0 ± 1.0	9.0 ± 1.4

wastewater. Change in TMP with filtration time during the continuous operation of the CEPS + DCMF process is presented in Fig. 10. When the TMP reached the critical value of 700 mbar, the fouled membrane was subjected to off-line chemical cleaning. After each filtration, the membrane was physically cleaned with a sponge and chemically cleaned by conducting immersing in 0.5 M NaOH solution for 15 h. At the end of the 1st and 2nd cycle of operation, the membrane was cleaned by immersing it in 0.5 M

Fig. 10 TMP profile as a function of time for continuous operation of CEPS + DCMF process



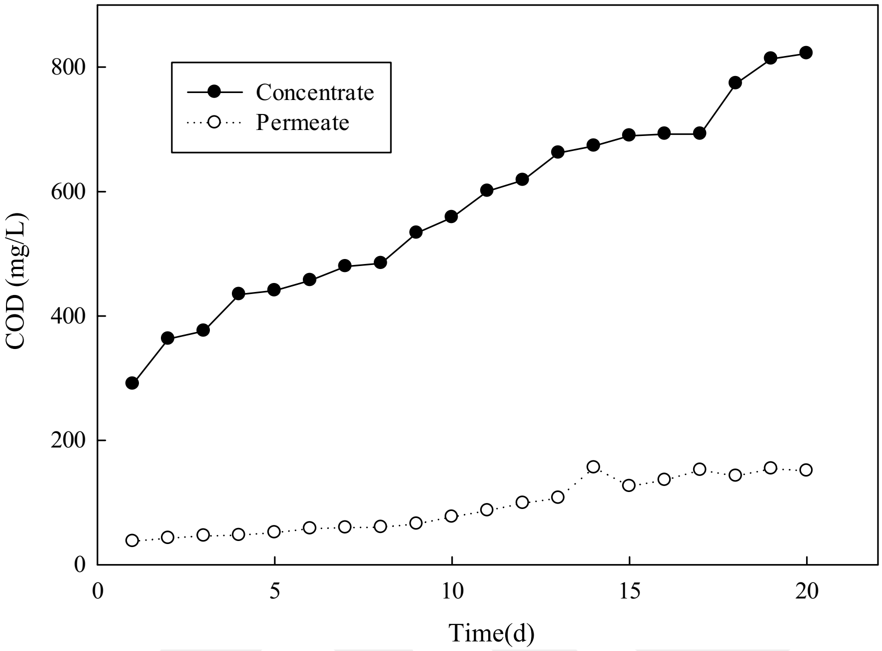


Fig. 11 COD concentrations in the concentrate and permeate of continuously operated CEPS + DCMF process

HNO_3 solution in addition to physical and NaOH cleaning procedure, due to a decrease in time of the filtration duration. It can be noticed that the trend of TMP change with filtration time varied for three different filtration cycles. From the beginning of the filtration to around 16 h (1st cycle), the TMP showed a gentle increase to around 300 mbar in the first 10 h but followed with a sharp increase to 700 mbar as the filtration time went from 10 to 16 h. Since the duration of the experiment gradually decreased in the first 6 filtrations, the HNO_3 cleaning procedure was also applied after the NaOH cleaning procedure.

The CEPS + DCMF process was operated with a concentration factor of 10 and COD concentration increased during the operation (Fig. 11). The COD concentration in the concentrate was 290 and 822 mg/L, initial and after 20 days of operation, respectively. Permeate COD concentration increased similar to the concentrate concentration as it increased from 38 to 142 mg/L at initial and after 20 days of operation, respectively.

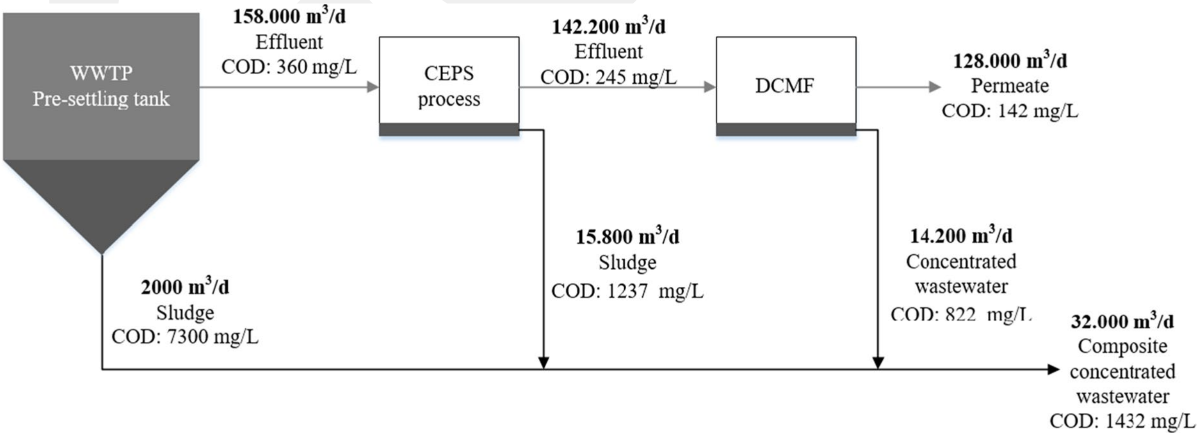


Fig. 12 Theoretical COD concentrations in different units of the proposed process used in mass balance and energy calculations

Table 11 Energy balance for electrical energy requirements and potential production with CEPS + DCMF process

Parameter	Value
Energy for permeate production	
Average TMP (m H_2O)[a]	4
Permeate flow rate (m^3/s)	1.48
Power requirement for permeation (kW)[b]	58.07
Required pumping energy for permeation (kWh/m^3)[c]	0.011
Energy for feeding pumping	
Reactor head loss (m)	0.1
Feed flow (m^3/s)	1.64
Power requirement for feeding (kW)[b]	1.61
Required pumping energy for feeding (kWh/m^3)[c]	0.0003
Energy for aeration	
Reactor head loss (m)	3
Air flow (m^3/sn)	14.8
Power requirement for aeration (kW)[b]	435.6
Required energy for aeration (kWh/m^3)	0.082
Electrical energy production potential from methane	
Organic matter amount of concentrate (kg COD/day)	45,824
Methane production (m^3 CH_4)[d]	16,038
Methane energy content (kWh/m^3)[e]	1.13
Electrical energy production from methane (kWh/m^3)[f]	0.37
Net energy production (kWh/m^3)	0.28
Electrical energy required for conventional treatment (kWh/m^3)	0.26

[a] For feeding pump head loss was assumed 0.1 m. Average TMP during long term operation (0.23 bar), equivalent to a hydraulic head loss E of 2.3 m for permeation.

[b] Energy requirement = QγE/1000, where Q (m^3/s) is flow rate, $\gamma = 9800$ N/m^3 and E (m) is head loss (Kim et al., 2011)

[c] Assumed energy transfer efficiency of 65% in conversion of electrical energy to pump energy(Kim et al., 2011)

[d] The CH_4 production potential of 1 g COD is around 0.35 L

[e] The heating value of 1 m^3 methane used in the calculation was 11.3 kWh (Wang et al., 2018)

[f] The energy conversion efficiency is accepted as 33%

The COD concentration was used to quantify the amount of OM in concentrated wastewater and to predict the potential for energy production in further anaerobic processes. The process flow diagram and material balance obtained considering the experimental results are provided in Fig. 12. The pre-settling tank flow rates were determined based on existing wastewater treatment plant data. Approximately 1.25% of the wastewater is removed as sludge in the pre-settling tank. The pre-settling tank effluent is fed to the CEPS process, and 10% sludge is removed. The COD concentration of the concentrated stream in this treatment process was calculated as 1432 mg/L. For each process, average COD concentrations were provided for mass balance calculations.

For the real-scale WWTP from which wastewater is obtained, the influent flow rate of the pre-settling tank is 160,000 m^3/day. According to mass-balance calculations provided in Fig. 12, 32,000 m^3/day concentrate will be generated corresponding to 45,824 kg COD/day. The most frequently used basic technology to recover energy from sludge is anaerobic digestion to generate methane. Hence, methane generation is considered in the energy generation calculations. On the other hand, the energy requirement of the novel DCMF process was also included in the energy balance calculations. Thanks to the proposed OM, recovery process 0.28 kWh/m^3 of energy can be produced. However, the energy consumption of the existing conventional wastewater treatment plant is 0.26 kWh/m^3. In other words, the application of CEPS + DCMF has a high potential for the pre-concentration of OM from municipal wastewater for further energy recovery to achieve energy-positive wastewater treatment. The required energy and the potential energy generation for the suggested process are illustrated in Table 11. The detailed study in the table shows that the suggested process is an energy-positive process, which can be safely used for the treatment of municipal wastewater.

4 Conclusions

In this study, a novel DCMF process configuration with PAM dosing was developed with the aim of concentrating OM from municipal wastewater for potential energy recovery. Different process configurations on fouling behavior of DCMF were investigated, and the system operating conditions were optimized in terms of OM recovery and membrane fouling. Compared to the PAM + DCMF configuration, the CEPS + DCMF was a promising process, and dosing of 0.5 mg/L PAM was effective for the pre-concentration purpose. pH did not have a significant effect on filtration performance. pH had no noticeable impact on the effectiveness of the filtration process. In the DCMF operations, TMP increased above 350 mbar after 220, 68, and 40th min, respectively, at 20, 30, and 40 LMH fluxes. Membrane fouling was reduced at filtration/backwash duration of 10/2 min. Thus, the formation of reversible fouling is controlled with the optimized backwash duration. COD concentration in concentrated wastewater was 444 ± 86 mg/L, 443 ± 73 mg/L, and 546 ± 34 mg/L, respectively, at recovery rates of 70, 80, and 90%. The long-term operation of CEPS + DCMF for 20 days, COD was reached 822 mg/L COD in concentrated wastewater. When evaluated with a holistic approach, according to the theoretical COD calculations, 1432 mg/L COD can be obtained in the concentrated stream by using the CEPS + DCMF process. If the CEPS + DCMF process were utilized as a real scale treatment, 0.28 kWh/m^3 of energy can be generated. These results indicated that CEPS + DCMF has a high potential for the pre-concentration of OM from municipal wastewater for further energy recovery studies.

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Data Availability The datasets generated during and/or analyzed during the current study are available from the corresponding author on reasonable request.

Declarations

Conflict of Interest The authors declare no competing interests.

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