Journal of Environmental Chemical Engineering 12 (2024) 114756

Contents lists available at ScienceDirect

Journal of Environmental Chemical Engineering

journal homepage: www.elsevier.com/locate/jece

Direct membrane filtration of municipal wastewater: Assessment of different fouling control strategies to enhance its energy, environmental and economic viability

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ARTICLE INFO

Keywords:
Direct membrane filtration
Chemical enhanced backwashing
Ultrafiltration membranes
Resource recovery
Municipal wastewater

ABSTRACT

This study assessed the viability of direct membrane filtration (DMF) to enhance resource recovery from municipal wastewater (MWW). A demonstration scale ultrafiltration plant equipped with industrial commercial membranes was operated for several months to evaluate its most suitable operating conditions. Different strategies were evaluated to mitigate membrane fouling, determining air sparging and classic backwashing as low effective methods when used in the DMF, suggesting they should be reduced to diminish operating expenses. Conversely, chemical-enhanced backwashing proved to be highly effective in mitigating fouling (membrane permeability recoveries of about 0.34 LMH bar^{-1} per g of NaOCl were obtained in this work), so that its use at low concentrations and high periodicities can be recommended. When employing a proper CEB protocol, filtration was possible for more than 45 days at low fouling rates (about 0.75 mbar day^{-1} when filtering at a transmembrane flux of 11.9 LMH), while a great permeate quality that meets EU discharge standards for non-sensitive environments was produced. An energy, carbon footprint and economy analysis of the system was performed to determine its viability after the application of the operating conditions obtained in this work. Optimistic results were achieved with net energy productions of about 0.47 kWh per m^3 of influent MWW, thanks to the particulated COD recovery. This represented net carbon footprint savings of about 0.19 kg CO_2-eq. per m^3 of influent MWW by increasing energy savings while avoiding the use of fossil fuels. Optimistic results were also obtained for the operating costs, with an estimated net profit of about €0.064–0.138 per m^3 of influent MWW. Unfortunately, membrane costs still represent the main bottleneck of this alternative, with high investment demands despite considering membrane acquisition costs only. However, thanks to the significant economic benefits achieved by this technology, membrane amortization could be greatly reduced, with an estimated payback period of between 2.6 – 5.6 years.

1. Introduction

Current economic models are widely criticized for being unsustainable for human development [1]. Indeed, the climate crisis, increasing water stress, and escalating energy and food crises are largely consequences of them. Because of these threats, a shift toward models based on the circular economy (CE) has become necessary [2], aiming to reduce the impact of human activity on the environment. Focusing on the water sector, municipal wastewater (MWW) has drastically changed its paradigm and is now identified as a source of essential resources, including reclaimed water, energy and nutrients (mainly nitrogen and phosphorus) [3]. Unfortunately, current facilities are unable to effectively recover all these wasted resources, requiring an update to implement CE fundamentals in this sector [4]. Current MWW management is primarily carried out by aerobic processes (*i.e.* conventional activated sludge (CAS)), which not only present significant drawbacks due to their high energy demands (up to 50 % of the total energy required for MWW treatment) and high sludge production [5], but also fail to recover all the potential energy contained in the influent organic matter (OM). Indeed, part of the incoming organics is degraded to CO_2 by aerobic bacteria, while the growing biomass is less biodegradable than raw MWW due to the increase in non-biodegradable compounds in

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https://doi.org/10.1016/j.jece.2024.114756
Received 25 July 2024; Received in revised form 6 November 2024; Accepted 10 November 2024
Available online 15 November 2024

the medium as microbes die and produce debris [6]. Given the considerable limitations and disadvantages of this technology, numerous researchers have proposed different alternatives to enhance MWW management (see for instance [4,7]). Among these options, the direct filtration of wastewater by membrane systems, commonly known in the literature as direct membrane filtration (DMF), is an attractive option due to all its potential benefits [8,9].

DMF consists of using a membrane system to filter raw MWW, generating a solids-free permeate rich in soluble nutrients and a concentrated sludge which can be valorized via anaerobic digestion (AD). The produced permeate is able to meet EU discharge standards for solids and OM [9,10], thus eliminating the need for aerobic treatments in the water line. This improves the system's energy savings by avoiding CAS's significant energy demands, while other indirect carbon footprint sources, such as fugitive N_2O emissions in conventional nitrification-denitrification process when nitrogen removal is required, can also be prevented [11,12]. This permeate could thus be used for fertigation when possible, directly recovering MWW's soluble nutrients content, or post-treated either by ion exchangers or osmosis membranes to concentrate the nutrients and produce commercial fertilizers [13]. Is important to highlight that wastewater treated by DMF (specifically UF) may not reach the removal ratios for micropollutants proposed in the revision of Directive 91/271. However, it seems clear that DMF will be able to reduce the concentration of, at least, some micropollutants such as pharmaceuticals [14] and some volatile organic compounds [15]. Moreover, it has proven very effective in removing antibiotic resistance genes [16] and microplastics [17]. The DMF can therefore help to reduce the intensity of quaternary treatments provided for in the new directive. The energy recovered is also significantly greater due to the higher amount of OM fed to the AD, significantly reducing MWW sludge production by replacing aerobic by anaerobic treatments. This alternative is especially attractive for small/medium installations in which AD is not possible due to low sludge production, thus boosting the resource recovery and favoring the decentralization of MWW treatment [8]. Fig. 1 shows a schematic diagram of MWW treatment after including DMF.

Despite all DMF's potential benefits, the severe membrane fouling when directly filtering MWW still needs to be addressed before considering this alternative as a potential competitor to other treatments [8].

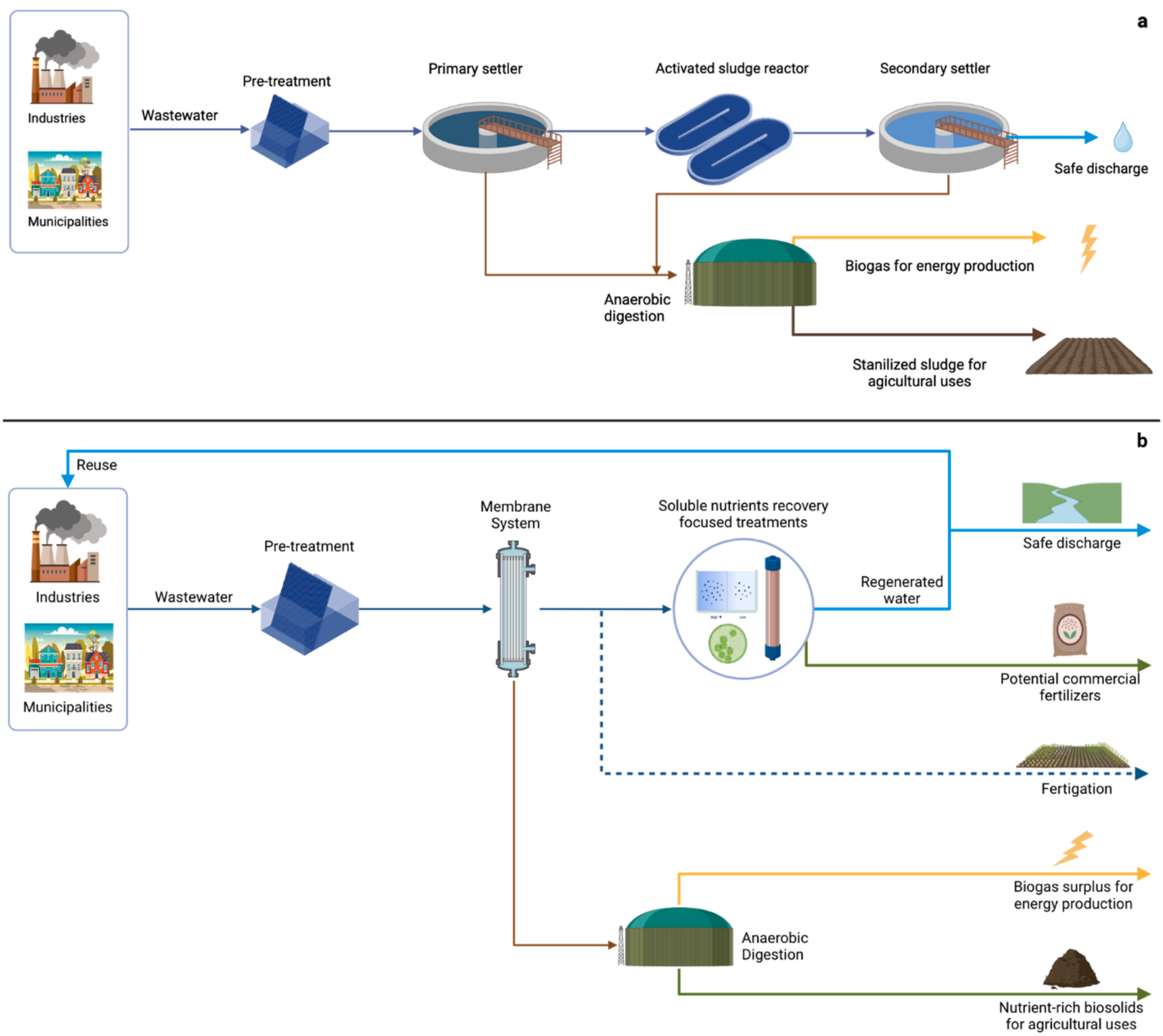

Fig. 1. Municipal wastewater treatment considering: (a) classic scheme and (b) DMF scheme.

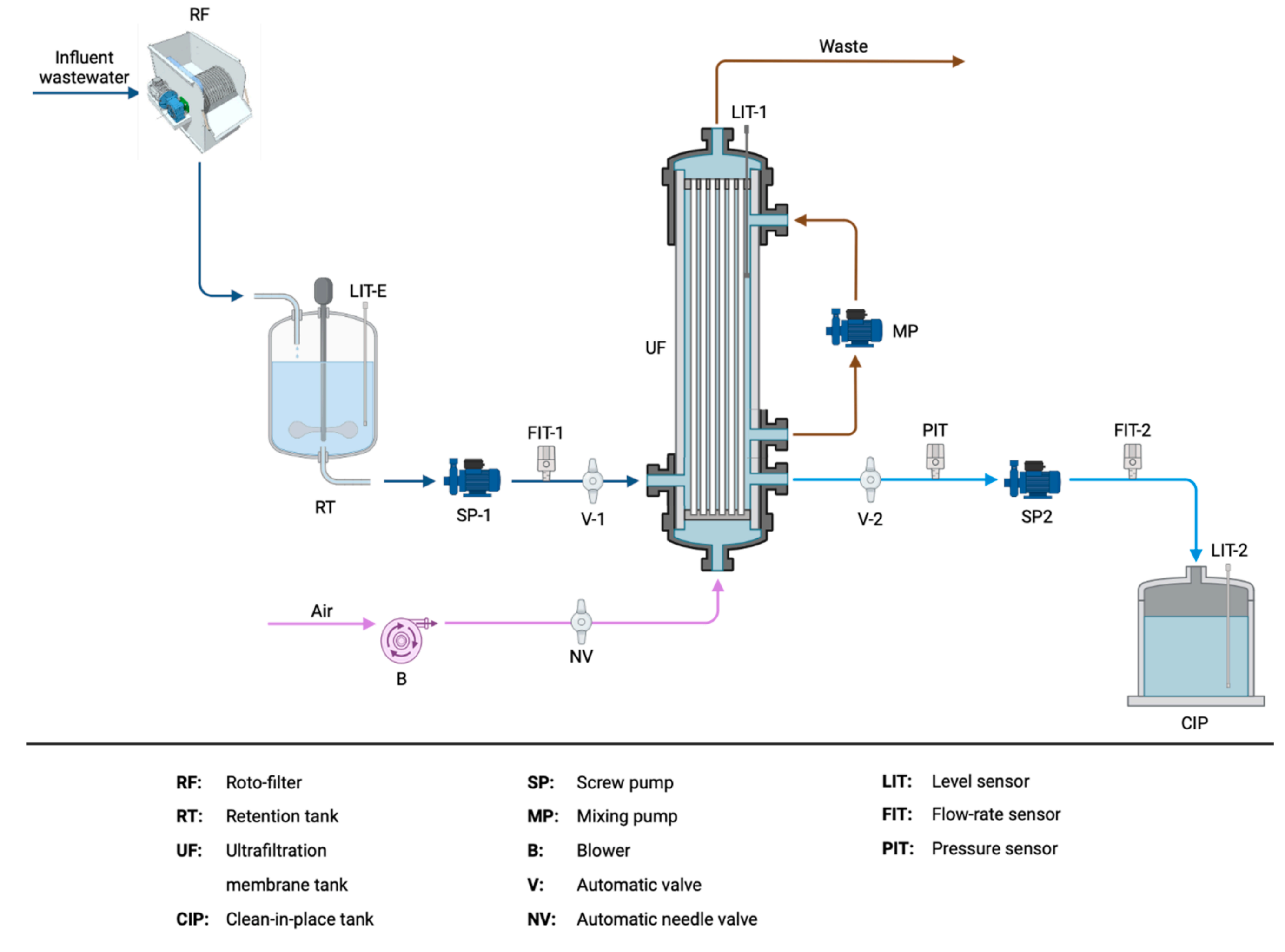

Fig. 2. Schematic diagram of the demo-scale DMF plant.

The application of moderate fluxes seems to be recommended to reduce fouling when directly filtering MWW, with irreversible membrane fouling sharply increasing as the treatment flux rises [10]. However, since membrane costs are still among the highest capital expenses in membrane-based systems [18], increasing the treatment flux to the maximum is essential to improve the viability of these alternatives from an economic point of view. Applying energy/cost-efficient fouling control strategies is thus key to achieving viable DMF treatments. Previous works indicated that fouling control strategies based on reversible fouling mitigation such as air sparging and backwashing do not significantly contribute to minimizing fouling [19]. Their application could even be a disadvantage, since removing deposited materials from the membrane (*i.e.* cake layer) may allow more soluble substances and colloids to reach the membrane surface, promoting irreversible fouling [10,19]. Indeed, irreversible fouling appears to control this kind of filtration, with OM identified as the main source, making up from 70 % to 90 % of all membrane fouling [10,20–22]. Other more specific fouling control strategies, such as chemical enhanced backwashing (CEB), *i.e.* the use of some chemicals mixed with water during periodical backwashing, could thus be greatly beneficial for DMF since the application of basic solutions could help to mitigate the organic fouling developed in the membrane. Previous research has studied the effectiveness of CEB during the DMF of MWW, reporting promising results [23,24]. Further studies on middle/long-term operations in demo-scale installations are thus required to confirm the results reported so far. The role of cake layer in preventing irreversible fouling when directly filtering MWW especially needs to be clarified to properly identify suitable fouling control strategies. The CEB protocol (intensity and periodicity) also needs to be properly evaluated to find the most efficient membrane cleaning at the lowest possible cost. Finally, the potential benefits of the proposed scheme, including energy savings, economic benefits and the impact on the carbon footprint need to be evaluated to properly identify the best operating conditions while recognizing the best scenarios for this technology.

The purpose of the present work was therefore: (1) to determine the effect of the cake layer on membrane fouling and optimize the reversible fouling control strategies applied in this study (air sparging and backwashing), (2) evaluate the effectiveness of CEB on irreversible fouling control and preliminarily optimize its application, and (3) assess the viability of the filtration process after applying the most suitable conditions determined in the previous two points. This feasibility of this alternative was evaluated from three different perspectives: the energy impact, economic impact and carbon footprint, in a fully-automated demo-scale plant equipped with commercially available equipment.

2. Materials and methods

2.1. Demo-scale filtration plant

The demo-scale plant consisted of a 567-L working volume membrane tank fitted with a commercially available industrial UF membrane module (PULSION® Koch Membrane Systems, 0.03-µm of pore size, 43.5 m^2 of filtration area). Filtration was conducted by vacuum by means of a screw pump (PCM M Series, EcoMoineau™), with the generated permeate collected in a 400-L working volume clean-in-place (CIP) tank to allow membrane backwashing when necessary. An additional screw pump (PCM M series, EcoMoineau™) was used to continually recirculate the contents of the membrane tank to ensure complete mixing, while air scouring was used to control the membrane cake layer (*i.e.* reversible fouling) by injecting air from below the membrane tank by means of a blower (G-BH7, Elmo Rietschle). The membrane tank was fed with the raw influent wastewater from the "Conca del Carraixet" wastewater treatment plant (WWTP) (Alboraya-Valencia, Spain) after classic pre-treatment (*i.e.* screening and sieving, desanding and degreasing). Supplementary screening of the influent with a 0.5 mm screen size roto-filter was carried out to protect the membrane module from large abrasive materials, storing the pre-treated influent in a 745-L working volume equalization tank to feed the membrane tank according to filtration requirements (hydraulic retention time of the equalization tank below 30 min). The stored fresh MWW was fed to the membrane module by means of another screw pump (PCM M Series, EcoMoineau™). Further information concerning the demo-scale plant and its operating protocol can be found in [19] while the characterization of the MWW fed to the membrane module is shown in Table S1 (Supplementary Material).

2.2. Operation and experimental plan

The demo-scale filtration plant was operated at a constant flux during all the experiments. The transmembrane pressure (TMP) was continually recorded to monitor the fouling occurring in the system. Filtration was carried out by consecutive filtration:relaxation (F:R) cycles, establishing a 180:40 s for each step in all the experiments. The demo-scale plant was operated with a pseudo-stationary TSS concentration in the membrane tank, modifying its value according to the experiment requirements. The concentrated sludge (waste stream) was continuously extracted from the membrane tank via overflow to ensure a stable TSS concentration, with the feed pump adjusted for that purpose. However, the membrane was operated with no waste production (*i.e.* infinite solids retention time (SRT)) at the beginning of each experiment to achieve the required TSS concentration in the least possible time and starting waste production on reaching the set-point. This infinite-SRT-operation lasted from between several hours to three days, according to the TSS, considering this filtration time as irrelevant to fouling due to its negligible duration (filtration experiments lasted for at least one month before chemical cleaning). Air sparging and permeate backwashing were used to control fouling during filtration, the former evaluated at three different intensities (specific air demand (SAD) of 0.21, 0.11 and 0.05 Nm^3 per m^2 of membrane area and h), while the latter was held at a flux of 10–5 L per m^2 of membrane area and h (LMH) for 60 s every 5, 20 or 80 F:R cycles, depending on the experiment (see Figs. 3 and 4).

Three blocks of experiments were carried out to: (1) determine the effect of the cake layer on membrane fouling and optimize the SAD and backwashing applied, (2) evaluate the effectiveness of CEB on irreversible fouling control and optimize its application, and (3) assess the viability of the filtration process in the middle/long-term after applying the most suitable conditions determined in the two former points. The first and second blocks of experiments were conducted at a significantly lower TSS concentration in the membrane tank (around 3.5 g L^{-1}) than that used in the last experiment (around 6.2 g L^{-1}) to promote the development of irreversible fouling and reduce the duration of the experiments. On the other hand, CEB cleaning was applied during the second block of experiments when membrane permeability fell below 60 LMH bar^{-1}, with the reagent, its concentration and its application time specified in Table 3. The CIP tank was used to prepare CEB cleaning according to the experimental demands, with its content substituted for tap water before adding the chemicals. Table 1 shows the membrane

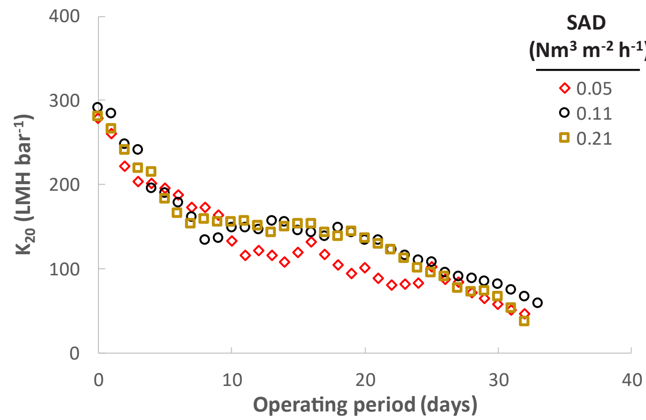

Fig. 3. Fouling evolution for the different SADs tested. Operating TSS concentration of about 3.5 g L^{-1} in all cases. A relatively constant value of about 5.7 min L g^{-1} and 146 mg DQO L^{-1} for the TTF and SMP concentration in the membrane tank sludge was determined in all the experiments.

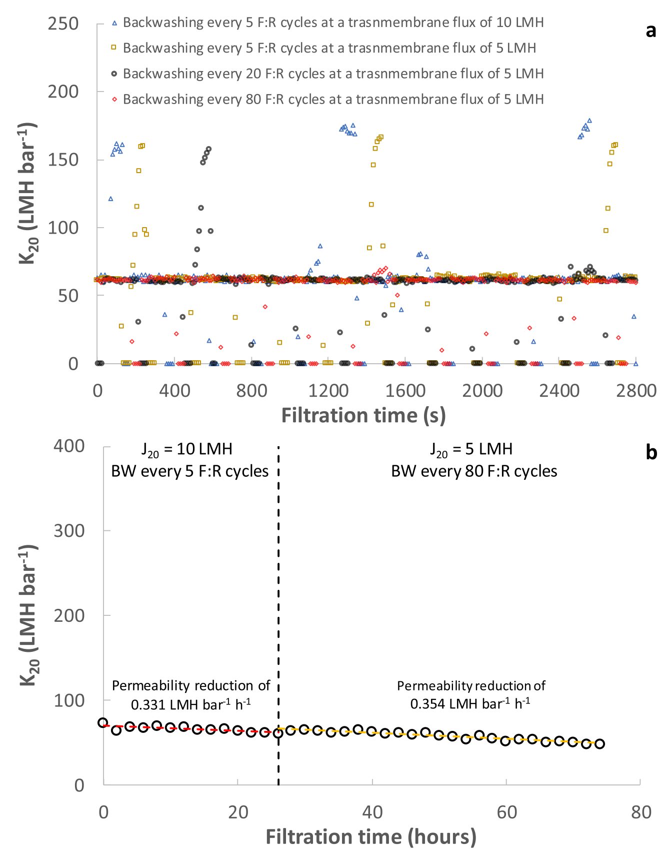

Fig. 4. Fouling evolution of the different backwashing protocols evaluated: (a) short-term and (b) middle-term. Operating TSS concentration and SAD of about 3.5 g L^{-1} and 0.05 Nm^3 m^{-2} h^{-1} in all cases. BW: backwashing.

Table 1
Operating conditions of each block of experiments.

Exp. block	Initial K_{20} (LMH bar^{-1})	J_{20} (LMH)	HRT (h)	SRT (d)	T (°C)	pH
1	293	12.1	1.08	0.53	15.9	7.6
2	304	12.3	1.06	0.52	19.2	7.5
3	312	11.9	1.10	0.95	20.6	7.8

Table 2
Permeate quality and sludge filterability in the first battery of experiments.

SAD ($Nm^3\ m^{-2}\ h^{-1}$)	TS		SMP		TTF
	($mg\ L^{-1}$)	(%)*	($mg\ DQO\ L^{-1}$)	(%)*	($min\ L\ g^{-1}$)
0.21	1221	17.6	64	15.9	6.63
0.11	1185	20.1	61	19.8	4.98
0.05	1203	18.8	66	13.3	5.42

* Percentage of TS and SMP captured by the used membrane.

Table 3
Membrane permeability recovery by CEB.

Exp.	Reagent dosed	Reagent concentration ($mg\ L^{-1}$)*	pH of the solution **	CEB time (min)	Recovered permeability ($LMH\ bar^{-1}$)
CEB−1.1	NaOCl	30	9.53	8	2.70
CEB−1.2		50	9.64		3.51
CEB−1.3		100	9.79		8.10
CEB−1.4		200	9.94		16.62
CEB−1.5		300	10.03		34.70
CEB−1.6		400	10.09		44.22
CEB−2.1	NaOCl + NaOH	50 + 398	12.00	8	23.21
CEB−2.2	NaOCl + NaOH	100 + 397			30.93
CEB−2.3	NaOCl + NaOH	200 + 396			34.02
CEB−3	NaOH	400	12.00	8	16.03
CEB−4	Citric acid	400	2.42	8	6.01
CEB−5.1	NaOCl + NaOH	50 + 398	12.00	12	20.43
CEB−5.2	NaOCl + NaOH			25	24.87

* Final concentration of each reagent reached in the membrane tank after the CEB.
** pH level measured in the CIP tank.

permeability at the start of each experiment, the transmembrane flux applied, the average temperature and pH, and the hydraulic and solids retention times (HRT and SRT) during each block of experiments. The presence of biological activity was not directly evaluated in the membrane tank, although due to the short SRTs used, it was assumed that no significant microorganism growth occurred. In fact, other studies determined that SRTs above 3 days are required to have a significant influence of the aerobic biomass on the OM recovered [10], while SRTs under 1 day were used in the present work (see Table 1).

2.3. Analytical methods and calculations

The influent MWW, membrane tank content and produced permeate were sampled twice a week to evaluate the system's solids and OM capture. Total and suspended solids (TS and TSS), total and soluble chemical oxygen demand (COD and SCOD), total nitrogen (TN) and total phosphorous (TP) concentrations were determined according to standard methods [25]. 0.45-µm pore size glass fiber membrane filters (Millipore, Merck) were used to obtain the soluble fraction of the collected samples. The soluble microbial products (SMP) concentration and the filterability of the membrane tank bulk were also determined once a week to ensure their invariability in the first and last block of experiments. Protein and carbohydrate concentrations were assumed as the major SMP contributors, determining their concentrations by a commercial total Protein Kit (Micro Lowry Peterson's Modification, Sigma-Aldrich) and the Dubois method [26], respectively. Sludge filterability was determined by the time-to-filter (TTF) method according to the protocol stablished in the standard methods [25].

The 20ºC-normalized membrane permeability (K_{20}) was calculated considering the temperature-normalized flux (J_{20}) and daily TMP average value (TMP_{ave}) recorded during filtration. The protocol used to calculate these variables can be found in [10,27]. Since continuous air scouring and periodic backwashing were used as fouling control strategies, in this work all the average K_{20} unrecoverable reductions after backwashing were considered as irreversible fouling. Similarly, the effectiveness of CEB was determined based on the recovery of membrane permeability after its application, calculated as the difference between the average K_{20} of three filtration cycles before and after the CEB (see Fig. S1 in Supplementary Material). Energy, economic and environmental balances were performed for the last experiment in order to determine the feasibility of the proposed system, considering energy recovery, capital and operating costs (CAPEX and OPEX) and carbon footprint. The protocol used to perform these analyses can also be found in [10]. The energy cost was estimated at €0.20 – 0.35 per kWh, based on current Spanish high-voltage electricity rates [18,28], while membrane acquisition was estimated as €30 per m^2 of membrane area, according to other studies using similar UF membranes [10,18] and data provided by the supplier (Koch Membrane Systems). Membrane lifespan was estimated by cumulative tolerance to basic (NaOCl) membrane cleaning. This study considered a maximum NaOCl exposure concentration of 50000 ppm h^{-1}, in line with data provided by the supplier (Koch Membrane Systems). The cost of chemicals was based on their market prices and data from other studies: €0.72 – 1.10 per L of NaOCl at a volumetric concentration of 15 %, €0.75 – 5.20 per kg of citric acid and €2.82 per kg of NaOH [18,29]. The fouling growth rate obtained in the last experiment was used to estimate membrane cleaning requirements (CEB and classic chemical cleanings) and membrane lifespan of the alternative. However, to obtain more realistic projections, a minimum membrane cleaning period of 8–12 months and a maximum membrane lifespan of 20 years were considered. A global warming potential (GWP) of 0.36 kg CO_2-eq per kWh of consumed energy was considered to calculate the carbon footprint based on the emissions ratio expressed in the EcoInvent database [30]. Finally, a volumetric flow rate of 30,000 $m^3\ d^{-1}$ of influent MWW was used as the basis for the system's CAPEX calculation. This value is similar to the influent flow rate of the "Conca del Carraixet" WWTP and also aligns with the values applied in other studies on CAS [31] and DMF [10], facilitating cost comparisons.

3. Results and discussion

3.1. Effect of the air sparging and permeate backwashing on fouling control

Fig. 3 shows the fouling growth rate obtained under the three increasing SADs used. No significant fouling mitigations were observed, despite increasing the air sparging intensity fourfold, barely identifying a slight filtration impoverishment during daily filtration when applying low SADs due to the increased reversible fouling. This was due to the accumulation of more material on the membrane surface, which promoted thicker cake layers and increased filtration resistance. However, this effect was not relevant to the development of irreversible fouling in the middle/long-term. Increasing the SAD was thus found inadequate for improving the DMF of MWW, since air sparging is an energy-intensive fouling control strategy [32,33], increasing filtration OPEX without any meaningful improvement in filtration or membrane lifespan. The use of low air sparging intensities [19], air pulsations (*i.e.* intermittent air injections) [34], air backflushing [35,36] or other non-air-related alternative methods, such as membrane vibrations [20] or rotations [37,38], could thus be suggested as better options to reduce the energy demands for reversible fouling control, since these techniques are much less energy-demanding than continuous air sparging [8]. However, it is important to highlight that these experiments were performed at a significantly lower TSS concentration than that recommended to prevent irreversible fouling (about 3.5 g L^{-1} in this case compared to the 6–10 g L^{-1} suggested in a former work [10]), aiming to reduce the experimental period to manageable time laps. Since reversible fouling could be a more serious problem when filtering more concentrated sludge or when treating other influents, the reversible fouling control strategies used should be adjusted depending on the case requirements.

Since cake layer thickness was found to be irrelevant to the evolution of middle/long-term fouling on the membrane, the effectiveness of classic permeate backwashing was also evaluated. Backwashing intensity and periodicity showed insignificant effects on both reversible (see Fig. 4a) and irreversible (see Fig. 4b) fouling control, obtaining similar profiles during short-term filtration. This was especially relevant, since these results were obtained when applying the lowest SAD tested in this work (*i.e.* 0.05 Nm^3 per m^2 and h). These results suggest that, in fact, the accumulation of material on the membrane did not play a dominant role. It is therefore suggested to reduce backwashing flux and periodicity as much as possible to enhance the water's treatment productivity while reducing the membrane area required for the task and thereby its CAPEX. In this case, moving from an F:R ratio of 5 at a backwashing flux of 10 LMH to a F:R ratio of 20 at a backwashing flux of 5 LMH represented an increase of the percentage daily filtration ratio of 0.75–0.81 %. This means increasing daily wastewater treatment by 655 m^3 per day, which significantly contributed to reducing the total membrane area demands of the system. However, as mentioned above, it is important to note that the effect of backwashing on reversible fouling was studied at relatively low solids concentrations (about 3.5 g L^{-1}), while irreversible fouling was only evaluated in the short-term (2 days). Better backwashing performance could be expected by increasing sludge concentration and in turn the amount of material forming the cake layer, while other long-term phenomena, such as the possible compressibility of the cake layer during continuous filtration, should also be considered when adjusting backwashing to suitable values. In fact, the results obtained in these experiments were used to identify the proper operating range conditions to apply in middle/long-term evaluations (see Section 3.3), not representing optimum values by themselves.

3.2. Effect of the cake layer on irreversible fouling mitigation

The results given in Section 3.1 seem to indicate that irreversible fouling during direct MWW filtration does not related to the cake layer formed onto the membrane surface. Indeed, irreversible fouling was not found to be influenced by changes in air sparging or backwashing intensity and periodicity, which strongly influence the amount of material reaching the membrane surface and the cake layer thickness. This would be in disagreement with other works' hypothesis which pointed to a potential beneficial effect of forming consistent cake layers during filtration to mitigate irreversible fouling [10,19,39–41]. This hypothesis is based on the possibility that thick cake layers prevent contact between the membrane surface and highly fouling pollutants such as colloids and/or soluble sticky substances (*e.g.* SMP). This may reduce major pore blocking or gel layer formation during filtration. However, since no meaningful changes in the irreversible fouling rate were observed in this work, despite altering the cake layer, its role in mitigating irreversible fouling could be considered as irrelevant in this case. These results were in line with the almost unaltered concentration of TS and SMP obtained in the permeate, regardless of the SAD employed, showing that the cake layer formed did not significantly improve the membrane retention capacity of these pollutants (see Table 2). A retention percentage of 15 – 20 % was obtained for both TS and SMP, showing that the membrane had a certain capacity to retain soluble organic compounds, as has been reported in other studies [10,42]. Neither did the TTF show a conclusive correlation. The slight sludge filterability changes observed were probably related to influent alterations rather than to the operating SAD (see Table 2). It can thus be assumed that sludge filterability did not significantly influence the obtained results. All this seems to indicate that, in fact, no sensible increment in the capture of colloids or other soluble sticky substances occurred, regardless of the cake layer. The relatively low operating TSS concentration (about 3.5 g L^{-1}) used in these experiments could of course have influenced the cake layer's capacity to prevent fouling, thus requiring higher solids concentrations to rule out this hypothesis. However, the results of Sanchis-Perucho et al. [10,19] suggest otherwise, since improvements in irreversible fouling were evident even at relatively low TSS concentrations (around 1 and 2.5 g L^{-1}), despite using similar SAD intensities and backwashing protocols to those used in this study at lower transmembrane fluxes (around 10 LMH). This seems to indicate that in fact the cake layer formed during filtration does not play a major role in controlling irreversible fouling. Indeed, given the evolution of permeability during each filtration cycle (see Fig. 4), there could not even be confirmed that an effective cake layer was formed under the conditions applied in these experiments. Therefore, other mechanisms seem to be involved in the irreversible fouling mitigations observed as raising the operating TSS concentration.

Another possible benefit of increasing the operating TSS concentration for mitigating irreversible fouling could be the capture of colloids and soluble substances by spontaneous flocculation. In this scenario, increasing the TSS concentration in the membrane tank would raise the interactions among particles in the bulk, promoting more collisions and potential additions between them, thus reducing the amount of free low-size particles in the bulk. However, when evaluating the particle size distribution of both, influent raw MWW and membrane tank bulk, an insignificant amount of small-size particles was found (*i.e.* smaller than the membrane pore size, see Sanchis-Perucho *et al.* [19]). This mechanism may thus be considered irrelevant in preventing pore narrowing and blocking, since no potential pollutants were found in the influent MWW in the first place. Results of a previous study [10] that found no significant differences in the permeate's SMP despite increasing the TSS concentration in the membrane tank from about 1–11 g L^{-1} are in line with this conclusion. Also, no meaningful differences in the permeate's TS or SMP were either observed in this study when increasing the TSS concentration in the membrane tank to about 6.2 g L^{-1} (see Table 4 in Section 3.4), also corroborating the aforementioned conclusion.

Despite all the above, it is important to highlight that mechanisms controlling fouling could be extremely complex and changing, especially when considering the wide diversity of pollutants in MWW [8]. Several additional matters can thus be suggested to complement this work and unveil the effects of cake layer thickness and TSS in the membrane tank on DMF irreversible fouling mitigation. TS and SMP concentrations in the permeate were used in this work for trying to elucidate potential enhancements on these pollutants retention capacity as altering the before mentioned variables. However, since small accumulations of colloids or other substances on the membrane may cause a considerable difference in the fouling growth rate, especially as the membrane is reaching high TMPs, this approach alone may not be enough to assess their impact. Indeed, the differences in mass balance may be too small to detect correlations with the permeate concentrations, while materials that could have been displaced on the membrane surface or pores, contributing to fouling, may be in a similar amount the ones captured in the cake layer when increasing its thickness, thus not displaying differences in the amount of these pollutants reaching the permeate but on the place where there are stocked. On the other hand, the particle size

Table 4
Permeate and sludge characteristics in the middle/long-term filtration.

Parameter	Stream	
	Waste	Permeate
TS (mg L^{-1})	7341	1228
TSS (mg L^{-1})	6180	-
DQO (mg DQO L^{-1})	9364	65
SMP (mg DQO L^{-1})	159	59
TN (mg N L^{-1})	434	47
TP (mg P L^{-1})	41.8	4.6
TTF (min L g^{-1})	6.02	-

distribution determination is not exempt from uncertainty. The laser diffraction method is limited when working with non-homogeneous materials since some of the solids characteristics, such as their shape and texture, significantly affect the output [43,44]. Thus, given the large diversity of materials, textures, shapes and sizes that can be expected in untreated MWWs the results provided by this technique may show a certain degree of inaccuracy. This technique has also given inaccurate results when working with fine particles due to their marked tendency to form agglomerations [44]. This self-agglomeration effect, often produced by electrical interactions between extremely small particles, may be even greater when filtering raw MWW due to the significant amount of sticky soluble components in these waters (see for instance Table S1 where is presented the SMP content in the influent raw MMW treated in this study). The laser diffraction method may therefore not be a suitable technique to determine the amount of colloids in raw MMW.

Further research is required to properly elucidate the mechanisms of the beneficial effects on irreversible fouling of increasing the TSS concentration in the bulk. Alternative methods to determine the particle size distribution such as X-ray CT-scanning are suggested to mitigate possible uncertainties due to fine particles agglomerations [44]. Indeed, colloids seem to be the main fouling promotors when directly filtering MWW, which would be in line with the good fouling mitigations observed when dosing coagulants [35,45,46]. Other mechanisms also need to be explored to understand the potential particle interactions and their impact on fouling. Lab-scale experiments with destructive methods, such as assessing the cake layer structure, porosity, and compressibility by SEM microscopy or determining the amount and nature of the mass attached to the membrane could be particularly useful to cover this aspect.

3.3. CEB effectiveness and preliminary optimization

Table 3 shows the membrane permeability recovery after each CEB applied. Exp. from CEB-1.1–1.6 were focused on determining the effect of increasing the punctual NaOCl concentration in the membrane tank (from 30 to 400 mg L^{-1}). The application of NaOCl was highly effective in recovering membrane permeability, achieving recoveries from 2.70 to 44.22 LMH bar^{-1} as increasing the applied NaOCl concentration (see Table 3). This was as expected since OM is usually the main fouling promotor during the DMF of MWW [10,20–22], with basic solutions, especially NaOCl, largely effective in removing those. A linear correlation was obtained between permeability recovery and raising NaOCl concentration during CEB when only NaOCl was used (see Fig. S2 in the Supplementary Material), indicating that it does not lose effectiveness at the relatively high concentrations evaluated in this study, despite the short reagent/membrane contact time used (about 8 min). Since a similar permeability recovery could be obtained regardless of the CEB concentration (around 0.34 LMH bar^{-1} per g of NaOCl), the reagent dose and periodicity could be adjusted according to operation necessities (*i.e.* fouling growth rate, reclaimed water productivity, etc.) or based in other demands.

Since NaOCl gave good results in recovering membrane permeability, exp. from CEB-2.1–2.3 were proposed to improve the performance of this chemical. As recommended by the membrane supplier, classic membrane cleaning can be improved by raising the pH of the feed solution to 12. Consequently, the pH of the CEB solution prepared in the CIP tank was increased in this set of experiments with NaOH to this pH value, with the same CEB protocol as before (see Table 3). Significant improvements were obtained in recovering membrane permeability thanks to the NaOH addition, increasing the CEB effectiveness by 6.6, 3.8 and 2.1-fold when adding 50, 100 and 200 mg L^{-1} of NaOCl, respectively (see Table 3). Due to the good results obtained by adding NaOH to the CEB solution, a CEB with this reagent only (exp. CEB-3) was performed to determine the isolated effects of the pH on membrane cleaning, in this case achieving a membrane permeability recovery of 16.03 LMH bar^{-1}. Likewise other studies that evaluated CEB effectiveness in DMF [24], cleaning with NaOCl gave a significantly better performance in controlling fouling than NaOH, achieving in this second case around 0.15 LMH bar^{-1} per g of NaOH used, about 2.3-fold lower than that obtained by NaOCl. Thus, although NaOH has a slightly lower market price than NaOCl, the latter was determined as a better option for CEB due to its higher effectiveness. Certainly, when considering the cost of each permeability unit recovered by each reagent, CEB with NaOH represents around €0.020 per LMH bar^{-1} recovered while NaOCl reduces the cost to about €0.018 – 0.012 per LMH bar^{-1} recovered, according to the results obtained in this study. Furthermore, NaOCl is also a more effective disinfectant than NaOH [24] which is as well an important benefit in DMF. Indeed, ensuring the inhibition of aerobic bacteria in the membrane tank improves energy savings by recovering all the OM while any pathogen reaching the permeate could also be removed by this reagent thereby enhancing the quality of the recycled water. Also, since pH levels above 12 are not recommended when operating PDVF membranes [47], the isolated use of NaOH during CEB would be extremely limited. Consequently, the combined application of NaOCl with NaOH was determined as the most suitable option. As for the chemicals concentration, it was appreciated a certain degree of non-linearity when the NaOCl concentration was raised during the 12-pH CEB experiments (see Fig. S2 in Supplementary Material), which, although slight, could indicate insufficient contact time between reagents and membrane. Since shorter backwashing times are preferred to raise as much as possible the water's production, CEBs with low reagents concentrations are indicated. It is also important to highlight that more periodical CEBs could also be used to prevent irreversible fouling, as they not only recover permeability but also play an important role in preventing material deposits on the membrane before they can be detected. Further studies on the optimal CEB concentration and periodicity will be performed to find the best conditions considering all involved costs (*i.e.* reagents acquisition, membrane replacements, etc.) and environmental impact.

The effectiveness of citric acid in CEB was also evaluated (exp. CEB 4), since inorganic fouling may also play a relevant role in the DMF of MWW [21,22,24]. However, a low effectiveness was observed in this work (see Table 3), achieving a permeability recovery of only 0.06 LMH bar^{-1} per g of citric acid used. These results confirmed the reduced influence of inorganic fouling in the DMF of MWW, in line with other studies that have also identified basic solutions as more effective in mitigating fouling compared to acidic ones [21,24]. Finally, to test the possible drop in reagent effectiveness due to the relatively short contact time with the membrane (about 8 min), two last experiments (exp. CEB-5.1 and 5.2) were conducted with longer backwashing times. Exp. CEB-2.1 was used to compare the contact time influence on the permeability recovery, achieving in all cases a final NaOCl concentration of 50 mg L^{-1} in the membrane tank. Despite increasing the contact time by about 1.5 and 3.1-fold in CEB-5.1 and 5.2 respectively, no meaningful increments in the permeability recovery were observed in these experiments (see Table 3) and thus it was concluded that the contact time established in this battery of essays was at least high enough to achieve effective cleaning at relatively low NaOCl concentrations.

Periodical CEBs (once or twice weekly) with low reagent concentrations (about 25–50 mg L^{-1} of NaOCl) at a pH of 12 were proposed to control irreversible membrane fouling from results obtained in this study. Furthermore, punctual citric acid CEBs (once every one or two months) could also be recommended to prevent inorganic fouling.

3.4. Middle/long-term operation and process feasibility

After determining the most suitable conditions to operate the demo-scale plant from those evaluated in this work, a last experiment was proposed to assess filtration performance and estimate the process viability. J_{20} was set at an average value of 11.9 LMH for its complete duration, while the operating TSS concentration was increased to

6.2 g L^{-1} to reduce irreversible fouling, as proposed in previous studies [10,19]. Air sparging and backwashing periodicity and intensity were set at 0.06 Nm^3 per h and m^2 and one in every 20 F:R cycles at a transmembrane flux of 5 LMH, respectively, to reduce the operating costs. Finally, the CEB protocol consisted of conducting an 8 min backwashing at a transmembrane flux of 5 LMH with 50 mg L^{-1} of NaOCl at pH 12 with NaOH once a week.

As can be seen in Fig. 5, filtration was possible for more than 45 days without serious irreversible fouling (about 0.75 mbar day^{-1}). The first reduction in membrane permeability occurred in the first 3 days, which consisted of the concentration period (*i.e.* increase of TSS concentration in the membrane tank to 6.2 g L^{-1}). However, since permeability was relatively stable after this first period, this reduction was associated with higher filtration resistance appearing as the TSS concentration was raised in the bulk, which did not affect fouling promotion. The fouling control protocol applied thus performed well in mitigating irreversible fouling. Indeed, according to the determined fouling rate, continuous filtration would be possible for more than 1 year without stopping the process for classic chemical cleaning. A high permeate quality was obtained thanks to UF, meeting the EU standards for non-sensitive environments, while the concentrated sludge, with about 9.4 gCOD L^{-1}, could be directly fed to AD to improve biogas production. Both effluents' characteristics can be found in Table 4. As in the first experiments (see Table 2), no sensible reductions in the permeate TS or SMP concentrations was observed in this last experiment either (see Table 4) despite increasing the TSS of the membrane bulk. Thus, as mentioned above, neither the cake layer nor the operating TSS concentration seems to affect permeate quality, which is controlled primarily by the membrane characteristics (mainly its pore size). A significant increase of the SMP concentration in the membrane tank over influent concentration was also found during filtration, which could be related to a slight microorganism activity. However, due to the short SRTs used in this work (under 1 day), their development was considered negligible. This was also supported by the TSS balance, which showed a proper closure (*i.e.* similar values for the influent and effluent mass fluxes), as can be seen in Table S2 in the Supplementary Material. The higher SMP observed in the membrane sludge was thus entirely attributed to the membrane's capacity to capture these compounds [10,42].

Regarding process viability, the DMF demonstrated remarkably lower energy demands and left a smaller carbon footprint compared to traditional MWW treatments, similar to findings reported in studies on AnMBRs (see Table 5). This highlights the great potential of this technology for upgrading MWW treatment, with the ability to compete in this aspect even against other notable emerging technologies. In the specific case of this study, the filtration energy requirements were significantly reduced thanks to lower air sparging needs. Indeed, when comparing the results achieved in this study with those obtained in a

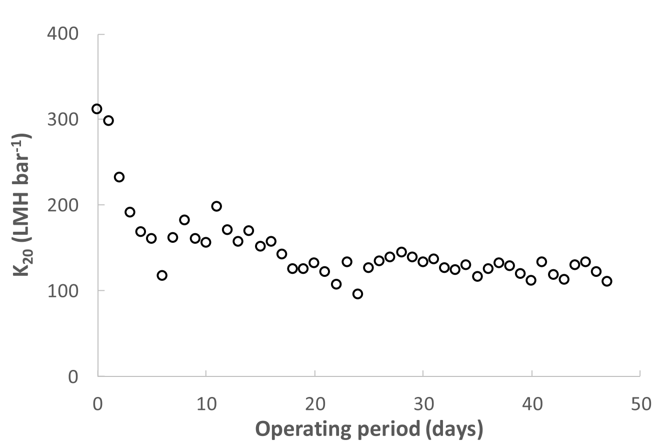

Fig. 5. Filtration evolution after applying the most suitable conditions established in Section 3.3.

Table 5
Energy demand and carbon footprint of different technologies for MWW treatment.

Treatment system	Energy demands (kWh m^{-3})	Carbon footprint (kg CO_2-eq m^{-3})*	Reference
DMF at demo-scale	0.07	0.025	This study
DMF at pilot/demo-scale	0.09 – 0.19	0.038 – 0.068	[10,48]
AnMBR at pilot/demo-scale	0.07 – 0.27	0.025 – 0.097	[49,50]
Conventional activated sludge	0.30 – 0.60	0.108 – 0.216	[51,52]
Extended aeration	0.34 – 0.82	0.122 – 0.295	[53]
Aerobic MBR	0.50 – 1.00	0.180 – 0.360	[54,55]

* Calculated from reported energy demands.

former work [10], where a J_{20} of 10 LMH and a SAD of 0.1 Nm^3 m^{-2} h^{-1} were employed, the energy balance was substantially improved, achieving in this case net energy productions of about 0.47 kWh per m^3 of influent MWW when considering the methane production by AD of all the recovered COD (see Fig. 6a). This also contributed positively to the carbon footprint of this technology, achieving a net CO_2 reduction of 0.19 kg CO_2-eq. per m^3 of influent MWW thanks to avoiding the usage of fossil fuels. In both cases, better results than those reported in the aforementioned work [10] were obtained (see Fig. 6a), showing that there is still room for highly improving the feasibility of this alternative. As previously mentioned, the use of low-energy fouling control methods, such as membrane vibration or rotation could be suggested to avoid air scouring. The system's OPEX was also improved by the operating conditions established in this work compared to the aforementioned one [10]. Although the CEB protocol raised the operating expenses due to higher chemical use for filtration, this was balanced by the lower membrane replacement demands and the higher energy recoveries thanks to reducing other fouling control strategies demands (*i.e.* air sparging and backwashing) (see Fig. 6b). Certainly, a potential economic benefit of around €0.064–0.138 per m^3 of influent MWW was obtained in this case, with the energy price completely controlling the potential benefits of this technology, they higher as the energy price increases. Besides, a significant improvement concerning this alternative's CAPEX was also achieved in this study compared to the above cited work [10] thanks to increasing the operating J_{20} and reducing backwashing flux and periodicity (see Fig. 6c). Considering the M€25.6 of a complete CAS CAPEX (equipment + direct and indirect construction costs) at a same treatment flow of 30,000 m^3 per day [31], the membrane acquisition cost alone would amount to about 15 %. Although this is still a significant amount, it represents a considerable improvement when factoring in the OPEX enhancement achieved in this work, resulting in payback periods of approximately 2.6 – 5.6 years when considering only the membrane acquisition cost, which could begin to be regarded as competitive.

These results demonstrate the significant potential of DMF for upgrading current MWW facilities, offering notable energy, carbon footprint, and economic benefits, while still leaving considerable room for improvement. Based on the results obtained in this study, implementing low-energy physical fouling control methods could represent a significant enhancement of the process viability, as it reduces the OPEX without significantly compromising fouling control. Increasing the operating flux even further could also entail meaningful improvements of the CAPEX, while the higher OPEX due to the greater fouling promotion could be palliated by increasing CEB periodicity and/or intensity. Further studies are required to continue enhancing the feasibility of this technology also considering life cycle cost (LCC) and life cycle assessment (LCA) of the process to properly determine the suitability of this alternative and best scenarios for its application.

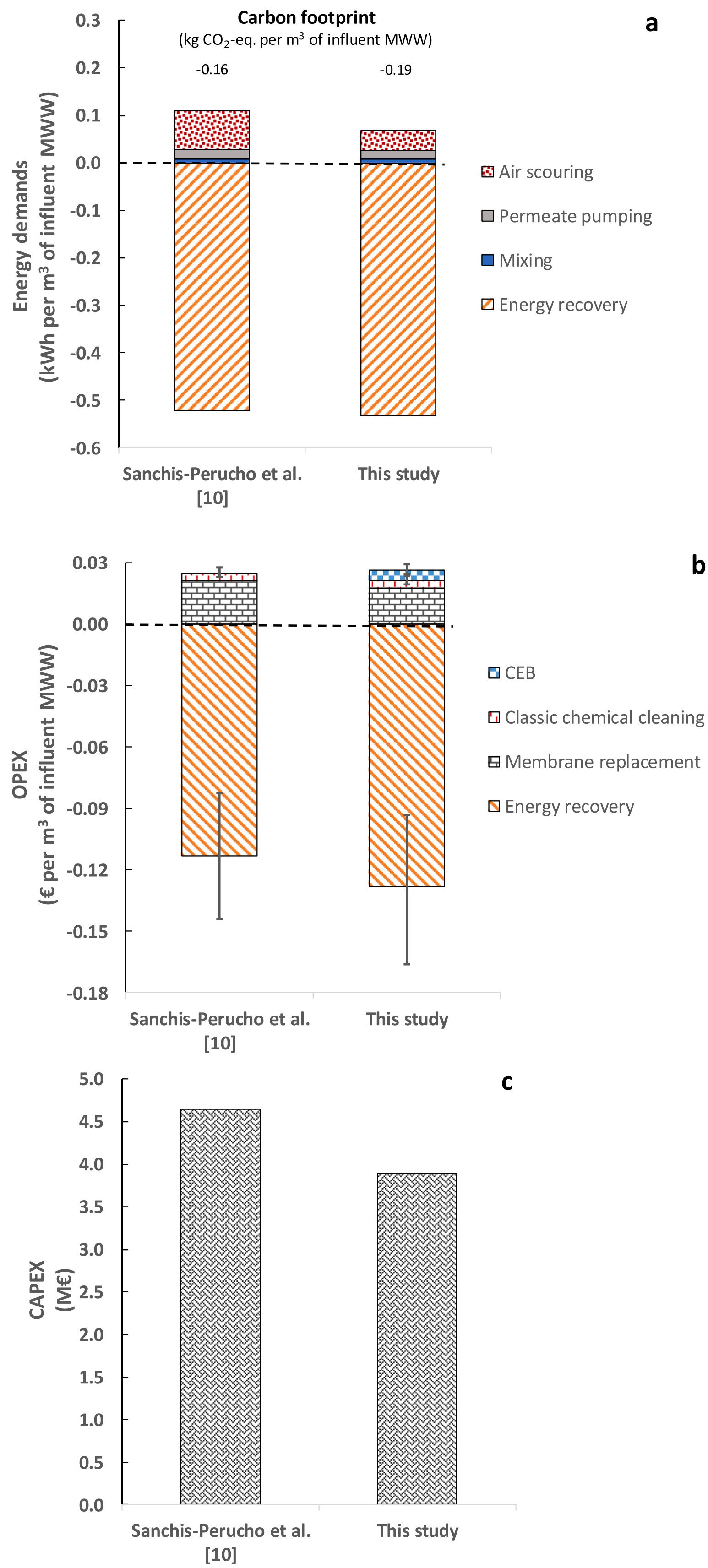

Fig. 6. Improvement of DMF feasibility after implementing the conditions established in Section 3.3: (a) energy and carbon footprint, (b) OPEX and (c) CAPEX. Note that data from Sanchis-Perucho et al. [10] has been updated with the costs considered in this work to allow for a proper comparison.

4. Conclusions

This work proves the great potential of DMF for enhancing resource recovery during MWW treatment while also establishing proper operating conditions to boost its feasibility. The main findings were as follow:

- o Air sparging and classic backwashing did not significantly impact reversible or irreversible fouling mitigation during the DMF of MWW. Minimizing their use would represent significant benefits during filtration, since air sparging is an energy-intensive method and could be substituted by other low-energy alternatives.
- o Cake layer does not seem to contribute to mitigating irreversible fouling in the meddle/long-term, nor does it affect permeate quality. Since a significant reduction in irreversible fouling is observed with increasing TSS concentration in the bulk, further research is needed to uncover the fouling mechanisms involved in the DMF of MWW and to propose appropriate countermeasures.
- o The CEB gave good results in controlling fouling, determining basic solutions as the best option in this case. Periodical CEBs at low NaOCl concentrations (about 50 mg L^{-1} reached in the membrane tank) and a pH of 12 with NaOH was determined as the most suitable option.
- o Filtration was possible for more than 45 days with a reduced irreversible fouling growth rate of about 0.75 mbar day^{-1} thanks to periodical CEBs. Continuous filtration could be sustained for more than a year without stopping the process for classic chemical cleaning, while achieving a high-quality permeate that meets EU standards for non-sensitive environments.
- o Good results were obtained for the viability of the process, achieving net energy productions of about 0.47 kWh per m^3 of influent MWW thanks to recovering all the particulate COD in the membrane module. This would represent a net carbon footprint saving of about 0.19 kg CO_2-eq. per m^3 of influent MWW thanks to boosting the energy recovery in MWW treatment while avoiding the use of fossil fuels. The OPEX also improved, estimating net profits of about €0.064–0.138 per m^3 of influent MWW. Unfortunately, membrane costs are still the main bottleneck of this alternative, with a high investment despite only considering the membrane acquisition. However, this can be balanced by the OPEX improvements, estimating in this work payback periods for the membranes amortization ranging between 2.6 – 5.6 years.

CRediT authorship contribution statement

Ángela Roig-Ferrer: Writing – review & editing, Investigation, Data curation. **Pau Sanchis-Perucho:** Writing – original draft, Supervision, Methodology, Data curation, Conceptualization. **Luís Borrás:** Writing – review & editing, Supervision, Resources, Project administration, Funding acquisition. **Laura García-Gabarda:** Writing – review & editing, Investigation, Data curation.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Acknowledgements

This research work was supported by the Spanish Ministerio de Economia, Industria y Competitividad via Fellowship PRE2018-083726. It was also made possible by the finance received from this Ministry during the Project "Aplicación de la tecnología de membranas para potenciar la transformación de las EDAR actuales en estaciones de recuperación de recursos." (CTM2017-86751-C1 and CTM2017-86751-C2). The authors are also grateful to the EPSAR (Entidad Pública de Saneamiento de Aguas de la Comunitat Valenciana) for its support to this work.

Appendix A. Supporting information

Supplementary data associated with this article can be found in the online version at doi:10.1016/j.jece.2024.114756.

Data Availability

Data will be made available on request.

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