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Wastewater treatment by membrane ultrafiltration enhanced with ultrasound: Effect of membrane flux and ultrasonic frequency

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Highlights

•Ultrasonic enhanced membrane ultrafiltration at two fluxes and two US frequencies.
•Enhancement of the treatment performance at the lowest US frequency of 35 kHz.
•US are more effective in fouling rate reduction at higher membrane flux (150 L/m^2 h).
•Higher OM and turbidity removals were obtained at 130 kHz.

Abstract

Membrane ultrafiltration is increasingly applied for wastewater treatment and reuse, even though membrane fouling still represents one of the main drawbacks of this technology. In the last years, innovative strategies for membrane fouling control have been developed, such as the combination of membrane processes with ultrasound (US). In present work, the application of membrane ultrafiltration and its combination with US were studied, evaluating the influence on the performance of the treatment and membrane fouling formation of two membrane fluxes, 75 and 150 L/m^2 h, along with two US frequencies, 35 and 150 kHz. The results observed showed that the combination of membrane ultrafiltration with US, respect to the filtration process alone, reduced membrane fouling rates to a greater extent at the higher membrane flux and lower US frequency applied, reaching a reduction of 57.33% at 150 L/m^2 h and 35 kHz. Furthermore, higher organic matter and turbidity removals were observed at higher frequency (130 kHz). The results obtained highlights the applicability of this combined process for the upgrading of membrane ultrafiltration and as an alternative option to conventional tertiary wastewater treatments.

Keywords

Fouling
Membrane flux
Sonication
Ultrasound
Wastewater reclaim

1. Introduction

Water scarcity along with its increasing demand have focused the attention on the development of best practices for its reuse through the application of advanced wastewater treatments, such as membrane filtration [1], [2], [3], adsorption [4], [5], [6], ultrasound (US) [7], electrochemical [8], [9] and advanced oxidation processes [10], [11], in order to reach high effluent quality. Among these treatments, membrane filtration represents a promising alternative to the existing technologies, since it increases the removal efficiencies through the physical separation of particulate and colloidal matter from wastewater [**12**].
However, membrane fouling still represents the main drawback for the widespread application of this technology. Fouling reduces the permeability of a membrane due to the accumulation of colloids, particles, macromolecules and salts on the membrane surface, which consequently decreases the flux, shortens membrane life, due to frequent chemical and physical cleaning, and raises the treatment costs [13], [14], [15].
Research studies have focused the attention on membrane fouling control through the combination of the membrane filtration with different processes such as adsorption [16], [17], electrochemical [18], [19], [20] and advanced oxidation processes [21], [22]. Furthermore, several studies in the last decades have been carried out on the application of US for the enhancement of membrane permeability and the overcome of membrane flux decline [23], [24], [25]. US is, indeed, widely used as a surface cleaning technique and is a promising method for membrane fouling control since no chemical cleaning reagent is needed, the permeate flux can be maintained during the filtration process, not only after cleaning, and filtration is not interrupted to perform cleaning [26], [27]. When US is introduced into water, it is subjected to a series of compression and rarefaction cycles, which lead to the formation of cavitation bubbles [26], [28], [29], [30]. Under compression cycle, the bubbles collapse and generate different physical and chemical phenomena including localized

hot spots with high temperature (up to 5000 K) and pressure (up to 105 kPa), high-velocity fluid movement and the formation of hydroxyl radicals ·OH due to thermal dissociation of water molecules [27], [31]. The removal of portions of fouling layers from the membrane surface and/or the avoidance of particles deposition can be also attributed to the generation of acoustic streaming, microstreaming, microstreamers, microjets and shock waves, associated with US and cavitation bubbles [27].

The enhancement of membrane filtration performance and the effective cleaning of fouled membrane through US application have been demonstrated by several researchers. Kobayashi et al. [24] applied US to remove fouling of ultrafiltration and microfiltration membranes which were used to treat peptone and milk aqueous solutions, respectively, at 60 kPa operating pressure and US frequencies of 28, 45 and 100 kHz observing that 28 kHz frequency reduced the formation of the fouled layer in both filtration systems. In the study of Gao et al. [26], the normalized permeate flux was increased from 0.21 without US to 0.7 with US applied at a frequency of 20 kHz and a power of 16 W to a cross-flow ultrafiltration system to investigate the US control of surface-water fouled ceramic membranes.

Alventosa-de Lara et al. [32] carried out a study for assessing the factors affecting the US-assisted cleaning of an ultrafiltration ceramic membrane fouled by dye particles and obtained an improvement of cleaning efficiency by decreasing US frequency from 80 to 37 kHz and at mixed wave application mode.

Shahraki et al. [33] investigated the influence of different sonication modes (continuous, pulsed, sweeping, and degassing) with different frequencies of US (37, 80 kHz and tandem) on permeation flux and fouling of flat sheet ultrafiltration membrane. They found that the decrease of US frequencies enhanced the permeation flux, with the best condition of filtration obtained at 37 kHz and pulsed mode corresponding to the fouling percentage and sonication effect factor of 10.53 and 187.4%, respectively [33].

Naddeo et al. [34] analysed the effect of US at different frequencies (35 kHz, 130 kHz) for enhancing ultrafiltration with polysulfone membranes of wastewater during a constant flux process. The authors found that the lower frequency was able to produce a lower transmembrane pressure (TMP) increase during cross-flow filtration while the removal of organic matter (OM) was higher at 130 kHz. In a more recent study, Yu et al. [35] investigated the intermittent application of US (3 min/10 min every 3 days at 38 kHz) to the ultrafiltration process of water treatment at a constant flux of 20 L/(m^2 h) in order to control membrane fouling, observing a decrease of both reversible and irreversible fouling, with a 50% reduction in TMP development over 60 days of operation.

To the best of the authors' knowledge, the effect of membrane flux was not investigated yet in the previous papers related to the combination of membrane ultrafiltration with US despite membrane flux is a key aspect in the operation of membrane ultrafiltration. Indeed, since the flux is the quantity of material passing through a unit area per unit of time [36], working at higher membrane fluxes reduces the need of a large membrane surface area, with a reduction of the footprint and capital cost required, or, at the same membrane surface area, allows higher flows to be treated. However, higher flux operation can increase the surface fouling by enhancing the convective force towards the membrane since the flux is also related directly to the driving force and to the total hydraulic resistance offered by the membrane [36], [37].
Therefore, the present work aimed to evaluate the influence of different membrane fluxes (75 L/m^2 h and 150 L/m^2 h) in the ultrafiltration process combined with US (USMe) applied to real municipal wastewater. The effect of different frequencies (35 kHz, 130 kHz) was also taken into account as a secondary aspect. Ultrafiltration tests alone (Me) without the irradiation of US were also carried out as a control tests.

2. Materials and methods

2.1. Experimental setup

The experimental setup, utilized in this study to carry out the tests, was reported in Fig. 1. A ultrasonic bath (Elma®TI-H-10 MF3 230V) with a volume of about 8.6 L was used as an ultrasonic source. The US bath was characterized by an electrical nominal power varying from 200 to 800 W, two frequencies (35 or 130 kHz) and an average ultrasonic power intensity from 0.3 to 1.1 W/cm^2.

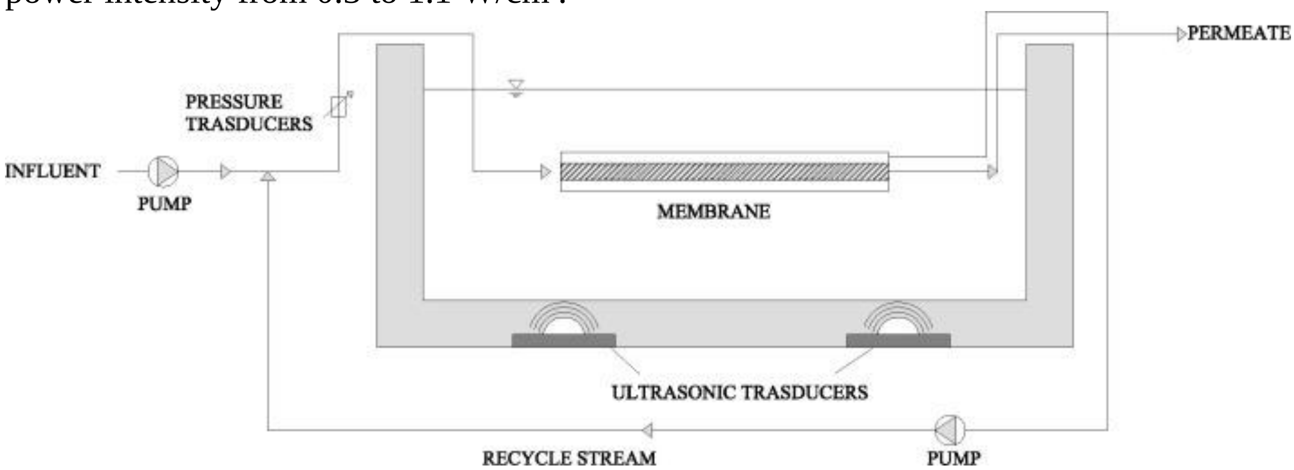

Fig. 1. Experimental setup of the USMe process.

The membrane unit was made of a single hollow fibre polysulfone ultrafiltrationmembrane (A/G Technology Corporation, USA) enclosed in a glass tube in order to collect the permeate with an inside-outside flow in a cross-flow configuration. The membrane, characterized by a filtration area of 6.6 cm^2 and a nominal pore size of 0.1 µm, was immersed at the centre of bath, filled with 5 L of DI water, and at a distance of 2.5 cm from the bottom according to previous studies [38], [39] in order to have a significant cavitational activity for controlling fouling without damaging the membrane. Real wastewater was collected downstream to secondary sedimentation tank of the wastewater treatment plant (WWTP) of Salerno (Italy) and used as a feed. A physical-chemical characterization of the influent wastewater treated by Salerno WWTP is reported in Table 1.

Table 1. Characteristics of influent wastewater from WWTP.

Parameter	Average value
COD [mg/L]	51.08 ± 4.94
Ammonia [mg/L]	3.8 ± 0.20
Nitrate [mg/L]	15.30 ± 0.25
Nitrite [mg/L]	0.280 ± 0.12
pH	7.91 ± 0.24
Turbidity [NTU]	3.28 ± 1.96
TSS [mg/L]	7.8 ± 2.80
UV_{254} [cm^{-1}]	0.15 ± 0.05

Two 323 S/D peristaltic pumps (Watson Marlow, UK) were utilized to feed the influent wastewater and recycle the streams.

2.2. Analysis

The standard methods 5130, 4030, 4020 [40] were used to analyse the total and soluble chemical oxygen demand (tCOD, sCOD), ammonia nitrogen (NH_4-N), nitrate nitrogen (NO_3-N), nitrite nitrogen (NO_2-N) and orthophosphate (PO_4-P). Total suspended solids (TSS) were determined in agreement with standard method 2090 [40]. The pH and temperature were analysed using a multiparametric probe (Hanna Instruments, HI769828).
Influent and permeate samples were collected for subsequent analysis of absorbance at UV_{254} and turbidity.
Organic matter (OM) was, indeed, characterized through UV_{254} which was measured using a Lambda 12 UV–Vis spectrometer (Perkin Elmer, USA). The turbidity was measured by a turbidimeter (HACH, Model 2100).

Membrane fouling formation was evaluated monitoring the trans-membrane pressure (TMP) variation over time through a PCE-932 full line pressure metre and a PS100 transducer (PCE Instruments, Italy) connected to a computer for the acquisition of the data.

2.3. Operating conditions

USMe tests were carried out at two membrane fluxes, 75 L/m^2 h and 150 L/m^2 h, in order to evaluate the enhancement effect of US on fouling formation at different membrane fluxes. Since the recycle ratio was one at both fluxes, there was a doubling of the feed flow at 150 L/m^2 h. The tests were performed in triplicate as to validate the results. Membrane ultrafiltration tests (Me) alone without US were conducted as control tests. The effect of US frequency was also considered. USMe tests were, indeed, conducted at both low (35 kHz) (USMe 35) and medium-high (130 kHz) (USMe 150) ultrasonic frequencies since, due to cavitation, mechanical forces are predominant at low frequencies while at medium – high frequencies there is the production of hydroxyl radicals and, consequently, sonolytic degradation [34]. The bath was set at the maximum electrical power of 800 W, which gave an average of 35 W/L and 29 W/L specific US density, respectively, for 35 kHz and130 kHz as measured by the calorimetric method according to previous studies [41], [42], [43]. US was applied continuously and the temperature inside the bath was maintained at 25 ± 2 °C by adding ice whenever necessary.

Before each filtration test, the TMP of DI water flow through the clean membrane was monitored according to the procedure reported in Secondes et al. [16]. The experiment with the influent wastewater was, then, performed and the filtration tests were stopped when the TMP achieved an approximate value of 20 kPa. After that, the membrane was completely cleaned according to the previous study of the authors [16]. TMP required to maintain the desired flux was used as the indicator of system fouling. The progress of the experiments is reported in terms variation of TMP over time and of the specific volume filtered (SVF) (L/m^2), defined as the cumulative volume filtered per unit area of membrane.

3. Results and discussion

3.1. Influence of US frequency

The results of US input frequency effects on membrane fouling formation are reported in Fig. 2. Each curve represents the average values of the three experimental tests performed for each process at a membrane flux of 150 L/m^2 h. As expected, the membrane fouling rate found was lower in the combined process USMe than in the Me process (Fig. 2).

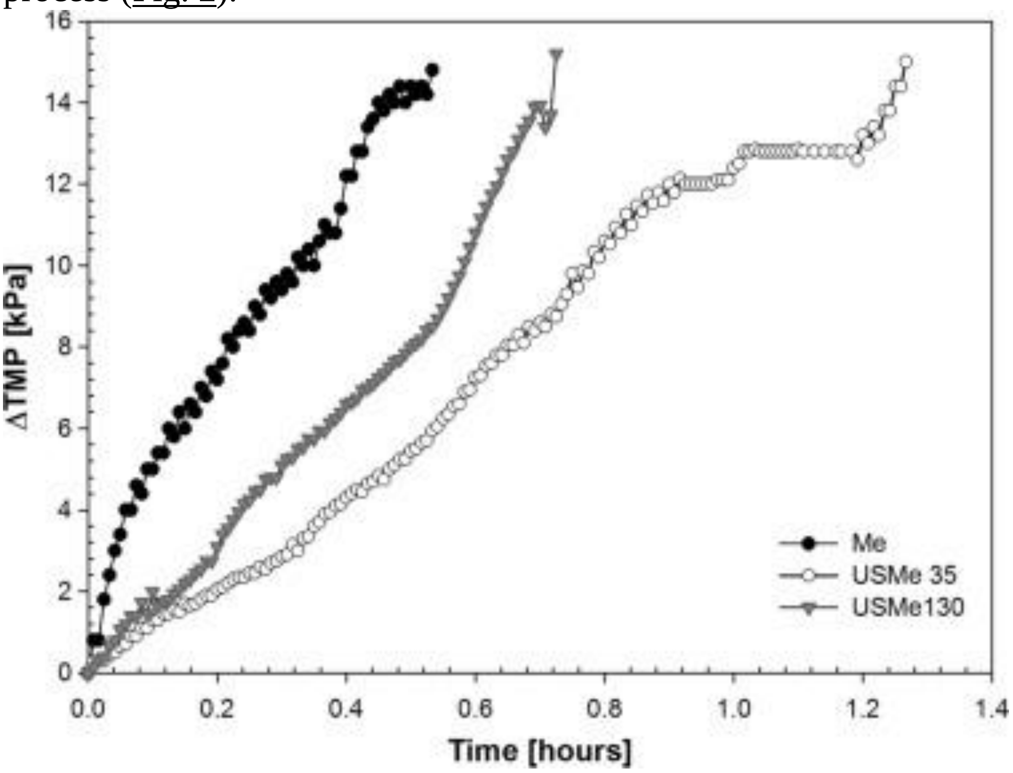

Fig. 2. TMP variation over time in Me, USMe 35 and USMe 130 processes at 150 L/m^2 h membrane flux.

In details, the membrane fouling rate decreased of 57.33% and 24.45% in the USMe 35 and USMe 130, respectively, compared to the control test (Me) as reported in Table 2. Therefore, the performance of the membrane ultrafiltration was enhanced when the US frequency was reduced from 130 kHz (USMe 130) to 35 kHz (USMe 35).

Table 2. Membrane fouling rate in Me, USMe 35 and USMe 130 tests and their percentage of reduction in the combined process (USMe) respect to the control test (Me) at the two membrane fluxes (75 and 150 L/m^2 h).

Membrane flux	**Fouling rate [kPa/h]**				
	Me	**USMe 35**	**% Reduction**	**USMe 130**	**% Reduction**
75 L/m^2 h	18.18	11.54	36.54	15.39	15.38
150 L/m^2 h	27.75	11.84	57.33	20.97	24.45

These results are in agreement with previous studies that observed an increase in the US-assisted cleaning efficiency of the fouled membranes with the decrease of the US frequency [31], [32], [34].

At low frequencies the bubbles produced through acoustic cavitation are larger and more energetically collapse cause stronger vibration, micro-jetting and localized turbulence leading to the detachment of fouling [32], [34], [44], [45]. Although higher frequencies lead to the collapse of more cavitation bubbles, they are smaller in size and collapse less energetically since the rarefaction (and compression) cycles are too short to allow a bubble to expand to a size sufficient to cause turbulence in the liquid [46].

As a result, high frequencies are not able to detach particles from the cake layer as efficiently as lower frequencies [31]. The violence of the collapses and the resulting turbulence are more significant than increased number of weaker collapses [31], [32]. Therefore, low frequencies are more effective in preventing membrane fouling.

3.2. Influence of membrane flux

As show in Table 2, the tests at 75 L/m^2 h showed lower membrane fouling rates in the process Me and in the combined process USMe at both frequencies of 35 and 130 kHz (USMe 35 and USMe 130), although at frequency of 35 kHz the fouling rates obtained for the two fluxes are comparable. At higher fluxes the hydrodynamic drag force overcomes repulsive forces which induces the deposition of foulants on the membrane surface and, thus, higher membrane fouling [47].

Interestingly, in the tests at the higher flux (150 L/m^2h) the application of US in the combined process USMe, compared with the control test (Me) at both frequencies, decreased more the membrane fouling rates than the same tests performed at the lower flux of 75 L/m^2 h (Table 2). The percentages of fouling rate reduction at the membrane flux of 150 L/m^2 h were, indeed, increased, due to the US application, of around 21% and 9% at the frequencies of 35 kHz and 130 kHz, respectively.

This can been also observed in Fig. 3 which shows the behaviour of ΔTMP as a function of the SFV at both the membrane fluxes (75 and 150 L/m^2 h) and US frequencies (35 kHz and 130 kHz). The TMP, indeed, grows with increasing specific volume filtered and, in particular, less quickly at the higher membrane flux equal to 150 L/m^2 h for both frequencies (Fig. 3a and b), although the lower frequency of 35 kHz resulted in better performance of the system (Fig. 3a). At the same SFV, the TMP observed was higher at a flux of 75 L/m^2 h than at 150 L/m^2 h. Considering the value of SVF reached at a ΔTMP equal to 15 kPa, in the USMe process the SVF was 94.9% and 48.7% higher at 150 L/m^2 h than at 75 L/m^2 h and for the applied frequencies of 35 kHz and 130 kHz, respectively.

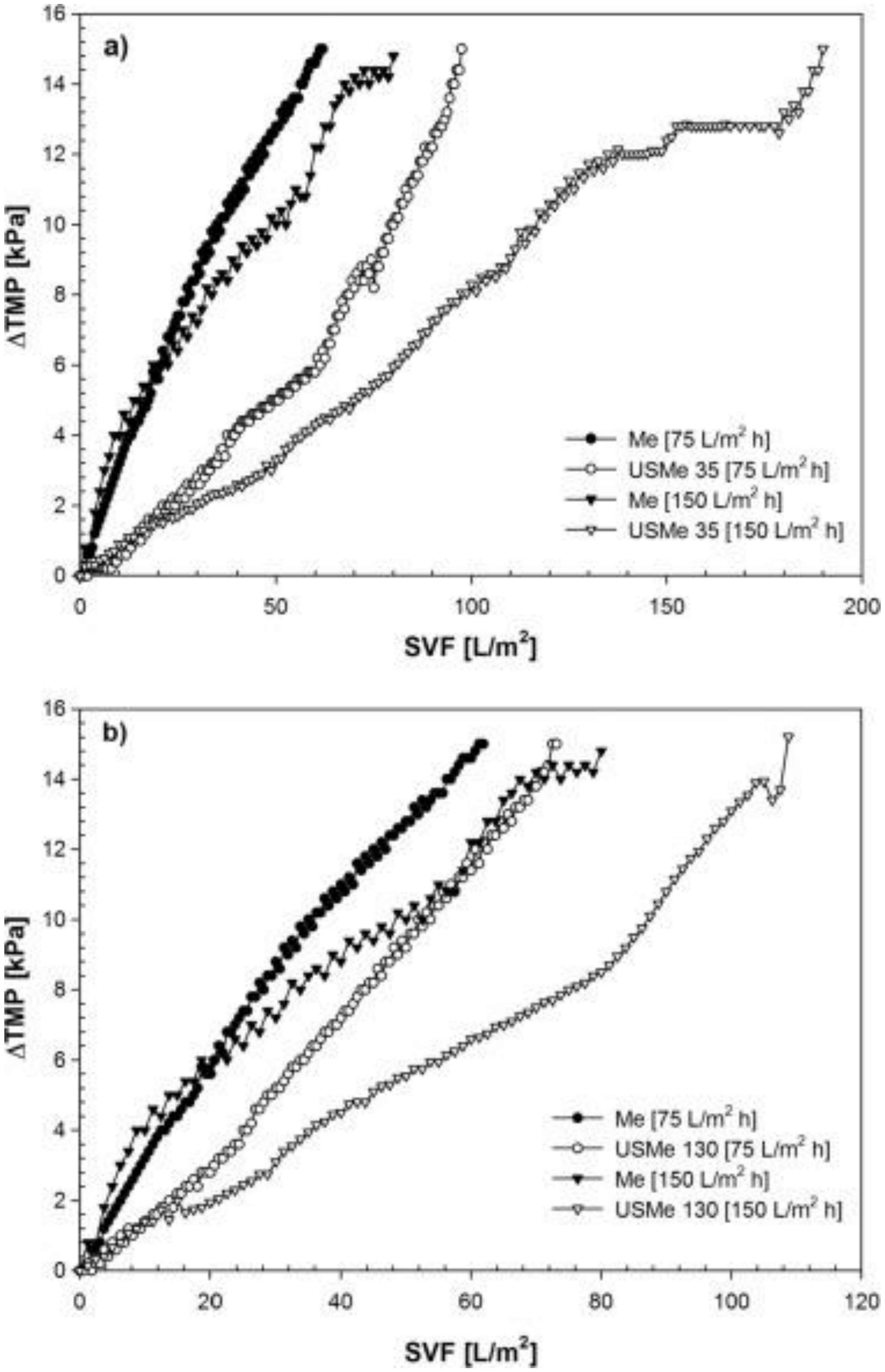

Fig. 3. TMP variation versus specific volume filtered (SVF) in Me and USMe processes at two membrane fluxes of 75 and 150 L/m^2 h: (a) at 35 kHz US frequency, (b) 130 kHz US frequency.

Therefore, the influence of US on membrane fouling is more effective at higher membrane fluxes. With the increase of the membrane flux, the cross-flow velocity also increases with a competition/combination between the effects of ultrasonication and of cross-flow flux [48]. The formation of the cake layer during cross-flow filtration results from a balance between the TMP and the shear-induced hydrodynamic force in the filtration channel, determined by the cross-flow flux and enhanced by ultrasonication [48].

Therefore, in the present study, this US enhancement was more evident at the higher flux applied due to the higher cross-flow velocity and, thus, higher shear-induced hydrodynamic forces. Indeed, the particles were detached from the membrane surface by the shearing action of cavitation mechanisms and continually carried away from the membrane surface by the increased feed flow [28]. The results obtained are also in agreement with a previous study [49], which found that when no US was used, the limiting flux was not influenced by the feed flow rate, but when US was applied the highest limiting flux was achieved with the highest cross-flow velocity.

3.3. Effect of USMe process on the effluent wastewater quality

The removal of the OM, in terms of UV_{254}, and turbidity at 150 L/m^2 h membrane flux and at both frequencies of 35 kHz and 130 kHz is reported in Fig. 4. The higher frequency of 130 kHz showed higher OM removal efficiency. At higher frequencies, the US energy is directed to the degradation of water molecules and the increased production of hydroxyl radicals [50].

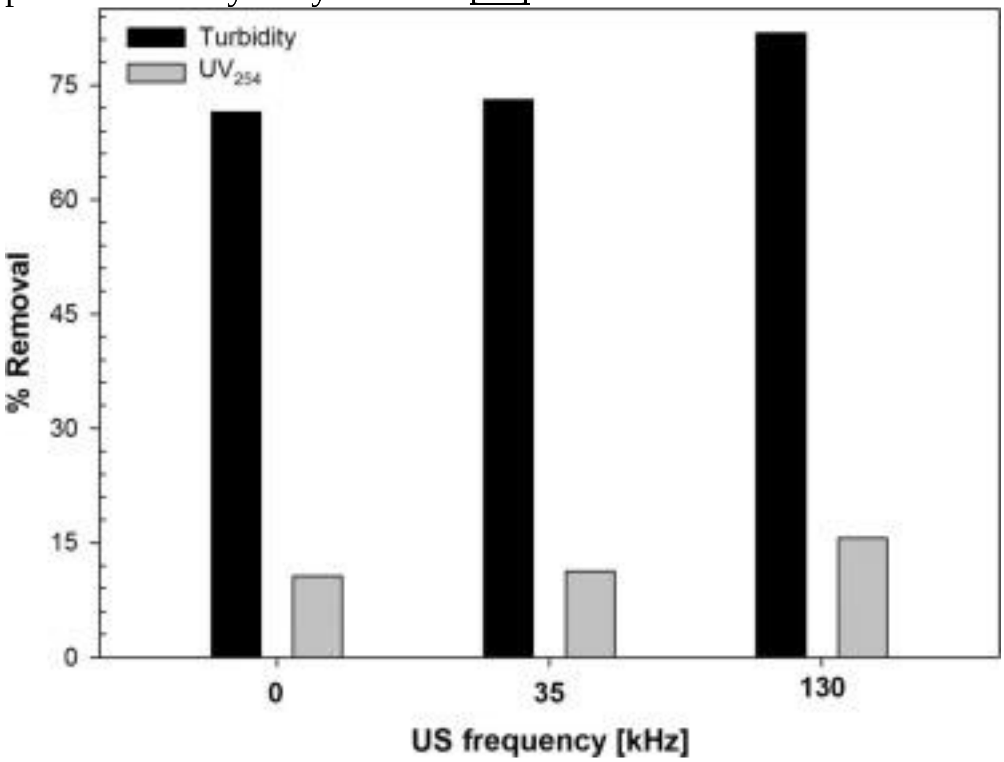

Fig. 4. OM and turbidity removal in the Me (0), USMe 35 and USMe 130 processes at a membrane flux of 150 L/m^2 h.

Turbidity has been evaluated as a physical parameter to assess the process performance. In the presence of US at the highest frequency (USMe 130), the effluent turbidity was lower than that without ultrasonication (Fig. 4). According to previous studies [51], [52], this can be attributed to the generation of microstreams and vibration which led to the aggregation of small particles to form large particles minimizing the effluent turbidity. The tests at 35 kHz showed a lower removal than those at 130 kHz and similar to the control test Me, due to the shattering the particles at this frequency.

4. Conclusions

In the present work, membrane ultrafiltration was combined with US irradiation (USMe) at two membrane fluxes (75 L/m^2 h and 150 L/m^2 h) and US frequencies (35 kHz and 130 kHz) using real municipal wastewater. Ultrafiltration tests alone (Me) were carried out as a control tests. The results observed showed that the performance of membrane ultrafiltration was enhanced when the US frequency was reduced from 130 kHz (USMe 130) to 35 kHz (USMe 35) at both membrane fluxes due to the stronger vibration and localized turbulence at lower frequency. Furthermore, the influence of US on membrane fouling is more effective at higher membrane fluxes since higher reductions of membrane fouling rates were found, up to 57.33% at 35 kHz. The increase of the cross-flow velocity at higher membrane flux allowed the detachment of the particles from the membrane surface by the shearing action of cavitation mechanisms. At a frequency of 130 kHz, higher OM and turbidity removal was found due to the production of hydroxyls radicals and weaker bubble collapse.

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