

Pilot system of microfiltration and reverse osmosis membranes for greywater reuse

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ABSTRACT

Nowadays, one of the most interesting issues for wastewater recycling is the on-site treatment and reuse of greywater. It is reclaimed for non-potable on-site purposes (i.e., irrigation and toilet flushing). A dual membrane process for greywater treatment, low-pressure microfiltration (MF) membrane followed by a reverse osmosis (RO) process. The MF pretreatment was able to tolerate unfavorable variations in feed greywater and presented high removal efficiencies of apparent color, turbidity, and suspended particles. Consequently, the RO membrane system could be operated at a higher permeate flux and lower frequency of chemical cleaning. It has been verified that the method has recorded removals of turbidity, apparent color, total suspended solids, linear alkylbenzene sulfonate, and organic matter parameters up to 90%. Results achieved in the present study revealed that the membrane process was a technically viable alternative for greywater treatment. The effluent quality emphasized the possibility of reusing domestic sewage for purposes other than consumption, such as car washing and toilet flushing.

Keywords: Domestic reuse; Greywater; Microfiltration; Reverse osmosis; Pilot scale; Treatment

1. Introduction

The main challenge for the maintenance and preservation of water resources is to minimize the discharge of domestic effluents. This is usually done by aerobic digestion, which is energy-intensive with a large footprint [1,2]. One of the options is domestic reuse. For this, it is necessary to separate the domestic effluent into greywater, produced from bathtubs, showers, hand basins, laundry machines and kitchen sinks, and blackwater, produced from the toilets [3,4]. Greywater usually allows easier treatment than blackwater, which contains higher organic matter load and

higher pathogen content. In addition, as the greywater is the major source of wastewater generated in households or office buildings [2,5], the source separation will greatly reduce the wastewater volume that must be diverted to biological treatment, as well as improve the operational conditions of the biological treatment by maintaining the biological oxygen demand (BOD₅) of the wastewater in relatively stable levels [6,7].

Some points relating to greywater still must be highlighted. Greywater is an important source of urban water that may be suitable for relatively easy on-site treatment and reuse [5,7]. Greywater reuse will decrease freshwater

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use, and minimize the demand and global costs on drinking water supplies [5]. Due to its characteristics, 30% of the organic fraction, and 9%–20% of nutrients, it may be a beneficial source of irrigation water in yards [5,8]. However, greywater has several factors that affect quantity and quality, before proposing a treatment system, it is necessary to characterize the greywater generated in the residence to avoid both under and over-design of the treatment system [9,10].

The treatment at some places is preferred to be done at household levels, where residents build their small treatment plants and reuse water for themselves [11,12]. The onsite household greywater treatment requires a process that can be easily operated and monitored by inhabitants, be compact, low cost, and produce good-quality effluent that is safe for reuse [12,13]. Greywater is generally reclaimed for various applications, such as non-potable reuse (flushing toilets, irrigation, washing cars, recharging aesthetically pleasing natures, or under groundwater systems) [14]. In terms of various applications, the reclaimed greywater should meet appropriate water quality standards or guidelines to ensure its safe and sustainable reuse [15,16].

Greywater treatment and reuse schemes have already been piloted in many countries around the world, employing different methods of treatment resulting in varying levels of system complexity and cost [17–20]. Thus, to meet greywater reuse standards, various membrane-based techniques have been widely adopted to treat greywater for producing water with superior quality [21]. Because this technology can be very efficient for allowing high rates of contaminants removal, the low necessity of chemical products to conduct the treatment, smaller area to implement the treatment unit, and lower production of residues, the membrane separation processes (MSP) can be more competitive in terms of costs in relation to a conventional treatment system [22]. A pilot system treating real greywater in a grey house with an ultrafiltration hollow-fiber submerged membrane bioreactor (MBR) achieved removals of approximately 87% of chemical oxygen demand (COD) and 80% of anionic surfactants, and total suspended solids (TSS) were reduced from 95 mg L⁻¹ in the influent to 8 mg L⁻¹ in the effluent [5]. However, there is still a lack of information on the behavior of membrane systems under real conditions in the case of single houses.

Thus, this paper aims to monitor a pilot system for on-site greywater treatment in a single household in Brazil, with a focus on treated greywater quality and membrane performance during 60 d of operation. The treatment system included an MSP with pressured microfiltration (MF) and reverse osmosis (RO) membranes. The evaluation of greywater treatment was conducted based on the monitoring of physicochemical parameters to assess greywater quality.

2. Materials and methods

2.1. Collection location

The treatment system was implemented in a 4 persons-household (useful area of 390 m²), located in Brazil. The residential sewer line was adapted to separate greywater from blackwater. Then, blackwater was directed into the sewer system, while greywater was directed into a 600 L

storage tank, a volume calculated based on the total of greywater generated during a whole day, thus guaranteeing the homogenization of the water derived from a variety of generating sources. The greywater included wastewater from baths, showers, hand washbasins, washing machines, and dishwashers.

2.2. Description of the membrane pilot plant

MF and RO trials were carried out on a pilot-scale membrane system. Fig. 1 presents a general scheme of the treatment process employed. The raw greywater was poured into the storage tank 1, which contained a submerged pump (B1) controlled by a level switch. This pump was inside an aluminum net with 1 mm mesh size openings to retain clothing fibers, hairs, and other residues. The greywater was then suctioned by a pressurized pump (B2) into the MF module (tank 2) and the permeate was conducted to the storage tank 3. The transmembrane pressure (TMP) and velocity were controlled by an electronic panel. The permeate from the storage tank 3 was pumped (B3) into the RO system which contained a spiral-wound cartridge. The permeate from the RO unit was then conducted into the storage vessel (tank 4), while the retentate/concentrated effluent was discarded.

MF system consisted of one module with two parallel hollow fibers submerged membranes. The MF was provided by the Brazilian Company PAM Membranes (Rio de Janeiro, Brazil). The fibers were made of polyamide material, distributed vertically, and fixed in the extremities of the cartridges. The upper extremity received the aperture of filtering fibers for the permeate exit. MF was operated in a cross-flow configuration. Details on the membrane cartridges are listed in Table 1.

RO system used for the tests contained a 6.3 cm diameter × 53.3 cm length stainless pressure vessel. Housed within the pressure vessel was a 6.1 cm diameter RO membrane provided by FilmTec™ membranes (Minnesota, USA) (Model Number TW30-2521), as described in Table 2.

Previous data collected about the used household cleaning products showed that chlorine was an element present in the commonly used chemicals. Thus, a carbon filter was installed in the piping of the RO supply to prevent the presence of chlorine in the RO module, as the RO membrane was intolerant to this element. The activated carbon may be used to remove chlorine with little degradation or damage to the carbon [23], so the carbon filter prevented possible membrane damage due to the presence of chlorine.

2.3. MF performance

To determine the performance of the MF, the pilot system was operated for a period of 60 d for 8 h a day at the residence under study, totaling 480 h of operation. Four cycles of 120 h of operation were performed and evaluated. The system was operated at a TMP of 0.50 ± 0.05 bar. The TMP remained constant, resulting in the reduction of permeate flux over time. The times for filtrate and backwashing pulses were 30 and 2 min respectively. There was no additional stirring in the vessel. Also, the permeate flux was monitored to determine the backwashing efficiency in

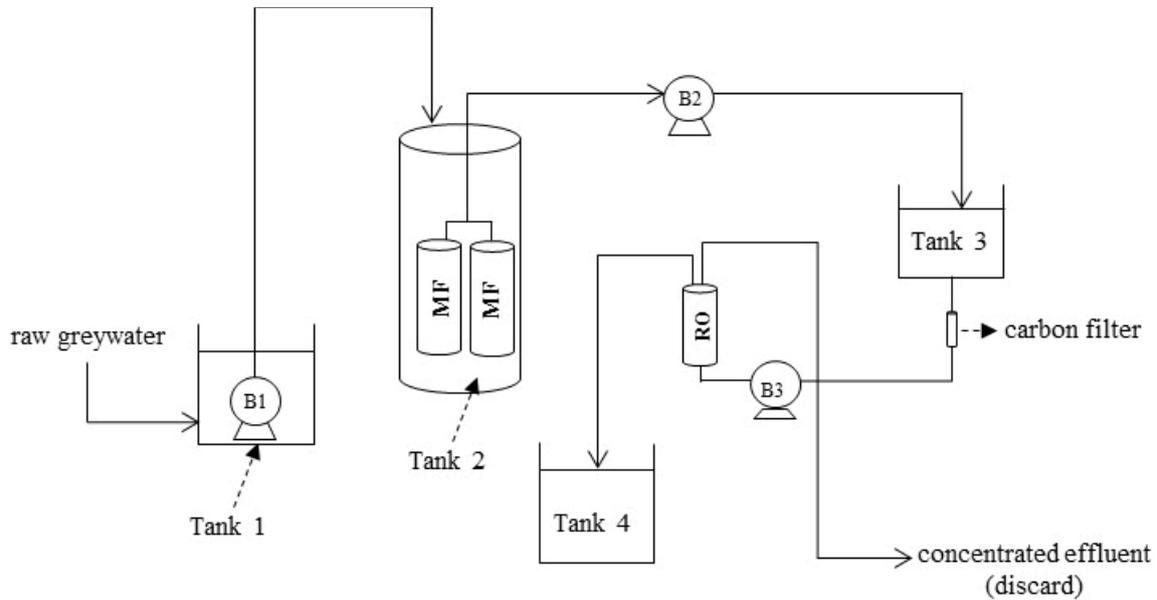


Fig. 1. Schematic representation of the pilot system B1 – Hydrobloc pump D300 X 32501 KSB submerged suction pump of the affluent. B2 – suction pump FLOJET series R3811 of the MF permeate. B3 – RO feed pump FLOJET series R3811.

Table 1
MF membranes specifications

Membrane property	Characteristic	
Manufacturer company	PAM Membranes	
Membrane configuration	Hollow fiber, outside-in flow	
Number of cartridges	2	
Mesh size	0.4 μm	
Packing density	500 $\text{m}^2 \text{m}^{-3}$	800 $\text{m}^2 \text{m}^{-3}$
Permeation area	0.5 m^2	1.0 m^2

the MF process and to evaluate the influence of the operational parameters on the MF membrane cartridges. At the end of the four cycles, the system was turned off.

Membrane cleaning was performed manually after each cycle of operation by taking out the MF membrane cartridges and cleaning the accumulated cake layer with tap water

jet until visible removal of the surface cake on the hollow fibers, followed by a 20 min backwashing with tap water at a pressure of 1 bar. Also, hydraulic permeability was determined. In the case of the permeability, recovery had not been achieved through backwashing, the chemical cleaning of the membrane cartridges would have been carried out using sodium hypochlorite diluted to 0.1% v/v in water.

2.4. RO performance

RO module was operated in batch mode for 10–12 h d^{-1} , depending on the volume of permeate produced by MF. This module was operated at a pressure of 6.0 ± 1.0 bar and a recovery rate of 90%. The permeate flux was monitored during its operation.

The hydraulic permeability of the module was assessed before starting treatment and after 60 d of operation. Subsequently, chemical cleaning was performed with the recirculation of alkaline NaOH solution at pH 10 for 10 min.

Table 2
RO membranes specifications

Membrane property	Characteristic
Manufacturer company	FilmTec™ membranes
Membrane configuration	Spiral wound
Element model number	TW30-2521
Membrane material	Polyamide thin-film composite
Dimensions of cartridges	6.1 cm diameter \times 53.33 cm long
Active membrane area	1.2 m^2
Maximum feed silt density index	5
Stabilized salt rejection	99.5%
Free chlorine tolerance	<0.1 mg L^{-1}

The recirculation was stopped, and the solution was allowed to act inside the module for 10 min. This procedure was repeated 10 times. And again, the permeability was investigated to determine the efficiency of chemical cleaning, in relation to the hydraulic permeability of the module.

2.5. Analytical procedures

The samples of raw greywater, MF permeate, and RO permeate were collected for characterization, at 15th, 30th, 45th, and 60th day of the process operation.

The samples were then transported to the Laboratory of Environment Management, Control, and Conservation of the Department of Chemical Engineering of the State University of Maringá for characterization. The conditions of sampling, conservation, and analyses followed the recommendations of standard methods [24].

The pH was measured using a DIGIMED equipment (Brazil), analytical instrumentation. Physical parameters were determined using the methodology described by [24]. The apparent color was determined using the platinum-cobalt method; turbidity was determined using the spectrophotometric method and total dissolved solids (TDS) and TSS were determined using the gravimetric method. The BOD₅ was determined using the BODTrak™ II methodology described by the HACH equipment (Hach Company, USA), with an accuracy of $\pm 1.0 \text{ mg L}^{-1}$, and the results were expressed in milligrams of oxygen consumed per liter of the sample during 5 d of incubation at 20°C. The COD was determined using the potassium dichromate oxidation in acidic medium, the methodology described by the standard methods [24]. The linear alquibenzene sulfonate (LAS) was determined through the spectrophotometric method of methylene blue, the methodology described by the Technical Brazilian Standard NBR 10,738/89 [25].

2.6. Pollutants removal efficiency and determination of the treated greywater quality

The parameter used to quantify the efficiency of MF and RO was solute removal (E). It was calculated as indicated in Eq. (1), where C_i and C_f were feed and permeate concentrations at the time of measurement, respectively.

$$E\% = \frac{C_i - C_f}{C_i} \cdot 100 \quad (1)$$

where C_i was the initial concentration of the analyzed parameters, C_f was the final concentration of the analyzed parameters and E was the removal efficiency (%).

3. Results and discussion

3.1. MF operating performance

The temperature of greywater oscillated from 18°C to 55°C according to the generating source as well as on the daily ambient temperature, which varied from 14°C to 33°C during operation.

In the first 10 min of each cycle, the TMP was increased and permeate flux was constant during filtration. When TMP

of $0.50 \pm 0.05 \text{ bar}$ was reached, it remained constant, which resulted in the reduction of permeate flux over time.

The decrease of permeate flux with time was monitored in order to obtain preliminary information about the fouling tendency of the membrane. The permeate flux was measured every hour. The behavior of the permeate flux as a function of operation time is shown in Fig. 2. The flux data plotted in Fig. 2 are the average daily flux measurements performed during the 8 h of operation.

It was observed in Fig. 2, that in the first hour of operation the fouling effect was more accentuated when the permeate flux decreased by 20% (23.3 to 18.7 $\text{L h}^{-1} \text{m}^{-2}$). After this period of operation, the flux reduction was more moderate. Four operational cycles can also be observed from the flux profile shown in Fig. 2. After each cycle, manual cleaning in the MF membrane cartridges was performed. This procedure allowed full recovery (1st and 2nd cycles) and partial recovery (3rd cycle of 96% and the 4th cycle of 90%) of the permeate flux in the MF module. This shows that the fouling occurred and could not be neglected in the process evaluation. The fouling is usually due to the accumulation and/or adsorption of organic matter in the pores and/or on the surface of the membranes, which leads to the decline of the permeate flux or increase of operating pressure with processing time, resulting in higher operating costs [26].

In order to control the decrease in the MF permeate flux, the backwashing pulses (2 min) were interchanged after 30 min of operation. This is the most common physical cleaning method to remove the particulate material of the pores and membrane surface aiming at reducing reverse fouling such as uneven or gel layers [27]. Considering these results for permeate flux decrease, the membrane system restriction was verified, which may interfere with the operational performance of the treatment and the permeate quality [28,29].

At the end of 480 h of operation, chemical cleaning was carried out. This allowed the recovery of 99.9% of the hydraulic permeability of MF membrane cartridges.

These results showed that greywater can be treated in the MF membrane. However, the duration of each MF cycle was relatively short.

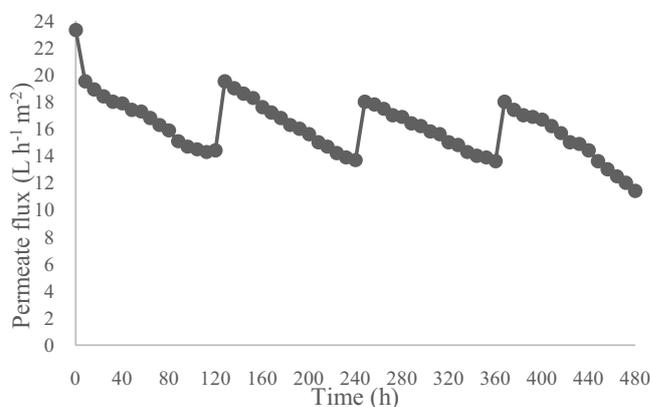


Fig. 2. The average permeates flux as a function of time for the MF.

3.2. Operational performance of RO membranes

The RO membrane assessed in this work was used to improve the greywater effluent feed which was pretreated by MF. RO is the process that ensures the highest water quality [30]. In this study, the RO system was operated during 60 d without any physical or chemical cleaning.

According to Fig. 3, the permeate flux presented a gradual decrease from 11.7 to 7.6 L h⁻¹ m⁻² (35% reduction), which occurred due to fouling characteristics to the MSP. The concentrate outflow was minimum in order to obtain the highest amount of permeate possible; therefore, it varied between 0.5 and 1.4 L h⁻¹. This oscillation derived from a pressure variation of 6.0 ± 1.0 bar. The higher the filtration pressure, the lower the amount of concentrate.

The reduction in hydraulic permeability for the RO membrane module was 30.5%, decreasing from 2.10 to 1.46 L h⁻¹ m⁻² bar⁻¹. After alkaline chemical cleaning, it was possible to recover the permeability in 96.2% (2.02 L h⁻¹ m⁻² bar⁻¹). It can be said that the reversible fouling was 26.7%, and the irreversible, 3.8%. According to [19], the main potential components of greywater fouling are particulate and inorganic-organic matter, dissolved organic matter, monovalent and multivalent salts, surfactants, and pathogens.

In short, the fouling progressed slowly, and the filtration operation of the spiral membrane module was stable. For the RO to function properly, it is important to carry out periodic chemical cleaning.

3.3. Efficiency at removing pollutants and establishing the quality of the greywater treated

Table 3 presents the average of the physical and chemical parameters determined along with the 60 d of assessed operation.

During the operation time, the average pH of raw greywater was 8.1 ± 0.8. The pH value was reduced at every stage of the treatment due to decreased LAS concentration values. RO permeate pH varied from 6.5 to 7.8.

Table 3 revealed that the MF achieved the removal of 71% of the apparent color, from 687 ± 537 to 200 ± 132 Pt-Co APHA (American Public Health Association), 62% of turbidity – from 188 ± 78 NTU to 71 ± 35 NTU, and 94% of TSS – from 136 ± 103 mg L⁻¹ to 8 ± 6 mg L⁻¹, a low apparent color, turbidity, and TSS filtrate were also obtained consistently. Although there was some variation in the physical

parameters of the feed, the MF was efficient as a pilot system for the pre-treatment of greywater, showing average removals of apparent color, turbidity and TSS concentration of 71%, 62%, and 94%, respectively. The RO coupling after the MF was essential to the treatment. The RO was responsible for the effluent polishing, which enabled the absence of suspended solids in the treated effluent, regarding all analyzed samples, as well as an average apparent color of 2 ± 3 UNITS Pt-Co APHA, and an average turbidity of 2 ± 1 NTU. The produced permeate was low in turbidity and free of TSS with excellent physical appearance. TDS were also removed significantly. Water produced from the RO operation presented an average TDS 258 ± 145 mg L⁻¹, rejection 72.8%. Excessive levels of TDS in reclaimed waters can result in fouling, may cause scaling, spots on car paint, and mortality to irrigated plants [31–33]. Contaminants removal from greywater by MF and RO membranes was similar to that obtained by the use of submerged spiral wound ultrafiltration membrane detailed by [34].

Evidenced by the results (Table 3), greywater treated from direct MF processes is not preferable for reuse in some applications that require high-quality water. Due to the porous nature of MF membranes, they have little capacity for organic removal, that can pass through the MF membranes and remain in the permeate [35]. This could lead to water treated with less microbiologically stable properties and possibly containing personal care products and household chemicals [36].

However, integrating MF to RO, the system produced greywater with excellent quality due to the non-porous nature of RO [21]. It was possible to obtain an efficiency of BOD₅ removal above 90%. The average BOD₅ concentrations for the raw and RO permeate were 288 ± 38 mg L⁻¹ and 4 ± 2 mg L⁻¹, respectively. BOD₅ removal potentially achievable by adopting the RO process can be up to 97.7% [8]. But, similar results were obtained by the use of a matrix of treatment trains including coarse filtration, MF, activated carbon, ultrafiltration, ultraviolet, and RO to treat greywater, obtaining 84.2% BOD₅ removal [37]. Also, the COD concentration in the treated greywater was 28 ± 9 mg L⁻¹, significantly lower than the COD concentration in the influent (613 ± 302 mg L⁻¹). The results showed that the combined MF and RO process is a promising technology that can be used to treat greywater. The quality of greywater treated in this study meets the guidelines for greywater reuse

Table 3
Greywater quality data from the pilot system operation

Parameter	Raw greywater	MF permeate	RO permeate
pH	8.1 ± 0.8	8.0 ± 0.7	7.0 ± 0.7
Apparent color, Pt-Co APHA	687 ± 537	200 ± 132	2 ± 3
Turbidity, NTU	188 ± 78	71 ± 35	2 ± 1
TSS, mg L ⁻¹	136 ± 103	8 ± 6	ND
TDS, mg L ⁻¹	1,123 ± 101	951 ± 204	258 ± 145
BOD ₅ , mg L ⁻¹	288 ± 38	202 ± 35	4 ± 2
COD, mg L ⁻¹	613 ± 302	255 ± 200	28 ± 9

ND – not detected

Table 4
The LAS concentration

Samples	Raw greywater (mg L ⁻¹)	RO permeate (mg L ⁻¹)	Removal efficiency (%)
15th day	18	0.3	98.3
30th day	40	0.3	99.3
45th day	72	0.4	99.4
60th day	45	1.6	96.4

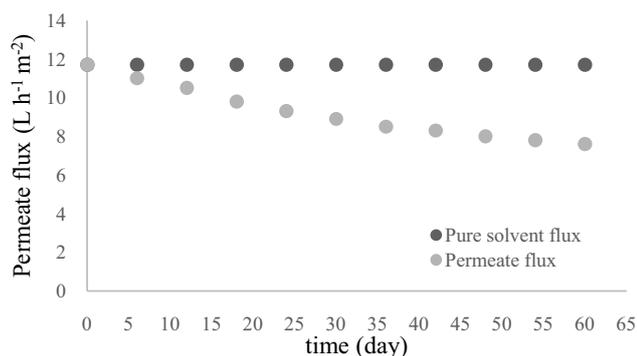


Fig. 3. Experimental data for permeate flux for the RO.

(unrestricted, restricted, environmental) [16,32] such as pH at 6–9, TSS ≤ 30 mg L⁻¹, BOD₅ ≤ 10 –30 mg L⁻¹, especially for irrigation and toilet flushing.

This technology requires a small footprint and could produce an effluent that is of high quality that may be suitable for reuse. This process showed to be more efficient than a biological process, for example, in treating greywater. An aerobic digestion unit integrated with hydrogen peroxide disinfection unit was studied for the purpose of greywater treatment to the standard for non-potable usage, achieving 88% removal of TSS and 68% of COD [13].

Table 4 indicates the removal of LAS in the RO permeate was higher than 96%. It was similar to the results obtained by [5] who used the treatment of ultrafiltration submerged hollow fiber MBR. However, LAS concentration increased during the treatment process. Accordingly, at the end of the 60 d of unit operation, the RO membrane saturated with the LAS, which was verified through the last permeate analysis. LAS concentration in the permeate at 60th day was 1.6 mg L⁻¹. The value found was four to five times higher than the remaining analyses – from 0.1 to 0.4 mg L⁻¹ on the pilot system. According to [34], this drop in rejection decrease may have been caused by the minimization of convective effects, that is, an increase in the polarization of concentration in the membrane surface may have occurred from dissolved ions accumulation, requiring a chemical cleaning of the RO membrane. Still, it may have been a result of the LAS passage through the membrane, reducing permeate quality.

Nonetheless, the RO permeate can be used in gardening and agriculture for irrigation and soil fertilization. Unless the water quality requirements regarding the fecal coliforms

counting are obligatory, the reclaimed greywater in this study can also be used for toilet flushing after disinfection.

4. Conclusion

The results of this study confirmed that the MF and RO membrane system was an effective method for greywater treatment and reuse with respect to physical impurities (apparent color, turbidity, and TSS), organic matter (BOD₅ and COD) and chemical (LAS).

From the pilot operation results, physical and chemical cleanings should be carried out periodically to guarantee the quality of the water treated and avoid irreversible fouling in membranes as well as operate in favorable hydrodynamic conditions regarding flux and rejection rates. This would be valuable both from membrane lifespan and operational aspects.

From an economic point of view, treating greywater for reuse with the MF and RO technologies could be another alternative. With MF pretreatment prior to RO, there are several benefits such as stable operation, extended membrane life, significant operator labor savings, and start-up times.

Based on the present results, MSP appears to be an effective alternative to traditional wastewater treatments for reducing the environmental impact and improving efficiency. In short, segregation of urban domestic effluent can benefit municipal sewage treatment systems for preventing the dilution of contaminants remnants eliminated by humans in the effluent and increasing the degradation potential of the compounds in biological processes involved in sewage treatment. Thus, domestic reuse is currently an important alternative, and measures such as to preserve and increase both consumptions and reuse efficiency postpone the upcoming scarcity and allow sustainable development.

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