Desalination and Water Treatment
www.deswater.com

doi: 10.1080/19443994.2014.999714

56 (2015) 3438–3446
December

Taylor & Francis
Taylor & Francis Group

Ultrafiltration fouling trend simulation of a municipal wastewater treatment plant effluent with model wastewater

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Received 15 July 2014; Accepted 21 November 2014

ABSTRACT

Secondary treatment effluents (STEs) from municipal wastewater treatment plants (MWTPs) require tertiary treatments to be reused in agriculture. Among tertiary treatment technologies, ultrafiltration (UF) has been proven to be a reliable reclamation process. Nevertheless, this technique has an important disadvantage: Membrane fouling. This phenomenon causes decline in permeate flux with time and increases the operational costs. Due to the fact that secondary effluents from MWTPs contain a large amount of different compounds and that there is certain variability in their composition, the use of a simplified model wastewater consisting of only few compounds may help to simulate better the UF fouling trend. The main STE components responsible for fouling membrane during UF tests are extracellular polymeric substances (EPS). These substances are mainly composed of proteins and polysaccharides; thus, they are commonly used to prepare model wastewaters. This work consisted in two parts. Firstly, a model wastewater was selected, attending to protein and carbohydrate content and chemical oxygen demand (COD), among different model solutions mimicking STE. Secondly, UF behavior of the selected model solution was compared with the behavior of the secondary effluent in the UF tests at different cross-flow velocities (0.8–1.2 m/s) and transmembrane pressures (TMPs) (62–100 kPa). The membrane used in the UF tests was UFCM5 Norit X-flow® hollow-fiber (HB). The model wastewater that represented the best the fouling trend of the STE had a composition of 15 mg/l of bovine serum albumin (BSA) and 5.5 mg/l of dextran. It was found that BSA contributed to long-term fouling, whereas dextran contributed to both long- and short-term fouling.

Keywords: Ultrafiltration; Model wastewater; Municipal treatment plant; Fouling

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Presented at the IX Ibero-American Congress on Membrane Science and Technology (CITEM 2014), 25–28 May 2014, Santander, Spain

1. Introduction

In the last few years, many wastewater treatment plants (WWTPs) are being upgraded by implementation of tertiary treatments that improve the quality of the biologically treated wastewater. The need for a tertiary treatment comes from the fact that high quality standards regarding suspended solids and pathogens are required for wastewater reuse. There are different techniques to carry out the wastewater reclamation. Among them, ultrafiltration (UF) has been proven to be a reliable process. In addition, UF has some advantages such as high permeate quality, no by-product generation, and high efficiency. Besides, it is easy to operate and economically feasible due to low energy consumption and to the small footprint [1–5]. However, UF processes, as other membrane processes, have an important disadvantage: membrane fouling [6]. As a consequence of fouling, the permeate flux decreases [7] (lower productivity) and the process increases its operating costs [7] (due to the increase in energy costs [8] and the need of frequent membrane cleaning) and its maintenance costs [9] (due to lower membrane lifetime [10]).

Currently, studies show that the best UF membranes for secondary treatment effluent (STE) from a municipal wastewater treatment plant (MWWTP) are hollow-fiber (HB) membranes [11,12]. This kind of membrane is widely used for large-scale water and WWTPs due to their high active surface/volume ratio [13].

The characteristics of a STE from a MWWTP are very variable because they depend on the efficiency of the secondary treatment, which will be influenced by wastewater characteristics and the type of the implemented biological treatment and their operating conditions. Thus, a correct modeling of the UF process may be an appropriate step for selecting the best operational conditions to minimize membrane fouling.

Soluble microbial products (SMP) as a part of extracellular polymeric substances (EPS) have been identified as the main membrane foulant [14]. They are released by the biomass in the biological process, and polysaccharides and proteins are their main components [14]. Thus, these substances have been used by different authors in the literature to model STE. Nataraj et al. [15] studied the fouling mechanisms with solutions containing a polysaccharide, and Nguyen and Roddick [16] used proteins and polysaccharides since they seemed to be the main responsible molecules for membrane fouling. These authors worked with xanthan, actigun CS11, and glucan. However, for the simulation of STEs, binary mixtures protein/polysaccharides have been more frequently used. Particularly, the behavior of bovine serum albumin (BSA)/dextran mixtures was reported by different authors [14,17,18].

Other authors include in their simulated solutions humic acids [3], although this kind of substances is more often used when fouling phenomena of UF membranes processing surface water are studied, since humic and fulvic acids are important components of the natural organic matter [19].

In order to achieve a synthetic model wastewater composition that could mimic the UF fouling trend of the HB membrane, different combinations and concentrations of model compounds were tested in this work. The model proteins used were whey protein concentrate (WPC) 45% and BSA, and the carbohydrates tested were dextran and xanthan. WPC has been also studied by other authors, but with the aim of studying the membrane fouling in applications of the UF to the dairy industry [20–22].

Besides, the UF fouling trend of the selected simulated wastewater was compared to that of the STE for different transmembrane pressures (TMPs) and cross-flow velocities (CFV). The experimental conditions were selected according to previous literature. Thus, low TMP were selected according to [10] and CFV between 0.59 and 2.96 m/s were chosen in the range proposed by Tasselli et al. [23] and Vincent et al. [24].

2. Materials and methods

2.1. Feed solutions

The feed solutions to UF module used were a STE from a MWWTP located in Valencian Region (Spain), and different model solutions consisting of either a binary mixture polysaccharide/protein or WPC at different concentrations. The proteins used were: BSA from Sigma–Aldrich, and WPC from Reny Picot (45% w/w). The carbohydrates used were dextran (250,000 Da from VWR International Ltd) and xanthan gum (from xanthomonas campestris, provided by Sigma–Aldrich). All model solutions were prepared using tap water, and proteins and polysaccharides were dispersed with gentle stirring. These model solutions were prepared before the UF tests and they were also stirred during the tests. Proteins and carbohydrates concentrations varied in the range of 10–18 and 5–9 mg/l, respectively.

It is important to note that BSA and WPC may form aggregates. Therefore, their particle size may increase. The aggregates of BSA have a particle size of 6–12 nm [14]. Besides, the isoelectric point of BSA is around 5.00 [25].

WPC may contain a variety of other components apart from proteins. Some of them are phospholipids, lipids, minerals, and sugars. The WPC can form aggregates which consist of proteins or a mixture of proteins with other components from whey [26].

Dextrans have hydrophilic properties and they have good water solubility, low toxicity, and certain inactivity [14].

Xanthan gum is an anionic polysaccharide [27]. In addition, xanthan gum has good water solubility [28].

Due to STE composition variability, different samples of STE were analyzed. The parameters measured were the concentration of proteins and carbohydrates and chemical oxygen demand (COD).

The COD was measured using the kits and a thermoreactor model "TR300" both from Merck. The proteins concentration was determined by a MicroBCA assay (Bicinchoninic acid protein assay micro) from Applichem. Carbohydrates concentration was determined by the *anthrone* (9, 10 dihydro-9-ketoanthracene) method (reagent from Panreac).

2.2. Particle size distribution (PSD)

Particle size of model foulants was measured. The equipment used to determine the PSD was a Zetasizer Nano-ZS 90 from Malvern. This equipment measures the particle size by laser diffraction.

2.3. Laboratory scale plant

Fig. 1 illustrates the scheme of the laboratory UF plant used in the experiments. The UF module was *Norit X-flow T/RX-300*:

This plant allows TMP and CFV to be fixed independently. Moreover, the temperature regulator ensures a constant temperature.

The HB membrane used was a *UFCM5* from *Norit X-flow*. The properties of this membrane, provided by the manufacturer, are shown in Table 1.

2.4. UF tests

During the tests, the temperature regulator kept the temperature constant. Data were logged in a programmable logic controller.

During the UF tests, the feed tank was stirred; the retentate and permeate were both returned to the tank, and the permeate flux was monitored.

Two series of UF tests were performed. The aim of the first set of experiments was to select the wastewater composition that better represented the STE UF performance. This first set of experiments was performed at a TMP of 70 kPa, a CFV of 1 m/s [29], and a temperature of 21 °C.

Once the best model wastewater was selected, the second set of experiments was carried out. In this set of experiments, TMP and CFV were varied to check

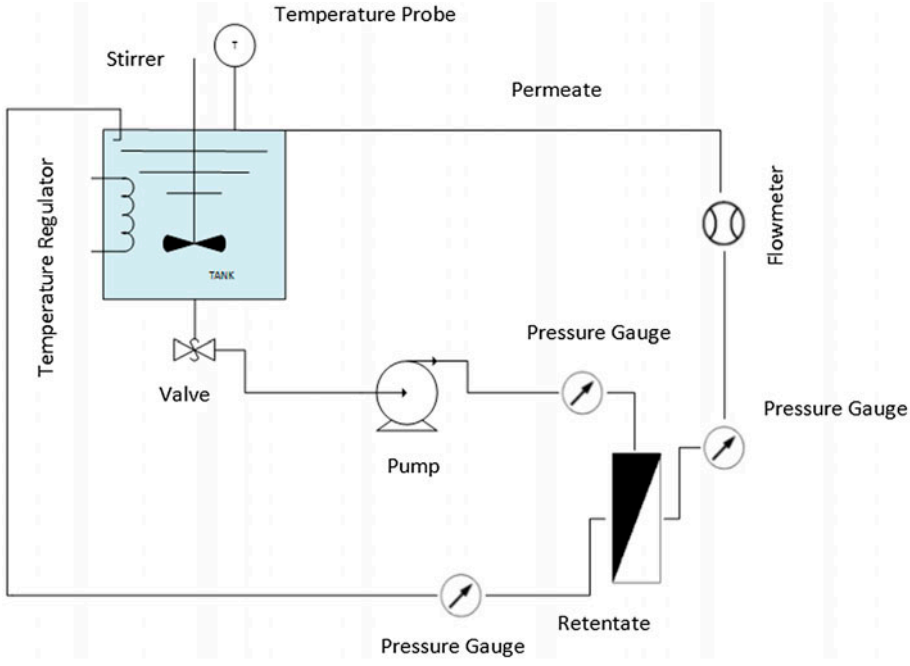

Fig. 1. UF laboratory scale plant scheme.

Table 1
Properties of the HB membrane

Diameter	1.5 mm
Molecular weight cut-off (MWCO)	200,000 Da
Active area	0.04 m^2
Material	Blend of polyethersulfone/ polyvinylpyrrolidone (PES/PVP)
Configuration	Inside–out
Charge	Negative
Hydrophilic properties	Yes
Pore size	21 nm
Isoelectric point	4.2

whether the selected model solution represented the STE for different experimental conditions. These experimental conditions are in the range of 62–100 kPa for TMP and in the range of 0.80–1.2 m/s for CFV.

In order to evaluate the effect of the operating parameters (TMP and CFV) on membrane fouling, the feed water composition should be the same for all the experiments. Due to the fact that the real wastewater has intrinsic variable composition, a model wastewater, which is capable of representing the behavior of STE, was proposed.

2.5. Membrane cleaning

The cleaning protocol was performed at the lowest TMP and the highest CFV achieved in the laboratory scale plant.

The cleaning protocol steps were as follows:

(1) A first rinsing of 30 min at 25°C with deionized water.
(2) A chemical cleaning with a cleaning solution consisting of 154 ppm of NaClO and 0.5 mol/l of NaOH (Panreac, Spain) in deionized water. The chemical cleaning was performed at 40°C.
(3) A second rinsing under the same conditions as the first rinsing.

The cleaning protocol was designed in order to clean the membrane fouled by the STE (real wastewater) and the synthetic wastewater.

After cleaning, the hydraulic permeability was evaluated to ensure that initial membrane conditions were restored.

3. Results and discussion

As explained above, several STE samples were analyzed for COD, protein, and carbohydrate concentration. Their mean concentration values were 38.9 mg/l for COD, 16.5 mg/l for proteins, and 7.3 mg/l for carbohydrates. These values are very similar to those obtained by other authors. Thus, Janssen et al. [30] reported in different samples 27.1, 19.5, and 23.3 mg/l for COD, and 16.9, 25.3, and 29 mg/l of SMP expressed as the sum of proteins and polysaccharides. The pH of STE was also measured and its value was 7.11. Then, model wastewater solutions were prepared by combination of different proteins and carbohydrates. Their concentrations were selected in a way that the measured values of proteins, COD, and carbohydrates of the simulated solutions were as similar as possible to those of the STE.

Membrane permeabilities before each UF test were not exactly the same because the cleaning efficiency could not reach 100% in all the tests. For this reason, the permeate flux of the membrane was normalized according to Eq. (1):

$$J_N = J \cdot \frac{R_0}{R_m} \quad (1)$$

In Eq.(1), "J" is the permeate flux obtained during the test, "J_N" is the normalized permeate flux, "R_0" is the resistance of the membrane before its first use, and "R_m" is the membrane resistance before each test. The values of R_0 and R_m were determined using deionised water.

The parameter "R_m" was calculated according to Eq. (2):

$$J_w = \frac{TMP}{\mu \cdot R_m} \quad (2)$$

where J_w is the pure water flux (with deionised water). The value of R_m was determined by a permeability test with deionised water, measuring the flux at different TMPs. Regarding R_0 value, this was determined when the membrane was new and the experimental procedure was the same as the one used to determine R_m.

Fig. 2 illustrates the evolution of the permeate flux with the time when the STE was ultrafiltered. As expected, a sharp flux decline occurred during the first minutes, meanwhile an almost constant flux was maintained in the rest of the experiment (approximately 24 L/m^2 h).

Data of the fouling tests with model solution can be found in Ref.[31].

Table 2 shows the comparison between every simulated wastewater and the STE from the point of view of the flux evolution with the time. In order to

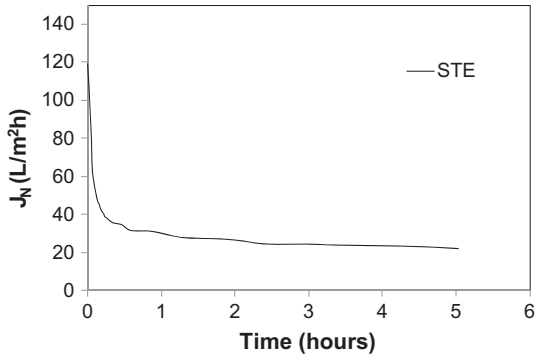

Fig. 2. Evolution of the normalized permeate flux of STE at 70 kPa and 1 m/s.

compare the performance of the simulated wastewater in the prediction of the STE behavior, flux measurements have been divided into two groups: Permeate fluxes in the initial part of the UF including the sharp flux diminution (initial decline) and the permeate fluxes measured when an almost constant flux was reached (steady state).

In this way, values of R-squared (R^2) and standard deviation (SD) calculated by comparing every simulated wastewater with STE can be observed in Table 2. R^2 total means the value of the regression coefficient calculated for all the measured fluxes in the UF tests, whereas R^2 initial decline and R^2 steady state were calculated for the fluxes of the initial decline and of the steady state, respectively. Identical nomenclature has been adopted for the calculated SDs.

R-square definition was applied to the flux vs. time curves of simulated wastewater and STE, which implies considering the sum of the squares of the differences of every pair of flux points for each time.

The model solutions consisting only of WPC 45% at concentrations of 10 and 13 mg/l showed smaller values of total R^2 than the other solutions, which were prepared with a mixture of proteins and carbohydrates. R^2 slightly increased with the concentration of WPC 45%. However, the addition of carbohydrates to the synthetic solutions led to a better fitness between flux data, what was due to the higher flux decline in the first part of the experiments.

On the contrary, the fitness of the steady-state fluxes became slightly worse for the solutions with carbohydrates addition. This led to similar global SD values for all the experiments with WPC, although considerable differences were found in the SD values calculated in the steady-state conditions for the different experiments.

The use of BSA instead of WPC improved the results since the highest total R^2 value was reached and the minimum SD value for the global data and especially for the steady-state data were obtained. Zator et al. [14] concluded in their study that the fouling trend did not only depended on the composition but on the particle size too. They explained that smaller particles produce less fouling and the fouling mechanisms for these particles were internal and external pore blocking, but they considered the internal fouling as the predominant mechanism.

The PSD measurement of WPC solutions (at different WPC concentrations) indicated that the mean diameter was slightly higher than 200 nm. Results have been expressed in scattered by particles intensity (%), which is the magnitude measured by the equipment, and in particle number (%), which is calculated from intensity measurements. In this way, a particle with large diameter detected by the apparatus absorbs a high intensity, but it loses its significance when the PSD in number is calculated. Fig. 3 shows the PSD analysis for WPC both in intensity and in number in %. The high size of WPC in comparison with the membrane pore size (20 nm) leads to a higher external than internal pore blocking.

Although xanthan used also presented particles with diameter values higher than 200 nm, xanthan solutions were polydispersed from the point of view of the PSD. If data are converted to number-weighted

Table 2
Values of R^2 and SD for UF tests (total, initial decline, and steady-state)

Composition	R^2 total	R^2 initial decline	R^2 steady-state	SD total (l/m^2 h)	SD initial decline (l/m^2 h)	SD steady-state (l/m^2 h)
15.75 mg/l WPC & 8.57 mg/l xanthan	0.981	0.978	0.897	25.991	27.029	8.883
15.75 mg/l WPC & 5.5 mg/l dextran	0.850	0.845	0.899	25.314	25.681	1.705
15.75 mg/l WPC & 7.315 mg/l dextran	0.953	0.950	0.897	27.616	28.539	1.039
15 mg/l BSA & 5.5 mg/l dextran	0.984	0.982	0.897	23.428	24.541	0.611
13 mg/l WPC	0.643	0.628	0.903	25.354	25.366	7.174
10 mg/l WPC	0.621	0.605	0.904	27.258	27.361	9.778

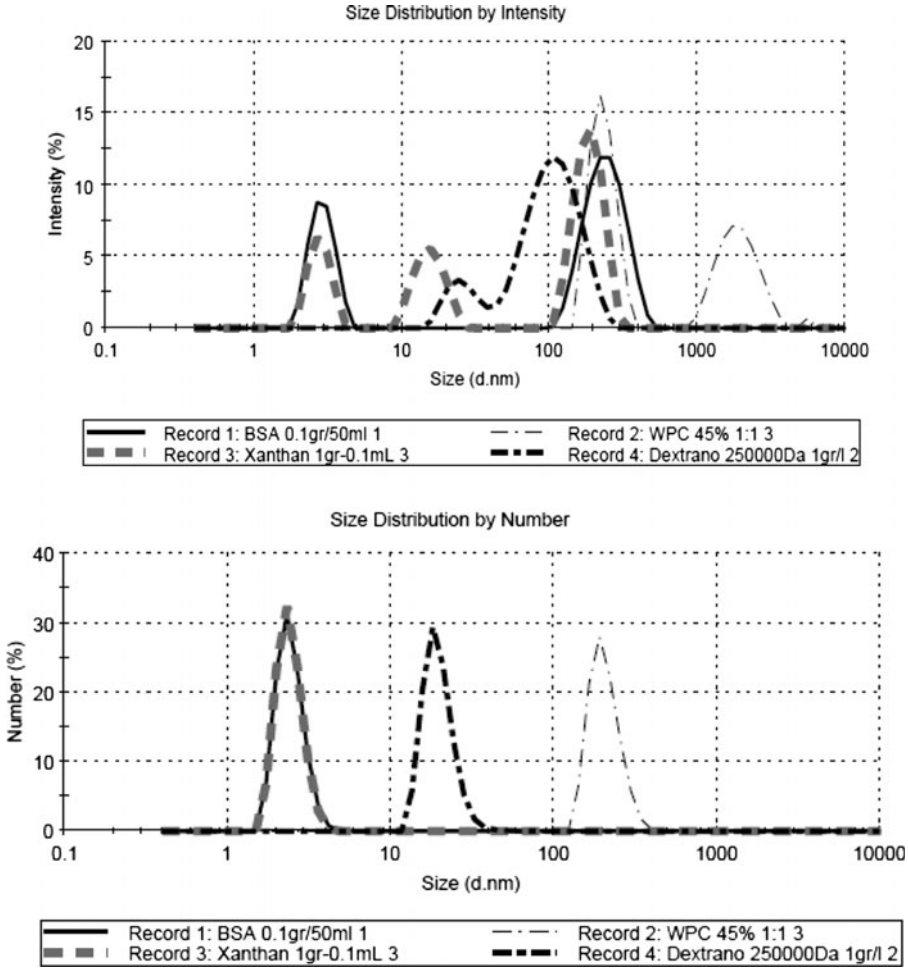

Fig. 3. Size distribution of BSA, WPC, Xanthan, and Dextran: (a) by intensity and (b) by number.

PSD, their analysis yields a peak at 2.5 nm, what could be the reason why internal pore blocking increased for simulated solutions with xanthan entailing a high flux diminution in the initial part of the UF tests. Fig. 3 shows the PSD analysis for the xanthan solution both in intensity and in number in %.

Concerning dextran, it should be commented that the fouling trend of the membrane with STE was mimicked with 7.3 mg/l better than with 5.5 mg/l when a same WPC concentration was used. The improvement in the fitting was due to the values obtained in the period of the initial flux decline. The effect of the dextran addition was very similar to that obtained with xanthan, though the particle diameter was higher (between 20 and 50 nm if particle number is considered. as shows Fig. 3).

The best model wastewater to simulate the STE behavior consisted of BSA (15 mg/l) and dextran (5.5 mg/l). BSA contributed to long-term fouling as it was mainly deposited on the membrane surface. This fact was observed in Fig. 3. BSA has two fractions with different particle size. This is due to the formation of agglomerates of BSA. Therefore, there are some particles of BSA with a higher particle size (200 nm)

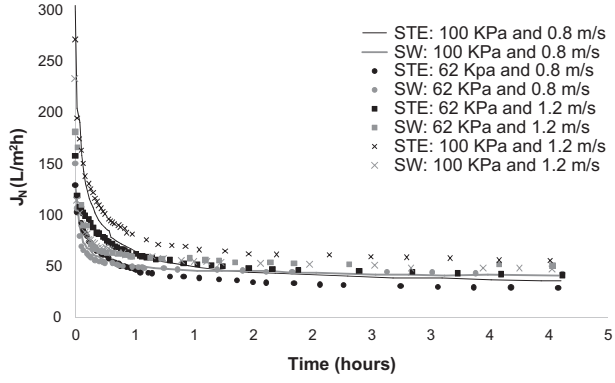

Fig. 4. Comparison between STE and model wastewater UF performance at different experimental conditions.

Table 3
Fitting accuracy of the selected model wastewater in front of the STE at different conditions

Operational conditions	R^2 Initial decline	R^2 Steady-state	SD Initial decline (L/m^2 h)	SD Steady-state (L/m^2 h)
1.2 m/s and 100 kPa	0.832	0.980	42.513	1.818
1.2 m/s and 62 kPa	0.901	0.963	26.221	3.780
0.8 m/s and 62 kPa	0.760	0.824	20.244	7.866
0.8 m/s and 100 kPa	0.806	0.980	36.628	3.143

than the membrane pore size (21 nm) and they contribute to external fouling because these particles are deposited over the membrane surface. However, the particles which do not form agglomerates have a lower particle size (3 nm) than the membrane pore size (21 nm) and they cause internal pore blocking. The addition of dextran contributed to a better fitting in the initial flux decline, contributing to internal and external pore blocking, since membrane pore and dextran have a similar size. This fact was also confirmed by the membrane retention to the dextran that was of 50.4%. Thus, this model wastewater reached the highest value of R^2 and the lowest values of SD.

Therefore, the selected model wastewater was the binary mixture BSA (15 mg/l)/ dextran (5.5 mg/l). This solution was ultrafiltered under different conditions of CFV and TMP and compared with STE UF at the same experimental conditions (Fig. 4). Table 3 summarizes the R^2 and SD calculated values for the data of the three comparison experiments.

R^2 and SD values showed that model wastewater was capable of correctly representing STE in the UF fouling trend in the steady state flux data. With the exception of the test at the lowest values of TMP and CFV, the R^2 value in the steady-state was above 0.96. Thus, it is confirmed that the selected simulated wastewater mimics STE at different operating conditions.

However, the behavior in the initial part of the UF is more difficult to simulate. Initial flux decline with simulated wastewater was always a little sharper than initial flux decline for STE. In Table 3, it can be seen that the deviations in the tests performed at high pressure are higher due to the fact that a higher pressure causes more fouling. In addition, the deviation is higher in the initial flux decline, not in the steady-state flux, because pore internal blocking occurs at the beginning of the UF tests. STE has a complex composition and model wastewater has only two components, so their UF performance was similar but it cannot be exactly the same. The tests were carried out with the same STE samples in order not to vary wastewater composition, but slight changes in STE composition occurred due to organic matter deposition on the membrane or carbohydrates degradation. This did affect to the behavior of the fouling trend in the initial part of the UF tests, and it explains the differences observed that led to R^2 values between 0.76 and 0.90.

4. Conclusions

UF experiments with simulated STEs from MWWTPs are of great importance for the study of the membrane fouling. The first goal to be achieved is the selection of a simulated wastewater mimicking STEs. After testing different protein and carbohydrates solutions, a model wastewater consisting of BSA (15 mg/l) and dextran (5.5 mg/l) was selected to model STE UF performance. Selection was carried out according to the better fitness both in the initial flux decline and in the steady-state parts of the experiments.

It has been proven that BSA formed aggregates whose particle size was higher than the membrane pore size; thereby, the BSA presence in the simulated solution exerted a great influence on long-term fouling. On the contrary, dextran had a similar particle size to the membrane pore size and this compound contributed to both long- and short-term fouling. PSD analysis helped to corroborate it.

It was very difficult to achieve high values of R^2 in the comparison of UF tests of simulated wastewater and STE for different operating conditions. Although fitness was high for the steady-state fluxes, the initial flux decline was more difficult to fit. This was due to the small changes in the STE rather than to the change in the operating conditions.

The optimal simulated wastewater cannot be extrapolated to other STE compositions, but the methodology can be applied to other studies.

Acknowledgments

The authors wish to gratefully acknowledge the financial support of the Generalitat Valenciana through the project "Ayudas para la realización de proyectos I+D para grupos de investigación emergentes GV/2013."

Nomenclature

J	—	permeate flux (L/m^2 h)
J_N	—	normalized permeate flux (L/m^2 h)
R_0	—	resistance of the membrane before the first use (m^{-1})
R_m	—	resistance of the membrane before each test (m^{-1})
μ	—	dynamic viscosity of the water (Pa·s)
s	—	slope of the permeability test (L/m^2 h·bar)
TMP	—	transmembrane pressure (kPa)
CFV	—	cross-flow velocity (m/s)
STE	—	secondary treatment effluent
MWWTP	—	municipal wastewater treatment plant
UF	—	ultrafiltration
EPS	—	extracellular polymeric substances
WPC	—	whey protein concentrate
BSA	—	bovine serum albumin
COD	—	chemical oxygen demand (mg/l)
PSD	—	particle size distribution (nm)
MWCO	—	molecular weight cut-off (Da)
HF	—	hollow-fiber

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