



## Control of fouling in MBRs through nanospheres addition

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### ABSTRACT

In the last decade, the membrane bioreactor (MBR) industry expanded and nowadays this technology is diffused worldwide for wastewater treatment. Nevertheless, membrane fouling is still a critical issue and most research is focused on this aspect in order to control the fouling phenomenon, such as the definition of foulant agents, which are mainly extracellular polymeric substances. One of the main drawbacks related to fouling in MBRs is the sudden jump of the transmembrane pressure, often attributed to the collapse of the fouling layer, which in turn leads to a reduction of the pores size. A potential solution to this problem can be the addition of particles as to reduce the compressibility of the fouling layer through the engineering of the cake structure. Aim of the present work is to test this hypothesis through the addition of nanospheres of different diameter in a hollow fiber MBR unit at lab scale. The nanospheres are inert and non-compressible, and have been chosen for their capability to form well-structured layers. In order to analyze the MBR filtration performance, a synthetic solution has been prepared, in which a foulant agent (sodium alginate) has been spiked. A filtration model has been provided as to interpret the results and their eventual dependence on diameter and concentration of the nanospheres. Results obtained at constant flux in dead-end mode show that the presence of particles changes cake resistance and leads to the formation of non-compressible fouling. However, the addition of particles leads to better filtration performances, no matter what the diameter or concentration may be. Nevertheless, further research is required with the aim of testing the hypothesis with real wastewater. This study may, therefore, enlarge the choice of membrane fouling reducers by taking into consideration their ability to form more structured fouling.

*Keywords:* Extracellular polymeric substances (EPS); Membrane bioreactor (MBR); Membrane fouling; Nanotechnology; Wastewater

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## 1. Introduction

Membrane bioreactors (MBRs) are nowadays considered as one of the best available technologies for municipal wastewater treatment. This aspect is confirmed by a steady increase in the number of MBR plants, of roughly 11% per year according to [1–3]. Several driving forces are responsible for this trend, such as more stringent limit values for macropollutants discharge into the environment, occurrence of xenobiotics in wastewater treatment plants effluents and constant migration toward urban areas, which in turn would lead to higher pollution loads in wastewater. In addition, MBR technology development is also due to the fact that membrane costs are decreasing [4]—therefore, lowering capital expenditure—and operational expenditure has been reduced as well, because of a reduction in energy demand subsequent to the implementation of new protocols. Nevertheless, membrane fouling is still a critical issue for the process, as cleaning measures are required in order to remove solids deposited on the membrane [5,6].

During the last few years, MBR fouling has been extensively studied by focusing on the different possible factors influencing it (such as sludge characteristics, operating conditions [7], hydraulic conditions [5], aeration [8], and membrane materials [9]). Research on fouling in MBRs provided water professionals with relevant information, and as a consequence operating procedures were introduced in order to control this phenomenon. Among them, module design and aeration conditions have been optimized; ON/OFF filtration cycles (relaxation), module backwashing, and chemical cleaning have been as well in an effort to mitigate membrane fouling [10].

On the other hand, it must be noted that knowledge on both fouling mechanisms and compounds that are responsible for its formation is still a key issue in MBRs. This lack of knowledge is due to the fact that wastewater is a complex suspension containing an array of micro-organisms and their metabolites, such as extracellular polymeric substances (EPS) and soluble microbial products (SMP), produced by the micro-organisms inside the membrane bioreactor. EPS and SMP compounds are the construction materials for micro-organism aggregates (such as flocs, biofilm, etc.) and consist of a large array of organic polymers (proteins, polysaccharides, nucleic acids, and humic substances) [11]. Different fouling mechanisms (namely pore blocking, cake formation, and adsorption) have been proposed by researchers in order to explain the way these metabolites affect membrane performance, but there is evidence that each compound can behave differently from the others. A

number of proteins and polysaccharides have been identified as EPS, and there is evidence that sodium alginate ( $\text{NaC}_6\text{H}_7\text{O}_6$ ) can be used as a model for EPS-related fouling development. Polysaccharides often form a significant component of EPS and have higher molecular weights and a wider molecular weight distribution than proteins. Alginate is a microbial polysaccharide, consisting of a linear copolymer composed of 1–4 linked [3-D-mannuronic acid, C-5 epimer and a L-guluronic acid in varying proportions. *P. aeruginosa* alginates have already been used to investigate the function of EPS in biofilms.

Concerning fouling, the most prevalent associated issue is a sudden jump of the transmembrane pressure (TMP). This TMP jump has been attributed to hydrodynamics [12], or to a sudden change in biomass or cake layer structure by compressibility effects [13,14]. Moreover, recently, it has been shown that the TMP jump could be due to an increase in EPS concentration at the cake layer-membrane interface, probably because of the death of bacteria [15]. This issue led to a wide production of studies that aimed to investigate the respective role of each fraction of the biofluid in terms of filtration resistance. The possible role and contribution of EPS is still not clear, even if several works suggested the biomacromolecular compounds as the most problematic ones in terms of fouling development, which are identified as protein substances and polysaccharides that may form a highly hydrated gel over the membrane [16–19]. In [10], it has been reported that even though these results are still controversial, there is a general agreement on the role played by carbohydrates and proteins as foulants in MBRs.

One of the most relevant aspects about TMP increase—in the hypothesis of constant flux—consists in the compression of the fouling layer. As previously demonstrated [20–22], the addition of adsorbent fine particles (activated carbon, zeolites) in MBRs allows to enhance filtration performances and to delay fouling [23–26]. Similarly, it has been discussed the introduction of using colloidal suspension to control fouling in MBRs [27]. In that research work, it has been shown that microfiltration membrane coated with nanoparticles (i.e. fullerene C60) could reduce both the microbial attachment and the respiratory activity during short-term filtration experiment. Authors proposed several patterns to explain the performance improvement: (a) adsorption of foulants to the powdered activated carbon (PAC) and thus, protection of the membrane surface from foulants deposition [21,28]; (b) scouring effect removing foulants deposited at the membrane surface [25,26]; (c) improvement of colloids and macromolecules

flocculation leading to stronger floc strength [27]; (d) bacterial inhibition by colloidal aggregates [27,29,30]; and (e) formation of a more porous and less-compressible cake layer [21,31].

Aim of this paper is to focus on this last statement and to investigate about how an addition of fine inert particles can impact on the filtration performances of the engineered fouling layer. More specifically, the proposed question is whether it is possible to engineer the fouling layer structure in an MBR to control fouling phenomenon. Thus, the present paper introduces an experimental study of the artificial structuring of a cake layer formed during the filtration of a synthetic solution (containing sodium alginate, a polysaccharide classified as EPS) with the addition of synthetic particles of different diameters and at different concentrations. Latex nanospheres were selected for this study; as elsewhere reported [32], the use of small synthetic particles could form well-structured particles array over the membrane surface during filtration. A recent study [33] showed that the addition of nanoparticles can improve MBR performance and this work aims to provide additional information about this topic. Such particles (deposited at the membrane) have been extensively studied in terms of processing and properties [32,34], but there is little information about their performance during a filtration process. Filtration experiments aimed to characterize the impact of the addition of the nanoparticles on the TMP increase.

## 2. Materials and methods

### 2.1. Chemicals

MBR influent was prepared using deionized water (Ultrapure Water, Millipore, US) and sodium alginate (Sigma–Aldrich, USA). The feed concentration for the polysaccharide was determined using the calibration curve at an absorbance  $\lambda_{480}$  (Perkin-Elmer, UV–vis Lambda 12, US), in order to obtain a D-Glucose concentration of  $20 \text{ mg L}^{-1}$ , according to research studies already published that indicate such a concentration as typical of urban wastewater [5,9,10,15]. The feed polysaccharide suspension was prepared adding  $20 \text{ mM NaCl}$  (Carlo Erba, Italy),  $1 \text{ mM NaHCO}_3$  (Nacalai Tesque, Japan), and  $64.1 \text{ mg L}^{-1} \text{ Na}_2\text{C}_2\text{O}_4$  (Sigma–Aldrich, US). After entering the reactor through the inlet tube, the solutions passes over the membrane, and permeate exits the system. In order to determine the sodium alginate size distribution, samples have been collected in the MBR system and analyzed using a Mastersizer (Malvern Instruments, Mastersizer 2000, US).

### 2.2. Nanospheres

In order to analyze the effect of the addition of inert particles on the cake resistance in MBRs, synthetic nanospheres (Duke Scientific, US) of different average diameter (300, 500, and 700 nm, given by the manufacturer) have been chosen. The tested elements consisted of polystyrene particles selected mainly because of their small diameter as well as their inert behavior. One of the most interesting properties of these particles is that they might pack, during filtration of fluids, at the membrane interface in regular arrays (i.e. hexagonal and/or cubic closest packing). Therefore, these particle layers have specific porosity (depending on the packing) and specific pore size, or interstitial spaces (depending on particle diameter). Each kind of nanosphere was used at two different concentrations ( $100$  and  $200 \text{ mg L}^{-1}$ ). The  $100 \text{ mg L}^{-1}$  concentration has been selected since it had been previously used [33]; moreover, in order to investigate the effect of a higher nanospheres concentration, the  $200 \text{ mg L}^{-1}$  has been tested as well. Characteristics of each kind of particles are given in Table 1. An analysis of the particles layer shows that smaller diameters provide narrower minimum pores and bigger particles have higher ratios to membrane porosity, while the nanospheres layer minimum pores to diameter ratio does not change whatever the used diameter.

As reported elsewhere [32], the pore size of such particle layers is primarily governed by the particle size. Thus, the pore size of the particles array, defined as the interstitial space between particles, can be calculated from simple geometry and is equal to 130, 210, and 300 nm for the 300, 500, and 700 nm diameters, respectively. Such pore size is always wider than the virgin membrane pore size ( $0.04 \mu\text{m}$ ).

In Fig. 1, the ultrafiltration membrane is shown when no nanospheres are added and fouling layer compression takes place, as well as the engineered fouling layer composed of nanospheres and EPS.

### 2.3. Experimental setup

A laboratory-scale aerobic MBR, consisting of a 5-L storage tank followed by a 2-L cylindrical reactor, was used for the treatment of the synthetic solution. The tank was equipped with a stirrer rotating at  $500 \text{ min}^{-1}$  (VELP, AREX, Italy). The membrane module employed was a hollow fiber MBR, ZW-1 (Zenon GE, US), of pore size  $0.04 \mu\text{m}$  and of surface area  $0.047 \text{ m}^2$  to retain solids in the reactor. The membrane has been produced in PVDF (Polyvinylidene fluoride).

The polysaccharide suspension was held in a 5-L feed tank equipped with a stirrer rotating at  $500 \text{ min}^{-1}$

Table 1  
Nanoparticles properties

|                                  |      | Nano300 | Nano500 | Nano700 |
|----------------------------------|------|---------|---------|---------|
| Mean diameter, $D$               | (nm) | 296     | 496     | 707     |
| Nanospheres layer min pores, $r$ | (nm) | 130     | 210     | 300     |
| Layer pores—diameter ratio       | –    | 2.30    | 2.38    | 2.33    |
| Layer pores—ZW-1 porosity ratio  | –    | 3.25    | 5.25    | 7.5     |

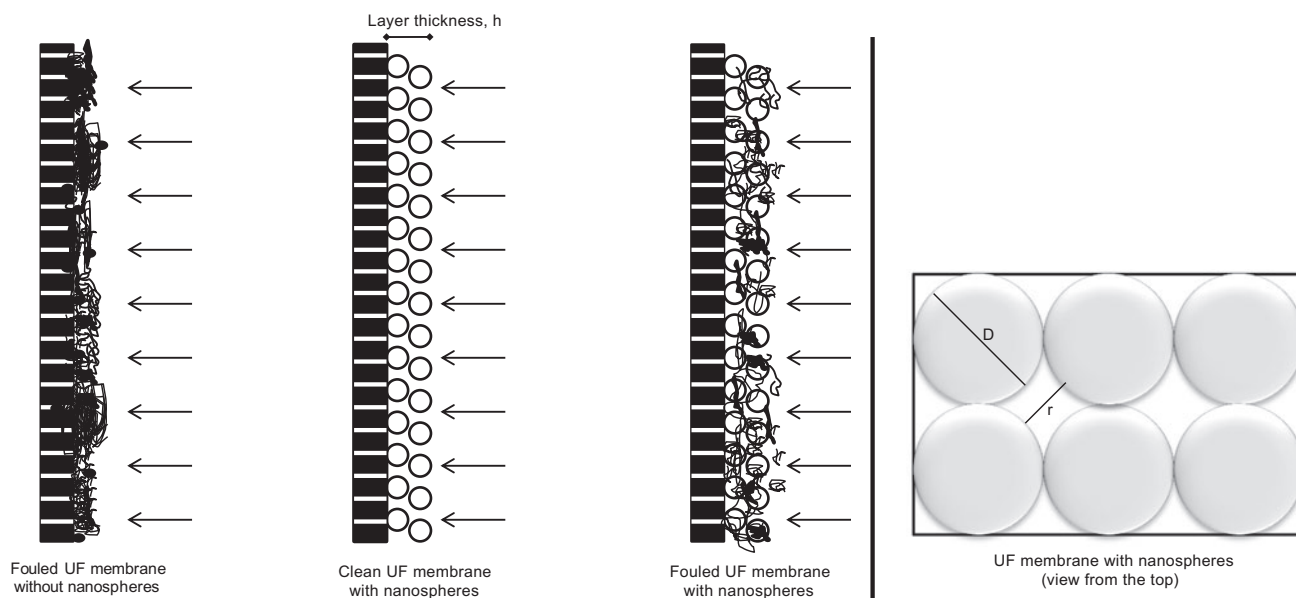


Fig. 1. Effect of nanospheres addition on fouling compressibility and detail of packed particles onto the membrane.

and was fed to the inlet port of the membrane module by a peristaltic pump (Watson Marlow 323, UK). An identical peristaltic pump was used to draw permeate from the immersed membrane unit. To keep permeate flux constant, subsequent adjustments of the pump velocity were made, measuring the permeate flow and accordingly modifying the pump RPM value.

In order to investigate the effect of nanospheres addition on the MBR filtration performance, experiments have been carried out using different nanospheres at a range of concentrations, keeping the same sodium alginate concentration in the feed solution. The experiments were performed at a constant flux of 20 LMH for 180 min, during which the TMP was continuously (every 2 s) monitored using a pressure transducer (PCE-932, PCE Instruments, Italy). Each diameter–concentration combination was repeatedly tested in order to assess the reproducibility of filtration tests. After each test, the MBR module was cleaned through soaking in sodium hypochlorite (Sigma–Aldrich, US) for 8 h and then backwashed

with deionized water. The initial value of the peristaltic pump was set at 8 RPM, and after chemical cleaning the flux recovery was tested using this value as a benchmark. A large storage tank (5 L) was connected to the filtration cell to allow longer filtration times. Experiments were performed at room temperature ( $20 \pm 3^\circ\text{C}$ ), which was monitored using a multiparameter probe (Hanna Instrument, 9828/4, US). The experimental setup is shown in Fig. 2.

### 3. Results and discussion

#### 3.1. Filtration of sodium alginate

Filtration experiments were performed at first with sodium alginate solutions according to the procedure described in Section 2.1.

After 180 min of filtration at 20 LMH, the TMP attains a value of roughly 0.9 bar, indicating severe membrane fouling. This observation is in line with the common assessment in the literature that

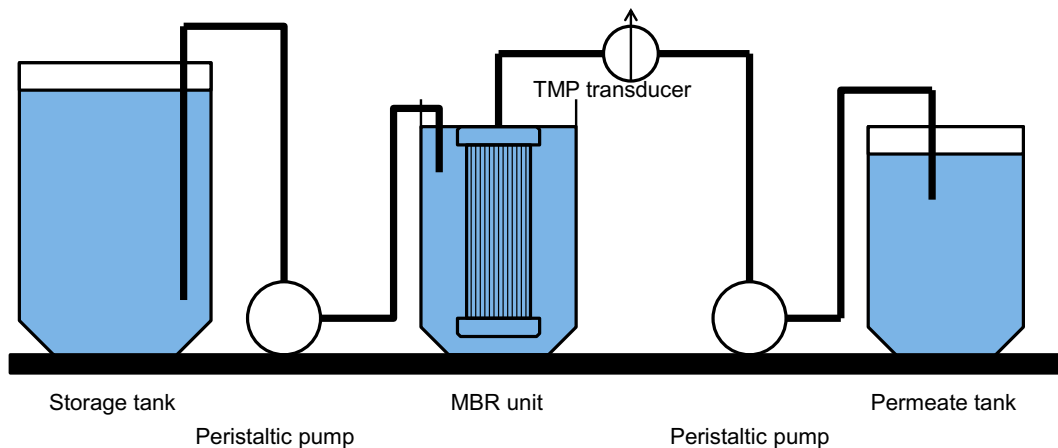


Fig. 2. Lab-scale MBR experimental setup.

polysaccharides are potentially one of the most significant fouling species in wastewaters and natural waters. Similar behavior has been observed with other types of organic foulants as well as colloidal particles. It has to be noted that the flux recovery after the chemical cleaning is almost total. This suggestion is in line with previous findings [35–37] regarding sodium alginate fouling in MBRs. During filtration of the synthetic solution, sodium alginate deposition on the membrane led to highly compressible fouling. This fouling was also easily removed through chemical cleaning and backwashing.

### 3.2. Effect of the nanoparticles addition on filtration performance

Once assessed the behavior of sodium alginate in terms of TMP increase, the nanospheres have been added to the MBR unit during the filtration of the synthetic solution in order to check whether the cake resistance could be reduced. A sharp decrease of the TMP trend has been confirmed for each investigated diameter, as shown in Fig. 3.

From the analysis of the TMP trends, it can be inferred that as a general assumption, the addition of nanoparticles reduced the pressure required overtime to allow the filtration of a fixed flow (20 LMH). The maximum reduction in TMP after a filtration time of 180 min was attained using the maximum diameter (700 nm) at the highest concentration (200 mg L<sup>-1</sup>). It must be noted that both diameter and concentration play a major role in cake resistance, and therefore, in TMP increase overtime; this aspect will be discussed in the next section. According to the results, the presence of nanospheres modifies the cake layer structure—more specifically, its compressibility—and makes

the fouling becoming non-compressible. This is also confirmed by geometrical considerations, as the 100 mg L<sup>-1</sup> concentration provided a layer thickness of 8.45 μm under the hypothesis that all the nanoparticles were deposited onto the membrane; the same approach was pursued for the 200 mg L<sup>-1</sup> concentration, which led to a nanospheres layer thickness of 17.07 μm.

Moreover, the size distribution of the sodium alginate solution is shown in Fig. 4. The polysaccharide presented a mean dimension during the experiment (180 min) of 213 nm.

This indicates a tendency to occlude all the pores at the top of the nanospheres layer when nanospheres with a diameter of 300 nm are added, since  $r = 130$  nm. On the opposite, larger diameters allow sodium alginate filtration through the nanosphere layer, reducing the TMP increase. This means, in turn, that lower compressibility values are attained when thicker nanospheres layers are formed onto the membrane.

### 3.3. Filtration modeling

In order to determine the resistance due to the membrane ( $R_m$ ), a set of filtration tests have been performed using only deionized water. The corresponding resistance ( $R = R_m$ ) was equal to:

$$R = \frac{TMP}{J\eta_w}$$

where  $J$  is the flux,  $\eta_w$  represents the dynamic viscosity of the water, and TMP is the monitored pressure. The resulting  $R_m$  was equal to  $1.8 \times 10^{11} \text{ m}^{-1}$ . This value is in accordance with other published studies [5].

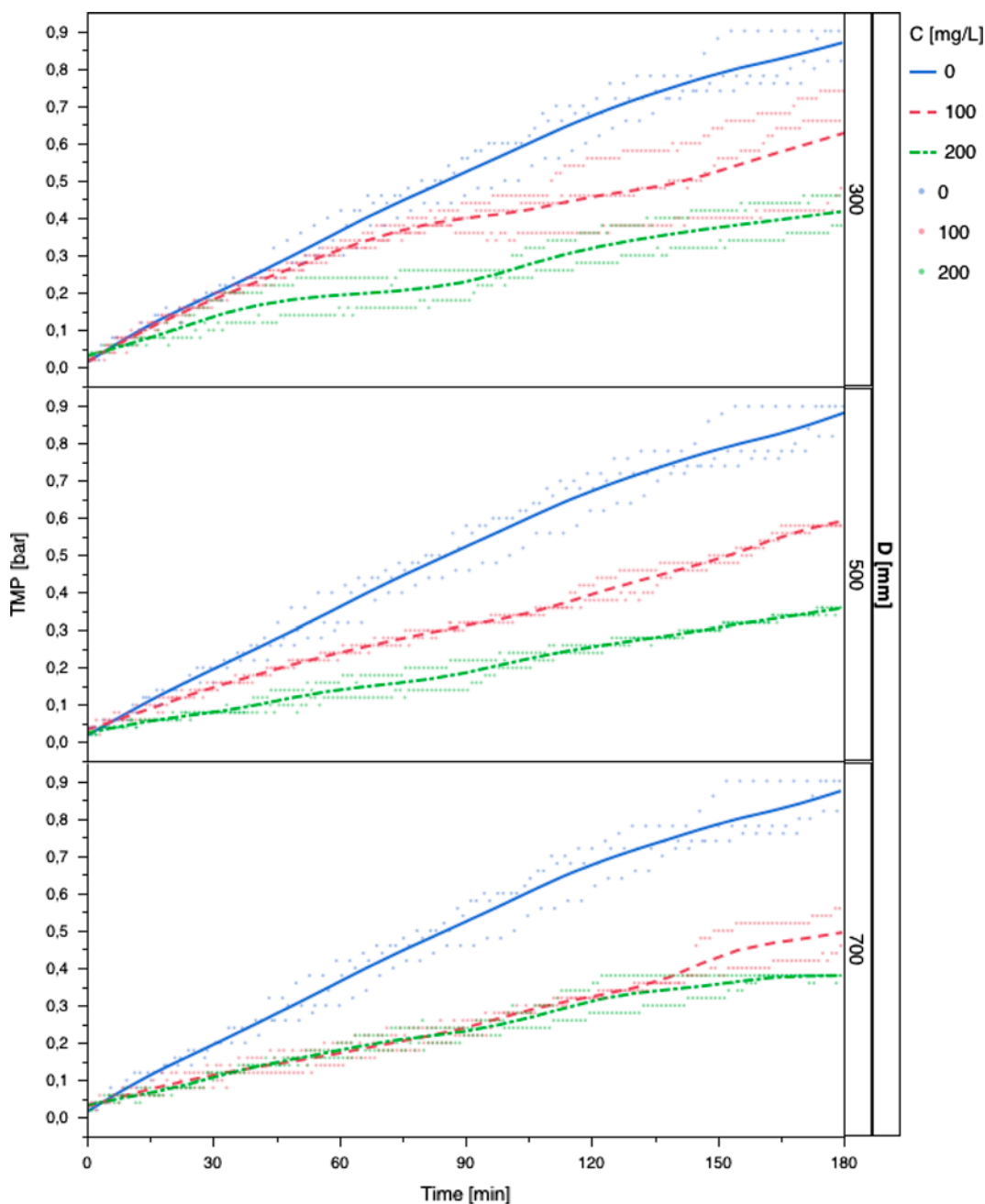


Fig. 3. TMP trends overtime at nanospheres different concentrations during the filtration of a synthetic solution containing sodium alginate ( $20 \text{ mg L}^{-1}$  as D-glucose): continuous lines represent average values, dots represent experimental data.

A set of preliminary filtration tests dosing nanospheres in deionized water and performed at the constant flux of  $20 \text{ LMH}$ , showed that the particle arrays induced an initial permeate flux decline not dependant on the particle size or concentration; then, the TMP trend kept steady.

The contribution in terms of resistance due to the addition of nanoparticles has been evaluated through

filtration of deionized water in which nanospheres (whose diameter is 300, 500, or 700 nm) at different concentration ( $100$  or  $200 \text{ mg L}^{-1}$ ) were dosed in the MBR module. Obtained TMP trends showed the same resistance, due to the fact that after a few seconds a steady value for TMP was attained ( $0.06 \text{ bar}$ ).

Whatever the particles, no difference in terms of TMP increase was observed. Moreover, at the same

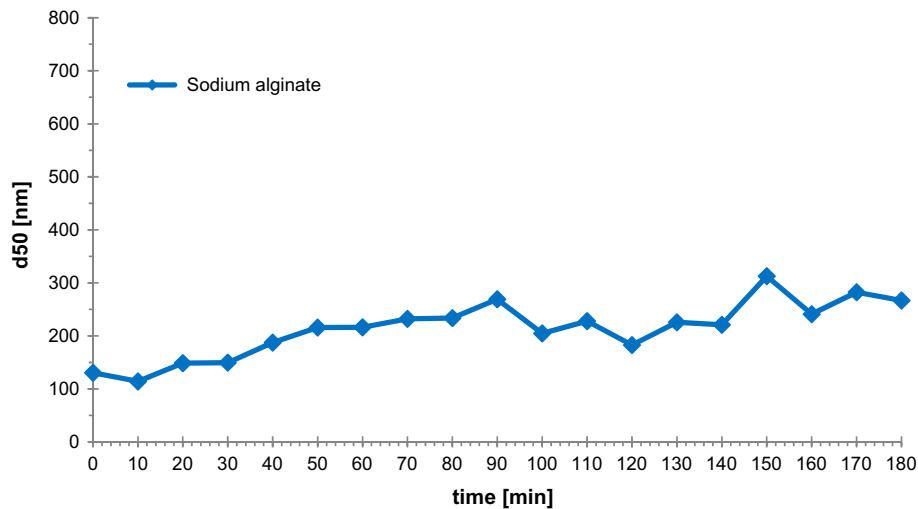


Fig. 4. Sodium alginate  $d^{50}$  (nm) overtime in the lab-scale MBR.

deposited mass (100 or 200 mg L<sup>-1</sup>) each particle type showed the same behavior in terms of resistance. As a consequence, it can be argued that TMP has no effect on the nanospheres layer resistance, since the formed layer is non-compressible. The flux recovery after membrane cleaning is total when deionized water is filtered dosing nanospheres.

The resulting resistance ( $R_n$ ), obtained from the previously explained equation in which  $R = R_m + R_n$ , is then equal to  $5.4 \times 10^{11} \text{ m}^{-1}$  for each tested diameter and concentration. This is probably due to the fact that the pores of the nanospheres layer are almost equal in terms of mean diameter, and therefore, no variation in resistance has been reported for deionized water filtration.

In a previous study [38], authors applied a sectional approach to account for the uneven cake formation in determining total filtration resistance. The authors divided the membrane surface into equal fractional areas, and calculated separate total resistances,  $R$ , for each section, which can consist of constant resistance  $R_0$ —due to membrane resistance  $R_m$  and nanoparticles layer resistance  $R_n$ —and time-dependant resistance due to cake formation,  $\kappa t$ . The total resistance in each section is then described as:

$$R = R_0 + \kappa \times t \quad (1)$$

where  $\kappa$  is a parameter depending on both nanospheres diameter and concentration.  $R_0$  is equal to  $R_m$  when no nanospheres are added, while in the opposite case  $R_0 = R_m + R_n$ .

The sectional resistance model was developed using a partially analytic approach. By dividing the

membrane into sections and considering the resistance in each section, the model accounts for uneven cake formation stemming from varying shear distribution along the membrane. Previous studies [38] were conducted using a submerged MBR, which filtered glucose-based synthetic wastewater using different sludge concentrations, filtration fluxes, and aeration intensities, and a similar model was employed. Comparison of the measured and computer-simulated transmembrane pressure over MBR operation time revealed that the model is able of capturing general trends and modeling of membrane fouling phenomenon.

From the comparison of the model with the experimental results, it has been possible to determine the value of  $\kappa$  as introduced in the model. In Table 2, the comparison between experimental results and developed model is reported in terms of  $R^2$ , from which it can be inferred that the model properly fits and can forecast TMP trends over time.

In Fig. 5 results in terms of  $\kappa/\kappa_0$ , being  $\kappa_0$  the empirical parameter when no nanospheres are added, are reported in function of nanoparticle diameter and used concentration.

Table 2  
Model/experimental results  $R^2$  for each tested combination

|                           | $R^2$ | $D$ (nm) |       |       |       |
|---------------------------|-------|----------|-------|-------|-------|
|                           |       | 0        | 300   | 500   | 700   |
| $C$ (mg L <sup>-1</sup> ) | 0     | 0.991    | –     | –     | –     |
|                           | 100   | –        | 0.921 | 0.983 | 0.968 |
|                           | 200   | –        | 0.942 | 0.984 | 0.971 |

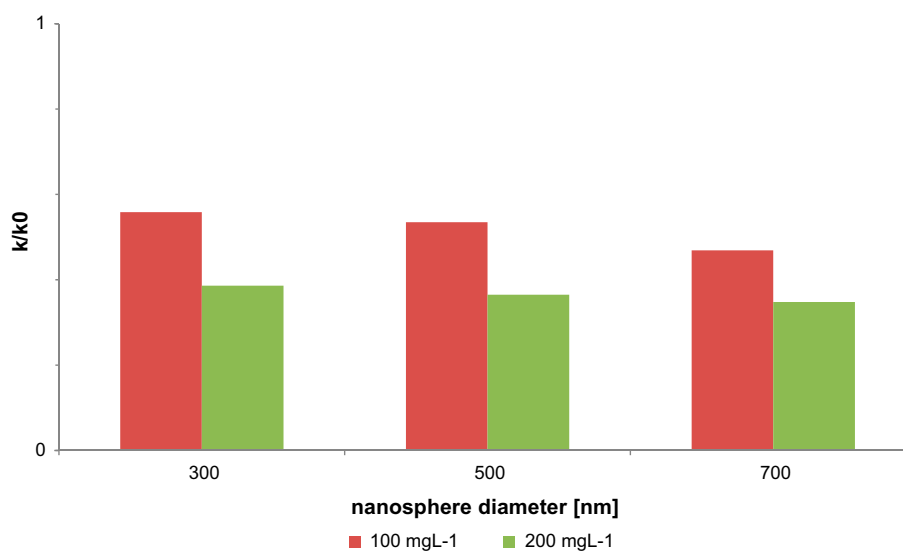


Fig. 5. Values of  $\kappa$  in function of both nanoparticle diameter and concentration,  $\kappa_0$  being the parameter for the experiment without nanospheres addition.

It can be observed that there is a direct correlation between TMP reduction and both nanospheres concentration and diameter; higher concentrations and larger nanospheres led to better filtration performance, in that larger pores allowed sodium alginate to filter through the engineered layer. This is in line with results reported elsewhere [33]; once extremely high concentrations are attained, there is no longer difference in terms of pressure reduction among different diameters, since the layer is thick enough to entrap a major share of the foulants. On the other hand, at lower concentrations (e.g. 100 mg L<sup>-1</sup>) diameters—and therefore pore sizes—still play a fundamental role.

A decrease in  $\kappa$ —and therefore in cake resistance—is reported when a concentration of 200 mg L<sup>-1</sup> is added to the MBR unit. This could be explained by the fact that the thickness of the layer played a major role in the filtration process. Moreover, the cake resistance is influenced by the pore size, in that higher diameters led to a stronger decrease in TMP.

#### 4. Conclusions

This study highlighted how the addition of nanospheres during MBR filtration may change the cake resistance, basically avoiding cake compression and subsequent pores diameter reduction. The presence of a foulant agent, namely sodium alginate, led to a sharp increase in TMP during filtration tests at constant flux. This compound led to a highly compressible fouling because of the influent passing through it, determining a reduction in size of its pores, and a consequent

increase in resistance. One of the major results of this study is that the fouling layer becomes non-compressible when inert particles are introduced in the reactor, and size of the pores can be kept constant over time. Notwithstanding the effect due to the presence of particles, this paper firstly shows that the formation of a non-compressible cake might be an important effect in the improvement of filtration performances. What is more, the formation of a more structured and engineered fouling layer may avoid the layer compression. A reduction in TMP overtime would, in turn, reduce the frequency of chemical cleanings; addition of nanospheres at a concentration of 200 mg L<sup>-1</sup> can beneficially extend filtration periods. This can, therefore, allow the MBR unit to work steadily, providing a high-quality effluent that would benefit the water environment as a consequence [39]. Subsequent water reclamation is therefore possible, reducing water stress in many areas [40–43]. Another finding proposed in this article consists in the dependence of the cake resistance on the diameter and concentration of the nanosphere introduced in the reactor; bigger elements provide larger pores in the cake and therefore attain lower TMP values. In addition, higher concentration of inert elements led to a more marked TMP decrease since the engineered layer thickness increases, preventing the foulant agent to deposit onto the membrane surface. Nonetheless, further research is needed in order to test the cake resistance reduction using aeration in the MBR unit, and therefore considering an additional shear stress in the model; subsequently, the system has to be tested during the filtration of a real



wastewater. The final step of this project will consist in a full-scale application for a sidestream MBR unit. In conclusion, these results show that if the fouling layer structuring is controlled and taken into consideration the amount of chemical compounds added into an MBR could be strongly reduced.

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