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ORIGINAL ARTICLE

Treatment of urban wastewater with pure moving bed membrane bioreactor technology at different filling ratios, hydraulic retention times and temperatures

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Abstract Studies investigating the functioning and possible utility of new wastewater treatment technologies are urgently needed if the requirements of European Directive 91/271/EEC are to be met. Here, moving bed biofilm reactor-membrane bioreactor (MBBR-MBR) technology was studied in a pilot plant of 445 L volume with ultrafiltration membrane (ZW-10) under 10 h and 24 h of hydraulic retention time (HRT) and three filling ratios (20 %, 35 % and 50 %) at temperatures between 2.5 °C and 17.3 °C. Biofilm density ranged between 1510±127 and 3775±247 mg/L carrier. Temperature was the operative variable with most influence in the behaviour of biomass and in organic matter and nitrogen oxidation whereas the filling ratio affected mainly biofilm density. Removal of organic matter and nitrogen increased with the amount of biofilm in the carrier. The amount of biofilm attached under the highest filling ratio was reduced as a consequence of increased collision between carriers, indicating that an optimum rate of filling ratio in this process can be determined. The organic matter removal rate reached 86.4 % and 91.5 % in terms of COD and BOD_5, respectively, and no less than 13.9 % and 13.7 % ammonia and total nitrogen content, respectively, was removed by the system.

Keyword Moving bed · Membrane bioreactor · Filling ratio · Temperature

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Introduction

Two of our society's biggest problems are water pollution and the wasteful use of freshwater resources (Popa et al. 2014). Increasing urbanization, industrial development, and changes in farming practices have caused a huge rise in the consumption of water resources as well as a deterioration in their quality (Wang and Tang 2014). Surface water pollution is a serious problem in many developed countries because of the enrichment of nutrients in water bodies, with discharge of excess organic pollutants, nitrogen and phosphorous substances into natural waters such as rivers and lakes (Plattes et al. 2006). Advanced wastewater treatment technologies are necessary to preserve water quality and to satisfy the limits imposed on the effluent by municipal wastewater treatment plants (WWTP) by the Water Framework Directive (Chave 2001). Water treatment may include mechanical, physical, biological, and chemical methods (Cerrone et al. 2011; Mamoukarris et al. 2014).

Although biological processes are a cost-effective and environmentally friendly alternative to the chemical treatment of wastewater (Mulkerrins et al. 2014; Trapani et al. 2010), these treatments result in continuous production of waste activated sludge, which is disposed of mainly inside treatment plant premises or landfill (Tricolici et al. 2014). The biological treatment currently used most extensively on a global basis is conventional activated sludge (CAS), in which all the biomass in the bioreactor is suspended (Guibaud et al. 2003). CAS has been used since the early 1900s and has become an effective system for organic carbon and nutrient removal in municipal wastewater plants (Kermani et al. 2008). This process is aerobic, requiring a supply of oxygen, and therefore involves higher aeration costs (Mehrdadi et al. 2006) and problems such as settling of the sludge lead to the requirement for large reactors and settling tanks, and biomass recycling (Pastorelli et al. 1999).

In order to improve CAS treatment, the combination of membrane technology with biological treatment using a membrane bioreactor (MBR) suggests an alternative solution for overloaded conventional wastewater treatment plants, replacing the settling tank with membrane filtration (Van der Roest et al. 2002). MBRs represent an attractive treatment technology in wastewater management because they produce a high quality effluent at a very low surface demand (Krzeminski et al. 2012) and reduce the number of pathogens present since the incorporated ultrafiltration membrane has the capacity to retain bacteria and some types of virus (Rodriguez et al. 2011). Indeed, MBRs can be operated at higher concentrations of suspended biomass, resulting in long sludge retention times even at smaller reactor volumes as well as lower sludge production, avoiding problems of sludge bulking (Ahl et al. 2006).

However, the biomass in the bioreactor can also be fixed on a carrier, forming a biofilm; processes using this biomass have proved to be reliable for organic carbon and nutrient removal without some of the problems of CAS (Ødegaard et al. 1994). Immobilization of biomass in the form of biofilms is an efficient method of retaining slow-growing microorganisms, such as nitrifiers, in continuous flow reactors (Kermani et al. 2008). Indeed, attached growth systems are generally considered less sensitive to toxic influents and variations in environmental conditions (Wang et al. 2005). Moving bed biofilm reactors (MBBR) consist of a process tank in which carriers are immersed and gradually colonized by the attached biomass on the protected surface on the inside. In MBBR systems, the carrier elements, which have a slightly lower density than water, move around the bioreactor freely and are kept in the tank by a sieve arrangement without the necessity for sludge recycling. This arrangement allows the carrier suspension to move freely. Movement of the carrier in the reactor is important to allow transport of substrates to the biofilm and to maintain low biofilm thickness by shearing forces, and therefore it is recommended that the filling ratio (the relation between the apparent carrier volume and the operative volume of the bioreactor) should be below 70 % (Rusten et al. 2006). Several studies (Germain et al. 2007; Davis et al. 2009; Kim et al. 2010) have demonstrated that, with MBBR, it is possible to obtain efficiencies in biochemical oxygen demand (BOD_5) and chemical oxygen demand (COD) of greater than 90 % and 85 %, respectively. Moreover, MBBR-MBRs are simple in operation, have a low risk of biomass loss and are less temperature dependent (Krzeminski et al. 2012), resulting a process that is inherently stable and resistant to organic and hydraulic shock loadings (Mehrdadi et al. 2006). The exceptional mixing conditions result in efficient mass transfer and elimination of the risks of liquid short-circuiting and clogging of the media with biomass or other solids (Welander et al. 1998).

The aim of the present research was to study the influence of the operative variables [filling ratio, hydraulic retention time (HRT) and temperature] on the behaviour of a MBBR-MBR in relation to organic matter removal and nitrification activity.

Materials and methods

Pilot-scale experimental plant

In this research, a pilot-scale experimental plant, located at the Puente de Los Vados WWTP in Granada (Spain), was used. A schematic diagram of the process configuration and pilot plant used is shown in Fig. 1. The influent was taken from the outlet of the primary settler. The experimental plant used in this research had a cylindrical bioreactor with an operative volume of 358 L in which the biodegradation took place and carriers were contained (the MBBR), and a rectangular tank with 87 L of operating volume in which three membrane units were submerged to the solid separation (the MBR). The ultrafiltration modules used were ZW-10 of ZENON ®, configured as an outside-in hollow fibre with a nominal membrane surface area of 0.93 m^2, a nominal pore size of 0.04 µm and an absolute pore size of 0.1 µm.

The typical operating transmembrane pressure of this module was 10–50 kPa with a maximum transmembrane pressure of 62 kPa. The carriers contained in the cylindrical reactor

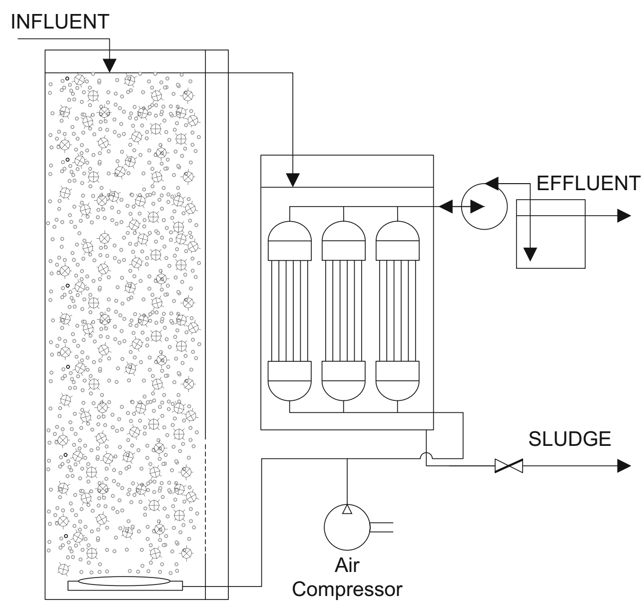

Fig. 1 Schematic diagram of the moving bed biofilm reactor-membrane bioreactor (MBBR–MBR) pilot plant. The influent from the primary settler is introduced in the MBBR tank where the carrier is contained and biodegradation takes place. The MBBR is followed by the MBR tank to solids separation in sludge flow (purge) and treated wastewater flow (effluent). The ultrafiltration membrane units worked in phases of 9.75 min of permeating and 0.25 min of backwashing

were K1 of Anoxkaldnes® at filling ratios of 20 %, 35 % and 50 %.

Operating conditions

Six cycles of operation were studied in relation to the filling ratio and HRT (Table 1). These cycles were ordered in three phases according to filling ratio in order to study the effect of the filling ratio on the behaviour of the system: phase I with 20 % (cycles 1 and 2), phase II with 35 % (cycles 3 and 4) and phase III with 50 % (cycles 5 and 6). In each phase, two HRTs [24 h (cycles 1, 3, and 5) and 10 h (cycle 2, 4 and 6)] were tested in a pure MBBR. The MBBR-MBR was operated at a flow rate of 45.5 L/h when HRT was 10 h and 18.96 L/h when HRT was 24 h. The average temperature of each cycle is shown in Table 1; this variable was not controlled due to the outdoor location of the pilot plant in order facilitate the scale up of the process to full scale (Leyva-Díaz et al. 2013). These operative variables fixed the suspended solids of the biofilm (BFSS) as shown in Table 2. The operation of the membrane applied was based on two different modes: continuous filtration during 9.67 min and periodic backwashing of 0.33 min. Air scouring of the membrane was applied continuously and the submerged membrane units were operated with a transmembrane pressure of between 0.3 bar and 0.5 bar at a constant flux using a suction pump in each cycle.

Analytical methods

The water samples were obtained for analytical determination every 24 h from the feed tank, biological reactor and permeate of the membrane. A sample (1 L) from each assayed point was conserved in the laboratory at 4 °C until physical and chemical analysis, and was analysed within 4 h of sampling.

The COD and BOD_5 were determined according to American Public Health Association, American Water Works Association and Water Environment Federation (APHA-AWWA-WEF) methods. The suspended solids (SS) were determined by gravimetric methods (APHA 2012). The pH was determined using a pH meter (Crison pH 25®) and conductivity was determined using a conductivity meter (Crison CM 35®). Tests on carrier samples were carried out according to Martín-Pascual et al. (2012).

Ammonium, nitrites and nitrates were determined by ionic chromatography using a conductivity detector (Methrom). Separation and dilution of the anions was carried out on a Metrosep A supp5 column using a solution of carbonate/bicarbonate as eluent, and sulphuric acid as the regenerate. Separation and dilution of the cation was developed with a Metrosep C 4 column using a solution of dipicolinic acid as eluent, and distilled water as the regenerate.

Statistical analysis

The data obtained were analysed using a computer-assisted statistics program, SPSS 21 for Windows. A least significant differences (LSD) test was used to measure the differences between the data obtained of temperature and COD, BOD_5, NH_4 and Nt removal rate for each cycle, which had at least seven data points. An analysis of variance (ANOVA) was used to assess the homogeneity of the variance, with a significance level of 5 % ($P<0.05$). The Shapiro–Wilk test was used as normality test of the data since the dataset was smaller than 2000 elements.

A multivariable analysis in Canoco for Windows version 4.5 was used to quantify the influence of the environmental variables (HRT, filling ratio and temperature) on the BFSS, organic matter removal (COD and BOD_5), nitrogen and ammonia removal. A Monte Carlo test of permutations (499 permutations) was performed, with a selected significance level of 0.05. The analysis represented 70.7 % of accumulated variance of the species and the totality of the cumulative variance of the relationship between the species and the variables.

Results and discussion

The average amounts of biofilm during the research varied between 1510±127 and 3608±341 mg/L carrier as shown in Table 2. Three homogenous subsets of Tukey's HSD of the

Table 1 Operative conditions [filling ratio, hydraulic retention time (HRT) and temperature] in the different phases and cycles checked in the main research. The average and standard deviation (SD) of the temperature data are shown with the homogenous subsets (indicated by lower case letters) of Tukey's honest significant difference (HSD) of the analysis of variance (ANOVA) test undertaken

Phase	Cycle	Filling ratio (%)	HRT (h)	Temperature (°C; average ± SD)
I	1	20	24	7.60±2.88 a
	2	20	10	4.60±2.61 a
II	3	35	24	17.03±0.55 b
	4	35	10	15.67±1.53 b
III	5	50	24	5.03±3.05 a
	6	50	10	2.50±1.5 a

Table 2 Average ± standard deviation of suspended solids of the biofilm (BFSS) with the homogenous subset of Tukey's (indicated by lower case letters) HSD of the ANOVA test undertaken

Phase	Cycle	BFSS (mg/L carrier; average ± SD)
I	1	3608±341 a
	2	1510±127 b
II	3	3775±247 a
	4	2529±77 c
III	5	1988±13 b
	6	1838±53 b

ANOVA test were needed to describe the variability in the BFSS, so the biofilm attached varied with the operative variable. The amount of biofilm affects the efficiency of the process in the removal of both organic matter and nitrogen and depends on the variables of the process. The thickness of the biofilm formed depends on the organic load, temperature and the concentration of dissolved oxygen (Levstek and Plazi 2009), stressing the importance of substrate entry, with greater load inputs leading to greater growth of attached biomass as well as affecting the C/N relationship (Bassin et al. 2012) as a result of competition for substrate availability between autotrophic and heterotrophic bacteria. It was observed through the different phases that biofilm density increases with the HRT independently of the other variables, possibly due to the fact that microorganisms could have more time to consume organic matter. The thick biofilm under high filling ratios presents greater activity, indicating an increased rate of removal of organic matter and nutrients per unit biofilm (Peyton 1996; Vieira and Melo 1999). However, a high filling ratio favours detachment of microorganisms from the biofilm, leading to a decline in biomass attached to the bioreactor (Gjaltema et al. 1997). In addition, fluidisation of the carrier requires a greater flow of air to suspend it, which incurs a cost overrun of the process (Wang et al. 2005). On the one hand, increased biofilm in the system for the same substrate means that microorganisms have less substrate and therefore less matter to purge; on the other hand, under the higher filling ratio, collisions between carriers occur at a higher rate, which can produce an increase in biofilm detachment, thus 35 % could be the optimum filling ratio. This aspect becomes clearer when comparing average density values between the phases under 35 % and 50 % of filling ratio, the biomass being lower at the higher concentration of carrier. Another aspect to consider in relation to the filling ratio was that, at the lowest percentage tested the variation in biofilm density as a consequence of the HRT was higher than seen in the experimental data, the amount of biofilm in the system increases with temperature. The highest density was detected in cycle 3 at 17.03±0.55 °C while the lowest density was in cycle 2 at an average temperature of 4.6±2.61 °C. Comparing attached biomass results with those obtained in previous research using a hybrid MBBR (Martín-Pascual et al. 2015) it was observed that the BFSS is lower under similar working conditions as a consequence of the lack of suspended biomass.

The organic matter removal rates in COD and BOD_5 obtained are shown in Fig. 2. The average removal of COD was between 67.05±1.14 (cycle 5) and 86.36±2.12 (cycle 3); regarding BOD_5, average rates ranged between 74.14±0.84 (cycle 6) and 91.54±1.17 (cycle 3). ANOVA analysis of the data showed that four and three different homogeneous subsets of Tukey's HSD were defined in COD and BOD_5 removal, respectively. These statistically significant differences show that the operative variables influenced organic matter removal rates. The effect of HRT was seen clearly in the data obtained, with higher rates as HRT increased. In relation to the filling ratio, no trend was shown under the other variables because the highest removal rate was obtained at a filling ratio of 35 %.

Under HRT of 24 h, the highest rate was obtained for the cycle with a medium filling ratio (35 %), coinciding with the cycle of higher temperature (17.03±0.55 °C); cycle 5 (7.60± 2.88 °C) presented a performance intermediate between that of 35 % and 50 % filling ratio (5.03±3.05 °C). With HRT of 10 h, the best rate was obtained in the cycle with higher average temperature (15.67±1.53 °C) while the remaining cycles obtained lower rates as a result of low temperatures (4.60± 2.61 and 2.50±1.50 °C for cycles 2 and 6, respectively). Moreover, it was observed that organic matter removal is reduced drastically at temperatures below 5 °C. The effect of temperature was more important than the presence of biofilm at low HRT, while filling ratio had a greater influence at high HRT.

Different removal rates of ammonium and total nitrogen were obtained in the present study (Fig. 2). Cycle 3 presented a high removal rate of ammonia under the highest temperature (17.03±0.55 °C), and the least efficient cycle in this consumption test was cycle 2 with the lowest concentration of carrier and an average temperature lower than 5 °C. The ANOVA test defined two different homogeneous subset of Tukey's HSD for both ammonia and total nitrogen removal. In relation to the above, no statistically significant differences were seen between 35 % and 50 % of filling ratio. This result could be due to the fact that the total biomass was very low as a consequence of the filling ratio and the possible inhibition of nitrification activity caused by the low temperature experienced during this cycle. A comparison of this technology with the hybrid MBBR tested by Martín-Pascual et al. (2015) shows than efficiencies are lower using pure biofilm; however, the reduction of suspended solids in the membrane tank improve its performance in relation to membrane fouling (Yang et al. 2009).

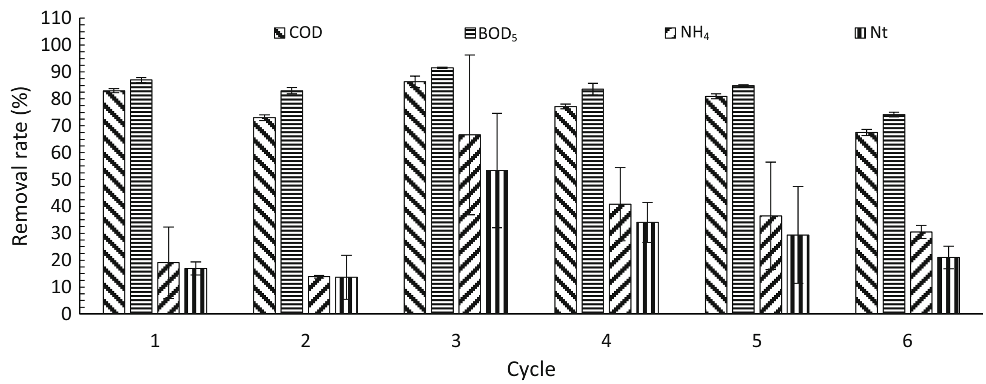

Fig. 2 Average and standard deviation of the COD, BOD_5, NH_4 and total nitrogen (Nt) removal in the different cycles of the research

The presence of statistically significance differences in the removal of organic matter and nitrogen, as shown by the ANOVA analysis, are due to variation in the operative variables (Table 1) or the concentration of BFSS (Table 2), or both. In order to analyse these factors, a multivariable analysis was performed. A biplot diagram of analysis of the redundancy of the multivariate analysis is shown in Fig. 3. A Monte Carlo test showed that filling ratio was the most influential variable affecting variability of the system under the conditions studied ($P = 0.09$). The variables most influencing BFSS were the HRT and temperature; the effect was positive, i.e. the higher HRT and temperature, the higher BFSS was observed. Temperature showed a positive correlation with BFSS and organic matter and nitrogen removal; biofilm density increases with temperature, caused by higher microbial activity. The HRT had a strongly positive influence on biofilm density and organic matter removal. Moreover, the statistical analysis revealed that filling ratio did not influence biofilm density under the conditions studied, because the BFSS under the highest filling ratio was lower as a consequence of collisions between carriers. However, filling ratio was slightly positively correlated with nitrogen removal, due to the higher presence of slow-growing microorganisms, such as nitrifiers, in the biofilm (Kermani et al. 2008).

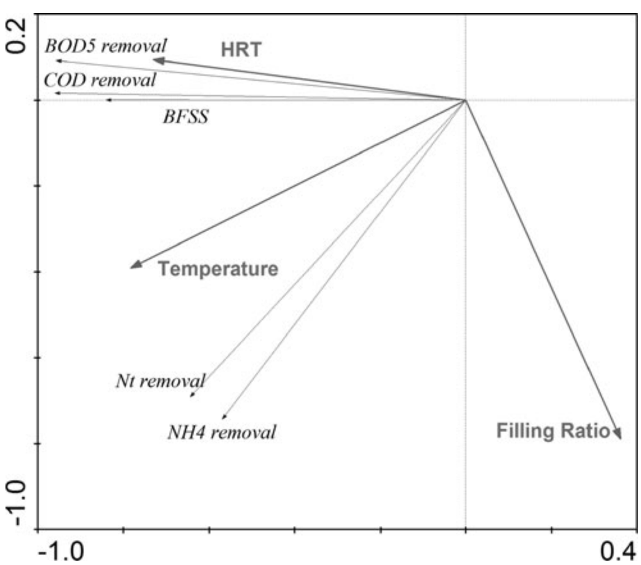

Fig. 3 Biplot diagram of analysis of redundancy in the multivariate analysis used to study the relationship between HRT, temperature and filling ratio as variables, and biofilm (BFSS), organic matter (COD and BOD_5) and nitrogen removal (NH_4 and total nitrogen) as species for the conditions tested

Conclusions

The results from this research were obtained in a moving bed membrane bioreactor system treating urban effluents under the following conditions: (1) 20 %, 35 % and 50 % filling ratio; (2) 10 and 24 h of HRT; and (3) temperatures between 2.5 and 17.0 °C. From these operating conditions, it can be concluded that:

- Biofilm density ranged between 1510±127 and 3775±247 mg/L of carrier, with attachment of biofilm being related positively with temperature and HRT, but becoming lower under the highest filling ratio as a consequence of increased collision between carriers, thus indicating that an optimum rate of filling ratio can be determined for this process.
- Organic matter removal increases with the amount of biofilm in the carrier, ranging between 67.50±1.14 and 86.36±2.12 % in COD removal and between 74.17±0.84 and 91.54±0.21 % in BOD_5 removal, with the best rates being obtained under the highest temperature (17 °C), 24 h of HRT and a filling ratio of 35 %.
- The highest removal rate of ammonia (66.61±29.70 %) took place in the cycle under the highest temperature (17 °C) and HRT (24 h). Nitrogen removal yield increased directly with the higher amount of biofilm attached in the bioreactor.

In view of these results, in the MBBR-MBR system, the highest efficiencies of organic matter removal and nitrogen oxidation took place in the cycle with higher BFSS under 24 h of HRT, 17.03±0.55 °C and a filling ration of 35 %.

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