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ANALYSIS OF THE OPERATING CONDITIONS IN THE TREATMENT OF COSMETIC WASTEWATER BY SEQUENCING BATCH REACTORS

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Abstract

The biological removal of organic matter from a cosmetic wastewater by sequencing batch reactors has been optimized by analyzing the influence of the hydraulic retention time as well as the inclusion of anoxic and/or aerobic stages in the process sequence. Hydraulic retention time of 5 d led to the highest total organic carbon and chemical oxygen demand removal regardless the sequence used. Although a completely aerobic sequence can be used for cosmetic wastewaters treatment, the alternation of anoxic and aerobic stages significantly improved the organic matter and nutrients removal. Total organic carbon and chemical oxygen demand removal efficiencies around 67 and 74 %, respectively, were achieved with a sequence which includes two anoxic stages of one hour at the beginning and the end of the cycle. Additionally, nutrients were satisfactorily removed when anoxic stages were included along the cycle, reaching nitrogen and phosphorous removals higher than 97 and 70 %, respectively. Under these conditions the flocs showed a moderate density, good mechanical stability and settleability.

Key words: Biological treatment, cosmetic wastewater, hydraulic retention time, reactor sequence, sequencing batch reactor

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1. Introduction

Cosmetic wastewaters are characterized by high values of chemical oxygen demand (COD), suspended solids, fats, oils, and detergents (Puyol et al., 2010). These effluents are frequently treated by means of coagulation/flocculation followed by pressure-flotation to separate the sludge (Meiners, 1994; Moggio, 2000). Nevertheless, the more stringent regulations concerning municipal and industrial wastewater disposal make necessary to implement new technologies and procedures for a more efficient treatment of this type of wastewaters. The application of several oxidation processes, such as Fenton oxidation (Bautista et al., 2007; Perdigón-Melón et al., 2010) and catalytic wet peroxide oxidation (Bautista et al., 2010) have been reported as possible strategies for reducing toxicity and increasing the biodegradability of the cosmetic wastewaters.

Biological systems are cost-effective treatments for many industrial wastewaters as compared to physico-chemical treatments. However, to analyze the potential application of a biological treatment to a specific wastewater it is necessary to assess its biodegradability since it can be unfeasible in some cases (Sanchis et al., 2013, 2014). In this sense, cosmetics contain non-readily biodegradable compounds including preservatives, surfactants, fragrances and co-solvents which make difficult the application of conventional biological processes to the wastewaters resulting from their manufacture (Perdigón-Melón et al., 2010). Furthermore, these wastewaters frequently vary in both flow and composition (change of manufacturing product, in-transition operation of the plant, washing etc.), which

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difficult the operation of conventional activated sludge systems (Venkata et al., 2001).

Nevertheless, some of the biological systems developed in the last decades have shown the potential of efficiently treating industrial wastewaters at relatively low investment and operational costs. Among them, sequencing batch reactors (SBR) have been claimed as an interesting alternative to conventional biological treatments, mainly because of their simple and flexible operation and the possibility of influencing the microbial system. It is possible to take more advantage of the flexibility of the SBR technology by conveniently adjusting the extent of the aerobic and anoxic stages to achieve higher carbon and nutrient removal.

The SBR process consists of five stages (filling, reaction, settling, discharge and idle) sequentially operating in the same tank. This technology is characterized by its efficiency and easy adaptation to variations of volumetric flows and pollutant concentrations, along with nutrients removal (Irvine et al., 1998; Mace and Mata-Alvarez, 2002; Monsalvo et al., 2012a).

The SBR technology has been successfully used to treat different kinds of effluents including domestic (Bernardes and Klapwijk, 1996), winery and brewery (Ling and Lo, 2001; Torrijos and Moletta, 1997), tannery (Carucci et al., 1999), paper pulp (Tripathi and Allen, 1999), dairy (Garrido et al., 2001), piggery (Bernet et al., 1999) and textile industry (Fongsatitkul et al., 2004) wastewaters as well as landfill leachates (Klimiuk and Kulikowska, 2005). However, there is a lack of information in the literature dealing with the application of this technology for the treatment of cosmetic effluents.

The aim of this work is to study the applicability of the SBR technology for the treatment of cosmetic wastewaters. The effect of using anoxic or aerobic conditions in the filling and reaction stages as well as the hydraulic retention time on the efficiency of the biological treatment are analyzed. For this purpose, organic matter measured as chemical oxygen demand (COD) and total organic carbon (TOC), and nutrients (N and P) removal from cosmetic wastewaters was evaluated to follow the performance of the system.

2. Materials and methods

2.1. Wastewater

The wastewater was collected from a Spanish cosmetic factory whose effluents were previously treated by means of coagulation–flocculation in the own industrial plant. The treatment included homogenization, filtration, coagulation, neutralization, flocculation and air-pressure flotation. The samples were stored in dark at 4 ºC immediately after reception from the cosmetic factory. A representative analysis of the wastewater as received in the lab after physical and chemical treatments in the plant is given in Table 1, together with the regionally allowable limits for industrial wastewaters discharge into the municipal sewer system (Law 10/1993). As can be seen, the pretreatment of these wastewaters does not satisfy the limits established for BOD_5 and COD.

Table 1. Representative analysis of the in-plant pre-treated cosmetic wastewater and discharge limit values

Parameter	*Average value*[a]	*Limit value*[b]
Fats and Oils (mg/L)	32 ± 8	100
Conductivity (µS/cm)	1135 ± 240	5000
BOD_5 (mg/L)	1285 ± 210	1000
COD (mg/L)	3220 ± 190	1750
TOC (mg/L)	825 ± 85	–
pH	7.3 ± 0.6	6.0 - 9.0
TSS (mg/L)	43 ± 13	1000
Ecotoxicity: T.U.[c] (5 min) T.U. (15 min)	 18.5 ± 1.8 15.9 ± 1.2	25

a Number of samples analyzed = 8; b Emission limit value for industrial wastewater discharges into the municipal sewer system according to the 10/1993 Act passed by the Community of Madrid; c Toxicity Units (T.U.)

The content of N and P of the cosmetic wastewater used in this work was negligible and those nutrients were externally added as ammonium sulphate and phosphoric acid, respectively. A COD:N:P ratio of 100:5:1 (w:w:w) was fixed and mineral salts were also added as micronutrients supply in a COD:micronutrients ratio of 1:0.05 (w:w). The mineral solution consisted on $FeCl_3$, $CaCl_2$, KCl and $MgSO_4$. All chemicals were purchased from Panreac and used without further purification.

Although the biodegradability of the wastewater can be evaluated by the BOD_5/COD ratio, the suitability of the treatment should be assessed with microbial population and working conditions similar to those to be used in the SBR. Recently, Polo et al. (2011) proposed a new biodegradability test to check in an easy and rapid way the behavior of a target compound or a wastewater in a biological reactor by means of respirometry, which is based on the direct relationship between the oxygen uptake rate (OUR), biomass growth and substrate consumption (Monsalvo et al., 2009; Tobajas and García-Calvo, 2000), since oxygen consumed by biomass is related to the microbial activity when oxidizing organic matter and ammonia as well as to endogenous respiration in aerobic processes (Corominas et al., 2006; Puig et al., 2005). In order to determine the specific oxygen uptake rate (SOUR) profiles, batch assays were carried out in a Liquid-Static-Static (LSS) respirometer. The method was designed on the basis of the typical operating conditions of an activated sludge process, measuring both the activity of the microorganisms as SOUR and organic matter degradation as TOC removal. Activated sludge was maintained in starvation and continuous aeration overnight in the reactors in order to eliminate any residual COD. Subsequently, cosmetic wastewater supplemented with ammonium chloride and phosphate buffer (pH 7.0) and micronutrients was incorporated.

The reactors were closed to avoid evaporation and continuously aerated during the whole reaction time to avoid oxygen limitation. The aeration system was automatically controlled by an electronic interface connected to the oxygen probes. The SOUR profile was obtained by interrupting the air supply and registering the DO decay within a range of 0.2 mg/L. The respirometer operated with two independent reactors simultaneously to guarantee reproducibility. The reaction vessels were placed in a thermostatic bath and continuously stirred by magnetic bars.

Tests were carried out for an organic load of 0.11 kg COD/kg VSS, 25 ºC and 8 h corresponding to the cycle length to be performed in the SBR. The biomass concentration was set at 350 mg VSS/L. This concentration was selected by preliminary tests in order to ensure a significant oxygen consumption detection at the lowest respiration rate and to prevent that the maximum OUR was greater than the oxygen transfer rate during the aeration period (Polo et al., 2011). The biodegradation of the cosmetic wastewater was followed from the TOC evolution and the SOUR profile.

2.2. Sequencing batch reactors

Four sequencing batch reactors of 15 L equipped with dissolved oxygen and pH probes were used (Fig. 1). Peristaltic pumps were used for feed and effluent discharge as well as for the addition of sodium hydroxide (NaOH) solution for pH control. Air was supplied by means of a flow compressor through a ceramic diffuser and mechanical stirring was also used. An air flow rate of 9 L/min was used to ensure an adequate dissolved oxygen concentration. Reactors were operated at room temperature (18 - 22 ºC). The seeding and seedling of the reactors were performed with activated sludge from a municipal wastewater treatment plant. The biomass concentration was fixed at 2000 mg VSS/L and the sludge age was maintained around 10 d.

All the experiments were conducted in cycles of 8 h. The influence of the organic loading rate was analyzed by modifying the hydraulic retention time (HRT) as follows: during the filling period a variable volume (0.5 – 2.6 L) of feed (wastewater supplemented with nutrients) was added to complete the working volume of 15 L. The experiments were carried out at HRT of 2, 3.5, 5, 6.5, 8, and 9.5 d, reaching organic loading rates (OLR) from 0.2 to 0.9 kg COD/kg MLSS.d. The length of each stage in the scheme of the cycle was also fixed as follows: filling (0.5 h), reaction (5.5 h), settling (1 h), draw (0.5 h) and idle (0.5 h). As shown in Table 2, four different sequences including anoxic and aerobic conditions were analyzed. According to the fixed time strategy previously reported by Monsalvo et al. (2012b) the reactors were operated for at least 2 weeks at each combination of operating conditions. Before the corresponding data collection, the stable performance of the bioreactor was ensured by checking the reproducibility of the time-evolution of the monitored parameters.

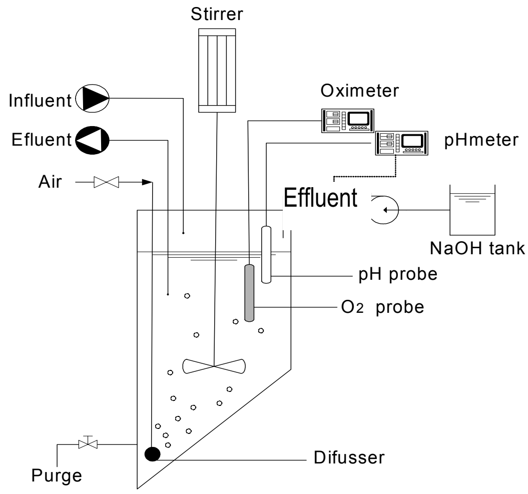


Fig. 1. Scheme of the sequencing batch reactors setup

Table 2. Distribution of the phases in the sequences used

Sequence	*Fill (h)*	*Reaction (h)*
1	0.5 (anox.)	5.5 (aer.)
2	0.5 (aer.)	1 (aer.) + 0.5 (anox.) + 3.5 (aer.) + 0.5 (anox.)
3	0.5 (anox.)	0.5 (anox.) + 4 (aer.) + 1 (anox.)
4	0.5 (aer.)	5.5 (aer.)

anox.: anoxic phase; aer.: aerated phase

2.3. Analytical methods

Previous to the analysis, cells and other inorganic and organic particulates were removed by centrifugation at 10,000 rpm for 10 min and the supernatants were filtered through 0.45 μm pore size filters. The degradation efficiency was evaluated by measuring the COD, TOC, nitrite, nitrate, ammonium and phosphate concentrations of the influent and resulting effluent. BOD_5 and COD determinations were carried out according to the 5210 and 5220 Standard Methods, respectively (SMEWW, 1995). TOC was determined using an OI Analytical Model 1010 TOC apparatus.

Ecotoxicity measurements of the pre-treated wastewater were carried out by the Microtox Acute Toxicity Test (SCI 500 Analyzer) using a freeze-dried preparation of the marine bacterium *Vibrio fischeri* as described in ISO (1998). Ammonium was determined by an ammonia-specific electrode (Crison, model pH 2002). The concentrations of nitrate, nitrite and phosphate were determined by anionic suppression IC (Metrohm, mod. 761 Compact IC) with a conductivity detector, using a Supp 5 column (25 cm long, 4 mm diameter) and a mixture of 1 mM $NaHCO_3$ and 3.2 mM $NaCO_3$ aqueous solution as the mobile phase. The results reported were the average values from triplicate runs. The standard errors were lower than 5% in all the cases. Scanning Electron Microscopy (SEM) images of the mixed liquor were obtained using a Hitachi S-3000N apparatus.

3. Results and discussion

The aerobic biodegradability of the cosmetic wastewater was followed from the evolution of TOC and the SOUR profile (Fig. 2). Two different fractions of the organic matter can be observed. A COD fraction which can be classified as readily biodegradable, that is completely degraded within the first two hours, and a second fraction that is slowly biodegraded and could be associated to refractory compounds that require a previous adaptation of the biomass or to intermediates formed within the first two hours. Besides, a fraction of the oxygen consumption can also be addressed to nitrification of the ammonia salts added.

There was no complete TOC removal, although the residual fraction (27.2%) was not toxic to the sludge since the biomass recovers the starting value of the endogenous respiration rate.

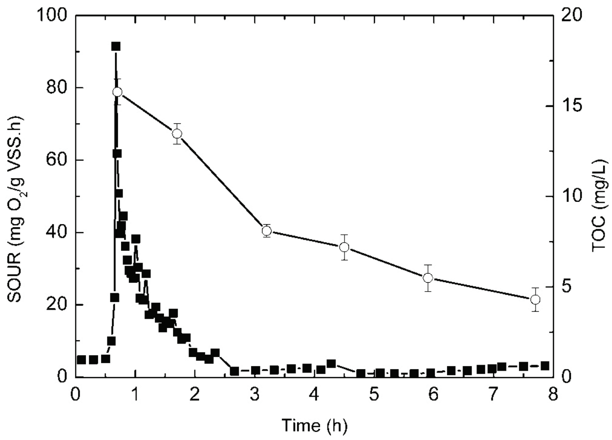


Fig. 2. Evolution of SOUR profile (■) and TOC (○) in the aerobic biodegradability test of cosmetic wastewater

Fig. 3 shows the influence of HRT (2 to 9.5 d) on COD and TOC removal efficiencies using a sequence with an anoxic filling followed by an aerobic reaction stage (sequence 1). As can be seen, the maximum removal of organic matter was achieved at 5 d HRT, which corresponds to an organic loading rate of 0.32 kg COD/kg VSS.d. This reveals that the operation of SBR under anoxic and aerobic conditions allows treating higher OLR of cosmetic wastewater than using an anaerobic SBR, where an OLR of 0.19 kg COD/kg VSS.d led to an inefficient performance of the reactor due to the presence of personal care products and disinfectants (de Oliveira et al., 2009). At higher HRT values, TOC and COD removal efficiencies decreased and stabilized at values near to 53.5 and 58.0 %, respectively.

The efficiency of the biological treatment decreased significantly at HRT values below 5 d. The effect of the extent and position of the anoxic and aerated steps in the removal of TOC and COD of the cosmetic wastewater was also tested but within a narrower HRT range according to the results obtained for sequence 1. As shown in Table 3, the sequence consisting of aerobic filling and reaction steps (sequence 4) led to the lowest TOC and COD removal whereas an improvement in the degradation efficiency was observed for combined anoxic and aerated stages, reaching values of COD and TOC removal of 74.1 and 66.7%, respectively, for the sequence 3. This operating strategy has been reported to be highly-efficient when treating wastewater with low concentrations of biodegradable organic matter (Monclús et al., 2009), being necessary for an effective biological nutrients removal (Bortone, 2009). An increase in COD and TOC removal was obtained in all the experiments when increasing HRT from 2 to 5 d. This effect is more pronounced for sequence 4.

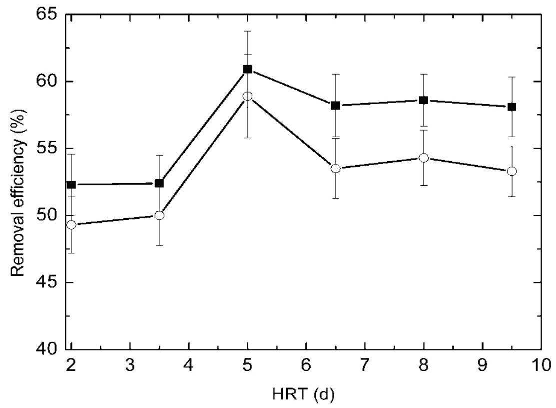


Fig. 3. COD (■) and TOC (○) removal efficiencies as a function of HRT using the sequence 1

Table 3. COD and TOC conversions (%) achieved with different sequences and HRT

	HRT (2 d)		***HRT (3.5 d)***		***HRT (5 d)***	
	X_{COD} (%)	X_{TOC} (%)	X_{COD} (%)	X_{TOC} (%)	X_{COD} (%)	X_{TOC} (%)
Seq. 1	52.3	49.3	52.4	50.0	60.9	58.9
Seq. 2	63.6	59.9	64.4	61.7	66.5	63.2
Seq. 3	66.4	62.5	67.8	64.1	74.1	66.7
Seq. 4	34.8	31.2	38.7	34.6	43.4	41.8

Several works found in the literature indicate that the optimal HRT for organic matter removal greatly depends on the composition of the wastewaters treated. Klimiuk and Kulikowaska (2005) reported that organic matter removal efficiency from a municipal landfill leachate with 1200-1600 mg COD/L a COD:N ratio around 100:8 and a BOD_5/ COD of 0.38, decreased at HRT below 3 d whereas no differences in the BOD_5 removal efficiency were found at HRT between 6 and 12 d. Ling and Lo (2001) analysed the effect of HRT and organic loading rate on the treatment of brewery wastewaters with COD values ranging from 700 to 4500 mg/L and a COD:N:P ratio close to 100:5:0.8 obtaining a maximum TOC removal of 97% at a HRT around 1.5 d with a loading rate of 3.1 kg TOC/m^3.d.

Fig. 4 shows the time-evolution of COD and TOC conversions along the cycle for all the sequences tested when operating at 5 d HRT. The use of aerobic or anoxic filling did not affect to mineralization, reaching TOC removal efficiencies lower than 7% in that stage. The use of a completely aerobic treatment (sequence 4) led to low COD and TOC removal efficiencies of 43.4 and 41.8%, respectively. The COD removal rate increased when using alternative aerobic and anoxic stages, thus sequences 2 and 3 showed the best results. The latter showed TOC and COD removal efficiencies around 74 and 67%, respectively, with an initial COD removal rate around 11 mg COD/g VSS.h, which is almost double than that obtained with the sequence 1. In the sequence 3 the anoxic period was extended up to 2 h along the cycle and the results suggest that part of the organic matter is easily removed in the absence of dissolved oxygen.

Higher TOC and COD removal efficiencies were achieved when the anoxic stage was placed at the beginning of the cycle, since the high availability of organic matter after the filling step allows the correct operation of the denitrifying bacteria. Actually, the treatment of this cosmetic wastewater under anaerobic conditions was found to be also efficient for the removal of most of the compounds present (Puyol et al., 2010). Therefore, the anoxic/aerobic coupled sequences showed to be much more effective than the aerobic/aerobic cycle for the treatment of these cosmetic wastewaters by SBR technology.

The effect of the anoxic and/or aerobic sequences on nutrients removal was also studied. Fig. 5 shows the N and P removal efficiency at 5 d HRT for all the sequences tested. N refers to dissolved N, including N-NH_4^+, N-NO_2^- and N-NO_3^-. As for COD and TOC, the absence of anoxic stages (sequence 4) gave rise to the lowest removal efficiencies with values of 55 and 45% for N and P, respectively. However, sequences combining anoxic and aerobic phases allowed N and P conversions higher than 95 and 70%, respectively. Although no significant differences were found regarding the N removal for sequences 1, 2 and 3, the alternation of aerated and anoxic steps in the reaction stage conducted to better results in terms of P removal. The enhanced phosphorus removal was due to phosphate accumulation during the anaerobic stage and utilization in the subsequent aerobic step of the sequence (Dassanyakee and Irvine, 2001).

Several studies have been reported in the literature on nutrient removal from wastewaters by SBR, some of them focused on the use and optimization of different phases (anaerobic, anoxic and oxic). Kargi and Uygur (2004) achieved the highest COD, ammonium and phosphate removal efficiencies (96, 87 and 90%, respectively), from a synthetic wastewater with a COD of 1000 mg/L and a COD:N:P ratio of 100:5:1.5 using an optimized 12 h cycle with Anaer/Anox/Aer/Anox/Aer phases. Recently, Coma *et al.* (2010) have pointed out the necessity of designing the length of the phases and optimizing the time-distribution of the feed in order to stabilize the system for long-term periods with high organic matter and nutrients removal efficiencies (88, 93 and 99% of carbon, nitrogen and phosphorous respectively for a synthetic wastewater with 560 mg/L of COD and a COD:N:P ratio around 100:7:1.2).

Debik and Manav (2010) obtained a COD, N and P removal of 91, 78 and 85%, respectively, from a domestic wastewater (average 500 mg/L of COD and COD:N:P ratio 100:12:1.5) by optimizing the length of Anaer (2h)/Aer (2h)/Anox (1h)/Aer (0.75h) phases for the reaction stage in an 8 h cycle. The morphology of the resulting flocs in each sequence was characterized by SEM at the end of the long-term experiment.

The SBR system allows the selection of flocculent bacteria, leading to good floc development, with a subsequent washout of filamentous bacteria in all cases. The flocs showed a moderate density facilitating the diffusion of dissolved oxygen and soluble organic matter into them. This enhances the contact between the wastewater and the biomass, allowing a better performance of the bioreactor.

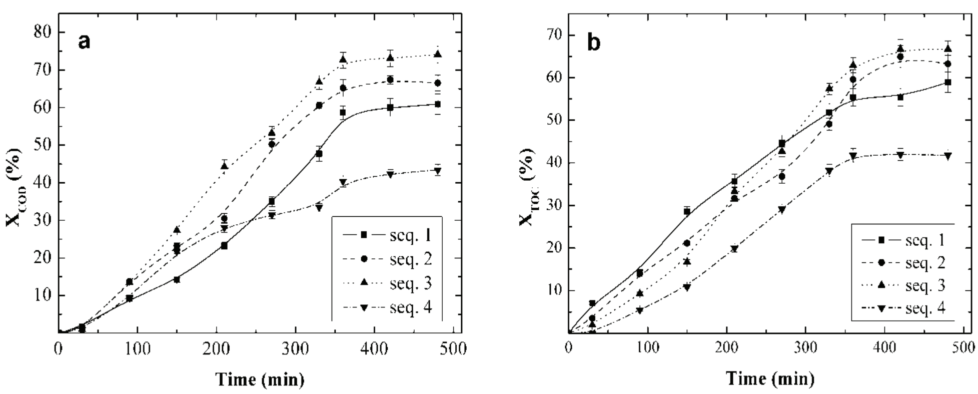


Fig. 4. Time-evolution of COD (a) and TOC (b) conversion during a cycle using a HRTof 5 d

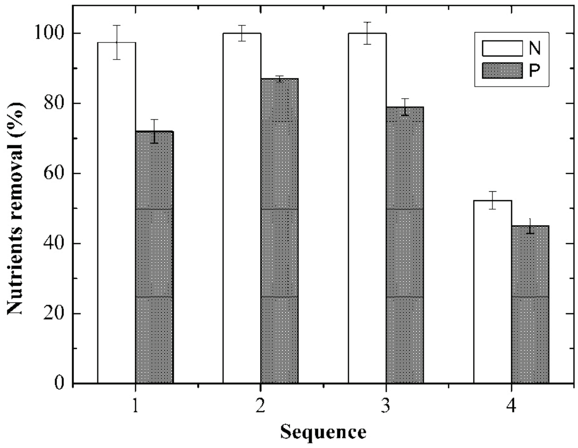
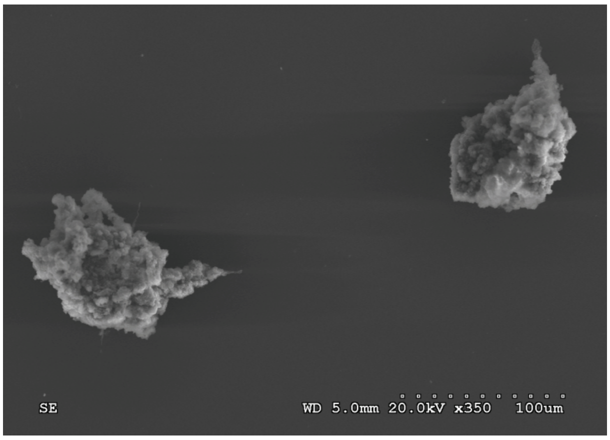
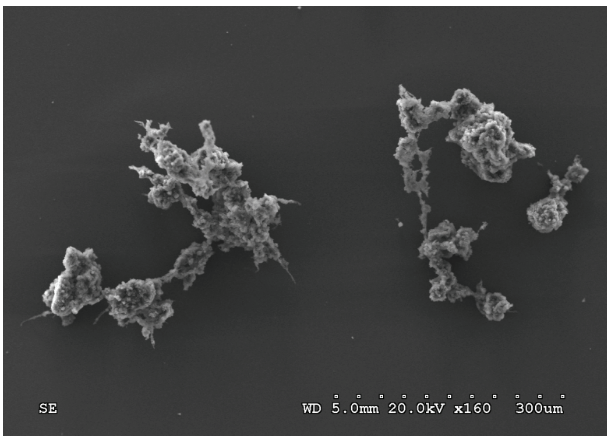


Fig. 5. N and P removal at 5 d HRT for different sequences

Additionally, the flocs showed a good mechanical stability, which prevents their breakdown by the mixing system. Thus, neither part of the flocs nor free living bacteria were observed in the supernatant along the experiments. However, the operating conditions used in this work led to some differences in the structure of the flocculent biomass as can be seen in Fig. 6.



a)



b)

Fig. 6. SEM images of flocules from SBR operated with sequence 3 (a) and 4 (b)

Large, round-like flocs with a diameter higher than 80 μm were observed when operating with anoxic stages (sequence 3). Filaments were no longer observed since filamentous growth can be easily controlled under these operating conditions. The use of a completely aerobic treatment (sequence 4) led to the formation of irregular flocs with a moderate presence of filamentous bacteria.

These microorganisms are known to render sludge more difficult to thicken and dewater and can also cause foaming problems in the subsequent digestion (Jenkins et al., 2004).

4. Conclusions

Cosmetic wastewater can be efficiently treated by SBR technology with alternative anoxic/aerobic stages, reaching higher removal efficiencies than that obtained by using an entirely aerobic cycle. The sequence including two anoxic steps at the beginning of the cycle and at the end of the reaction stage showed to be the best in terms of COD and TOC removal. The use of sequences which include at least an anoxic stage allowed achieving N and P removal efficiencies above 90 and 70%, respectively.

The reduction of phosphorous was increased by alternation of aerated and anoxic steps during the reaction stage. The highest TOC and COD removal efficiencies were obtained at 5 d HRT for all the sequences tested. The use of a completely aerobic treatment led to the formation of irregular flocs with a moderate presence of filamentous bacteria.

The inclusion of anoxic stages reduced their presence improving the sludge settleability. Thus, SBR can be considered as a potential alternative for the biological treatment of cosmetic wastewaters.

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