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Sequencing batch reactor technology coupled with nanofiltration for textile wastewater reclamation

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ABSTRACT

Textile wastewaters are characterized by high organic matter concentration and colour presence. Conventional treatments do not remove completely the colour since the aerobic bacteria cannot degrade the azo-bond of the reactive dyes. However, their elimination is a requirement for wastewater reuse. In this study, it is proposed the reuse of textile wastewater as process water by a hybrid process combining a sequencing batch reactor (SBR) process with nanofiltration (NF) membranes. The aim is to evaluate the colour removal yield in the SBR and to study the influence of the addition of NF retentate on the SBR feed. The laboratory SBR was operated in cycles of 20 h and was fed with a solution containing a mixture of three reactive dyes: Remazol Yellow RR, Remazol Blue RR and Remazol Red RR. Every day colour and COD removal efficiencies were determined. The NF retentate was stored in order to mix it with the synthetic wastewater for the SBR feed. Colour removal yield ranged from 85 to 90% for the red and blue dyes and from 70 to 75% for the yellow one when the SBR feed was only the textile synthetic wastewater. However, when the SBR feed was the mixture of 50% synthetic wastewater and 50% of NF rejection the colour removal efficiency was reduced between 10 and 15%.

1. Introduction

Owing to water scarcity textile industries are upgrading their wastewater treatment plants (WWTPs) with the aim of achieving a final effluent that can be reused.

When the first WWTPs for the textile mills were erected, conventional treatments, both physico-chemical and biological were implemented in order to meet the legal standards. The current tendency is the use of biological processes, since they achieve higher efficiencies, preparing the wastewater for further reclamation and reuse. In this way, although some authors have proposed treatments for the raw wastewater that do not include biological processes [1,2], in the last years most of the works have reported about systems based on biological processes that prepare the wastewater for further reclamation and reuse [3].

A typical raw wastewater from a dyeing, printing and finishing textile industry is characterized by high COD and conductivity values together with colour presence.

Conventional biological wastewater treatment processes reduce significantly the wastewater COD. However, these processes do not eliminate the colour and conductivity is hardly modified by a biological process. Minimizing the COD, eliminating the colour and reducing the conductivity value are of paramount importance in order to reuse the final effluent. Thus, the combination of different techniques is required.

The use of biological and membrane processes can be appropriate for achieving the aforementioned aims. In this way, Sahinkaya et al. [4] studied the combination of a conventional activated sludge (CAS) process and nanofiltration (NF) for treating wastewater from a Denim textile mill. 75% of the colour was eliminated in the CAS process due mainly to its adsorption onto the sludge flocks and NF membranes retained around the 65% of the wastewater conductivity. In the same way, in former works of our research group [5] NF was used as a treatment for the secondary effluent from a textile mill. Other authors also studied this processes combination [6,7].

However, membrane fouling may produce a fast decrease in the process performance [8,9]. A biological process degrading the reactive dyes could guarantee a less fouling tendency in a subsequent membrane stage.

Reactive dyes are normally azo-based chromophores combined with different types of reactive groups, e.g., vinyl sulphone, chlorotriazine. They are characterized by binding to the cotton fibres through covalent bonds [10].

It is well known that biological mineralization of azo-dyes requires the integration of an anaerobic process that degrades the azo-bond to aromatic amine intermediates and an aerobic process for their total degradation. [11,12]. Melgoza et al. [13] studied the degradation of a synthetic wastewater containing the colorant DB79. These authors observed that the DB79 was biotransformed to amines in the anaerobic stage decolorizing the wastewater. The

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amines formed were mineralized in the aerobic phase. The increase of toxicity in the anaerobic stage due to the amines formation and the wastewater detoxified after the aerobic treatment were also studied.

In this work a Sequencing Batch Reactor (SBR) process is proposed as biological treatment. The difference with a CAS process lies on the operation in cycles, which consist of a series of phases, including reaction and sedimentation [14,15].

The number of phases and their characteristics depend on the treatment objective. In the literature, studies of degradation of different industrial wastewaters (for example from tanneries and from chemical and petrochemical industries) by means of SBR technology have been reported [16,17]. In addition, it has been reported that the selection of microorganisms that are able to degrade synthetic compounds is enhanced by the dynamic conditions that are typical of periodic systems like SBRs [18,19].

Concerning colour removal by means of SBR processes, Venkata Mohan et al. [11] studied the degradability of an azo dye (C.I. Acid Black 210) using cycle periods of 24 h, alternating anoxic and aerobic phases at room temperature. A 100% of colour removal was achieved for volumetric organic loads of 0.56 and 0.75 $KgCOD/(m^3 d)$. Özer Çınara [20] compared the results obtained from three cycle durations (48 h, 24 h and 12 h). It was observed that the best performance in terms of colour removal and aromatic amine degradation was achieved with a 24 h cycle.

Shaw et al. used a 6 phase anaerobic/aerobic sequencing laboratory scale batch reactor for treating a synthetic textile wastewater including polyvinylalcohol and an azo-dye [21]. Gonçalves et al. [22] evaluated an anaerobic/aerobic system for the treatment of wool dyeing effluents. These authors worked with cycles of 24 h and studied the influence of the variation of the aerobic phase time.

Other authors worked with the SBR technology with only aerobic reaction phases but combining it with other techniques in order to eliminate the wastewater colour. Thus, Sirianuntapiboon and Sansak [23] combined the SBR technology with adsorption with granular activated carbon (GAC), inoculating both acclimated sludge and GAC in the reactor. These authors achieved colour removal efficiencies between 94 and 99% for the direct blue 201 dye. Sheng-bing Hea et al. used zeolite powder instead of activated carbon [24]. Harrelkas et al. [25] compared the degradation of two textile dyes (azo and phtalocyanine dyes) using a combination of photocatalysis on supported TiO_2 with an SBR operated aerobically.

In this work the treatment of a simulated textile wastewater was carried out with a laboratory scale SBR in order to eliminate COD and colour. Moreover, the influence of the conductivity on the reactor performance was studied with the aim of evaluating the possibility of recycling the NF reject stream to the SBR.

There is hardly literature on the biological degradation of the Remazol dyes used in this research work. Onli Kapdan and Ozturk reported about the biological degradation of the Remazol Red RR [26]. In addition, for these dyes type Demir et al. [27,28] proposed their biodegradation by means of White rot fungus *Phanerochaete chrysosporium*, in contrast to the method proposed in our work.

Table 1
Dyestuff properties (VS: vinyl sulphone, MCT: monochlorotriazine) [29].

Dyestuff	Remazol Yellow RR	Remazol Red RR	Remazol Blue RR
Structure	Monofunctional	Bifunctional	Bifunctional
Reactive group	VS	MCT/VS	VS/VS
Azo-bonds number	1	1	2

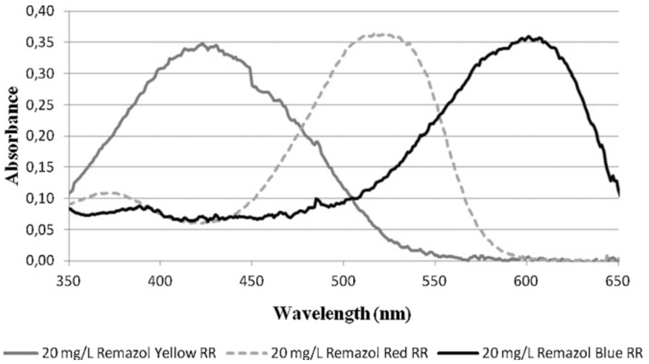

Fig. 1. Absorbance spectra of the dyes at the working concentration (20 mg/L).

Although the biological treatment of azo-dyes and the combination of aerobically operated SBRs have been previously reported, as explained above, the combination of SBR under alternate anaerobic-aerobic conditions and NF has not been previously studied. In this process combination, the fouling of the NF membrane will be potentially reduced due to the previous azo-dyes degradation.

2. Materials and methods

2.1. Dyes

The dyes used for the simulated wastewater solution were Remazol Yellow RR, Remazol Red RR and Remazol Blue RR from DyStar. Remazol are vinyl sulphone fiber-reactive dyes. The main characteristics of the three dyes used are summarized in Table 1 [29]. Their absorbance spectra at the working concentration (20 mg/L) can be observed in Fig. 1.

2.2. Simulated wastewater

The simulated wastewater had the following composition: 400 mg $C_6H_{12}O_6$ L^{-1}, 270 mg CH_3COONH_4 L^{-1} (equivalent to 600 mg COD L^{-1} and 50 mg N L^{-1}), 84 mg K_2HPO_4 L^{-1} (equivalent to 15 mg P L^{-1}), 25 mg $CaCl_2{\cdot}2H_2O$ L^{-1}, 36 mg $MgCl_2{\cdot}6H_2O$ L^{-1} and

Table 2
Characteristics of the prepared solutions for the SBR feed (STW = simulated textile wastewater, NFR = nanofiltration rejection).

Synthetic wastewater (SW)	Days of use of the synthetic wastewater	COD of the SW (mg/L)	Conductivity of the SW (mS/cm)	STW/NFR (L/L)	Feed COD (mg/L)	Feed conductivity (μS/cm)	pH	NT (mg/L)	PT (mg/L)
1	1–10	645	1183	5.0/0.0	645	1183	7.5	46	13.2
2	11–21	678	1200	5.0/0.0	678	1200	7.3	42	19.6
3	22–31	646	1241	5.0/0.0	646	1241	7.4	44	13
4	32–43	640	1150	5.0/0.0	640	1150	7.1	46	13
5	44–55	642	1237	5.0/0.0	642	1237	7.1	49	16.3
6	56–60	644	1130	4.0/1.0	525	1283	7.6	NM	NM
6	61–65	644	1130	3.5/1.5	407	1340	7.6	NM	NM
7	66–70	638	1146	2.5/2.5	325	1480	7.7	NM	NM

NM, not measured.

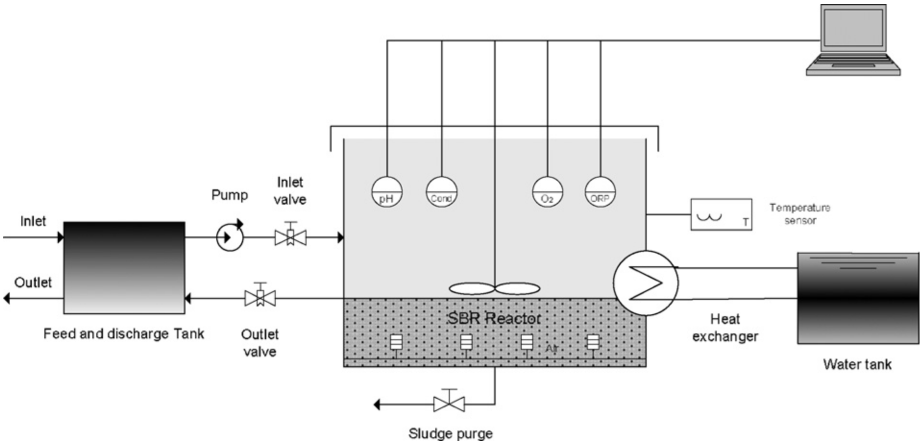

Fig. 2. SBR pilot plant used in the laboratory.

20 mg L^{-1} of each Remazol dyestuff. Chemicals were dissolved in tap water.

During the experiments seven different solutions were prepared according to the above mentioned recipe. The measured characteristics for these solutions, the period of their use in the experiments and the composition of the SBR feed solutions can be observed in Table 2.

2.3. Wastewater analysis

Colour was determined by measuring the absorbance values of the samples at three wavelengths (440, 520 and 600 nm), corresponding to the maximal absorbance of yellow, red and blue colours, respectively. These measures were performed with a Hewlett Packard model 8453 spectrophotometer. By measuring the absorbance values of solutions containing mixtures of the three dyes at different concentration ratios, it was determined that the absorbance values in the mixtures at the three wavelengths corresponded with dyes concentrations of approximately 10% lower than the real concentrations. This interference error was practically constant, independently of the dye concentration; thereby the results of colour removal expressed in % were not altered.

COD was determined by means of cell tests from Merck after 0.45 μm filtration. Mixture liquor suspended solids (MLSS) and mixture liquor volatile suspended solids (MLVSS) were measured following APHA [30].

2.4. Laboratory SBR and NF plants

A 25 L volume reactor was used as SBR for the experiments. The SBR operation was controlled by a computer equipped with a SCADA program and the transmitters were DL 421,422, 423,424 from Honeywell for pH, ORP, conductivity and dissolved oxygen, respectively. The electrodes were Meredian II for pH, ORP and conductivity and DL5000 for dissolved oxygen from Honeywell. A scheme is shown in Fig. 2.

Feed was carried out with a peristaltic pump (D-25V from DINKO) and the effluent was gravity withdrawn. Mixing was provided via a Heidolph mechanical stirrer. Air was supplied by means of an air blower and was diffused through porous ceramic diffusers into the reactor.

Temperature was maintained at 36 °C by means of a thermostatic bath in order to favour the anaerobic azo-dye degradation.

The NF laboratory plant was equipped with a pressure vessel for a spiral wounded membrane of 2.5 in. of diameter. The membrane tested was NF 270 from Dow Chemical, whose main characteristics can be seen in Table 3. The initial operating conditions were a feed flow rate of 400 L/h, a transmembrane pressure of 6 bar and a temperature of 25 °C.

Table 3
Characteristics of the NF 270 2540 membrane.

Manufacturer	FILMTEC, DOW Chemical
Type	Spiral wound membrane
Material	Polyamide thin film composite
Cut-off	155 Da[a]
Average pore diameter	0.84 nm[b]
Active area	2.6 m^2
Permeate flow rate[c]	3.2 m^3/d
$MgSO_4$ rejection[c]	>97%
Maximum operating pressure	41 bar
Maximum operating temperature	45 °C
pH range	2–11

[a] Ref. [31].
[b] Ref. [32].
[c] Test conditions: 4.8 bar, 25 °C, 15% of permeate recovery 2000 ppm of $MgSO_4$.

2.5. Methodology

Two series of experiments were carried out in the laboratory SBR. In the first one, activated sludge taken from a municipal wastewater treatment plant was used as seed in the SBR for the process start-up. In the second series of experiments, the simulated wastewater was mixed proportionally with the reject stream of the NF membrane treating the SBR effluent to increase the conductivity gradually.

The operation of the SBR in both series of experiments was carried out according to the data of Table 4. The volume exchange ratio (VER) is defined as the quotient between the fill volume and the reaction volume. The hydraulic retention time is defined for SBR similar to continuous flow activated sludge systems as V_T/Q

Table 4
SBR phases duration in the experiments.

Cycle steps	Duration
Anaerobic fill phase	0.2 h
Anaerobic reaction phase	9 h
Aerobic reaction phase	8 h
Settling phase	2 h
Draw phase	0.3 h
Idle phase	0.5 h
Total cycle time (T_C)	20 h
Reaction volume	15 L
Volume Exchange ratio	1/3
Hydraulic retention time (HRT)	2.5 d

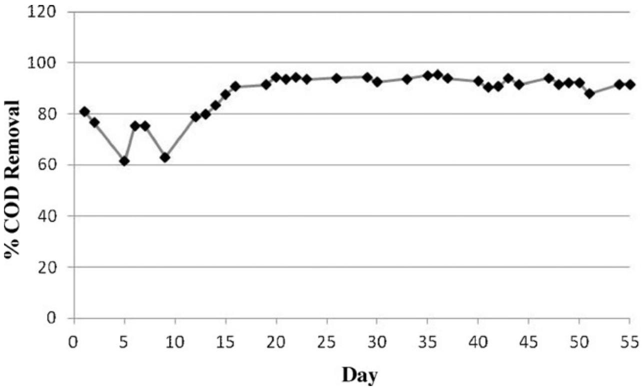

Fig. 3. Evolution of COD removal in the first series of experiments.

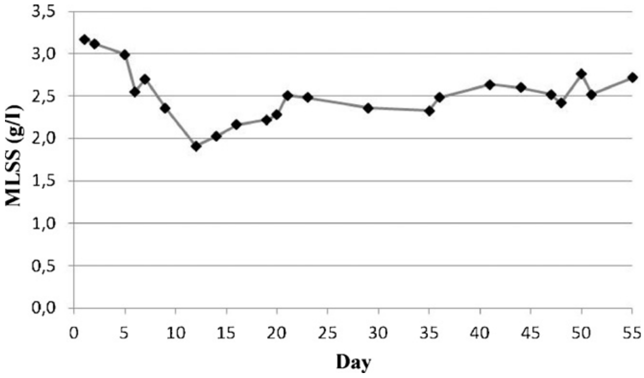

Fig. 4. Suspended solids in the mixture liquor in the SBR.

and can be also expressed as the total cycle time (T_C) divided by the VER.

In the last two weeks of the first series of experiments, SBR supernatant was stored in order to be used as NF feed. Previous to NF, the SBR supernatant was filtered with a 5 μm cartridge filter. At the beginning, the NF plant was operated in a total recirculation mode, i.e. recirculating both permeate and reject streams to the feed tank. Once the steady state was reached, 5 L of permeate were withdrawn in order to increase the feed concentration. It was repeated periodically every for hours until a volume concentration factor (VCF) of 1.7 was achieved. The VCF is defined as the relation between the initial water volume and the remaining water volume in the feed tank after the permeate extractions. Every day, 4 L of SBR supernatant were added to the NF feed tank, taking from the system 1.5 L of permeate and 2.5 L of reject stream. In this way, the feed concentration was maintained approximately constant during NF. The withdrawn rejection samples were mixed with the synthetic wastewater to feed the SBR.

3. Results

3.1. Series 1

Fig. 3 shows the COD removal performances in the operation period of the reactor with simulated wastewater. It can be observed that, after initial fluctuations, the COD removal increased gradually during the first 16 days of operation, reaching a value higher than 90%. During the rest of the operation, the COD removal yield was maintained above 90% (except from day 51th).

The evolution of the MLSS demonstrates that the start-up of the reactor was approximately two weeks. Thus, it can be observed an initial decrease in the MLSS concentration because of the biomass acclimation period and a subsequent increase, once the acclimation was realised (Fig. 4). Lourenço et al. [33] reported higher acclimation times but this was due to the higher dye concentrations used.

Concerning the MLVSS, it has to be mentioned that the percentage of volatile solids in the sludge was almost constant during the SBR operation (80–82%).

Colour elimination was evaluated at three different wavelengths, corresponding to the maximum of absorption of each dye. It was observed (Fig. 5) that the initial elimination values increased considerably after the first days of operation. From these initial days onwards, colour removal was practically constant except for 4 days (between days 20th and 24th). During those days, a decrease in colour removal, which reached values very similar to those obtained in the first days of operation, was detected. This was due to an operation problem that entailed a lack of anaerobic phases in three cycles.

It must be highlighted that the yellow dye elimination was always lower compared with those obtained for the other two dyes. This difference was approximately of 10%. Thus, the initial colour removals at the wavelengths of 520 and 600 nm were approximately of 80% in both cases, meanwhile the colour removal at 440 was round 70%. The lower yellow colour elimination is caused by the more complexity of the yellow dye molecule. In fact, its toxicity in terms of EC50 (Daphnia Magna, 48 h) is 100 mg/L meanwhile red and blue dyes have lower toxicities, according to the safety data from the dyes provider. The maximum performance values were 87.5% for the blue dye, 88.4 for the red and 76.2 for the yellow one.

These results are similar to those obtained by Lourenço et al. [34], working with Remazol Brilliant Violet 5R (up to 90% elimination efficiency) and Remazol Black B (up to 75% removal efficiency). In the same way, Sponza and Isik [35] reported 86% of Direct Black 38 dye removal efficiencies operating a UASB reactor with a HRT of 2.9 days. However, higher Remazol Red RR removal efficiencies were reported by Kapdan and Ozturk working at a temperature of 28 °C in a SBR [26].

Figs. 6 and 7 show the variation of COD and colour, respectively, in the anaerobic reaction phase of a cycle in the last period of operation in the series 1. This experiment was replicated three times with the same results.

It can be observed how COD lowered sharply in the first 2 h of the anaerobic phase, what was mainly due to the organic matter adsorption onto the biomass flocks and to the quick assimilation of the rapidly biodegradable organic matter by some aerobic bacteria that are subjected to stress conditions in anaerobic phases. After that, COD was constant until the 5th hour, increasing slightly at the end of the period probably due to the release of residual material from the cells.

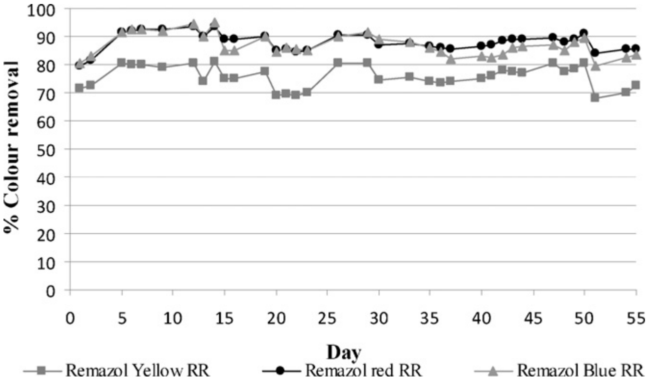

Fig. 5. Colour removal yield in the SBR.

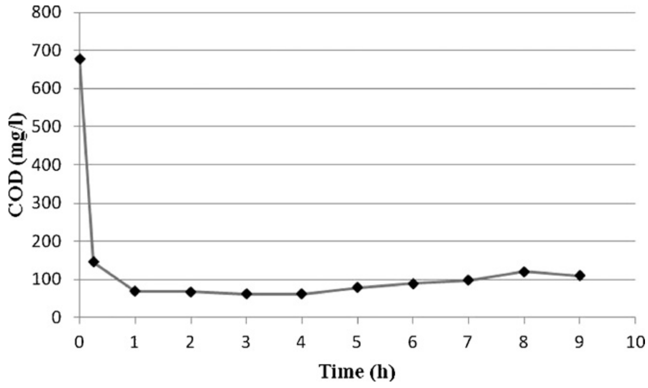

Fig. 6. Kinetics of COD removal in the anaerobic phase of the SBR.

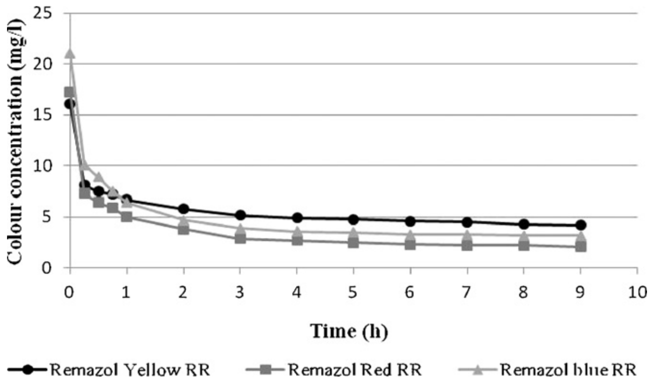

Fig. 7. Kinetics of colour removal in the anaerobic phase of the SBR.

Mineralization of the COD was carried out in the aerobic phase. It includes the COD adsorbed on the flocks, the COD taken up and stored internally in the cells and a part of the soluble COD remaining after anaerobic stage. During the aerobic phase, soluble COD lowered from around 100 mg/L to approximately 50 mg/L.

Colour was almost completely removed in the anaerobic phase. Its elimination in the aerobic phase was negligible.

In order to check if first-order decolourization kinetics with respect to dye concentration could be applied in the anaerobic phase, the values of $\ln(C/C_0)$ were plotted against the time in the anaerobic phase. An example for the three dyes can be observed in Fig. 8.

Concerning the kinetic of the dyes degradation it can be seen that after the initial decrease due to dilution and adsorption onto the flocks, their concentrations decreased gradually. This behaviour is the same for the three dyes. It can be noticed that there are two successive periods with first order kinetics. Lourenço et al. [36] observed a similar behaviour. The kinetic coefficients are summarized in Table 5.

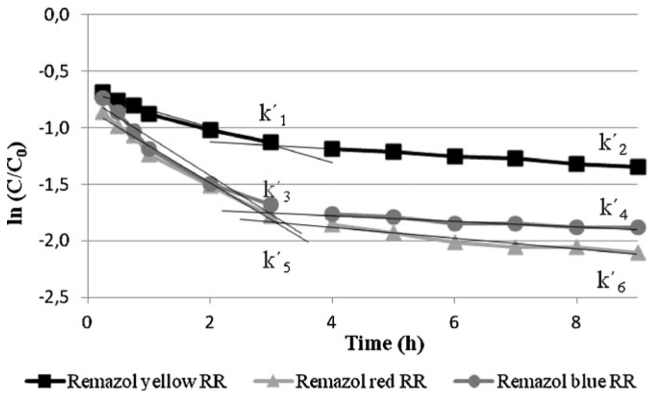

Fig. 8. Evaluation of the kinetic coefficients for the decolourization of the dyes in the anaerobic phase of the SBR cycle.

Table 5
Apparent first-order decolourization rate constants (h^{-1}) ($T = 36\,^\circ C$).

Dye	First period	Second period
Remazol Yellow RR	$k'_1 = 0.156$	$k'_2 = 0.328$
Remazol Red RR	$k'_3 = 0.028$	$k'_4 = 0.029$
Remazol Blue RR	$k'_6 = 0.156$	$k'_6 = 0.328$

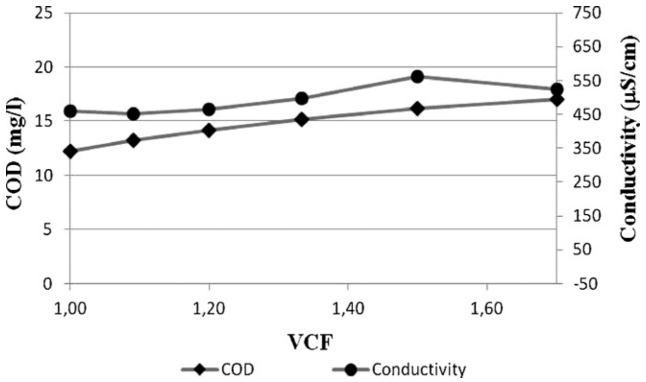

Fig. 9. COD and conductivity values measure in the NF permeates at different VCF.

It must be mentioned that the study about the further degradation products of the azo-dyes was not an objective of this work. Detailed information about degradation of the aromatic amines compounds can be found in [37]. Their biodegradation depends on the position, type and number of substituents in the aromatic ring.

3.2. Series 2

In this series of experiments, the SBR feed solution was the mixture of simulated wastewater and the rejection stream of a laboratory NF plant that treated the SBR effluent.

Concerning the NF experiments, Fig. 9 illustrates the COD and conductivity values measured in the permeate samples for the different VCF tested according to the methodology. It can be observed that the COD and the conductivity measured values increased with the VCF as expected. However, they were lower than 25 mg/L and 750 µs/cm respectively, even for the maximal VCF tested. During the continuous operation at VCF = 1.7, the flux was maintained practically constant (45 L/(m^2 h)) at the operating transmembrane pressure, what indicated that membrane fouling was not detected.

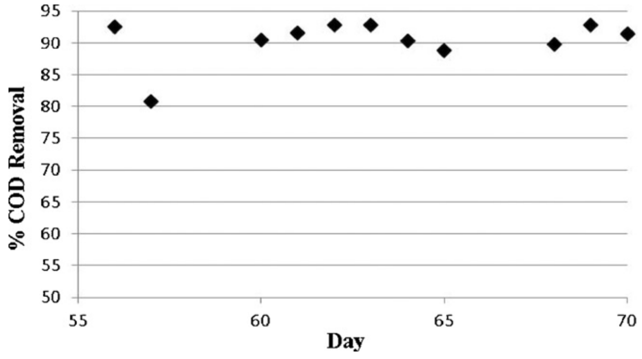

Fig. 10. COD removal yield in the experiments with increasing conductivity.

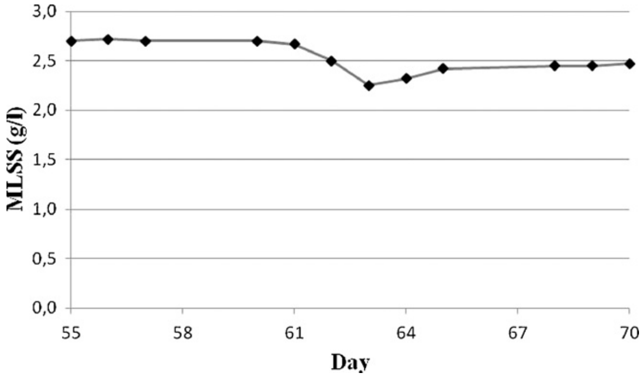

Fig. 11. Suspended solids in the mixture liquor in the SBR in the series 2.

Fig. 10 illustrates the variation of the COD removal performance in this experiments series. The origin of the graph coincides with the last operation day of the series 1. It can be observed that in spite of an initial decrease, COD removal efficiencies were very similar to those obtained with only textile simulated wastewaters. These results were expected since the effect of the NF recirculation was the dilution of the SBR feed in terms of COD. Thus, the organic load (kgCOD/kgMLVSS d), i.e. the relationship feed/microorganisms, was lower, what usually implies a higher performance in the biological process. In this way, it can be concluded that the negative effect of the partial biomass inhibition could be hardly observed due to the decrease of the organic load.

The evolution of the MLSS in this experiment series can be observed in Fig. 11. At the beginning the MLSS concentration did not vary, but from day 61th to 63th MLSS tended to diminish. It has to be commented that in the day 61th the volume of the NF reject stream in the SBR feed was increased up to 1.5 L. From the day 64th a slight increase in the MLSS concentration was observed.

On the contrary, the conductivity increase of the SBR feed affected considerably to the colour removal efficiency in the anaerobic phase (Fig. 12) in spite of the colour diminution in the SBR feed by dilution after mixing simulated wastewater with NF rejection. For mixtures of 50% of simulated wastewater and 50% of NF rejection (conductivity value = 1480 μS/cm), the yellow dye elimination decreased down to 65%. Concerning the blue and red dyes, their removal efficiencies decreased a 10% in comparison with those obtained for a mixture of 3.5 L of simulated wastewater and 1.5 L of NF rejection. The values of the global removal efficiencies were around 70%.

These results lead us to comment that for an industrial operation of the process special attention should be paid to the conductivity fluctuation in the reactor. An equalization tank would be necessary, above all to avoid a deterioration of the colour removal efficiency.

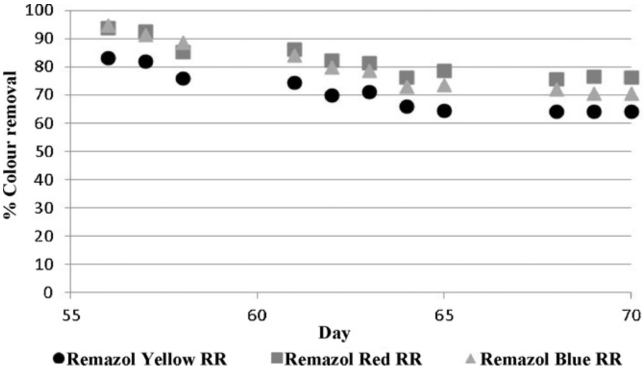

Fig. 12. Dye removal efficiencies in the experiments with increasing conductivity.

Globally it can be stated that the combination of SBR and NF presents advantages in comparison with other technologies in textile wastewater reclamation. NF is the technique that makes possible the production of reclaimed wastewater with enough quality. With the membrane used in this work around 60% of the wastewater conductivity was eliminated. A previous biological process is necessary to prepare the wastewater for the NF stage and by means of a SBR both the biodegradable COD and the colour can be eliminated alternating anaerobic and aerobic phases. Besides, the dyes degradation entails a positive effect on the membrane performance since fouling will be reduced.

4. Conclusions

Textile wastewater reuse is only possible if organic matter and conductivity are reduced and colour is removed. In order to achieve this, a combination of a biological treatment including anaerobic and aerobic conditions and nanofiltration is proposed.

A SBR alternating anaerobic and aerobic phases is suitable for colour removal. In this way, for a simulated textile wastewater, yields of almost 90% were achieved in the removal of the reactive dyes Remazol Blue RR and Remazol Red RR. Lower yields were obtained in the Remazol Yellow RR removal (the maximum yield was 76.2%).

The acclimation of the sludge was of 16 days at the working concentration. COD removal efficiencies were higher than 90% during all the reactor operation.

Two successive periods with first order kinetics are proposed for the anaerobic degradation of the dyes according to the experimental data.

The mixing of the simulated wastewater with rejection of a nanofiltration laboratory plant treating the SBR effluent was performed in order to study the influence of the conductivity increase on the SBR performance. At an industrial scale, the NF rejection should be partly treated by the SBR.

Results of the combined feed showed that percentages of NF rejection higher than 40% imply non acceptable colour removal efficiencies (below 70% for the red and blue dyes and below 60% for the yellow one).

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