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Effect of variability on the treatment of textile dyeing wastewater by activated sludge

A. Alinsafi [a,b], M. da Motta [c], S. Le Bonté [b], M.N. Pons [b,*], A. Benhammou [a]

[a] *Laboratoire d'Automatique et d'Etude des Procédés, Faculté des Sciences Semlalia, Avenue Prince Moulay Abdellah, Marrakech, Morocco*
[b] *Laboratoire des Sciences du Génie Chimique, CNRS-ENSIC-INPL, 1, rue Grandville, F-54001 Nancy Cedex, France*
[c] *Departamento de Engenharia Química, UFPE, Av. Prof. Arthur de Sá, s/n, Cidade Universitária, Recife, 50.740-521 PE, Brazil*

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Abstract

Using a pilot scale continuous system with activated sludge, the effect of the variability of non-pretreated synthetic textile wastewater containing reactive dyes on the pollution bio-removal and on the characteristics of activated sludge (sludge volume index, floc size and shape, filamentous bacteria abundance) has been monitored. Off-line batch respirometry tests have been used in parallel to assess the toxic effects of some of the reactive dyes. Experiments were run by increasing the concentration of a single dye (Run 1) and by feeding with a non-repetitive (Run 2) or a repetitive (Run 3) sequence of dyes. In all runs dyes were added to a synthetic wastewater of constant composition. Although the biomass was significantly upset by some of the dyes, it was able to cope up with the variations of their chemistry and remained active throughout the experiments and could contribute to COD removal. The dyes were partially adsorbed but not degraded by the micro-organisms.

Keywords: Filamentous bacteria; Image analysis; Morphology; Settleability; Spectrophotometry; Toxicity

1. Introduction

Wastewater discharged from a textile plant shows large fluctuations in terms of quantities and pollution load, depending upon customer orders, types of manufactured textile materials and production schedules.

Taking into example the case of a cotton knits plant, a number of processes are associated with textile production. They include the weaving of the fabric and its preparation by bleaching, dyeing and finishing and they generate different types of wastewater (Fig. 1).

Textile wastewater contains considerable amount of suspended solids and of weakly biodegradable substances such as additives, detergents, surfactants and dyes. It exhibits highly fluctuating pH, high temperature and COD concentration [1]. In consequence, these effluents present aesthetic and environmental problems by absorbing light in receiving bodies (river, lakes, etc.) and by interfering with aquatic biological processes.

Even when equalization tanks are installed to damp the variations of the incoming wastewater, its treatment has still to deal with severe disturbances. A biological step is often included in the treatment sequence because it is considered inexpensive and efficient towards COD removal. However, even in the case of physical and/or chemical pre-treatment, these disturbances can affect the

* Corresponding author. Tel.: +33 3 83 17 52 77; fax: +33 3 83 17 53 26.
E-mail address: Marie-Noelle.Pons@ensic.inpl-nancy.fr (M.N. Pons).

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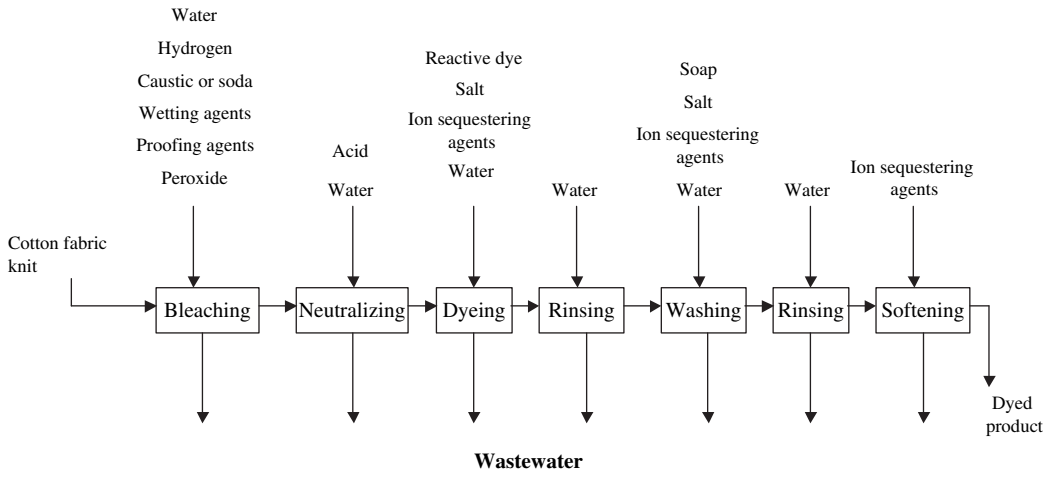

Fig. 1. Typical flowsheet of a textile cotton knits plant.

biomass. Due to their slow growth the micro-organisms have no time to adapt their metabolism to the fast wastewater composition variations. For example the maximum specific growth rate reported for heterotrophic bacteria for municipal wastewater is about 4–8 day^{-1} at 20 °C [2].

Methods for dealing with textile wastewater consist of various biological, physical and chemical treatment methods that can be applied separately or be combined [3–5]. Chemical treatment employing inorganic coagulants especially, not only alum ($Al_2(SO_4)_3$) [6] and polyaluminium chloride [7] but also dolomite ($CaMg(CO_3)_2$) [8], magnesium chloride ($MgCl_2$) [9] and bittern [10,11] has been found to be efficient for colour removal in textile and tannery wastewater. Adsorption employing activated carbon [12] and various other adsorbents such as silica [13,14] or wood [15] has been quite widely studied and applied. Advanced oxidation processes (AOPs) based on titanium dioxide either in aqueous suspension [16–18], in combination with activated carbon [19,20] or deposited as a thin-film deposited on a glass surface [21] have been proposed. Electrochemical methods have been investigated directly to treat textile wastewaters from several dyeing and finishing mills [22] or by combination with photocatalysis [23]. The combination of ozonation, chemical coagulation, and activated sludge processes can be very effective due to the capability of ozonation to decompose large and complex dye molecules into smaller ones [24]. Ozonation can be substituted by electrochemical oxidation [25]. Park and Lee [26] have tested the combination of a fluidized bed biofilm preceding an FBAS-C or succeeding it (C-FBAS) to treat dyeing wastewater: a better treatability was achieved with FBAS-C than C-FBAS in terms of the total sludge production. Bioprocesses are often used as a polishing step after dyeing wastewater chemical or physical pretreatment.

In biotreatment by activated sludge, the dyes are adsorbed on the biomass, more than they are effectively degraded [27–29]. Sorption is considered as the first mechanism for total or partial removal of dyes by biomass [30–33]. It is referred to as bio-elimination and its efficiency depends widely on the type of dye, i.e. on its molecular structure, colouring group and solubility [27,34].

Recent studies focus on the study of the adsorption of reactive dyes on dried activated sludge and the capacity of the biosorbent to bind to the dye as a function of initial pH, dye type and concentration [35]. Others have attempted to use some specific bacterial genera such as *Aeromonas* sp., *Pseudomonas luteola* or *Escherichia coli* [36] as biosorbents. A facultative anaerobic consortium has been tested alone [37] or in combination with activated sludge [38]. Species such as algae [39] or white-root fungi, e.g. *Coriolus versicolor* (alone [40] or in combination again with activated sludge [41]) or *Phlebia tremellosa* [42] have been proposed. Generally speaking biomaterials are under investigation for sorption of dyes as of many other pollutants [43–45].

Whatever the disturbances and the treatment process, the pollution levels on effluent discharged to a receiving water body (river, lake, etc.) considered for re-use on the industrial site should be maintained within strict limits. The paper focuses on the investigation of the effect of dyeing wastewater on the behaviour of an activated sludge system, and especially on its stability.

2. Materials and methods

The experimental set-up is composed of an aerated reactor (38 l) and a clarifier (9 l). The sludge is recycled from the clarifier by a peristaltic pump (recycle rate of 1.5 l/h). Five gas diffusers are located on the floor of the reactor. The clarifier is equipped with rotating wall and

surface scrappers. Bacteria are fed with artificial wastewater corresponding to a chemical oxygen demand (COD) of 400 mg/l for Runs 1 and 2 and of 200 mg/l for Run 3. One litre of concentrated substrate [46] contains 628 g of meat extract (Viandox®, Amora, Dijon, France), 33.82 g of sucrose, 27.06 g of NH_4Cl and 3.68 ml of phosphoric acid corresponding to a concentration of 20 000 mg COD/l. The synthetic wastewater (feed rate 1.5 l/h) at 400 mg COD/l (200 mg COD/l) is obtained by dilution of 100 ml (50 ml) of concentrated substrate in 50 l of tap water and by addition of commercial reactive dyes: Drimarene K (Clariant, La Défense, France), Procion (DyStar, Frankfurt, Germany) (Table 1) and Dylon (Plymouth, England) dyes. Each dye is characterised by its wavelength corresponding to the maximum of its UV–visible absorption, although it can exhibit secondary absorption bands.

The first run (Run 1) has been performed with an increasing concentration of reactive Drimarene Blue DR KBL CDG: 3 days at 10 mg/l, 3 days at 25 mg/l, 8 days at 50 mg/l, then no dye. For the second run (Run 2) the biosystem was fed sequentially by a series of Drimarene K reactive dyes, each at a constant concentration of 50 mg/l. Finally for the third run (Run 3) an alternate feeding of the three Dylon reactive dyes (green, yellow and grey) at an average concentration of 138 mg/l was employed.

For Run 2, the nature of the dye in the feed was changed every 24 h and the dye concentration was kept constant (50 mg/l). There was no repetition except at the end when dyes C1 and C2 were re-injected on days 15 and 16. In order to calculate the concentration of each dye present in the system at a given time, a system of linear algebraic equations was solved:

$$\mathbf{A} = \mathbf{B} \times \mathbf{C} \quad (1)$$

where $\mathbf{A}(n)$ is the array of absorbances measured at the n wavelengths in the range 360–700 nm, $\mathbf{B}(n,m)$ the matrix of coefficients relating the absorbance at the n wavelengths to the concentrations of the m dyes, and $\mathbf{C}$, the array of dye concentrations.

For each run the reactor was inoculated with activated sludge obtained from a municipal wastewater treatment plant and the biosystem was adapted to the synthetic substrate without any dye for a period of at least 8 days. The synthetic wastewater, the mixed liquor and the clarifier supernatant were regularly sampled. After filtration (pore diameter 7 μm) their UV–visible spectrum in the range 200–800 nm was determined on a SECOMAM Anthelie Light spectrophotometer (Domont, France). Their COD could not be measured due to interferences with sample colour.

The settleability characteristics were measured daily in a 2 l cylindrical column. After sludge settlement, the supernatant turbidity (in FTU) was measured at 450 nm with a DR 2000 spectrophotometer (Hach, Loveland, Colorado). The sludge concentration (MLSS) was measured by centrifugation of a sample aliquot for 15 min at 3000 rpm and by drying the solid residue for 24 h at 105 °C. Microscopic inspection was also conducted daily and coupled to automated image analysis to evaluate the filamentous bacteria abundance and the size and shape (in terms of their roughness) of the flocs. An optical microscope (Dialux 20, Leitz, Wetzlar, Germany) equipped with a video camera (Hitachi CCTV model HV-720E(F), Horten, Norway), connected to a PC via a Matrox Meteor (Montréal, Canada) grabbing board was used. A drop of mixed liquor was carefully deposited and covered with a cover slip. A series of at least 70 images (magnification ×100) was grabbed by a systematic examination of the slide. If necessary a second slide was used. A procedure, called FlocMorph V.0, has been developed with Visilog 5 (Noésis, Les Ulis, France) [47]. The flocs are characterised by their projected equivalent diameter (D_{eq}) and their roughness, expressed by the fractal dimension (D_f) of their projected contour. The number of filaments (N_f) and their total length (L_f) per image are also given.

In parallel, acute toxicity tests have been conducted in batch respirometers [48] by comparison of the respiration of municipal wastewater with and without dye, based on the amount of oxygen consumed during 15 min by bacteria. For these tests fresh municipal

Table 1
Feeding schedule of Run 2

Dye	Code	λ_{max} (nm)	Day	Dye	Code	λ_{max} (nm)	Day
No dye			0	Blue P herd	C10	616	10
Green	C1	661	1	Red DR BT KBL CDG	C11	515	11
Violet	C2	549	2	Orange DR KGL CDG	C12	398	12
Yellow DR K4G	C3	422	3	No dye			13
Blue DR K2LR CDC	C4	614	4	Red DR K4BL CDG	C13	542	14
Orange DR K3R CDG	C5	488	5	Green	C1	661	15
Yellow P hexl	C6	419	6	Violet	C2	549	16
Blue DR KBL CDG	C7	591	7	No dye			17
Yellow DR KR	C8	437	8	No dye			18
Crimson P hexl	C9	547	9	No dye			19

DR = Drimarene, P = Procion.

activated sludge, not adapted to any dye, was used. Batch reactors, of 2 l working volume, were used. An SOS Pack 2 probe (Orbisphere, Hach Ultra Analytics, Vésenaz, Switzerland) connected to a PC allows to monitor the dissolved oxygen concentration with respect to time, in response to pulse addition of domestic wastewater (0.36 l) spiked with dyes to 1.84 l of activated sludge. The following mass balance can be written (Eq. (2)):

$$\frac{dC}{dt} = K_l a(C_s - C) - OUR_{end} - OUR_{exo} \quad (2)$$

where $K_l a$ is the oxygen mass transfer coefficient (s^{-1}), C is the dissolved oxygen concentration (mg/l) in the reactor liquid phase, C_s is the dissolved oxygen concentration at saturation (mg/l), OUR_{exo} (mg/l/s) is the exogenous uptake rate, resulting from the consumption of rapidly biodegradable substrates and OUR_{end} (mg/l/s) is the endogenous oxygen uptake rate, resulting from the consumption of more slowly biodegradable substrates as well as storage materials (maintenance). The volume of oxygen consumed for the first 5 min after the injection of the wastewater sample, $V(O_2)$, is calculated by integration of OUR_{exo} with respect to time. The three phases reported in Fig. 2 are successively used to evaluate OUR_{end} (phase II), $K_l a$ and C_s (phase III) and OUR_{exo} (phase I).

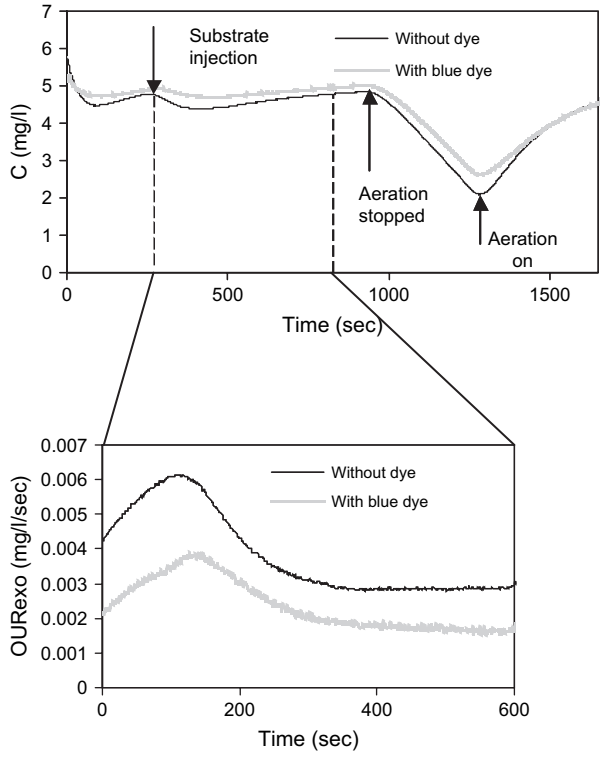

Fig. 2. Dissolved oxygen concentration in respirometer and exogenous uptake rate with and without blue DR K2LR CDG reactive dye added to the wastewater sample.

3. Results

3.1. Acute toxicity

The level of acute toxicity with respect to the activated sludge was investigated by respirometry for some of the dyes (C2, C4, C5, C6 and C12) used in Run 2.

Fig. 2 presents the curves of dissolved oxygen concentration and exogenous uptake rate for the blue DR K2LR CDG dye (C4) at 50 ppm. After substrate injection, the decrease in dissolved oxygen concentration and the corresponding increase in the exogenous uptake rate are the indication of the degradation of the easily biodegradable organic matter. When the substrate contains the dye, the increase in OUR_{exo} is less important than without, due to the dye inhibitory action.

Fig. 3 gives the percentage of inhibition of bacterial activity, calculated by the decrease in the volume of oxygen consumed observed when dyes are added to the domestic wastewater. A reduction of up to 45% was observed, but it does not seem to be related to the dye concentration. The reduction depends strongly on the kind of dye. By examining the absorbance spectra at the beginning and at the end of respirometry tests, a decrease in the colour was observed. Considering that the test duration was limited to 15 min, it was found plausible that the dyes were adsorbed on the sludge and probably not really degraded.

3.2. Feeding with increasing concentration of a single dye (Run 1)

In Run 1, the concentration of the blue dye was slowly increased after stabilisation of the reactor. Time

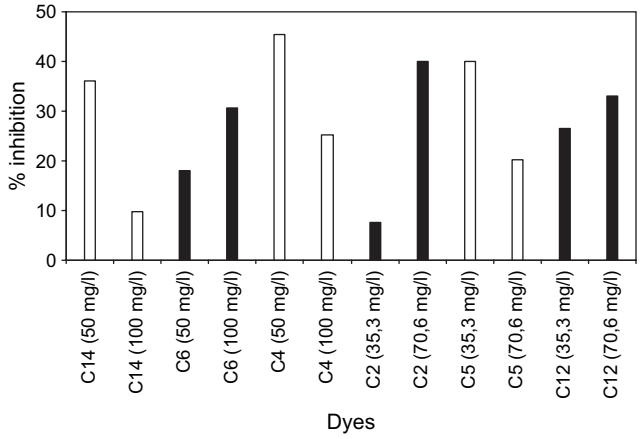

Fig. 3. Percentage of inhibition on bacterial activity for some reactive dyes.

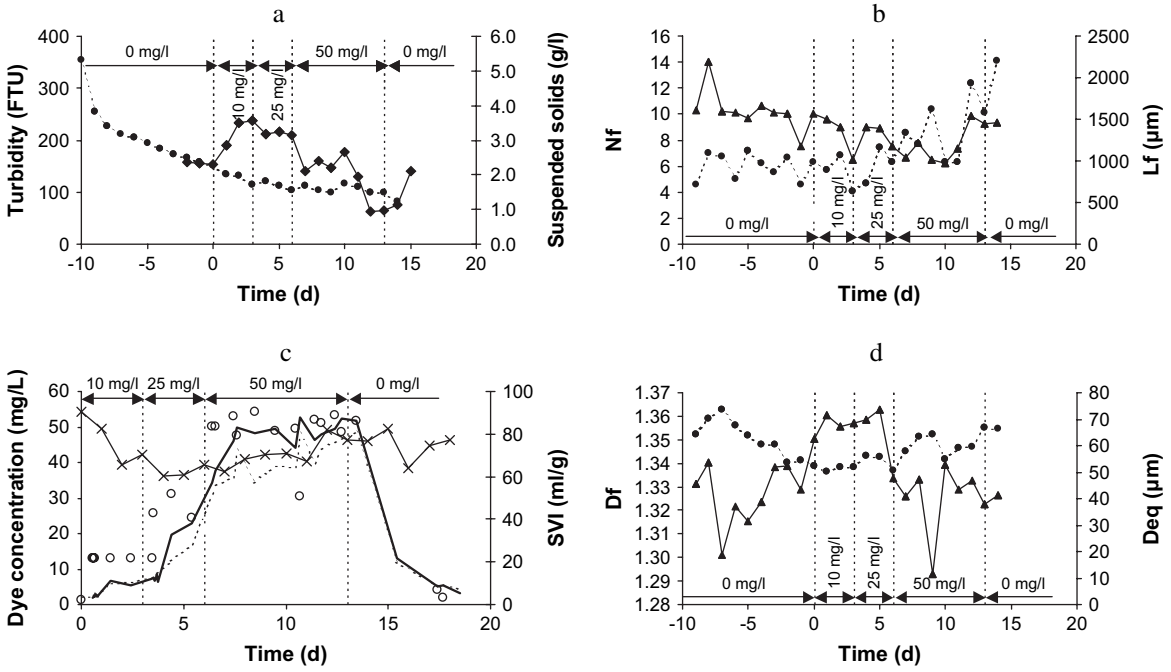

Fig. 4. Run 1, a: clarifier supernatant turbidity (◆), mixed liquor suspended solids (●); b: number (N_f) (▲) and total length (L_f) (●) of filaments per image; c: dye concentration in feed (○), bioreactor (- - -) and clarifier (—), sludge volume index (—×—); d: floc equivalent diameter (●) and fractal dimension (▲).

zero is the first day of dye addition to the feed. The fate of the blue dye was easily quantified by the changes in absorbance at 591 nm. A decrease in the dye concentration in the filtrated mixed liquor and in the clarifier supernatant was observed for dye concentrations in the feed of 10 and 25 mg/l (Fig. 4). However for the higher dye concentration, no decrease was observed. In fact the dye concentration in the clarifier supernatant was equal to the dye concentration in the feed, although it was slightly lower in the mixed liquor. This suggests that a part of the dye is adsorbed on the sludge in the reactor and is desorbed in the clarifier. During this run, no macroscopical detrimental effect of the dye on the sludge could be observed. The mixed liquor suspended solids concentration decreases regularly, at the same rate that during the stabilisation period without any dye. The slow loss in solids is due to the nature of the synthetic substrate. The filament abundance increases slightly when the dye concentration is set at 50 mg/l, but no filamentous bulking is observed, the sludge volume index remaining in the range 60–80 ml/g, which is quite acceptable. The fractal dimension stays in the range 1.29–1.36, and does indicate significant changes in the floc roughness. It was difficult to use turbidity to monitor the experiment as the presence of the dye interferes very much with the measurement.

3.3. Feeding with a non-repetitive sequence of dyes (Run 2)

On the injection day of a new dye, the dye concentration in the clarifier and in the reactor first increases and then decreases gradually (Fig. 5), once the feed of that given dye stopped. This is due to the

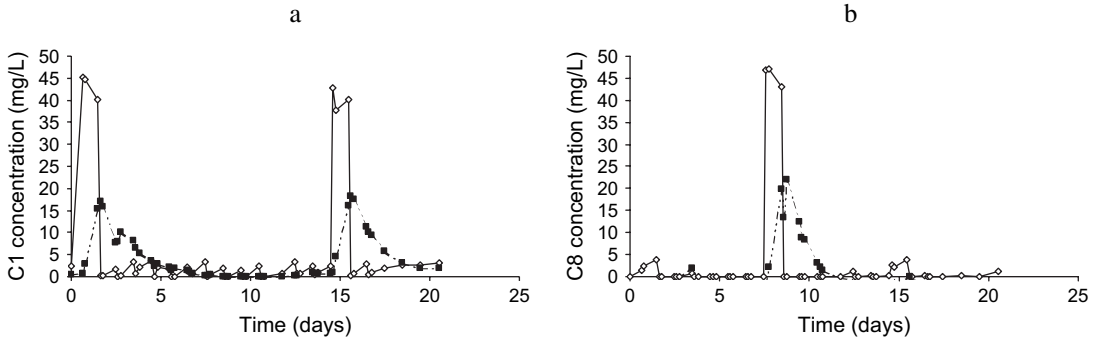

Fig. 5. Example of dye concentration monitoring during Run 2. C1 (a) and C8 (b).

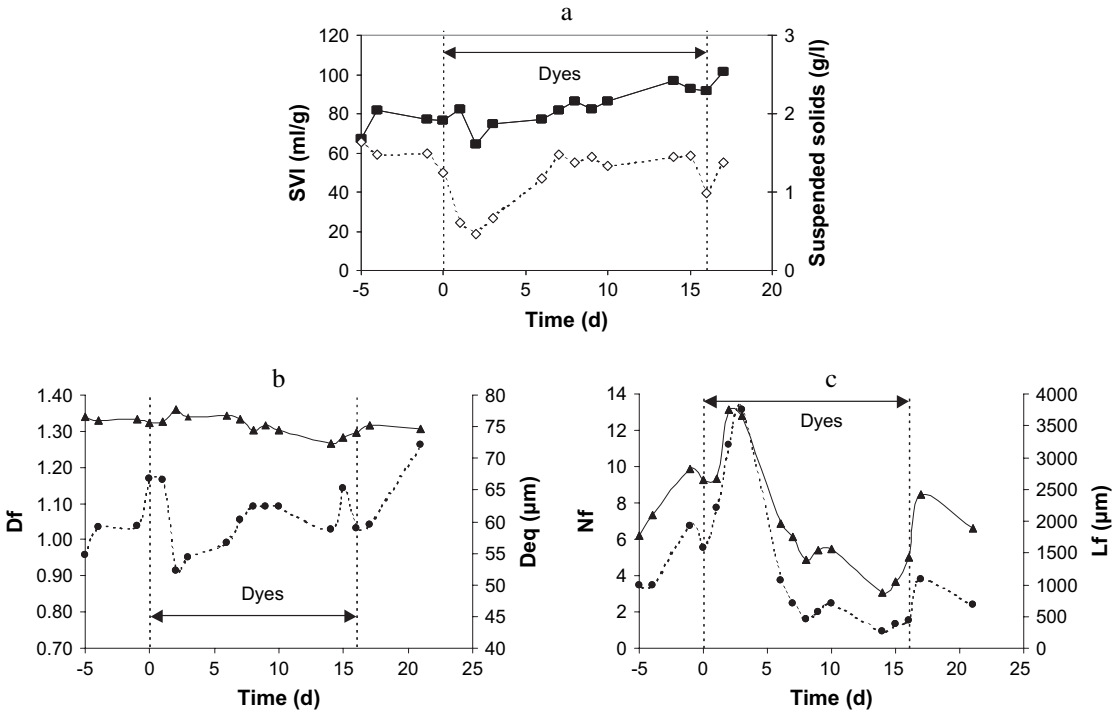

Fig. 6. Run 2, a: sludge volume index (SVI) (■) and mixed liquor suspended solids (◇); b: floc projected diameter (●) and fractal dimension (▲); c: number (N_f) (▲) and total length (L_f) (●) of filaments per image.

hydrodynamics of the total system. The global colour of the clarifier supernatant remained very intense during the whole experiment as the adsorption capacity of the sludge is reached. For several dyes they seemed to be present before they were really injected. This is due to the interferences between dyes which exhibit absorption bands at similar wavelengths. Each commercial dye is in fact the mixture of different but unknown chromophores. Furthermore some interference with the natural colour of the meat extract cannot be excluded. Before the dyes were injected on day 1, the filament abundance was slightly increasing, although the SVI was stable (Fig. 6). A severe change in the sludge state can be observed after day 1 with an increase in the filament abundance and a decrease in the MLSS concentration. The image analysis method quantifies the abundance of the filaments outside the flocs. Those inside the flocs constitute a kind of backbone to which zoogleal bacteria are attached. Dye addition seems to have induced a sudden defloculation. It has to be stressed that all the other ingredients of the feed were maintained constant. The sludge tends to recover from that shock with an increase in the MLSS concentration and a decrease in the filament abundance in the next days. Both parameters stabilised at values slightly lower than before dye addition. The re-injection of dyes C1 and C2 induces a new shock at the end of the experiment: the filament abundance increases again and the MLSS concentration decreases. When wastewater without dye is injected again the system tends to go back to its initial state. The floc size remains rather constant along the experiment. The floc fractal dimension, which characterises the roughness of the floc varies in agreement with the filament abundance: the floc gets rougher when the filament abundance increases. The SVI was stable, probably because neither the floc size nor the filament abundance varied to a great extent. It was again found difficult to monitor the turbidity of the clarifier supernatant during the experiment due to interference with the dye colour at 450 nm.

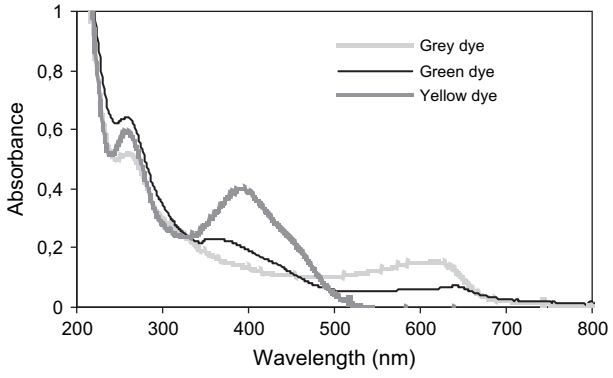

Fig. 7. UV/visible spectra of Dylon reactive dyes.

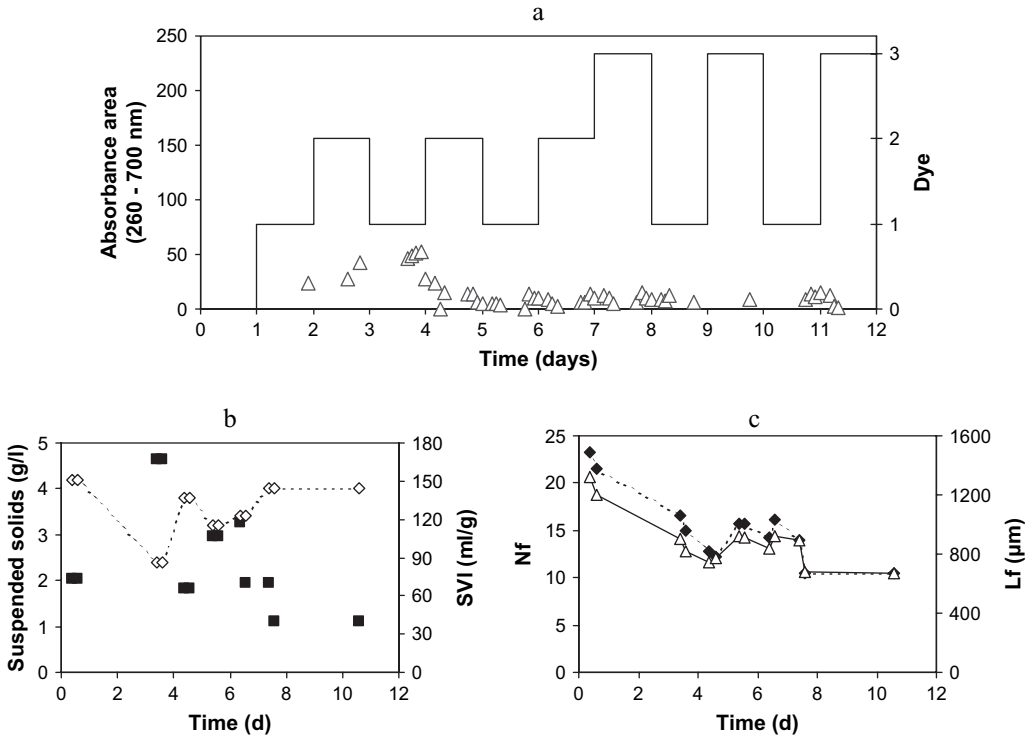

Fig. 8. Run 3, a: feeding schedule (1 = yellow, 2 = green, 3 = grey) and colour of the clarifier supernatant (△); b: mixed liquor suspended solids (◇) and sludge volume index (SVI) (■); c: number (△) and total length (◆) of filaments per image.

3.4. Feeding with a repetitive sequence of dyes (Run 3)

For Run 3, three Dylon dyes (green, yellow and grey) at an average concentration of 138 ppm were alternatively used for a period of 24 h. The yellow dye was injected every two days. The remaining days either green (in the first half of the experiment) or grey (in the second half) was injected. The concentration of each dye in this case could not be calculated as in Run 2 because the UV/visible absorbance spectra of the three dyes have a high similarity as shown in Fig. 7. They are probably based on very similar chromophores. Colour is globally monitored by the area underneath the absorbance curve between 260 and 700 nm. With this estimation method the feed colour varies between 50 and 250, depending upon the dye. Fig. 8a shows a decrease in colour in the clarifier supernatant, which was not observed in Runs 1 and 2. That is due to probably a very good adsorption of the Dylon dyes on the activated sludge, more than to a real degradation. A decrease in the mixed liquor suspended solids concentration together with an increase in the sludge volume index was observed at the beginning of the dye sequence. Then the system stabilised before the switch to grey dye. The shock is not so large than at the beginning of the experiment as the yellow dye is still injected regularly. During this experiment a constant decrease in the filament abundance was observed. The temporary change in the settling properties might be related in the present case more to an alteration of the exopolymers, in response to the toxic shock induced by the presence of the dyes.

4. Conclusion

The effect of the variability of composition of a synthetic textile wastewater on activated sludge has been monitored. The sludge is certainly upset by the dyes due to their toxicity: it does not have enough time to adapt to their chemical nature between each change but it remained active throughout the experiments. Almost no biodegradation with respect to dyes was observed. The dyes in Runs 1 and 2 are adsorbed on the sludge in the bioreactor and desorbed in the clarifier. In Run 3, the wastewater colour is strongly reduced at the outlet of the system but it is unclear whether it is due to absorption or due to real degradation.

If the wastewater treatment purpose is just a decrease in the colour, this can indeed be achieved by adsorption. However, the dye molecules stay entrapped in the sludge, which has limited adsorption capacity and whose disposal is problematic. Further experiments will now be performed with physically pretreated textile

wastewater of variable composition, in order to increase its biodegradability and decrease its colour.

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