



## BIOLOGICAL DENITRIFICATION OF A TEXTILE EFFLUENT IN A DYNAMIC SAND FILTER

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### ABSTRACT

Post-denitrification of a pre-treated textile effluent was tested in a pilot-scale dynamic up-flow sand filter, which has been used as a biofilm reactor, together with filtration of suspended solids (SS) and decolorization. The potential application of the reactor as a three-in-one unit (decolorization, filtration and denitrification) has been successfully tested. Biomass growth and the sloughing of biological film did not prevent the removal of high concentrations of influent SS. Both pilot- and bench-scale tests confirmed that the intrinsic denitrification kinetics was zero-order, corresponding to a half-order removal rate if nitrate concentration is lower than  $10 \text{ mgN l}^{-1}$ . Zero-order and half-order kinetic constants have also been calculated. At low nitrate loading rates (up to  $2 \text{ kgN m}^{-3} \text{ d}^{-1}$ ) the filter followed the ideal plug-flow hydrodynamic model. In the lower part of the filter, zero-order kinetics fitted denitrification removal rates, while in the upper part of the filter denitrification followed half-order kinetics. At nitrate loading rates higher than  $2.5 \text{ kgN m}^{-3} \text{ d}^{-1}$ , nitrogen bubbles developed and partially mixed the reactor. Consequently, flatter concentration profiles were detected in the reactor and denitrification followed half-order kinetics along the entire filter. © 1998 Published by Elsevier Science Ltd. All rights reserved

### KEYWORDS

Biofilm; denitrification; filtration; kinetics; up-flow dynamic sand filter.

### INTRODUCTION

High concentrations of organic and ammonium nitrogen are often present in textile effluents and must be removed to comply with strict authorised limits (i.e.  $10 \text{ mg l}^{-1}$  total N in sensitive areas). When conventional, existing activated sludge processes cannot be easily retrofitted to achieve nitrogen removal, post-denitrification processes with an external carbon source can be an interesting alternative. Moreover, when strict limits on COD and suspended solids have to be achieved, robust filtration units are required and have to face occasional bulking sludge overflow from the biological treatment. In the specific case of effluents from the textile dyeing industry, a noticeable coloration, basically reddish, persists after the biological treatment and can be removed by cationic polyelectrolytes (amino resins) with a high degree of decolorizing power. In this paper the possibility has been evaluated of associating filtration, decolorization and post-denitrification with addition of an external carbon source in one single treatment unit.

## DESCRIPTION OF THE PILOT PLANT

The dynamic filter used in the experiments is shown in Figure 1. Water is brought into the filter through the feeding pipe (1) which ends with a diffusing cone (2), placed in the lower part of the filter. Filtered water outflows from the upper part of the tank through a spillway (3). In this way a movement of water from the lower to the upper part is created through the sand bed. (4). At the same time, sand proceeds slowly downwards. At the bottom an air-lift (5) carries the polluted sand to the upper part (6) where the suspended solids are separated from the clean sand which falls down on the upper part of the bed (7). In this way a cyclical movement is obtained and the filter can be operated continuously. Part of the solids accumulated in the sand bed during filtration are removed in the ascending pipe of the air-lift by the high turbulence. The remaining impurities are removed in the washing area (6) by a weak upward current of filtered water, caused by the different levels existing between the spillway of backwashing water (8) and the free surface of filtered water (9).

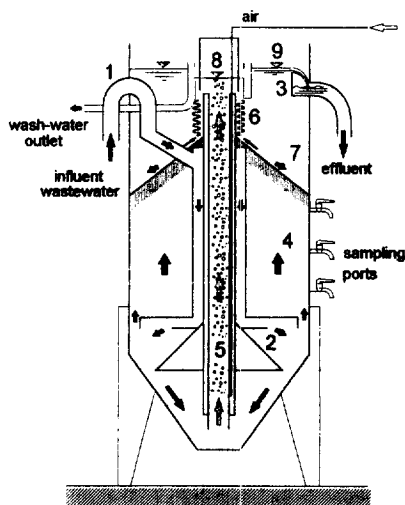


Figure 1. Schematic of the dynamic sand-bed filter used in the experiments.

Concentrated solutions of acetic acid and/or a hydroalcoholic mixture, sodium nitrate, and/or decolorizing polymer, according to the phase of the experiment, were added to the influent wastewater. If needed, a known amount of recycle sludge was added in order to simulate an overload of suspended solids escaped from the settler. The pilot-scale reactor, made of stainless steel with a diameter of 0.96 m (section=0.7 m<sup>2</sup>), was 4.05 m high; the sand layer was about 2 m high (1.4 m<sup>3</sup>). During the experiments, the hydraulic loading of 10 m h<sup>-1</sup> has been maintained as it was shown as optimal by previous experiments (Koopman *et al.*, 1990; Hultman *et al.*, 1994). Effectiveness of washing depends mainly on the air flow blown into the central air lift. Air flow was kept at about 27 l min<sup>-1</sup> (pressure of about 300 kPa). The volume of wash water ranged 8 to 10% of the daily volume of treated water.

## MATERIALS AND METHODS

Experiments have been organised into three phases. In the first one decolorization and filtration efficiencies without biomass growth were determined. In this paper, the second and third phase are described, where two different organic substrates have been dosed (acetic acid and hydroalcoholic solution at 25% methanol and 50% alcohols up to C-8) in order to allow the development of the denitrifying heterotrophic biomass.

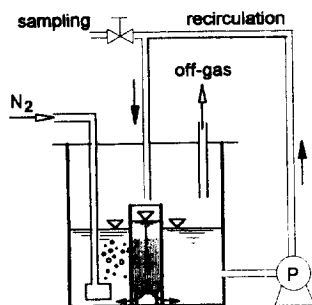


Figure 2. Bench-scale reactor.

For filtration purposes, a fine quartz sand would have been the best choice as filter medium. However, basaltic sand ( $d_{60}=2.6$  mm;  $d_{10}=2$  mm; specific surface area= $2720$  m<sup>2</sup>, Olthoff *et al.* 1985) was chosen as filter medium because its rough surface favours the attachment of the biofilm and its high specific surface area allows an optimal utilisation of the filter volume. Influent and effluent 24-hours composite samples were collected by an automatic sampler. Nitrates were preserved by adding HgCl<sub>2</sub> (40 mg l<sup>-1</sup>) in the sampling bottle. Analyses of suspended solids, absorbance at various wavelengths, nitrate nitrogen and total COD were performed according to the official Italian IRSA-CNR methods for waters and wastewaters. Filtered COD was determined after filtration over GF/C membrane. TKN and nitrous nitrogen were determined occasionally. Spot samples have been taken at different heights from three sample ports. Temperature and dissolved oxygen were measured on site. After immediate filtration over 0.45 m membrane, the samples were sent out for the remaining analysis. Biomass, as volatiles solids, was calculated as the difference between the weight of the colonised supports after drying at 105°C and the weight after combustion at 600°C. For more details, see Longhi and Mattioli (1995) and Canziani and Bonomo (1996).

Data on process kinetics were also obtained from the bench-scale reactor shown in Figure 2. In a 0.5 l cylindrical container a second container is inserted. This is filled with sand (34.8 cm<sup>3</sup>) extracted each time from the pilot plant. The bottom of the smaller cylinder is formed by a small meshed net allowing water flow and supporting the sand bed. A solution of sodium acetate (300 mg l<sup>-1</sup>) and nitrate (30-35 mgN l<sup>-1</sup>) flows through the sand bed with the same hydraulic loading as in the pilot plant. Water circulation is kept by a peristaltic pump with a capacity of 10.8 l h<sup>-1</sup>. Water flows from the pump feeding pipe into the internal cylinder, where a water level of some millimetres is formed above the sand bed; after the filter, the solution is collected into the external cylinder and the cycle starts again. Anoxic conditions were ensured by sparging nitrogen gas into the tank. A magnetic stirrer ensured complete mixing. The colonised sand, taken from the pilot plant was brought to the lab within an hour and the tests started after waiting for about 2 hours, enough for the biomass to become acclimatised. Concentration of nitrate and carbon were the same as in the pilot plant. At regular intervals, 7.5 ml filtered samples were taken using a syringe with 0.2 µm membrane, in order to stop any possible biological activity.

## RESULTS AND DISCUSSION

### Filtration and decolorization

The filter removed suspended solids up to 120 mg SST l<sup>-1</sup>, and the effluent concentration never exceeded 30 mg SS l<sup>-1</sup>. The highest removal efficiency (>90%) has taken place at an influent concentration between 80 and 120 mg SS l<sup>-1</sup> (Figure 3) with a hydraulic loading rate of 10 m h<sup>-1</sup>. In case of higher influent concentration (i.e. loading rates >1 kgSS m<sup>-3</sup> h<sup>-1</sup>), removal rates decrease and the selfcleaning system is not sufficient. Nevertheless, the slight expansion of the sand bed has always prevented clogging of the filter even in the occurrence of loadings higher than 1 kgSS m<sup>-3</sup> h<sup>-1</sup>. On the other side, the rough basaltic sand was not permitted to obtain appreciable filtration efficiencies at influent SS values lower than 30 mg l<sup>-1</sup>, especially when sand is colonised by the biological film. In this case, at influent SS between 20 and 35 mg l<sup>-1</sup>

<sup>1</sup>, no removal can be observed (Figure 4a) or a slight increase could be observed (up to 10 mg l<sup>-1</sup>), caused by the contribution of the sloughing biomass in the upper part of the filter, still far from the air lift. Nevertheless, on the occurrence of SS concentration peaks the filtrating action is remarkable, as shown in Figure 4b. A considerable portion of the loading is removed in the lower portion of the filter. Also, sludge flocs incorporate the excess biomass and are kept back in the upper part as well.

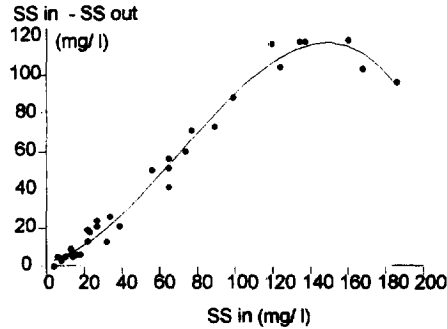


Figure 3. Suspended solids removal vs. influent concentration (test without biomass growth). The interpolating curve is:  $(SS_{in} - SS_{out}) = a + bSS_{in} + cSS_{in}^2 + dSS_{in}^3$   $a = 2.192$ ;  $b = 0.2133$ ;  $c = 0.0127$ ;  $d = -0.00006$ . Determination coefficient:  $r^2 = 0.986$ .

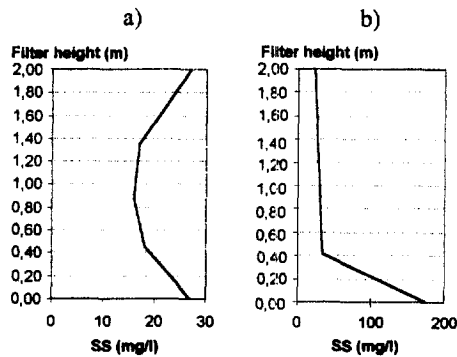


Figure 4. Filtration with biomass growth: a) normal operation: b) operation with an overload from the secondary settler.

Adding a decolorizing polyelectrolyte directly into the filter, without preliminary mixing, did not hamper the decolorizing efficiency and flocs are retained in the filter bed without causing any inconvenience because the biological denitrification process. Decolorization tests have been made at different wavelengths with optimal dosage of the polymer determined with a jar-test (20-30 mg l<sup>-1</sup>). A 10-cm deep tank filled with effluent resulted colourless when diluted 10 times, according to Italian regulations.

### Denitrification

Two sets of experiments have been performed. In the first one, between September and December 1994, three runs have been carried out, dosing acetic acid as carbon source (Figure 5). Three runs have been performed also in the second set, when a hydroalcoholic mixture has been used as the carbon source. Due to partial denitrification of the effluent in the industrial plant, it was necessary to dose sodium nitrate in the pilot plant in order to investigate high loading conditions. In order to take into account all oxidising agents present in wastewater, the contribution of the different oxidising species has been assimilated to an equivalent nitrate nitrogen load by means of the following expression:

$$NO_3^- - N_{eq} = NO_3^- - N + 0,60 NO_2^- - N + 0,35 O_2$$

where the coefficients, obtained from stoichiometric calculations (see for example Koopman *et al.*, 1990) show how much nitrate nitrogen is required to oxidise the same amount of COD by the unit mass of nitrous oxide or of oxygen. This equivalence is useful only for dosage calculations, not as far as kinetic evaluations are concerned, where every substrate has to be considered separately. While nitrous nitrogen was always negligible ( $<0.4 \text{ mg l}^{-1}$ ), dissolved oxygen in the influent wastewater averaged  $4.1 \text{ mg l}^{-1}$  and could not be neglected.

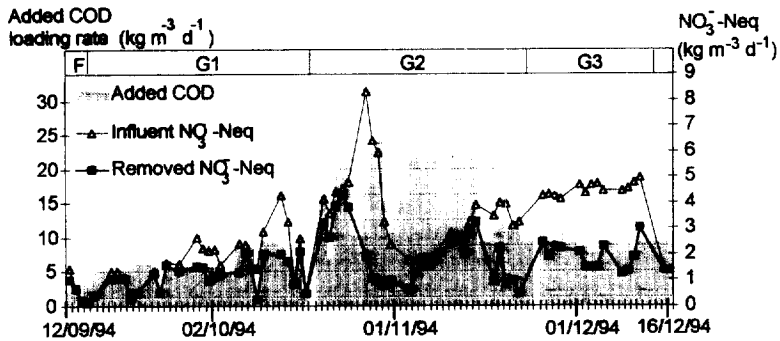


Figure 5.  $\text{NO}_3\text{-N}_{\text{eq}}$  removed and COD added during denitrification tests with acetate dosage. The ratio between the unit of the two vertical axes is equal to the stoichiometric ratio between COD and equivalent nitrates ( $3.79 \text{ gCOD per g of NO}_3\text{-N}_{\text{eq}}$ ): in this way, the added COD can be directly related to the theoretically removable equivalent nitrates. Legend: Run F = start-up; Run G1 = stoichiometric dosage of acetate; Run G2 = excess dosage of acetate; Run G3 = low dosage of acetate.

From Figure 5 it can be observed that between 29/10 and 7/11 and between 21 and 25/11/1994 in the pilot plant a severe inhibition of the heterotrophic denitrifying activity has occurred, while any adverse effect on denitrification in the industrial plant was not observed, where both nitrification and denitrification proceeded regularly. Also,  $\text{O}_2$  concentration along the reactor, even in the presence of substrate and of biomass, did not change or has shown a slightly decreasing trend between influent and effluent.

It has been supposed that some chemical compounds in the industrial effluent could have selectively inhibited some of the enzymatic activities typical of the specific denitrifying micro-organisms developed in the pilot scale reactor. Here, a bacterial population that was highly efficient in a quick and complete utilisation of only one organic substrate (acetate) has been selected. In similar conditions it can be inferred that nitrate reduction reactions occur according to the same sequence of intermediate reactions: if a certain enzyme is blocked, the entire process can stop. As a partial confirmation of what has been assumed, it was observed that in the test carried out with a hydroalcoholic mixture (i.e.: diversified substrates) never any inhibitory phenomenon has been observed. The filter has proved to be able to withstand periods without dosage of carbon substrate or nitrates or both, restoring its usual denitrifying activity immediately after feeding has started again.

At low volumetric loadings, only the lower part of the filter is active and no nitrogen gas bubbles have been observed from the top of the filter. Bubble formation was probably avoided because only a small portion of the filter (the lower layers) as heavily loaded. On the other hand, as it happened in the next set of tests, when the height involved in the denitrifying activity is higher because of the higher loadings applied, a remarkable quantity of gas (1-2 cm wide bubbles) was clearly visible at the top of the filter. The rising of gaseous nitrogen did not upset the sand filter but increased the head losses (from 30 to about 60 cm).

The consumption of acetate (expressed in terms of COD) increased linearly with nitrate removal (Figure 6). The slope represents the average stoichiometric ratio observed with a value of approximately 4.9, against a theoretical value of 3.79 (Profazier and Mezzanotte, 1995). In the experiments with dosage of

hydroalcoholic mixture, a ratio of 4.6 has been observed. From the theory of biological reactions in fixed biomass systems (e.g. Harremoës *et al.*, 1982), the transition between nitrate and organic carbon occurs according to the following expression:

$$\frac{S_C^*}{S_{NO_3-N}^*} = \frac{D_{NO_3-N}}{D_C} \cdot f_{C/NO_3-N}$$

where  $S^*$  is the substrate concentration at the biofilm/liquid interface (index C stands for Carbon) and  $f$  is the stoichiometric ratio. Therefore, it could be argued that the ratio between the diffusion coefficients can be equal to  $4.6/3.79=1.21$  and to  $4.9/3.79=1.29$  if the hydroalcoholic mixture or, respectively, sodium acetate is used as carbon source.

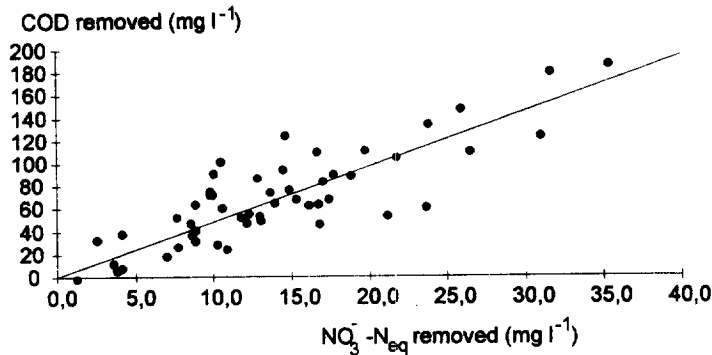


Figure 6. Filtered COD removed vs. equivalent nitrates removed during denitrification tests with acetate dosage. The slope of the regression line is 4.856 ( $r^2=0.7$  and  $n=51$ ).

The amount of fixed biomass is evenly distributed along the filter and, on average, it is equal to 1.2% of the weight of sand. Continuous washing of the sand through the airlift device removes part of the fixed biomass and keeps the biofilm thin. At a specific surface of  $2720 \text{ m}^{-1}$  and assuming a density of  $50 \text{ mg SSV cm}^{-3}$  and that the biofilm covers 100% of the sand grains, biofilm thickness can be estimated about  $150 \text{ }\mu\text{m}$ .

From the spot samples taken from the sample ports along the filter, the trends of  $\text{NO}_3\text{-N}$ ,  $\text{O}_2$  and filtered COD were obtained. At low loading rates (up to  $1.5 \text{ kgNO}_3\text{-N m}^{-3} \text{ d}^{-1}$ ) only a small portion of the filter (the first 45 cm) is actively working, as it can be seen from Figure 7A. The remaining part is not utilised and works as a reserve in case of loading peaks. For this purpose, the action of the air-lift is crucial because it ensures sand circulation and thus a homogeneous circulation over the whole bed, which may effectively contribute to the biological denitrifying process. In a different way, in a fixed bed, the upper layers, being not much exposed to significant concentrations, became poorly colonised, and, in case of loading peaks, they may not be as active as the lower layers. In Run G3, profiles obtained under conditions of excess of COD (Figure 7B) show that the whole height of the filter contributes to nitrate removal, which results therefore almost complete. Very high loading rates were applied in the last experimental phase (summer 1995), when a hydro-alcoholic mixture was used as the carbon source. Concentration profiles along the filter were completely different (Figure 8). High temperature and high loading have helped high nitrate removal rates (up to  $5 \text{ kgNO}_3\text{-N m}^{-3} \text{ d}^{-1}$ ) and this has caused, as previously stated, the formation of an impressive bubbling of gaseous nitrogen which, mixing the liquid phase, has in fact modified the reactor hydrodynamics. While before the plug-flow model could be applied, the best fit of data in presence of bubbling is the one which adopts the model of four completely mixed reactors in series. Bubbling appeared to be evident in the interval  $2\text{-}2.5 \text{ kg NO}_3\text{-N}_{\text{removed}} \text{ m}^{-3} \text{ d}^{-1}$ .

### Calculation of the process kinetic constants

Profile data and data deduced from lab-scale tests have been worked out in order to check whether the overall process kinetic was zero-order (i.e. complete penetration of the biofilm by nitrates) or 1/2-order (partial penetration; Canziani and Bonomo, 1996). The mass balance of nitrates in the bench-scale reactor yields:

$$(dS/dt) V_w = -r_a \cdot a_w \cdot V_s \quad (1)$$

where  $S$  is the limiting substrate concentration,  $V_w$  is the volume of water ( $m^3$ ),  $r_a$  is the reaction rate ( $gN m^{-2} d^{-1}$ ),  $a_w$  is the specific surface area of the filter media ( $m^{-1}$ ) and  $V_s$  the volume of colonised sand ( $m^3$ ). Assuming that carbon is dosed in excess and that the biofilm is completely penetrated by nitrates, then  $r_a = k_0 \cdot L$  and equation (1) becomes:

$$(dS/dt) V_w = -k_0 \cdot L \cdot a_w \cdot V_s \quad (2)$$

After integration, the expression of the nitrate concentration becomes:

$$S = S_0 - k_0 \cdot L \cdot a_w \cdot V_s / V_w \cdot t \quad (3)$$

and defining  $t_c = V_s / V_w \cdot t$  as the time of real contact between water and the biomass in the reactor, we get the dimensionless expression:

$$S/S_0 = 1 - (k_0 \cdot L \cdot a_w \cdot t_c) / S_0 \quad (4)$$

which represents a straight line in the plane  $[t_c/S_0; S/S_0]$  with a slope  $m = k_0 \cdot L \cdot a_w$  and intercept  $i = 1$ .

In the same way, for a partly penetrated biofilm with an overall reaction rate  $r_a = k_{1/2a} S^{*1/2}$ , where  $k_{1/2a} = (2D k_0)^{1/2}$ , the corresponding expression is:

$$(S/S_0)^{1/2} = 1 - k_{1/2a} \cdot a_w \cdot t_c / (2C_0)^{1/2} \quad (5)$$

which represents a straight line in the plane  $[t_c/(S_0)^{1/2}; (S/S_0)^{1/2}]$ . Similar mass balances can be calculated in order to find the solutions for continuous-flow reactors, where contact time in the reactor is expressed as  $t_c = h/q$  (filter height 'h' divided by the hydraulic loading 'q').

In tests carried in the experimental phase characterised by acetate dosing, both kinetic models fit the real data (Figure 9). In the calculations it has been assumed that all the sand was evenly covered by the biological film. Due to the fact that the filter was working as a plug-flow reactor, it is possible that the part near the inlet, with high nitrate concentration, presented a completely penetrated biofilm, while, in the upper part, with low nitrate concentration, the biofilm, (which was homogeneous in the whole sand bed) could be partially penetrated. Results obtained from lab tests (Figure 10) have confirmed that, when all the biofilm was operating at high nitrate concentrations in the bulk liquid (thanks to the continuous effluent recirculation) overall zero-order kinetics best fitted the experimental data, with  $k_0 L$  ( $20^\circ C$ ) =  $0.041 gNO_3-N$

$\text{m}^{-2} \text{h}^{-1}$ . Had temperature been the only reason for different values, the coefficient of Arrhenius= $[k_0L(20^\circ)/k_0L(14.8^\circ)]^{1/(20-14.8)}=(0.041/0.030)^{1/5.2}=1.062$  would have been obtained.

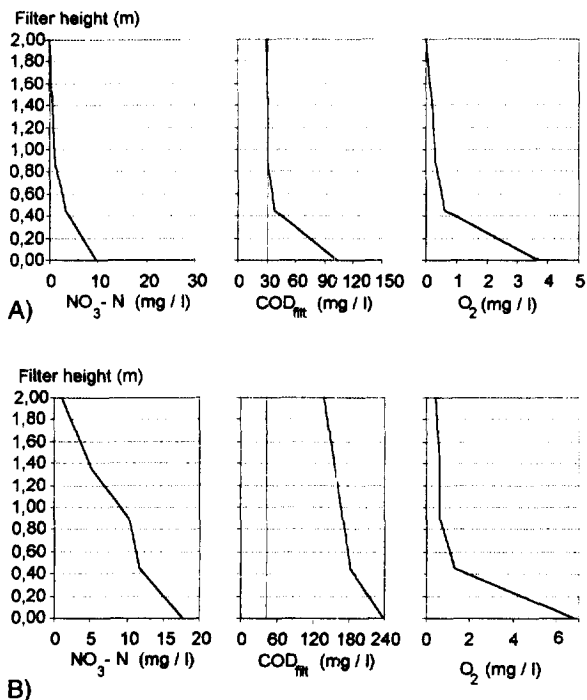


Figure 7. Concentration profiles of  $\text{NO}_3\text{-N}$ ,  $\text{O}_2$  and filtered COD along the filter: A): run G1, day 12/10; B) run G2, day 14/11. The vertical line in the COD diagram shows the concentration in the influent before dosing the carbon source.

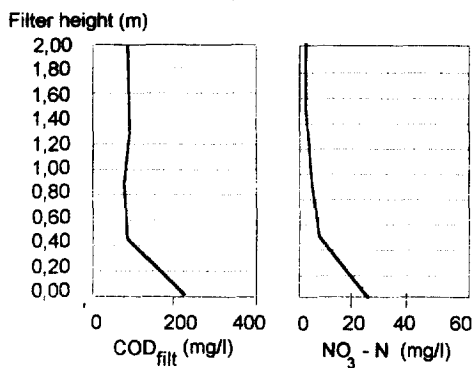


Figure 8. Concentration profiles of nitrates and filtered COD (12/07/95,  $T=25^\circ\text{C}$ , nitrate removal= $3 \text{ kgNO}_3\text{-N m}^{-3} \text{ d}^{-1}$ ). The gradient is flat because of the mixing effect caused by the bubbles of gaseous nitrogen.



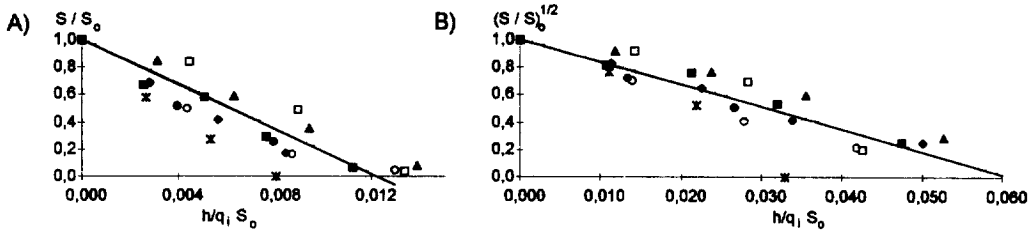


Figure 9. Tests performed on the pilot plant (sodium acetate as carbon source): 9/11/94 (●); 11/11 (▲); 14/11 (■); 15/11 (\*); 16/11 (◆); 19/12 (○); 20/12 (□); average T=14.8°C. A) biofilm is assumed to be completely penetrated by nitrates: zero-order constant= $k_0L=0.030$  (gNO<sub>3</sub>-N m<sup>-2</sup> h<sup>-1</sup>); r<sup>2</sup>=0.831; B) biofilm is assumed to be partially penetrated by nitrates: half-order constant  $k_{1/2a}=0.0120$  (gNO<sub>3</sub>-N<sup>1/2</sup> m<sup>-1/2</sup> h<sup>-1</sup>); r<sup>2</sup>=0.854.

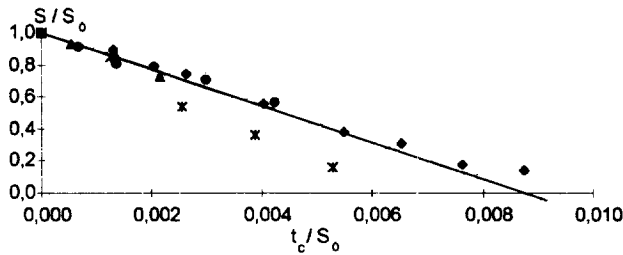


Figure 10. Tests performed on the bench-scale plant (acetate dosage), assuming a zero-order reaction rate: 9/11/94 (●); 11/11 (▲); 14/11 (■); 15/11 (\*); 16/11 (◆); 19/12 (○); 20/12 (□); average T=20°C. Zero-order overall constant= $k_0L=0.041$  (gNO<sub>3</sub>-N m<sup>-2</sup> h<sup>-1</sup>); r<sup>2</sup>=0.911.

In tests carried out in 1995 instead, the average concentration of the filter was close to the effluent concentration, because of the mixing induced by the nitrogen bubbles. Low concentration evenly distributed along the entire filter permits us to assume an overall 1/2-order reaction rate, typical of a partially penetrated biofilm. In the interval between 20 and 26.5°C the dependence of the constant  $k_{1/2}$  on temperature is shown in Figure 11 and allows us to calculate a factor=1.04 (r<sup>2</sup>=0.95; n=6).

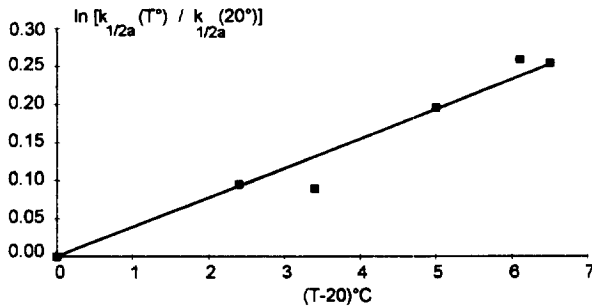


Figure 11. Half-order kinetic constant ( $k_{1/2a}$ ) vs. temperature in the tests with dosage of a hydroalcoholic mixture (1995).  $k_{1/2a}(20^\circ)=0.0135$  (gNO<sub>3</sub>-N<sup>1/2</sup> m<sup>-1/2</sup> h<sup>-1</sup>); a 4 CSTRs hydrodynamic model has been found as the best fit for the calculations.

### CONCLUSIONS

Biological denitrification has provided reliable even in combination with dosage of a decolorizing polyelectrolite. The actual dosage of organic substrate is higher than the stoichiometric ratio (3.8 gCOD/gNO<sub>3</sub>-N), equal to 4.9 with acetic acid and 4.6 with a hydroalcoholic mixture. The removal efficiency has been kept above 90% for loadings up to more than 5 kg NO<sub>3</sub>-Neq d<sup>-1</sup> and concentrations up to

40 mg l<sup>-1</sup> at a temperature of about 26°C. The elaboration of experimental data either onsite or in the lab has confirmed the hypothesis of intrinsic kinetic of zero order. For nitrogen removal rates higher than 2 kg NO<sub>3</sub><sup>-</sup>-N m<sup>-3</sup> d<sup>-1</sup> the development of bubbles of N<sub>2</sub> gas changes the hydrodynamic of the reactor from plug-flow to four CSTR in series. For concentrations of nitrate in the reactor lower than 10 mg l<sup>-1</sup> it may assumed the hypothesis of partial penetration of the biofilm with  $k_{1/2}(20^{\circ}\text{C})=0.012\text{-}0.014 \text{ gNO}_3\text{-N}^{1/2} \text{ m}^{-1/2} \text{ h}^{-1}$ .

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