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Nitrogen removal via nitrite in a sequencing batch reactor treating sanitary landfill leachate

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ABSTRACT

The present paper reports the results of the application of a control system, based on artificial intelligence concepts, for the automation of a bench-scale SBR treating leachate generated in old landfills. Attention was given to the nitritation and denitritation processes in order to enhance the nitrogen removal efficiency. Nitrification and nitrogen removal were usually higher than 98% and 95%, respectively, whereas COD removal was approximately 20–30% due to the low biodegradability of organic matter in the leachate from old landfills; therefore, external COD was added to accomplish the denitrification process. Adjusting the length of the oxic phase, almost complete inhibition of the nitrite oxidizing organisms was observed. The results confirm the effectiveness of the nitrite route for nitrogen removal optimisation in leachate treatment. A significant saving of approximately 35% in external COD addition was achieved.

1. Introduction

Sanitary landfill leachates are a great environmental concern because of the high pollutant concentration. Leachate characteristics present high variation due to several factors among which the main are the landfill operation, waste age and climatic conditions. Refuse decomposition in landfills follows a series of phases in which chemical, physical and biological properties of the buried wastes undergo to large variations. During landfill aging, the concentration of organic compounds into leachate normally decreases and becomes less biodegradable, whereas ammonia concentration tends to increase. As a consequence, leachates generated in old landfills usually present a low biochemical oxygen demand (BOD) to total Kjeldahl nitrogen (TKN) ratio (Berge et al., 2005; Kjeldsen et al., 2002; Lema et al., 1988).

Sanitary landfill leachate treatment is usually accomplished by multistage systems using chemical, physical and biological processes (Lema et al., 1988). Among several biological treatment systems, sequencing batch reactors (SBRs) have been successfully applied for leachate treatment (Mace and Mata-Alvarez, 2002). When the BOD/TKN ratio is low as in leachate from old landfills, biodegradable organics must be added for biological denitrification causing a considerable increase of the operational costs (Dedhar and Mavinic, 1985; Shiakowski and Mavinic, 1989).

Biological nitrogen removal is usually accomplished via nitrification and denitrification processes. During nitrification process, ammonia is biologically oxidized to nitrate (with nitrite as intermediate) which is than reduced to nitrogen gas using organic matter as electron donor during the denitrification processes. Because ammonia oxidation is normally the rate-limiting step, nitrite does not usually accumulate during the nitrification process (Henze et al., 1997). Nevertheless, nitrite accumulation has been occasionally observed (Andreottola et al., 1997; Beline et al., 2007; Fux et al., 2006), especially for concentrated wastewater where high ammonia concentration can inhibit the nitrification processes (Anthonisen et al., 1976).

In recent years, there has been a great interest in using nitrite as shortcut for nitrification and denitrification processes. Indeed, the main advantages of the nitrite pathway are the decrease in oxygen consumption and the reduction of organic matter demand (Alleman, 1984; Lai et al., 2004; Peng et al., 2008; Turk and Mavinic, 1989; Yamamoto et al., 2008). Over the last decades large efforts have been carried out to study several operational conditions (e.g., low dissolved oxygen concentration, selective inhibition, temperature) to accumulate nitrite and inhibit nitrite oxidation (Abeling and Seyfried, 1992; Hellinga et al., 1998; Randall and Buth, 1984; Turk and Mavinic, 1989). Although selective inhibition of nitrite oxidizing organisms has been described, biomass acclimation that prevented stable nitrite built-up has been sometimes observed (Turk and Mavinic, 1989; Villaverde et al., 2000). Recently, several innovative biological processes have been developed for nitrogen removal from highly concentrated wastewaters using the nitrite route (reviewed by Villaverde, 2004).

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Several authors have reported that the nitritation and denitritation processes can be managed using SBRs: in fact, nitrite accumulation can be sustained adjusting the length of the oxic phase (Sauter and Alleman, 1980). Recently, ammonium-rich wastewaters have been successfully treated in SBR systems via the nitrite route. Although the nitrite route has been studied for different type of high-strength wastewaters (Li et al., 2004; Peng et al., 2004), most of the applications are related to sludge dewatering effluents (Fux et al., 2006).

During the last decade large efforts have been carried out on wastewater treatment monitoring, control and automation (Olsson G., 1999). Several applications have been reported on using dissolved oxygen (DO), pH and oxidation reduction potential (ORP) for monitoring and control of wastewater treatment plants, both in continuous and batch processes (Al-Ghusain et al., 1994; Charpentier et al., 1998; Spagni et al., 2001, 2007). In batch reactors, DO, pH and ORP have been frequently used for phase length optimization (Battistoni et al., 2003; Cheng et al., 2000; Ra et al., 1998). Due to the high variations in influent characteristics and the complex dynamics of the biological processes artificial intelligence techniques such as fuzzy logic (Marsili-Libelli, 2006; Marsili-Libelli et al., 2008) or neural networks (Cho et al., 2001; Luccarini et al., 2002) have been developed for SBR control and automation.

The objective of this study was to evaluate a real-time control system for nitrogen removal in SBRs treating raw leachate generated in old landfills. The control system is based on DO, pH and ORP signals and applies fuzzy logic to determine the appropriate switching commands to adjust the length of the anoxic and oxic phases and the amount of external chemical oxygen demand (COD) added for denitrification. Particular attention was given to the nitritation and denitritation processes in order to enhance nitrogen removal via nitrite, reducing the energy required for ammonia oxidation and the amount of the external COD added for nitrogen reduction.

2. Methods

2.1. Experimental set-up

The study was conducted using a laboratory-scale SBR. The reactor, with a maximum working volume of about 24 L, was operated at 20 ± 1 °C in a thermostatic room for almost 300 days.

The SBR was controlled by a PC-based control system which could be operated either locally or remotely via the internet. Feeding, effluent and sludge withdrawal were provided by peristaltic pumps, mixing by a mechanical stirrer, whereas air was supplied by an aquarium blower. The SBR was operated with a full-cycle divided in series of 4 sub-cycles, followed by one hour of settling. The effluent was drawn during the last three minutes of the settling phase to reach the minimum reactor volume of 15 L. During the last minute of the fourth sub-cycle a small amount of mixed liquor was also drawn to control suspended solids concentration in the reactor: the sludge withdrawal maintains a mean solid retention time (SRT) of about 20–25 days. Every sub-cycle started with an anoxic–anaerobic phase followed by an oxic phase. During the first minutes of the anoxic–anaerobic phase (i.e. every sub-cycle), leachate (flow of 1.2 L/h) was added to the tank to a loading rate of approximately 0.1 $\text{gTKN}/(\text{L} * \text{d})$ and 0.15 $\text{gCOD}/(\text{L} * \text{d})$. Due to the variation of the leachate strength the amount of feed (and consequently the time of feeding with a constant flow) was modified in order to obtain an approximately constant load.

During the first 80 days of experimentation, the SBR was run with a fixed timing of the anoxic and oxic phases in order to assess the performance of the plant for COD and nitrogen removal. During this period, the SBR was operated with a cycle of 24 h divided in 4 sequences of 2 and 3.75 hour of anoxic and aerobic phases respectively. Afterwards, a fuzzy supervisory control system adjusted the length of the phases, operating the pumps, the blower and the stirrer.

Due to the characteristics of the leachate used in the present study (Table 1), a concentrated solution (20 g/L) of sodium acetate trihydrate (equal to 9.4 gCOD/L assuming a relation of 0.47 gram of COD per gram of salt) was added during the anoxic–anaerobic phase (at a flow rate of 0.14 L/h) after the feed phase, in order to supply the required biodegradable COD for denitrification. Acetate was added after the feed phase because the leachate addition can affect the ORP and pH signal (Spagni et al., 2007) and this could be misinterpreted by the control system. The added acetate was not included in the calculation of the organic load to the SBR.

Because of the low leachate phosphorus concentration (relative to COD and nitrogen), a concentrated solution of KH_2PO_4 was added into the SBR to maintain phosphate concentration in the reactor approximately at 0.5–1.0 mgP/L.

2.2. Seeding sludge

The activated sludge from the experimental set-up implemented by Spagni et al. (2007) was used as a source of seeding sludge, which had been acclimated for almost two years to the same leachate considered in this study. That SBR was managed with fixed phase duration switched by digital timers to study extensively the nitrogen removal and the possibility of using DO, pH and ORP signals for the development of a control system, which has been implemented and applied in this work. More details on the relationships between biological nitrogen removal processes and DO, pH and ORP signals are reported in Spagni et al. (2007).

2.3. Control system

The lab-scale plant was supervised by a control system composed of a fuzzy inferential system developed for this application in the LabView 7.1 (National Instruments, Austin, TX, USA), which could be operated either locally or remotely through the Internet. The implemented fuzzy supervisory system was introduced to identify and manage the correct switching sequence of the set-up, which performed the phase-end detection and managed the on/off switching of the blower, mixer and pumps (filling, acetate addition, sludge and effluent withdrawal). The control system acquired the signals from the process using on-line sensors (DO, pH and ORP) through an analogue/digital converter (PCI 6024E, National Instruments, Austin, TX, USA). In addition to the fuzzy automated mode the actuators could be operated manually with a specified duration. The rules of the control systems are based on the results obtained during preceding experimentation and described elsewhere (Marsili-Libelli, 2006; Marsili-Libelli et al., 2008; Spagni et al., 2001, 2007). Upon acquisition, the data are

Table 1
Main leachate characteristics

	Unit	Mean	Max	Min	SD[a]
TSS	mg/L	240	760	85	282
VSS	mg/L	160	540	43	213
$Alk_{4.3}$	meq/L	131	162	94	26
pH	–	8.08	8.23	7.93	0.21
K_{20}	mS/cm	16.5	18.5	15.33	1.12
CODt	mg/L	2055	2623	1769	282
CODf	mg/L	1937	2432	1688	253
TKN	mg N/L	1319	1530	1008	168
NH_4^+-N	mg N/L	1199	1406	933	175
Ptot	mg P/L	8.0	9.7	4.8	2.2
PO_4^{3-}-P	mg P/L	6.8	8.6	2.2	2.1

[a] Standard deviation (nine measurements).

validated and denoised using a wavelet filter, then numerical derivation is performed and a fuzzy inference algorithm is used to detect the end of the current phase (more details about the control system are reported in Marsili-Libelli et al., 2008).

2.4. Nitrification activity

Two nitrification tests were carried out during the experimental period managed by the control system, in order to evaluate the ammonia and the nitrite oxidation rate with leachate. The first and the second nitrification test were conducted on days 91 and 202, respectively, from the beginning of the experimentation, and therefore, after 11 and 122 days from the application of the fuzzy supervisory control system. The aim was to evaluate the activity trend during the experimentation of the two micro-organism groups (ammonia oxidising and nitrite oxidising organisms).

A well-defined amount of leachate was added to 300 mL of activated sludge in 500 mL glass bottles, in order to approximately reproduce the loading rate to the lab-scale SBR. One test (the second) was carried out adding NH_4Cl salt (with the leachate) in order to increase the amount of ammonia in the bottles and, therefore, increase the duration of the experiment. The bottles were continuously aerated during the tests maintaining the DO concentration above 6 mg/L. Nitrification activity was estimated interpolating the trends of ammonia, nitrite and nitrate concentrations. Specific nitrification ($\mathrm{mg\,N\,g^{-1}\,VSS\,h^{-1}}$) activity was calculated relative to the mixed liquor volatile suspended solids (VSS).

2.5. Leachate characteristics

Raw leachate was obtained from a municipal landfill located in the northern Italy. Landfill leachate was stored at 4 °C to reduce degradation and replaced every 20–30 days.

BOD to COD ratio measured during three years was approximately of 0.2 confirming the low organic matter biodegradability. Most of the samples presented volatile fatty acids concentrations below the instrumental detection limits (Spagni et al., 2007). Heavy metals were present (data not shown) at typical range concentrations of municipal landfill leachates (Lema et al., 1988). Table 1 reports some characteristics measured for the raw leachates used in the study. The leachate used in the study was characterized by a high nitrogen concentration relative to that of COD. For this reason external COD (acetate) was added to accomplish denitrification process. In general, the leachate characteristics present the usual high variability of leachates of old municipal sanitary landfills that are still in operation.

2.6. Analytical methods and on-line instrumentation

The bench-scale plant was on-line monitored for pH, dissolved oxygen (DO) and oxidation–reduction potential (ORP) using WTW instruments. It was also monitored for total suspended solids (TSS), volatile suspended solids (VSS), ammonia, nitrite, nitrate, total Kjeldahl nitrogen (TKN) total phosphorous (Ptot), orthophosphate ($PO_4^{3-}-P$), total chemical oxygen demand (CODt), filtered COD (CODf), alkalinity to pH 4.3 ($Alk_{4.3}$) and conductivity at 20 °C (K_{20}) according to the Standard Methods (APHA, 2005). Sample filtration was carried out using Whatman GF/C filters.

2.7. COD and nitrogen removal efficiency

Due to the low biodegradable COD present in the leachate utilised in this study, a concentrated solution of acetate was added as carbon source for denitrification. Even if the acetate solution was quite concentrated (see above), a partial dilution was expected due to the small reactor volume and the small amount of leachate added (especially for high strength leachate). Therefore the COD and nitrogen removal efficiency was calculated taking into account the effect of dilution.

3. Results and discussion

3.1. COD removal

The effluent CODt and CODf concentrations were roughly related to the leachate strength: indeed, the higher the influent COD concentration, the larger the effluent COD (Fig. 1). The high effluent COD concentration confirms the low biodegradability of the leachate utilised for the study. The high CODf/CODt ratio (average of 0.93) measure in the effluent demonstrates the good sludge settling characteristics. In fact the average effluent TSS concentration was 110 mg/L, whereas the mixed liquor TSS varied approximately from 2.5 and 3.5 g/L.

COD removal efficiency (calculated taking into account the effect of dilution caused by the external COD addition) showed large variations during the experimentation and on one occasion (approximately on day 220) reached negative values (Fig. 2). The negative COD removal efficiency (that means that, considering the dilution effect of the external COD addition, the effluent COD concentration is higher than that of the influent) was caused by the high variability of the leachate characteristics. The high hydraulic retention time (HRT) caused a sort of "memory" in the reactor and, therefore, when the COD had a sudden decrease, the effluent concentration was still influenced by the liquid present in the reactor. This effect is well visible on day 220 when COD in the influent decreased from 2600 to 2100 mg/L. During the entire experimental period, the COD removal efficiency was approximately 20% as average, confirming the low biodegradability of the leachate generated in old landfills.

3.2. Nitrogen removal

During the first 80 days, when the plant was operated with fixed phase duration, nitrogen removal was achieved via the nitrification/denitrification path (Fig. 3). Indeed, during this first experimental period, nitrate represented the major part of the total nitrogen in the effluent. On the contrary, nitrite was always below 0.5 mg N/L, with the exception of a transitional period, approximately on day 60, during which ammonia and nitrite slightly accumulated in the reactor.

On day 80, the fuzzy control system was put in operation to control the length of the phases. With the application of control

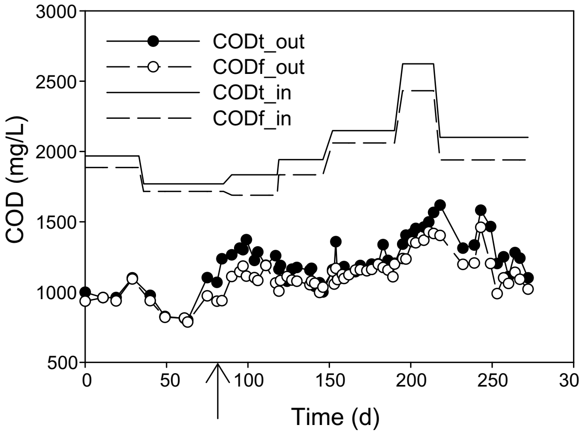

Fig. 1. Influent (_in) and effluent (_out) CODt and CODf. The arrow indicates the switch on of the control system.

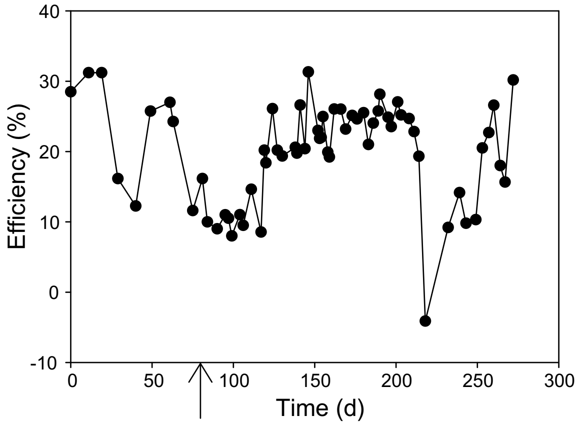

Fig. 2. COD removal efficiency. The arrow indicates the switch on of the control system.

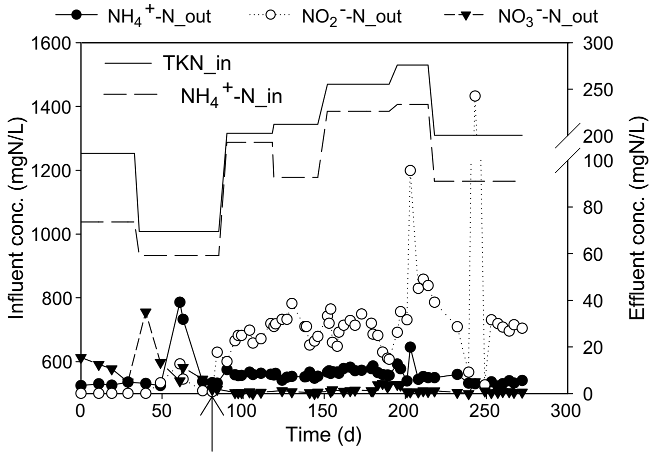

Fig. 3. Influent ammonia and TKN concentration (left axis); effluent ammonia, nitrite and nitrate concentration (right axis). The arrow indicates the switch on of the control system.

system, nitrite was immediately accumulated in the SBR, whereas nitrate decreased to concentration below 1.0 mg N/L (Fig. 3). The nitrite accumulation observed approximately on day 200 and on day 240 were caused by problems with the instrumentation and a break down of the external COD pump.

It can be noticed that after the nitrite peaks caused by the faults, the control system was able to decrease the nitrite concentration in a few days once the pump was repaired. It is also noteworthy that, in spite of the very high concentration of nitrite during some cycle, nitrate accumulation was never observed, confirming the effectiveness of the treatment system in selecting ammonia oxidizing bacteria over nitrite oxidizing organisms.

In spite of the high variation of the influent ammonia and TKN concentration, during the entire experimental campaign the SBR generally showed good nitrification efficiency, consistently in excess of 98% (Fig. 4).

The average nitrogen removal was of 95% during both the first (fuzzy control off) and the second experimental periods (fuzzy control on) (Fig. 4). The lower nitrogen removal values are related to the nitrite peaks (Fig. 3) as described above.

3.3. Nitrification activity

Two nitrification tests were conducted on day 11 and on day 122 days under the supervision of the fuzzy control system.

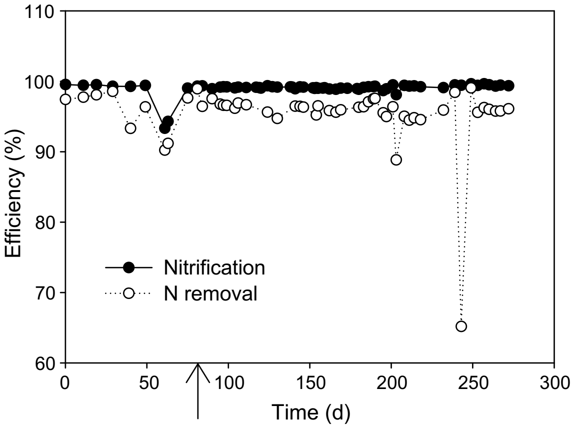

Fig. 4. Nitrification and nitrogen removal efficiencies. The arrow indicates the switch on of the control system.

Specific ammonia oxidation rates for the first and second nitrification test were 12.6 and 4.9 mg N/(VSS*h), respectively. The high variation in nitrification rate during biological leachate treatment was previously reported (Kim et al., 2006; Spagni et al., 2007). Specific nitrite oxidation (obtained by interpolating the nitrate concentration) varied from 0.1 to 0.15 mg N/(VSS*h), confirming the very low activity of the nitrite oxidizing organisms (Fig. 5).

The results showed the almost complete inhibition of the nitrite oxidizing bacteria activity. This very low nitrite oxidation rate observed during the first nitrification test (Fig. 5a) was completely unexpected. In fact, this reduction in the nitratation process (nitrite oxidation to nitrate) cannot be explained by the population dynamics of the nitrifying bacteria, because 11 days (1/2 of the SRT) are not a sufficient time for bacteria selection. Therefore, other mechanisms of nitrification inhibition should be investigated.

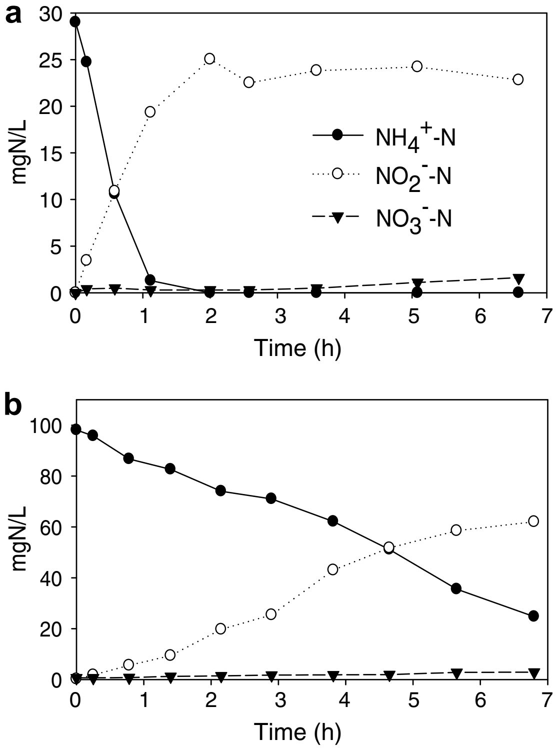

Fig. 5. Results of the first (a) and second (b) nitrification test.

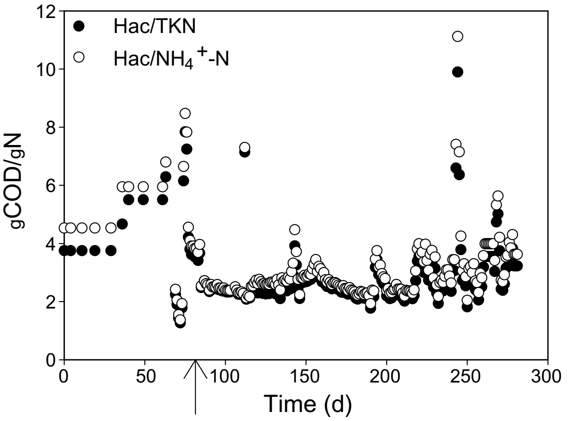

Fig. 6. External COD added to TNK and ammonia ratios. The arrow indicates the switch on of the control system.

The results confirm that nitrite built-up can be sustained adjusting the duration of the oxic phase in batch reactors. Moreover, the nitrite build-up seems to be stable because no acclimation by the nitrite oxidizing bacteria (Turk and Mavinic, 1989; Villaverde et al., 2000) was observed, at least for the duration of the experimentation. Indeed, the nitratation process was almost completely absent even after about four months of operation with the supervisory control system (Fig. 5b).

3.4. External COD to nitrogen ratio

During the entire experimental campaign the ratios of the external COD added to TKN and $\mathrm{NH}_4^+ - \mathrm{N}$ loaded with the influent were measured. Fig. 6 shows that the use of the nitrite path considerably decreased the amount of external COD (acetate) required for the denitrification process. The measured Hac/TNK ratios were of 4.15 and 2.73 gCOD/gN during the first and the second experimental periods, respectively. Similarly, the measured $\mathrm{Hac}/\mathrm{NH}_4^+ - \mathrm{N}$ ratios were of 4.64 and 2.98 gCOD/gN during the first and the second experimental periods, respectively. Therefore, the application of the supervisory control system utilised during the second experimental period, originated a saving of approximately 35% of the external COD addition which is close to the theoretical value of 40% (Alleman, 1984; Abeling and Seyfried, 1992; Turk and Mavinic, 1989).

4. Conclusions

This study has confirmed the viability of the SBR process equipped with a monitoring and automation system based on artificial intelligence concepts for biological treatment of leachates resulting from old landfills.

The results confirm the effectiveness of the nitrite path for nitrogen removal optimisation: in fact, a significant saving (approximately 35%) of the external COD required to accomplish the denitrification process was achieved.

The developed control system appears to be effective and stable: indeed, almost complete inhibition of the nitrite oxidizing organisms was observed, whereas acclimation to the changed operational conditions was not observed.

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