KINETIC EVALUATION AND PERFORMANCE OF PILOT-SCALE FED-BATCH AERATED LAGOONS TREATING WINERY WASTEWATERS

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Abstract

Winery wastewater was treated in two pilot-scale aerated lagoons operating in fed-batch mode. A first lagoon of 27.2 m^3 working volume was gradually filled with wastewater with an average COD content of 8700 mg/L. Given that with the flow-rate used (790 L/day) this first lagoon completed its working volume after 30 days of starting, part of the liquid contained in the first lagoon was transferred to another adjacent second lagoon of 10.8 m^3 working volume. Therefore, the experiment continued in the second lagoon for another additional 24 days using an influent with a COD content of 18700 mg/L at a flow-rate of 170 L/day. After the 21st day, a maximum COD removal efficiency of 91% was achieved, and this value was maintained virtually constant until the end of the experiments. A mathematical model based on three differential equations solved simultaneously allowed the kinetic parameters of the system to be obtained.

Keywords: Assessment; kinetics; fed-batch lagoon; aerobic treatment; winery wastewaters.

1. Introduction

Wineries and other grape processing industries generate large volumes of wastewater annually, estimating amounts of about 1-3.5 litres of wastewater per litre of wine produced (Andreattola et al., 2002; Malandra et al., 2003; Alister et al., 2005; Vlyssides et al., 2005; Mosteo et al., 2006). This wastewater originates from various washing operations during the crushing and pressing of grapes, as well as the rinsing of fermentation tanks, barrels and other equipment or surfaces, from the washing of bottles and from cooling (Petruccioli et al., 2002; Malandra et al., 2003). Due to the high organic load (with a COD in the range of 1800-25000 mg/L) and the large volumes with a pronounced seasonal variability, the environmental impact of this effluent is noticeable; the different wine-making technologies used may also account for the marked variability in the amount and the composition of this wastewater (Petruccioli et al., 2000; Malandra et al., 2003). In addition, all the typical substances of grapes and wine (organic acids, sugars, alcohols, etc.), the residues from the wine-making (yeasts, bacteria, clarifying and fining agents, etc.) and sterilising agents used for the treatment of tanks and winery rooms (Fumi et al., 1995) can all be found in the winery effluents (both in suspension and solution).

In the last two decades, a large number of small wineries have disappeared in favour of large, often cooperative wineries that process large amounts of grapes in short periods of time. As a consequence, the volumes of the daily wastewater have increased considerably during the grape-harvesting and wine-making periods (Petruccioli et al., 2002; Vlyssides et al., 2005). Although the high organic load of this wastewater would recommend the application of an anaerobic treatment for removing its polluting content, several problems have been found in the application of anaerobic processes. This is due to its seasonal nature, its variable volumes and compositions and the difficulties in the monitoring and controlling of the process by specialized personnel (Malandra et al., 2003).

Several criteria should be considered when deciding on a treatment system for winery wastewater. These include an eco-friendly process that is flexible enough to handle various concentration loads and characteristics; low capital and operating costs. The system should require minimal personal attention, and the desired degree of degradation should be achieved without a need for dilution with water (Malandra et al., 2003). Many methods, both chemico-physical and biological, have been assayed to reduce the organic load of the winery wastewater. Among the former methods were evaporation-condensation with or without combustion, microfiltration, ultrafiltration and reverse osmosis. Among the latter were anaerobic digestion using UASB reactors and aerobic treatment using attached film systems, conventional or multistage activated sludge processes, rotating biological contactors, jet-loop reactors, etc. (Fumi et al., 1995; Petruccioli et al., 2000 and 2002; Malandra et al., 2003). However, most of these methods have the following characteristics in common: they are relatively expensive, they are not applicable in all situations, and they are not always capable of withstanding fluctuations in the hydraulic and pollution load. In order to solve some of these problems various techniques are being studied at present as alternatives to, or improvements on, the above-mentioned conventional methods in order to see if they are efficient and appropriate.

Fed-batch operation of an aerated lagoon is a recent concept in wastewater treatment technology (Dinçer, 2004). Fed-batch operation involves the slow addition of highly concentrated wastewater into an aerated lagoon with no effluent removal until the tank is full. The aerated lagoon usually contains a large volume of highly active and dense microorganisms at the beginning of operation with slow feeding. Concentrated or toxic wastewater is diluted inside the reactor, resulting in less inhibition and higher COD removal rates. This type of operation is widely used in the fermentation/bioprocessing industry with feed-back control in the case of substrate inhibition. Fed-batch operation is different from sequencing batch operation. Concentrated feed wastewater is added slowly or intermittently into the aerated lagoon in fed-batch operation as compared with complete batch operation in a sequencing batch reactor. In fed-batch operation, liquid volume in the lagoon increases linearly with time, as it is a process which lacks a stationary phase and which has process variables that are not constant (Costa et al., 2004). Fed-batch systems also have the advantage that the repressive effect caused by the presence of carbon sources is avoided because carbonaceous compounds which may be present are rapidly removed by the microorganisms being cultivated. In addition, fed-batch processes usually have reduced medium viscosity and the effects of toxic components are also limited by dilution (Costa et al., 2004; Dinçer et al., 2004).

Despite the occurrence of several research works reported in the literature regarding the use of laboratory-scale fed-batch aerated lagoons treating different industrial wastewaters (Bali and Sengul, 2002; Dinçer, 2004; Kapley et al., 2007) containing some toxic compounds (e.g. 4-chlorophenol, boron, nitroaromatic substances, etc.), kinetic studies and performance evaluation of full or pilot-scale aerated fed-batch lagoons treating winery wastewater have not been reported up to now.

The aim of this paper was to assess the overall performance of two pilot-scale fed-batch aerated lagoons treating winery wastewaters under real industrial operating conditions, studying the kinetic behaviour of these aerated systems with the objective of designing analogous purification systems.

2. Materials and methods

2.1. Pilot fed-batch aerated lagoons

Given that the experiment aimed at reproducing full-scale operational conditions, the pilot fed-batch aerated lagoons were installed in a wine industry located at Molina, Chile (Southern Chile, 35 degrees south, 71 degrees west longitude and 240 metres about sea level).

During the experiment a first lagoon of 27.2 m^3 working volume was gradually filled. This lagoon had an inclined base and a variable depth of between 1.70 and 1.82 m. Its length and width were 2.98 m and 4.89 m, respectively. During the operational period the lagoon was filled to reach 10 cm below the upper edge. Given that with the flow-rate used this first lagoon completed its working volume after 30 days of starting, part of the liquid contained in the first lagoon was transferred to another adjacent second lagoon of 10.8 m^3 working volume and a depth of between 1.21 m and 1.36 m. The length and width of this second lagoon were 2.89 m and 2.96 m, respectively. As a consequence, the experiment continued for an additional period of 24 days. Therefore, the total experimental period was 54 days. The first lagoon was filled with the effluent from the above-mentioned typical winery industry during the vintage period. During the filling-up process, the feeding pump was not strictly operated in continuous mode, but the feeding was added intermittently by pulses by using a timing device to simulate real operating conditions. To be specific, to fill the first tank 7 pump pulses per day with a duration of 4 minutes per pulse were used. Therefore, an average flow-rate of 790 L/day was used during the first phase of the experiment carried out during the first 30 days. During the second phase of the experiment (days 31-54), the flow-rate was adjusted to 170 L/day (6 pulses/day each lasting 1 minute) in the second lagoon. Therefore, the experiment was extended another 24 days.

The chemical oxygen demand (COD) values of the winery wastewaters used for feeding the first and second lagoon were 8700 and 18700 mg/L, respectively. The COD of the feeding of the second lagoon was higher as consequence of the change in the processing of white to black grapes, which originated most concentrated winery wastewaters. For the aeration of the lagoons, three Jet-Mix model superficial aerators were used. The Jet-Mix aerators inject a flow of atmospheric air under the liquid surface at a high rate, which produces a horizontal flow to maintain the solids in suspension, increasing the levels of dissolved oxygen. Table 1 summarizes the technical specifications of the aerators used in this study.

2.2. Experimental procedure

Given that the Jet-Mix aerators require a minimum operating depth in the lagoon of 35 cm, for the first three days of the experiment a Venturi pipe was used as an alternative system. Therefore, an initial volume of effluent of 3.47 m^3 was added to the lagoon 1 (35 cm deep). During these first 3 days the aspirated flow-rate passed through a Venturi system. The use of this system was not necessary in the second lagoon because the initial liquid height allowed to operate directly with the Jet-Mixers.

The above-mentioned three Jet-Mixers were used during the rest of the experiment. The Jet-Mixers were started-up manually and switched off with the aim of achieving a dissolved oxygen concentration in the wastewater higher or equal to 2 mg/L.

The first lagoon was operated during the first 30 days of the experiment, while the second operated between the 31^{st} and 54^{th} days. Prior to filling the first lagoon, the winery wastewater was subjected to the following initial steps: roughing down, sedimentation and homogenization.

2.3. Chemical analyses

COD and pH were analysed according to the Standard Methods numbers 5220C and 4500, respectively (APHA, 1998). Dissolved oxygen was measured by using a portable dissolved oxygen sensor (DO30G Yokogawa Electric Corporation). Microscopic observations were made using a visible optic microscope.

pH, COD and dissolved oxygen concentrations were measured daily in triplicate samples and the results expressed as means. Therefore, all the values plotted in the next Figures correspond to the average values obtained for each case.

3. Results and discussion

3.1. pH evolution

Figure 1 illustrates the variation of the inlet pH and pH in the aerated lagoons with time during the two stages of the experimental study. The inlet pH or pH of the winery wastewater is acidic in both cases with average values of 4.3 and 3.6 during the first and second stages respectively.

Given that between the generation of wastewater and its treatment, several hours go by in which the wastewater is not aerated. It is likely at this stage for an anaerobic digestion process to start, transforming part of organic matter into volatile fatty acids of two to five carbon atoms; this fact would justify the acidic nature of the wastewater prior to being subjected to the aerobic treatment. From the 5th day of initiating the aerobic treatment, a continuous increase in the pH of the first lagoon was observed, and from the 15th day of treatment the pH was always alkaline achieving an average value of 7.6. This was due to the alkalinity generated by the oxidation of the organic matter to CO_2.

3.2. Microbiological characterisation

The microscopic observations made in the lagoon contents confirm that the operating conditions were adequate for the correct performance of this system. During the first days of the experiments a predominance of yeast was observed, detecting also the presence of the protozoa *Arcellas sp.* When the pH achieved values close to 7.0, *Chilodonella cucullulus* was also detected, whereas when the system achieved a pseudo-equilibrium state, the protozoa *Opercularia sp.* was also present.

Previous reports have demonstrated that yeasts play an important role in the biodegradation of winery wastewaters in rotating biological contactors (RBC) (Malandra et al., 2003). Yeast and bacterial species displayed a dynamic population ship in the biofilms developed on the RBC discs. One of the yeasts isolated (MEA5) was also able to reduce the COD of synthetic wastewater by 95% within 24 h under aerated conditions (Malandra et al., 2003). A microbiological characterisation was also carried out after 5 months of operation in a high-rate multistage activated sludge system treating winery wastewater (Petruccioli et al., 2000). The bacterial species isolated from this reactor liquid belonged for the most part to the genus *Pseudomonas*, well known for the ability of several of these members to degrade aromatic compounds and to produce exo-polysaccharides that play an important role in floc formation. *Saccharomyces cerevisiae* was also isolated in this reactor; this yeast is always present in winery wastewater, particularly during vintage and played an important role in the degradation process (Petruccioli et al., 2000). *Saccharomyces cerevisiae* was also present in the microbial consortia developed in jet-loop type reactors treating winery and olive oil wastewaters (Petruccioli et al., 2002; Eusebio et al., 2005). Finally, a phylogenetic analysis of 5'-partial 16S rDNA sequences detected in the microbial consortium developed in a fed-batch reactor treating wastewater containing nitroaromatic residues revealed that Proteobacteria (α, β, and γ) and Firmicutes were the major phyla in a microbial community (Kapley et al., 2007).

3.3. Dissolved oxygen

The dissolved oxygen concentration varied with time achieving values of 0.5 and 8.0 mg/L as extremes, 2 mg/L being the average value reached. The experiments were designed to achieve dissolved oxygen concentrations equal to or higher than 2 mg/L. However, as the control of the aeration system was carried out manually, by supplementing the low level Jet-Mixer (JM-005) when necessary, these oxygen concentrations were not reached in some cases (especially during the first 10 days of the experiments). As consequence of this phenomenon, a decrease in the substrate removal rate was observed in these specific cases.

Similar dissolved oxygen concentrations (about 2 mg/L) were reported in aerobic treatments of winery wastewater and different industrial wastewaters containing aromatic compounds using conventional or multistage activated sludge systems and fed-batch reactors (Fumi et al., 1995; Petruccioli et al., 2000; Bali and Sengul, 2002; Dinçer, 2004).

3.4. Temperature

Given that the duration of the total experimental phase was 54 days and the season in which the experiments were carried out was March to May, the ambient temperature experienced a slight variation with the operation time with 17 ºC and 25 ºC as extreme values. This meant that the experiments were prevented from being performed at isothermic conditions. No relationship between the increase in temperature and the decrease in the concentration of dissolved oxygen was observed. As a consequence, the deficit in the dissolved oxygen concentration may be attributed to the use of an aeration system with insufficient capacity.

Similar and higher operating temperatures (around 30 ºC) (Fumi et al., 1995; Petruccioli et al., 2000) were used during the aerobic treatment of winery wastewaters using high-rate multistage and free or immobilized activated sludge bioreactors operating in continuous mode. However, while the average COD removals obtained in the first case (98%) were higher than those obtained in the present work, in the second case (90%) the efficiencies were very similar to those achieved in our experiments using fed-batch aerated lagoons (Figure 2).

3.5. COD removal efficiency

Figure 2 illustrates the variation of the COD removal efficiency (%) as a function of the operation time. COD removal efficiency can be defined as the quotient between the difference of the initial COD and effluent COD concentrations and the initial COD concentration (Pelillo et al., 2006).

During the first 10 days of the assay, the COD removal efficiency was low (around 12%-20%) as a consequence of the initial biomass acclimatization process to the substrate with a predominance of yeasts. In addition, the aeration system (Venturi tubes) used during this period did not supply enough oxygen to facilitate the metabolism of the microorganims responsible for the degradation of this wastewater. Between days 11 and 20, an exponential increase in the COD removal efficiency with time was observed, showing a high affinity of the microorganisms for the substrate. The microscopic observations revealed a predominance of bacteria over the yeasts during this period. It can be considered that all the most easily biodegradable compounds (simple sugars, acids, alcohols, etc.) are degraded during this time period. This behaviour was foreseeable given that the COD/BOD_5 ratio was 2.1 ± 0.2. Finally, after the 21st day, a maximum average COD removal efficiency of 91% was achieved, and this value was maintained virtually constant until the end of the experiments, indicating that a small fraction of the COD of this wastewater is not biodegradable or is resistant to the aerobic degradation. As was also observed, an electrical failure in the aeration system happened on the 41st day causing a 76% decrease in the COD removal efficiency. The system recovered again up to efficiencies of 91% after the above-mentioned operation time.

Lower COD removal efficiencies (61%) were reported after a period of two months in a fed-batch reactor treating wastewater containing nitroaromatic compounds (Kapley et al., 2007). In the same way, removal efficiencies of 43% were detailed in rotating biological contactors treating winery wastewaters at hydraulic retention times as low as 1 hour (Malandra et al., 2003). In contrast to this, COD removal efficiencies of around 90%, similar to those obtained in the present research work, were achieved during the aerobic treatment of winery wastewaters using multistage and jet-loop activated sludge reactors operating in continuous mode (Petruccioli et al., 2000 and 2002).

3.6. Mathematical model of the process

For the formulation of the mathematical model of the process, the following hypotheses were assumed:

- The volumetric flow-rates and the inlet CODs for the two stages or phases of the experimental work were considered constant.
- Although the influent winery wastewater is not sterile, the contribution of the microorganisms of the influent to the total biomass content present in both lagoons is negligible.
- It is assumed that the temperature in each lagoon is virtually constant, with complete mixed conditions in both cases.
- Finally, it is assumed that a fraction of the inlet COD is not biodegradable as can be seen in Figure 2. Therefore, the experimental values of inlet and effluent COD must be corrected by subtracting the non-biodegradable fraction.

These hypotheses are applicable to fed-batch processes and were described in different texts and previously reported works (Dunn et al., 2003; Dinçer, 2004; Costa et al., 2004; Kapley et al., 2007). Taking these assumptions into account, and making COD and microorganism balances, the following mathematical model can be established by using the following differential equations:

$$\mathrm{d}V/\mathrm{d}t = F \qquad (1)$$

$$\mathrm{d}S/\mathrm{d}t = (F/V)\,(S_0 - S) - [(\mu_m(S - S_{nb})/(K_S + (S - S_{nb})) - K_d](X/Y) \qquad (2)$$

$$\mathrm{d}X/\mathrm{d}t = [[\mu_m(S - S_{nb})/(K_S + (S - S_{nb})) - K_d] - (F/V)]X \qquad (3)$$

where: V is the lagoon volume (L or m^3); F is the volumetric flow-rate (L or m^3/day); t is the operation time (days); μ_m is the maximum specific microbial growth rate (1/days); S_0 and S are the influent and effluent substrate concentrations (mg or g COD/L) respectively; S_{nb} is the non-biodegradable substrate concentration (mg or g COD/L); X is the cellular or biomass concentration (mg or g volatile suspended solids, VSS/L); Y is the cellular yield coefficient (g VSS/g COD); and K_S is the saturation constant (mg or g COD/L).

To obtain equations (2) and (3) which correspond to the substrate and microorganism balances, the following equations for expressing the substrate removal rate (r_s) and microorganism growth rate (r_x) were used (Dunn et al., 2003):

$$r_s = [\mu_m(S - S_{nb})/(K_S + (S - S_{nb})) - K_d](X/Y) \quad (4)$$

$$r_x = X[\mu_m(S - S_{nb})/(K_S + (S - S_{nb})) - K_d] \quad (5)$$

For all these equations, substrate concentrations (S_0, S and S_{nb}) were expressed as soluble COD.

The values of the kinetic parameters (μ_m, Y, K_S and K_d) were determined from the experimental results of substrate and biomass concentrations (Figure 3) simultaneously solving differential equations (1), (2) and (3) using the 2008 MathCad software (version 14.0) (MathCad, 2008) with the aim of obtaining a minimum value of the sum of the squares of the differences between the experimental and theoretical values. The following initial conditions were considered for initiating the software calculation:

- First stage: initial volume of the first lagoon, 3.47 m^3; initial influent COD, 8700 mg/L; initial flow-rate, 790 L/day; and initial biomass concentration, 900 mg VSS/L.

- Second stage: initial volume of the second lagoon, 5.1 m^3; initial influent COD, 18700 mg/L; initial flow-rate, 170 L/day; and initial biomass concentration, 2115 mg VSS/L.

Therefore, the values for the kinetic parameters μ_m, Y, K_S, and K_d as well as the corresponding standard deviations obtained using the MathCad software (version 14) were: 0.28 ± 0.01 1/days, 0.26 ± 0.02 g VSS/g COD, 175 ± 9 mg COD/L and 0.12 ± 0.01 1/days, respectively. Finally, the non-biodegradable substrate concentration (S_{nb}), which was obtained from the maximum COD removal percentage achieved during the last 25 days of the process was 790 ± 39 mg COD/L. Similar values of the kinetic constants to those obtained in this research work were reported in the literature for aerobic treatment processes of similar wastewaters (Ramalho, 1983; Dunn et al., 2003; Dinçer, 2004).

The proposed equations (1-3) were validated by comparing the theoretical curves obtained with the corresponding experimental data from the two phases of the experimental work. Figure 3 shows the comparison of the experimental data of the CODs and biomass concentrations of the aerated lagoons with the corresponding theoretical values obtained by the equations (2) and (3), respectively. The minor deviations obtained in all cases (lower than 10%) demonstrate the suitability of the kinetic model proposed and suggest that this model describes the aerobic degradation of winery wastewater in fed-batch lagoons very accurately and that the kinetic parameters obtained represent the activity of the microorganisms effecting the aerobic degradation of this waste. In any case, the proposed mathematical model can be considered as a "black box" which allows fitting the experimental data adequately and represents the overall performance of the system.

4. Conclusions

Winery wastewater was biologically degraded using pilot-scale aerated lagoons operating in a fed-batch mode. A maximum COD removal efficiency of 91% was achieved after the 21st day of operation, and this value was maintained virtually constant.

A mathematical model based on three differential equations solved simultaneously allowed the kinetic parameters of the system to be obtained. The kinetic constants were obtained and the proposed equations were used to simulate the aerobic process and to obtain the theoretical values of the aerated lagoon CODs. Deviations between the theoretical and experimental values equal to or lower than 10% were obtained.

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Table 1

Technical specifications and characteristics of the three Jet-Mix aerators used in this study.

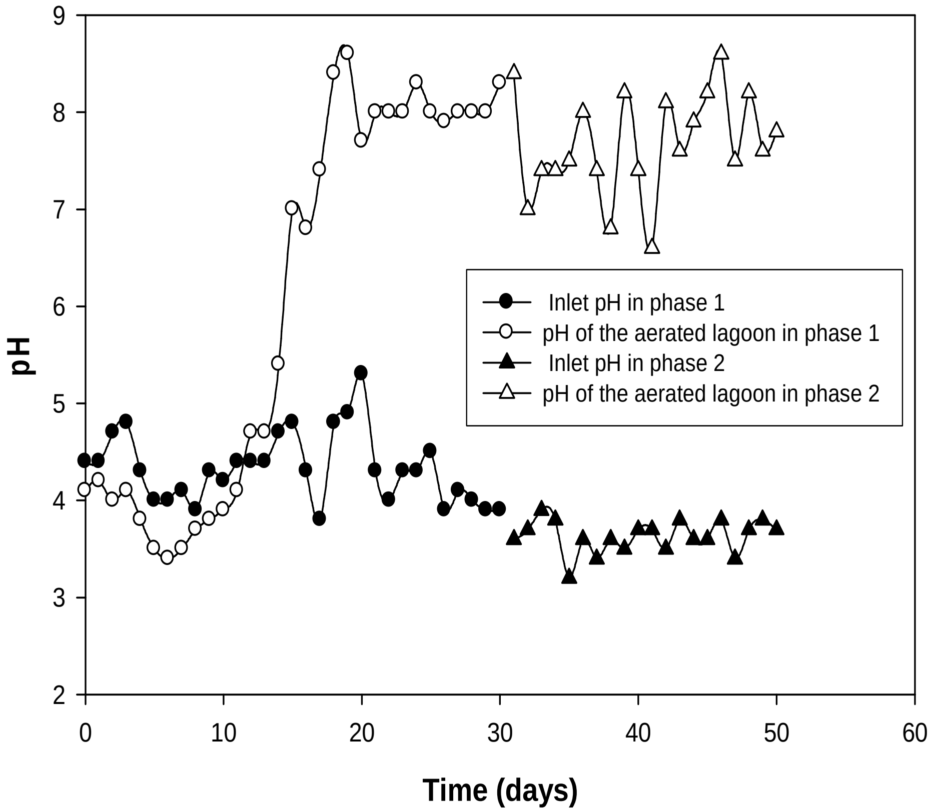
Jet-Mix Model	Pump Engine (kW)	Oxygen Transfer Rate (kg O_2/h)	Mixing Volume (m^3)	Operating Deep (m)
JM-005	0.40	0.48	40-100	0.3-1.5
JM-010	0.75	0.90	80-200	0.8-2.5
JM-020	1.50	1.80	160-400	0.8-2.5

Figure captions

Figure 1. Variation of the inlet pH and pH in the aerated lagoons with time during the two stages of the experimental study.

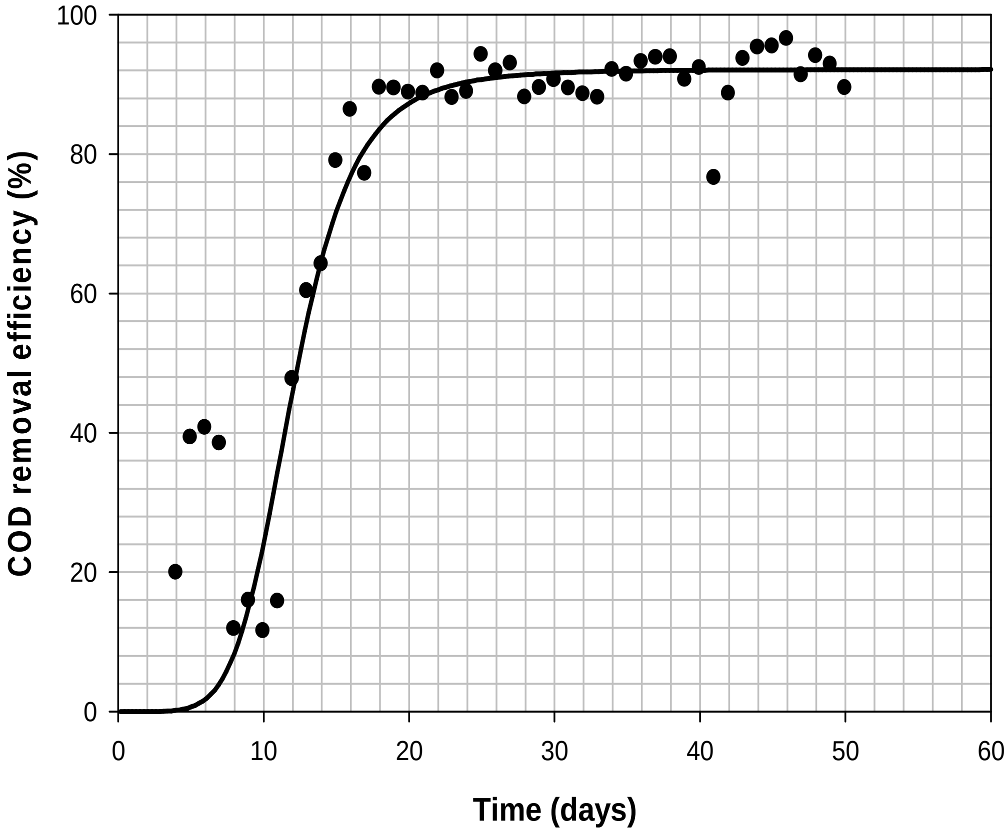
Figure 2. Variation of the COD removal efficiency with operational time during the experimental study.

Figure 3. Variation of the experimental and simulated COD and biomass concentration (X) values of the aerated lagoons during the two stages of the experimental study.



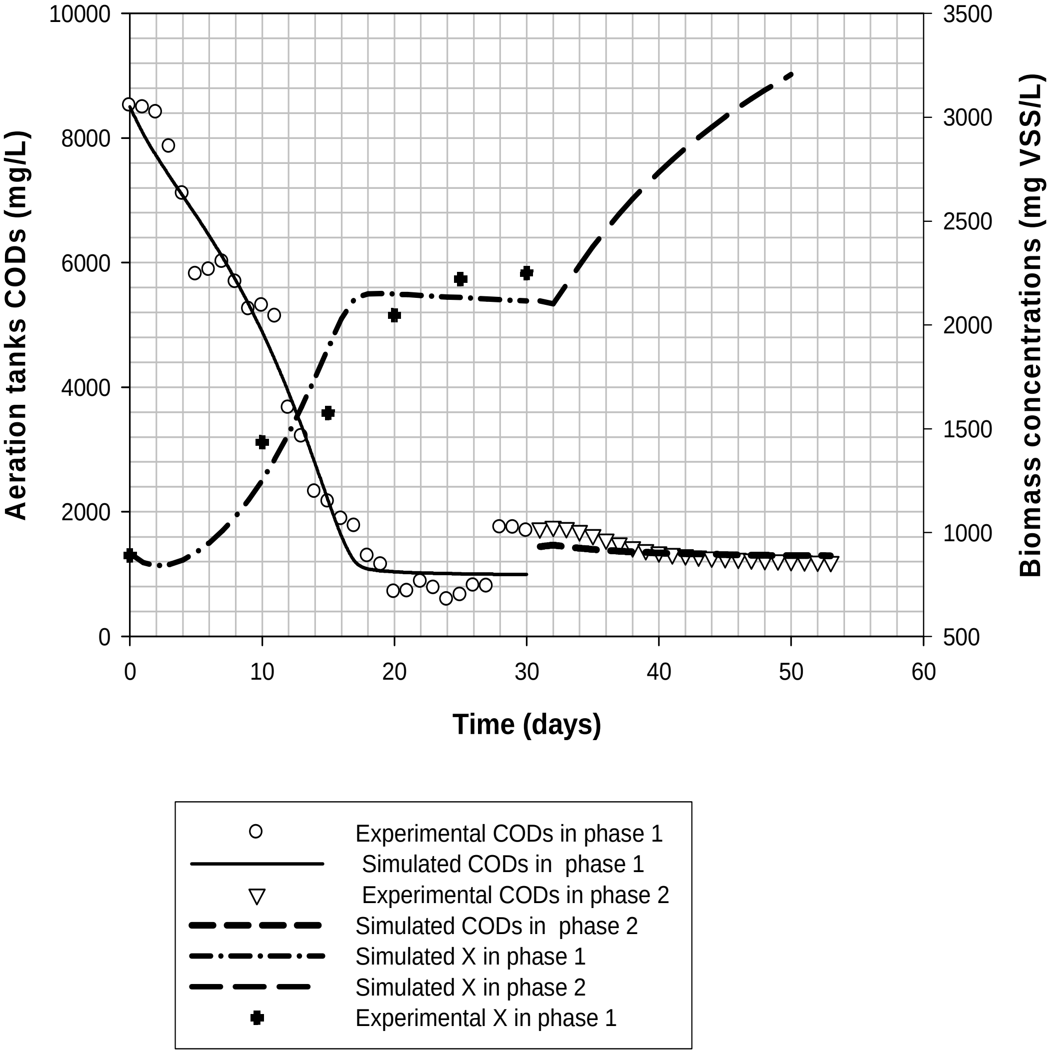
pH
Time (days)
Inlet pH in phase 1
pH of the aerated lagoon in phase 1
Inlet pH in phase 2
pH of the aerated lagoon in phase 2
2
3
4
5
6
7
8
9
0
10
20
30
40
50
60

Figure 1



100
80
60
40
20
0
COD removal efficiency (%)
0
10
20
30
40
50
60
Time (days)

Figure 2



Aeration tanks CODs (mg/L)
Biomass concentrations (mg VSS/L)
Time (days)
0
2000
4000
6000
8000
10000
500
1000
1500
2000
2500
3000
3500
0
10
20
30
40
50
60
Experimental CODs in phase 1
Simulated CODs in phase 1
Experimental CODs in phase 2
Simulated CODs in phase 2
Simulated X in phase 1
Simulated X in phase 2
Experimental X in phase 1

Figure 3