

Sequencing batch reactor performance treating PAH contaminated lagoon sediments

Andrea Giordano^{a,*}, Loredana Stante^a, Francesco Pirozzi^b, Raffaele Cesaro^b, Giuseppe Bortone^c

^a ENEA – PROT-IDR, Environmental Technologies, Water Resource Management Section, Via Martiri di Montesole 4, 40129 Bologna, Italy

^b University of Naples Federico II, Department of Hydraulic and Environmental Engineering Girolamo Ippolito, Via Claudio 21, 80125 Napoli, Italy

^c Emilia-Romagna Region, Environmental Directorate, Via dei Mille 21, 40100 Bologna, Italy

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Abstract

The applicability of sediment slurry sequencing batch reactors (SBR) to treat Venice lagoon sediments contaminated by polycyclic aromatic hydrocarbons (PAHs) was investigated, carrying out experimental tests. The slurry, obtained mixing tap water and contaminated sediments with 17.1 mg kg⁻¹ TS total PAHs content, was loaded to a 8 l lab-scale completely stirred reactor, operated as a sequencing batch reactor.

Oxygen uptake rate exerted by the slurry, measured by means of a DO-stat titrator, was used to monitor the in-reactor biological activity and to select the optimal operating conditions for the sediment slurry SBR.

The PAHs removal efficiency was evaluated in different operating conditions, obtained changing the hydraulic retention time (HRT) of the lab-scale reactor and adding an external carbon source to the slurry. HRT values used during the experiments are 98, 70 and 35 days, whereas the carbon source was added in order to evaluate its effect on the biological activity. The results have shown a stable degradation of PAHs, with a removal efficiency close to 55%, not dependent on the addition of carbon source and the tested HRTs.

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1. Introduction

Large volumes of dredged materials have to be removed periodically from harbours, ports and waterways mainly to maintain and/or increase shipping depth. U.S. Environmental Protection Agency (EPA) estimated that approximately 10% of the sediment underlying the surface water in USA is heavily contaminated by toxic pollutants representing a potential risk for fishes and humans [1].

Sediment contamination involves both inorganic and organic compounds with a wide range of concentrations; among

organic pollutants, polycyclic aromatic hydrocarbons (PAHs) are the most common [2].

PAHs include 70 natural and anthropogenic organic compounds constituted by aromatic rings, ranging between two and seven, and mainly derived from petroleum activities. In the XX century there was a great increase of anthropogenic production of PAHs by combustion of fossil fuel. Due to their toxicity, 16 PAHs were listed by U.S. Environmental Protection Agency as priority pollutants, which should be monitored in aquatic and terrestrial ecosystems.

In harbours, rivers and estuaries the PAHs accumulation rate greatly exceeds their natural degradation rate: this mainly occurs because PAHs are hydrophobic and sorb tightly to sediment particles in saturated aqueous environments [3]. Generally, an increase in the size and angularity of PAHs molecule results in a concomitant increase in the hydropho-

* Corresponding author. Tel.: +39 051 6098698; fax: +39 051 6098309.

E-mail addresses: andrea.giordano@bologna.enea.it (A. Giordano), loredana.stante@bologna.enea.it (L. Stante), francesco.pirozzi@unina.it (F. Pirozzi), racesaro@unina.it (R. Cesaro), gbortone@regione.emilia-romagna.it (G. Bortone).

bicity and electrochemical stability [4], which represent the two primary factors contributing to the persistence of high molecular weights PAHs in the environment [5]. Therefore, PAHs are relatively persistent and recalcitrant in sediments and more difficult to be degraded under natural conditions than most of the other organic contaminants [6].

Several isolated microorganisms are able to remove PAHs [7–9] with different kinetic rates depending on environmental conditions and PAHs molecular weight, number of rings and molecule structure.

PAHs biodegradation may occur both aerobically and anaerobically. In particular, when oxygen is not available, some microorganisms can use Mn(IV), nitrate, Fe(III), sulphate and carbonate as alternative electron acceptor [10].

Surfactant addition is a practical method to enhance the biodegradation of hydrophobic PAHs [11], which are scarcely available to microorganisms. Mineralization of organic compounds is also favoured by biosurfactant addition [12].

The most widely used technologies for bioremediation of PAH contaminated soils and sediments are landfarming, bioventing, air sparging and bioslurry reactor [6,13,14]. In particular, bioslurry reactor is a biological ex situ technology, offering optimal process control and high biodegradation rate [14,15]. The biological process is carried out in a reactor where the dredged sediments are mixed with external water. If needed, external addition of nutrients is provided. Solids concentration typically ranges between 5 and 50%, depending on the mixing and aeration equipments and on contaminant toxicity. Bioslurry treatment has been tested in different types of reactors, including: batch system [16]; sequencing batch reactor (SBR) [17]; continuous-flow completely stirred tank reactor (CSTR) [18].

Recent results [18] have shown, at least for soil contaminated by hydrocarbons, that SBR are able to reach higher efficiencies than the other above-mentioned systems.

The SBR functioning cycle includes three temporal steps: during the Fill step, the slurry is fed into the reactor; in the Reaction step the system works as a batch reactor, performing the biodegradation process; the final step consists of the extraction of a fraction of treated slurry from the reactor (Draw step) and the replacement with an equal volume of untreated slurry.

The most important operating factors influencing the SBR removal efficiency are the hydraulic retention time (HRT), the volume of slurry replaced at the end of each cycle, the solids concentration and the mixing speed. The HRT and the volume of slurry replaced per cycle can be adjusted to face different contaminant loads to the reactor.

This paper describes experiments carried out using a lab-scale aerobic SBR, fed with contaminated sediments from Venice lagoon (Italy). The Venice lagoon is one of the most superfund sites in Italy (listed among the main sites in needs of reclamation by the Italian Law. 426/1998), as some lagoon areas have been used as dumping sites for urban and industrial waste since the beginning of 1900 [19]. It was es-



Fig. 1. Lab-scale SS-SBR.

timated that $7.0 \times 10^6 \text{ m}^3$ of sediments must be dredged by Venice channels, whose $1.5 \times 10^6 \text{ m}^3$ are polluted by heavy metals, pesticides, PAHs, etc. [20].

Initially, the biological activity in the lab-scale SBR was investigated examining the depletion rate of two added organic substrates (glucose and lactose). Then, the total PAH removal efficiency and the oxygen uptake rate (OUR) were evaluated in different SBR operating conditions. The addition of lactose in the reactor as biosurfactant stimulator [21,22] to enhance PAH removal efficiency was also investigated.

2. Materials and methods

2.1. Sediments

Sediments used for the experimental tests were collected from the treatment plant Alles located in Fusina (near Venice), where sediments dredged from the lagoon are de-watered after removal of particles with diameter larger than 2 mm. The granule size distribution was evaluated according to the Standard of American Society for Testing and Materials (ASTM) D42 protocol [23].

The slurry (200 l) was prepared mixing sediments and tap water to reach a 10% solids concentration (weight of dry sediment/weight of slurry) and was stored at 4 °C to stop the biological activity. Before feeding the slurry into the reactor, it was maintained, for about 20 min, in the thermostated chamber to restore the temperature.

2.2. Bioreactor

The study was carried out in the thermostated chamber (at $21 \pm 0.3 \text{ }^\circ\text{C}$) using a 8 l pyrex-made reactor with a 17.1 cm diameter and 47.0 cm height (Fig. 1), upper closed with a stainless steel flange to ensure the seal. The reactor operating level was set at 41.0 cm (leaving 6 cm of freeboard); thus the reactor working volume was 7 l. The reactor was equipped

Table 1
SBR operating conditions during the four experimental phases

Phase	Cycle length (d)	HRT (d)	Volume replaced per cycle (l)	Number of cycles
I	14	98	1	21
II	7	70	0.7	36
III	3.5	35	0.7	46
IV ^a	3.5	35	0.7	46

^a At the beginning of each treatment cycle 0.5 g of lactose were added.

with a DO probe (cell OX325, WTW), a pH probe (Mettler Toledo) and a magnetic stirrer with a variable speed impeller, working at 300 rpm and placed in the flange central position.

Oxygen was supplied by an air flow of 50 Nl h⁻¹ and distributed through a fine bubble diffuser at the bottom of the reactor. The air flow was forced to pass through a humidifier before entering the reactor, in order to minimize evaporation.

Off gases firstly were piped to an air-cooled condenser and then collected to an amberlite resin trap (XAD2 Supelco) to catch volatile organic compounds. Peristaltic pumps (Cellai 503U) were used for fill and draw operations.

2.3. Experimental design

The experiments were grouped into four phases with different operating conditions (Table 1). In all cases the times to fill and draw the lab-scale SBR were lower than 3 min (as a result the entire cycle length was devoted to react) and no adapted microorganisms (bioaugmentation) were added to the fed slurry to enhance the biological activity.

Phase I was aimed at starting the process and evaluating the slurry biological activity. Its duration, cycle length and HRT were set to 294 days, 14 days and 98 days, respectively. In these conditions, the duration corresponds to about three HRTs, usually considered sufficient to reach steady state conditions [24]. The slurry volume replaced per cycle was equal to 1 l.

At the beginning of the two last treatment cycles, two carbon sources (3.65 g of glucose and 3.45 g of lactose) were added and their degradation trend was monitored in order to detect the presence of biological activity.

The further three phases were carried out to evaluate PAHs removal efficiencies and OUR trends in different operating conditions.

In particular, during Phase II, the treatment cycle length, the HRT and the volume of slurry replaced for each cycle were adjusted to 7 days, 70 days and 0.7 l (10% of working volume), respectively.

During Phases III and IV the treatment cycle length was shortened to 3.5 days while the volume of slurry replaced for each cycle was the same as the previous Phase II.

Moreover, during Phase IV, 0.5 g of lactose was added at the beginning of each treatment cycle to stimulate bio-surfactant production and increase the sediment microbial community activity.

2.4. Chemical analyses

Fed and treated slurry were sampled during each treatment cycle and analysed. Total organic carbon was measured, on both dried (TOC_D) and filtered (0.45 μm, TOC_F) samples, using a Shimadzu TOC analyser (model VPN cph). Total and volatile solids (TS and VS, respectively), total Kjeldhal nitrogen (TKN), total phosphorous (P_t), chlorides and colony forming units (CFU) were measured according to standard methods [25].

The slurry fed into the reactor and the slurry drawn at the end of each cycle were analysed for PAHs determination according to the *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods* (EPA, SW-846) [26]. Briefly, Soxhlet extraction of the organic compounds was carried out on 5–10 g dried sediments according to EPA 3540C method, silica-gel clean-up was performed on the Soxhlet extract according to EPA 3630B method, and finally the eluted fraction was analysed by GC/MS according to 8270C method using an Agilent Technologies 5973 gas-chromatograph, equipped with a 5970B mass selective detector and a 30 m × 250 μm thickness 0.25 μm, DB-5 fused silica capillary column (J&W Scientific), operating in the selected ion monitoring (SIM) mode.

2.5. DO-stat titrator

The DO-stat technique was applied with the final scope to monitor the OUR trend versus time during the treatment cycles of Phases II, III and IV in the SBR.

DO-stat titration tests were performed on 250 ml slurry samples collected from the SBR. Generally, one sample per day was taken; only during 24 h after feeding new slurry, the number of samples per day was increased to two or three in order to better follow the OUR trend, excluding the first 2 h to avoid chemical interferences on the measurements [27]. During DO-stat titration, DO set-point was set to the saturation value to avoid oxygen liquid/gas transfer. Titration tests were performed in the same thermostatic chamber (21 ± 0.3 °C) where the lab-scale reactor was placed.

DO-stat titrations were carried out using an automated system (Fig. 2), MARTINA (Multiple Analyte Reprogrammable TITration Analyser, Spes srl), which is able to collect and record pH, DO, temperature and ORP data (minimum frequency 1 per s) and convert set-point titrations into DO sig-

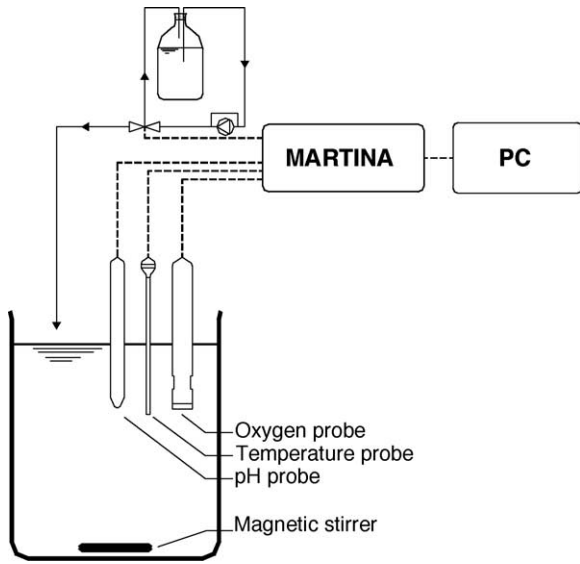


Fig. 2. Scheme of the DO-stat.

nals. The DO was maintained at its set-point value by spiking a diluted (0.09–0.18 M) H₂O₂ solution. OUR data, related to each sample (OUR_S), were calculated according to the following formula:

$$OUR_S = r_{tit} M_{H_2O_2} 16 V_S^{-1} \rho^{-1} \quad (1)$$

where OUR_S is expressed as mgO₂ kg⁻¹ slurry h⁻¹; r_{tit} the titration rate (ml H₂O₂ h⁻¹); M_{H₂O₂} the H₂O₂ solution molarity; 16 the stoichiometric coefficient for H₂O₂ decomposition to oxygen (mgO₂ mol H₂O₂⁻¹) (i.e.: H₂O₂ → 0.5O₂ + H₂O); V_S the volume of slurry (l); ρ the slurry density (kg l⁻¹).

For Phases III and IV, due to the lack in the literature of relationships suitable to fit analogous data, the experimental OUR values have been fitted with the following relationship:

$$OUR = OUR_{max} 10^{-kt} + OUR_{end} \quad (2)$$

where OUR_{max} is the maximum OUR value; k the kinetic constant; t the time.

This relationship hypothesises that OUR is the sum of two terms, related to the exogenous respiration (assumed to follow a decreasing first order kinetic) and to the endogenous respiration, respectively.

3. Results and discussion

3.1. Fed sediment characterisation

The granule size distribution of sediments (Fig. 3) shows 59 and 40 wt.% of silt and clay, respectively, with negligible sand content. In Table 2 the fed slurry contaminant concentrations are listed: according to the Italian law, the dredged sediments with total PAHs level of 17.1 mg kg⁻¹ TS are classified as waste rather than a valuable solid material. As expected,

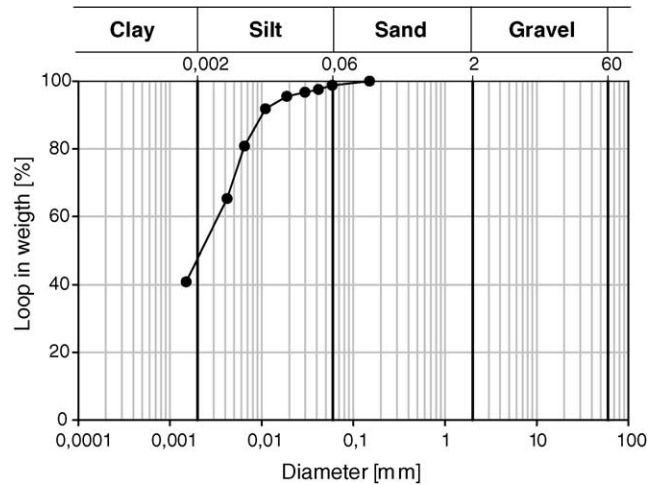


Fig. 3. Granule size distribution curve of contaminated sediments.

Table 2
Fed slurry contaminant concentrations

Total PAH (mg kg TS ⁻¹)	17.1
TOC _D (g kg TS ⁻¹)	86.5
TKN (g kg TS ⁻¹)	2.3
P _T (g kg TS ⁻¹)	1.2
Chloride (g kg TS ⁻¹)	1.6
CFU (CFU kg TS ⁻¹)	3.7 × 10 ⁷
TS% (w w ⁻¹)	10.5

due to the aged Venice channel contamination, the main PAH fraction was characterised by 5–6 rings (44%), followed by the fraction with four rings (33%) and the fraction with 2–3 rings (23%).

3.2. Phase I

Figs. 4 and 5 show the concentrations of the externally added organic substrates (glucose and lactose) in course of time, together with the TOC_F concentrations. Both substrates were degraded before the end of the cycle (14 days), when

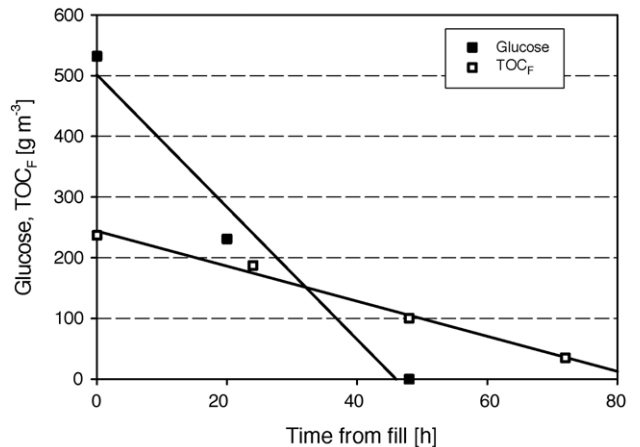


Fig. 4. Glucose vs. time in the SBR treatment cycle during Phase I.

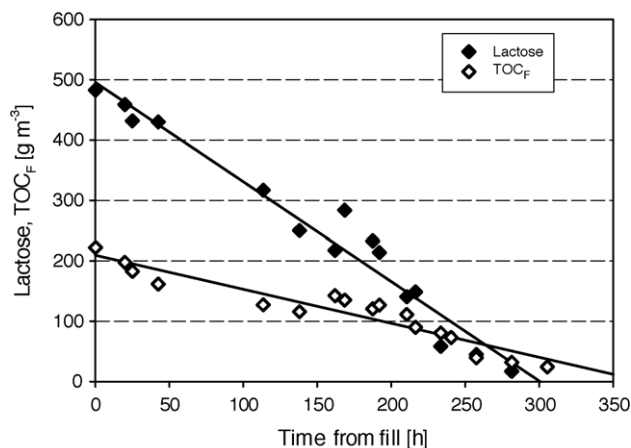


Fig. 5. Lactose vs. time in the SBR treatment cycle during Phase I.

TOC_F values lower than 35 g m⁻³ have been detected. However, whereas glucose was completely degraded in about 80 h (less than 1/4 time of the treatment cycle length), lactose was completely degraded only at the end of the cycle.

The biodegradation of both substrates started immediately after the feeding phase, although glucose and lactose have a different biodegradability behaviour, confirming the presence of sediment biological activity and biomass adaptation to different carbon sources.

The substrate degradation rates (calculated as the slope of the temporal depletion) were 11.1 and 1.6 mg l⁻¹ h⁻¹ for glucose and lactose, respectively.

TOC_F degradation rates were 2.9 and 0.5 mg l⁻¹ h⁻¹ for glucose and lactose, respectively: the first value is five times greater than the second, according to the results obtained by Pitter and Chudoba [28], working with activated sludge.

3.3. Phase II

All parameters measured on fed and treated slurry during Phase II are summarised in Tables 3 and 4. In Fig. 6 total PAH values versus time are represented. In particular, the plotted values are related to the last six cycles exclusively, because the previous cycles were necessary to achieve the steady state conditions, corresponding to three HRTs [24]. The points represent the mean values, whereas the bars are referred to measurement replicates. The data show a global removal efficiency of 55% (from about 21.2 to 9.6 mg kg⁻¹

Table 3
TOC_D and CFU average concentrations in untreated and treated slurry during Phases II, III and IV

Phase	Slurry	TOC _D (g kg ⁻¹ TS)	CFU (CFU kg ⁻¹ TS)
II	Untreated	84.5 ± 1.7	3.7 × 10 ⁷
	Treated	77.4 ± 4.0	1.2 × 10 ⁸
III	Untreated	86.5 ± 1.9	2.5 × 10 ⁷
	Treated	78.9 ± 1.7	1.9 × 10 ⁸
IV	Untreated	86.5 ± 1.9	2.5 × 10 ⁷
	Treated	81.7 ± 1.6	4.4 × 10 ⁸

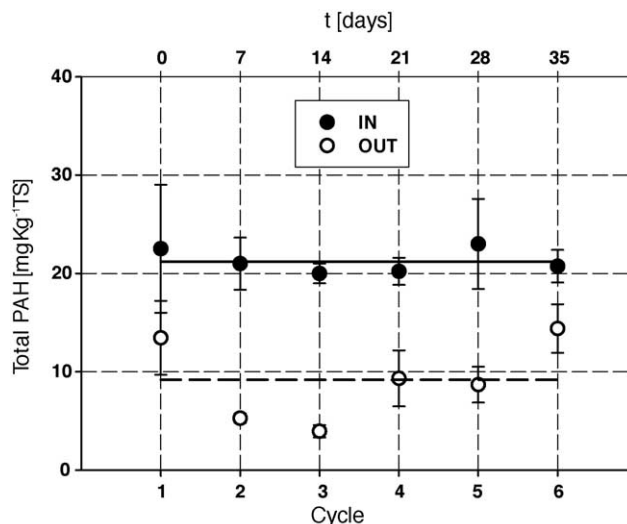


Fig. 6. Average and standard deviation of total PAH measured in treated and untreated slurry during Phase II.

TS) and a decreasing removal efficiencies corresponding to the increase of aromatic rings (Fig. 7). In fact, the concentrations of 2–3 rings PAHs in the fed and treated slurry were about 3.7 and 1.1 mg kg⁻¹ TS, respectively, with an average removal efficiency of 70%. A very close result was observed on four rings PAHs, with a removal efficiency of 64%, corresponding to initial and final concentrations of 7.2 mg kg⁻¹ TS and below 3 mg kg⁻¹ TS, respectively. Finally, PAHs with 5–6 rings, which are the main fraction, showed the lowest removal efficiency, close to 43%, with initial and final concentrations of 10.3 and 5.9 mg kg⁻¹ TS, respectively.

The above cited results on PAHs removal have to be attributed to the microbial activity since the analysis on resin trap, where off-gas reactor was collected, showed PAH concentrations below the analytical instrument detection limits, indicating negligible volatilisation and chemical reactions.

From Table 3 a one order of magnitude increase of CFU is observed corresponding to a TOC_D decrease during each cycle equal to 7.1 g TOC_D kg⁻¹ TS.

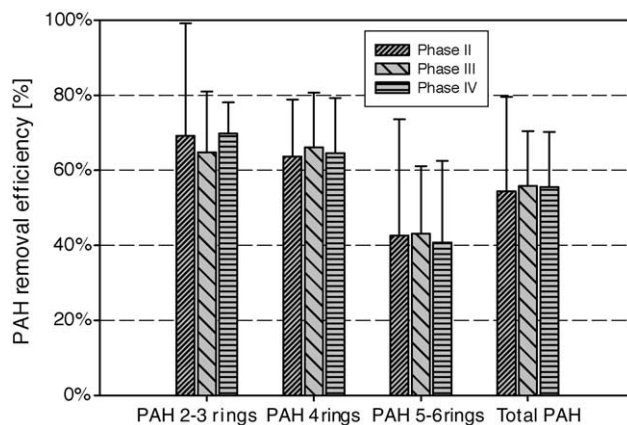


Fig. 7. PAH removal during Phases II, III and IV.

Table 4

PAH average concentrations in untreated and treated slurry during Phases II, III and IV (\pm standard deviation)

Phase	Slurry	PAH 2–3 rings (mg kg ⁻¹ TS)	PAH 4 rings (mg kg ⁻¹ TS)	PAH 5–6 rings (mg kg ⁻¹ TS)	PAH total (mg kg ⁻¹ TS)
II	Untreated	3.7 \pm 1.1	7.2 \pm 0.5	10.3 \pm 1.7	21.2 \pm .2
	Treated	1.1 \pm 1.2	2.6 \pm 1.1	5.9 \pm 3.2	9.6 \pm 5.3
III	Untreated	4.0 \pm 0.5	5.3 \pm 0.9	7.1 \pm 1.2	16.4 \pm 1.8
	Treated	1.4 \pm 0.6	1.8 \pm 0.8	4.0 \pm 1.3	7.2 \pm 2.4
IV	Untreated	4.0 \pm 0.5	5.3 \pm 0.9	7.1 \pm 1.2	16.4 \pm 1.8
	Treated	1.2 \pm 0.3	1.9 \pm 0.8	4.2 \pm 1.5	7.3 \pm 2.4

The OUR related to cycles of Phase II decreases from an initial value of 6–8 mgO₂ kg⁻¹ slurry h⁻¹ to a fairly constant value of about 1–1.5 mgO₂ kg⁻¹ slurry h⁻¹ (Fig. 8), assumed to be the endogenous respiration level (OUR_{end}).

These results indicated that the most exogenous activity was over after 2 days so the cycle length was shortened from 7 to 3.5 days.

3.4. Phase III

In Table 4 total PAH average values are summarised whereas in Fig. 9 PAH values versus time in the fed and treated effluent slurry from the SBR during the last 16 cycles of Phase III are plotted. As in previous cases, the values measured during cycles in the range 1–30 were not considered as they are not related to steady state conditions.

The PAH concentrations had just been reduced from 16.4 to 7.2 mg kg⁻¹ TS by means the microbial activity. The concentrations of PAHs with 2–3 rings in the fed and treated slurry were 4.0 and 1.4 mg kg⁻¹ TS, respectively, with an average removal efficiency of 65% (Fig. 7). For four rings PAHs, a removal efficiency of 66% was obtained, from 5.3 mg kg⁻¹ TS to about 2 mg kg⁻¹ TS. Lowest removal efficiency was observed for 5–6 rings PAHs, close to 43%, with concentrations from 7.1 mg kg⁻¹ TS to about 4 mg kg⁻¹ TS.

As in Phase II, a one order of magnitude CFU increase was observed and a corresponding 9% TOC_D decrease was measured.

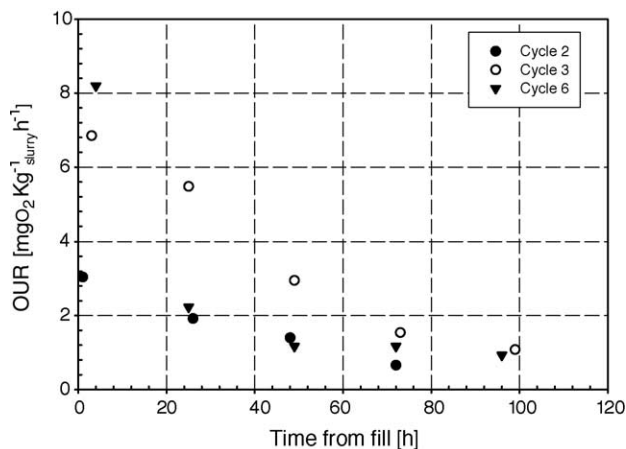


Fig. 8. OUR vs. time during three SBR cycles of Phase II.

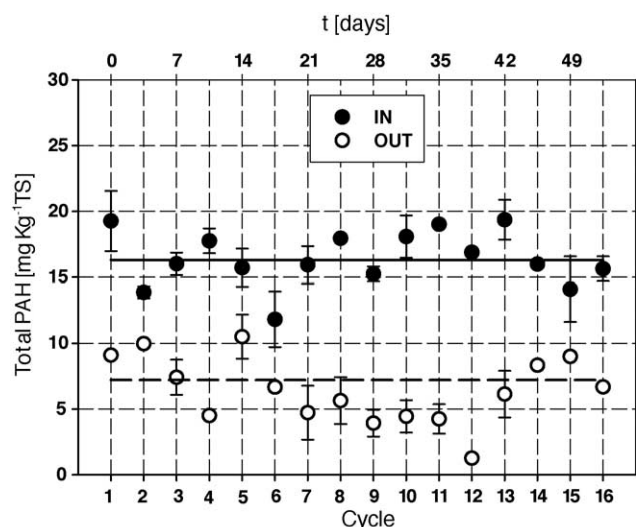


Fig. 9. Average and standard deviation of total PAH measured in treated and untreated slurry during Phase III.

In Fig. 10 the OUR_S values (ranging between 9 and 2 mgO₂ kg⁻¹ slurry h⁻¹, close to the Phase II values) and the fitted OUR curve are reported.

The parameters of relationship 2 (and the related 95% confidence levels) were estimated using the least square error criterion ($R^2 = 88.3\%$) to fit the OUR_S data, obtaining:

$$\text{OUR}_{\text{end}} = 1.9(1.4\text{--}2.4) \text{ mgO}_2 \text{ kg}_{\text{slurry}}^{-1} \text{ h}^{-1}$$

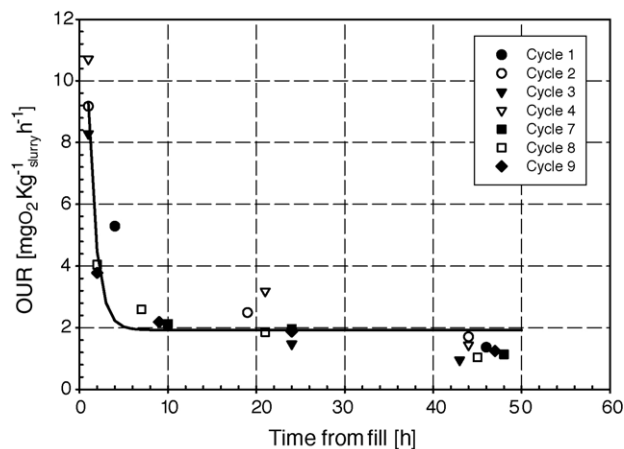


Fig. 10. OUR vs. time during seven SS-SBR cycles of Phase III.

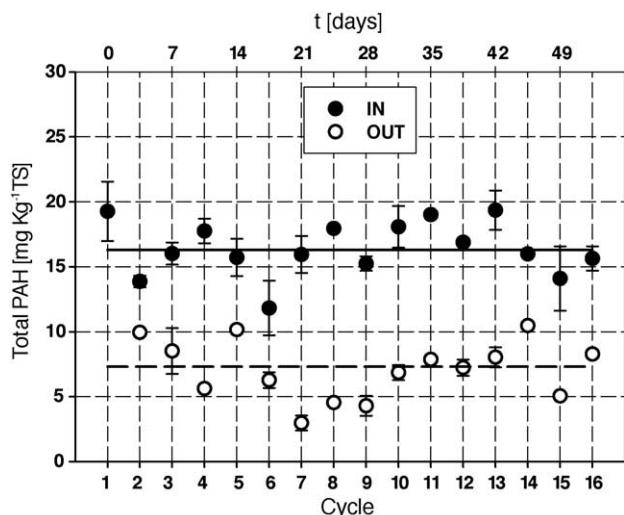


Fig. 11. Average and standard deviation of total PAH measured in treated and untreated slurry during Phase IV.

$$\text{OUR}_{\max} = 21.3(7.8\text{--}34.9) \text{ mgO}_2 \text{ kg}_{\text{slurry}}^{-1} \text{ h}^{-1}$$

$$k = 0.5(0.2\text{--}0.7) \text{ h}^{-1}$$

In terms of total PAH removal efficiency, a 56% value has been evaluated (Fig. 7). This result is close to those obtained during the Phase II, confirming that the length cycle shortening has no significant effect on PAHs removal because the endogenous conditions are reached before the end of the cycle length.

3.5. Phase IV

Fig. 11 shows PAHs versus time in the fed and treated slurry during the last 16 cycles of Phase IV. The total PAH concentrations had just been reduced from 16.4 to 7.3 mg kg⁻¹ TS, with a 56% removal efficiency close to those obtained in the previous phases: no significant enhancement of the reactor performances were observed, although the added lactose was biodegraded during each cycle.

In Fig. 7 the removal efficiencies observed for PAH with different rings are shown, whose values are equal to 70, 64 and 41% for 2–3 rings, 4 rings and 5–6 rings, respectively, whereas the related concentrations are reported in Table 4.

Once more, a CFU increase (one order of magnitude) and a TOC_D decrease were observed. The related concentrations are listed in Table 3.

OUR_S measured during Phase IV are shown in Fig. 12, they have been fitted by means of relationship 2, obtaining the following parameter values and related 95% confidence levels ($R^2 = 94.7\%$):

$$\text{OUR}_{\text{end}} = 4.7(4.0\text{--}5.5) \text{ mgO}_2 \text{ kg}_{\text{slurry}}^{-1} \text{ h}^{-1}$$

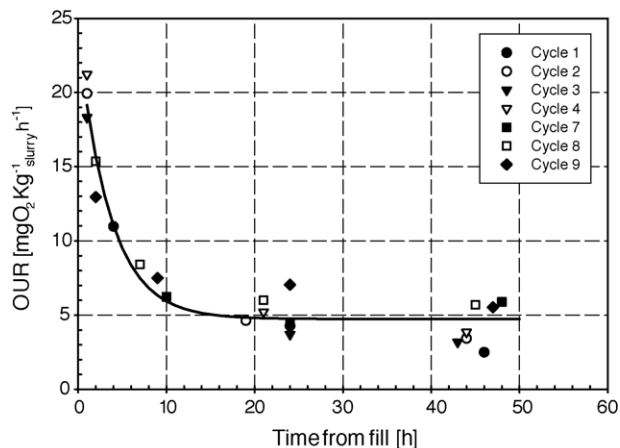


Fig. 12. OUR vs. time during seven SS-SBR cycles of Phase IV.

$$\text{OUR}_{\max} = 19.0(15.9\text{--}22.1) \text{ mgO}_2 \text{ kg}_{\text{slurry}}^{-1} \text{ h}^{-1}$$

$$k = 0.1(0.1\text{--}0.2) \text{ h}^{-1}$$

From the initial maximum value, the OUR decreased to a fairly constant value of about 5 mgO₂ kg⁻¹ h⁻¹, which was assumed to be the endogenous respiration level (OUR_{end}).

As expected, the OUR_{max} and OUR_{end} were higher than the values obtained in the previous phases since the lactose added to the SBR increased both the exogenous and endogenous oxygen uptake rates; moreover, lactose addition implies an increase of the time required to reach endogenous conditions, from about 10 h (measured in Phase III) to approximately 24 h in Phase IV.

4. Conclusions

This paper shows experimental tests carried out with a 81 lab-scale SBR loaded with PAHs contaminated lagoon sediments and operated in different conditions.

Oxygen uptake rate, measured by DO-stat titration technique, has enabled the monitoring of the in-reactor biological activity, providing also useful information in the selection of the reactor operating conditions. OUR profile, modelled as the sum of two terms, related, respectively, to exogenous and endogenous respiration, is useful for the aeration equipment design in order to avoid air pumping waste.

A total PAH efficiency removal close to 55% was achieved for long (98 days), middle (70 days) and short (35 days) HRT of the SBR; moreover, although the addition of lactose (external carbon source) in the SBR has increased the biological activity, as clearly shown by the OUR test results, no further improvement of the PAHs efficiency removal was observed.

The results show that the investigated biological treatment system can be pursued, with respect to the dredged sediment used, to meet the PAH standards required by the Italian law for their beneficial reuse as, e.g., building materials.

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