

## AnSBBR Applied to a Personal Care Industry Wastewater Treatment: Effects of Fill Time, Volume Treated Per Cycle, and Organic Load

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**Abstract** A study was performed regarding the effect of the relation between fill time, volume treated per cycle, and influent concentration at different applied organic loadings on the stability and efficiency of an anaerobic sequencing batch reactor containing immobilized biomass on polyurethane foam with recirculation of the liquid phase (AnSBBR) applied to the treatment of wastewater from a personal care industry. Total cycle length of the reactor was 8 h (480 min). Fill times were 10 min in the batch operation, 4 h in the fed-batch operation, and a 10-min batch followed by a 4-h fed batch in the mixed operation. Settling time was not necessary since the biomass was immobilized and decant time was 10 min. Volume of liquid medium in the reactor was 2.5 L, whereas volume treated per cycle ranged from 0.88 to 2.5 L in accordance with fill time. Influent concentration varied from 300 to 1,425 mgCOD/L, resulting in an applied volumetric organic load of 0.9 and 1.5 gCOD/L.d. Recirculation flow rate was 20 L/h, and the reactor was maintained at 30 °C. Values of organic matter removal efficiency of filtered effluent samples were below 71% in the batch operations and above 74% in the operations of fed batch followed by batch. Feeding wastewater during part of the operational cycle was beneficial to the system, as it resulted in indirect control over the conversion of substrate into intermediates that would negatively interfere with the biochemical reactions regarding the degradation of organic matter. As a result, the average substrate consumption increased, leading to higher organic removal efficiencies in the fed-batch operations.

**Keywords** AnSBBR · Personal care industry wastewater · Fill time · Feed volume · Organic load

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## Nomenclature

A	inner cross-section area of the reactor, $\text{cm}^2$
BA	bicarbonate alkalinity, $\text{mg CaCO}_3/\text{L}$
TVA	total volatile acids concentration, $\text{mg HAc/L}$
$C_I$	influent organic matter concentration, $\text{mg COD/L}$
$C_{EF}$	organic matter concentration of filtered effluent samples, $\text{mg COD/L}$
$C_{ET}$	organic matter concentration of unfiltered effluent samples, $\text{mg COD/L}$
$\text{CH}_4$	methane concentration, $\text{mmol/L}$
$\text{CO}_2$	carbon gas concentration, $\text{mmol/L}$
$C_S$	organic matter concentration of filtered samples, $\text{mg COD/L}$
$C_X$	biomass concentration in the reactor (solids per liquid volume treated per cycle), $\text{g TVS/L}$
$C'_X$	biomass concentration in the reactor (solids per mass of inert support), $\text{g TVS/g-foam}$
$\text{RSOL}_F$	removed specific organic load for filtered effluent samples, $\text{mg COD/g TVS.d}$
$\text{RSOL}_T$	removed specific organic load for unfiltered effluent samples, $\text{mg COD/g TVS.d}$
AVOL	applied volumetric organic load, $\text{mg COD/L.d}$
$\text{RVOL}_F$	removed volumetric organic load for filtered effluent samples, $\text{mg COD/L.d}$
$\text{RVOL}_T$	removed volumetric organic load for unfiltered effluent samples, $\text{mg COD/L.d}$
$M_{\text{TVS}}$	total mass of total volatile solids, $\text{g TVS}$
Q	recirculation flow rate, $\text{L/h}$
TSS	concentration of total suspended solids, $\text{mg/L}$
VSS	concentration of volatile suspended solids, $\text{mg/L}$
TS	concentration of total solids, $\text{mg/L}$
TVS	concentration of total volatile solids, $\text{mg/L}$
$t_B$	batch fill time, $\text{min}$
$t_{FB}$	fed-batch fill time, $\text{h}$
$t_C$	cycle length, $\text{h}$
$V_I$	total influent volume, $\text{L}$
$V_B$	batch influent volume, $\text{L}$
$V_{FB}$	fed-batch influent volume, $\text{L}$
$V_{RT}$	volume of liquid medium in the reactor, $\text{L}$
$V_{RS}$	volume of liquid medium in the reservoir, $\text{L}$
X	biomass amount in the reactor, $\text{g TVS}$
$v_S$	superficial velocity of the liquid medium, $\text{cm/s}$
$V_U$	volume of liquid medium in the system, $\text{L}$
$\varepsilon_F$	organic matter removal efficiency of filtered effluent samples, %
$\varepsilon_T$	organic matter removal efficiency of unfiltered effluent samples, %
$\phi$	bed porosity, %

## Introduction

Application of anaerobic sequencing reactors (ASBR) to the treatment of industrial wastewaters has been shown to be very interesting, especially when effluents are intermittently generated. Operational flexibility of these reactors allows removal of toxic and refractory compounds, which can be furthermore improved by altering feed strategy to fed batch. In this way, it is possible to maintain low concentrations of these

compounds in the reaction medium and, hence, reduce the inhibitory effects on substrate degradation. In addition, operating the reactor in fed-batch mode enables treatment of effluents containing elevated organic loads, absorbing peaks of volatile acids production that normally destabilize the process [14].

An alternative that has been investigated to improve the configuration of anaerobic sequencing batch reactors is the immobilization of biomass on polyurethane foam (AnSBBR), which increase solids retention and eliminates the settling step [9, 10]. The major advantage of this support, however, is the contact surface between the biomass and the liquid medium. Ratusznei et al. [9] proposed a configuration of a mechanically stirred anaerobic sequencing batch reactor containing immobilized biomass (AnSBBR) on polyurethane foam, with which they treated 0.5 L of synthetic wastewater based on sucrose, soluble starch, cellulose, meat extract, soy bean oil, and salts, with 485 mg COD/L, in 8-h cycles at 30 °C. In 10 days of operation, the system attained stability with organic matter removal of up to 86%.

With regard to agitation of the medium, an alternative is to implement recirculation of the liquid phase [2–5]. This system presents great operational flexibility because the volume to be treated may be increased by using a side reservoir. In this way, [3, 8] achieved increase in AnSBBR performance on treating synthetic medium based on glucose and sucrose with 500 mgCOD/L. Efficiency reached 95% as a consequence of the improved contact between substrate and biomass. The same justification was used by [7] to explain the gain in efficiency on treating hyper saline wastewater in the same type of reactor.

When the removal of toxic and refractory compounds is studied, it is necessary to assess the action of other factors that directly affect feed strategy to minimize the impact on the microbial community. Within this context, [1] investigated the ratio between the volume of wastewater fed per cycle ( $V_A$ ) and volume of liquid medium in the system ( $V_U$ ), treating synthetic medium based on sucrose, soluble starch, cellulose, meat extract, soy bean oil, and salt, with approximately 500 mgCOD/L. The results showed that, at  $V_A/V_U \geq 0.50$ , organic matter removal efficiency for filtered and unfiltered samples was 84% and 79%, respectively, whereas at higher dilutions of the influent, i.e., at  $V_A/V_U \leq 0.25$ , these efficiency values were lower, around 80% and 74%, respectively. The researchers also observed that, at higher dilutions, it became difficult to control the variation in volume of the AnSBBR due to the formation of extracellular polymer (ECP) likely of microbial origin.

Toxic compounds that have been investigated include anionic surfactants, commonly used in personal care, cosmetics, and pharmaceutical industries. Cserháti et al. [6] studied the biodegradability and environmental impact of these surfactants and concluded that they have an ambiguous role in the environment, since they may have toxic effects on living organisms and promote decomposition of organic pollutants and oxidation of inorganic compounds. According to the authors, there is still much research to be done, since the relation between chemical structure, physical-chemical properties, biodegradability, and impact caused by the surfactants has still not been elucidated.

Within this context, the focus of this work was to assess the application of an AnSBBR with liquid phase recirculation and immobilized biomass to the treatment of wastewater from a personal care industry. Therefore, a study was performed regarding the effect of the relation between fill time, volume fed per cycle and influent concentration at different applied organic loadings on the efficiency and stability of the reactor. Reactor behavior over the course of the operation cycle was also assessed to obtain useful information for scale-up.

## Materials and Methods

### Experimental Set-Up

The AnSBBR, schematized in Fig. 1, consisted of a three-compartment acrylic cylinder with outer diameter of 100 mm and wall thickness of 3.5 mm. Stainless steel 314 perforated sheet divided the 560-mm-high center compartment in five isometric stages to minimize bed compaction. A 35-mm-high compartment at the lower part of the reactor favored distribution of the wastewater, and a 65-mm-high one at the upper part functioned as biogas collecting chamber. The recirculation system consisted of a peristaltic pump and a 300-mm-high acrylic cylindrical side reservoir with the same diameter and wall thickness as the reactor. Flow rate was adjusted with the aid of a 100-ml graduated tube. Charge and discharge were carried out by diaphragm pumps, which, like in the recirculation operation, were turned on and off by an automatic timer system.

### Inoculum and Inert Support

The inoculum came from an Up-flow Anaerobic Sludge Blanket (UASB) reactor treating wastewater from a poultry slaughterhouse, with total solids and total volatile solids of 49 and 42 g/L, respectively. The material selected for immobilization of the inoculum [13] was polyurethane foam cut in 1.0-cm cubes with apparent density of 23 kg/m<sup>3</sup> and porosity close to 95%. Solids attached to the inert support were measured in each experimental condition according to methodology proposed by [10].

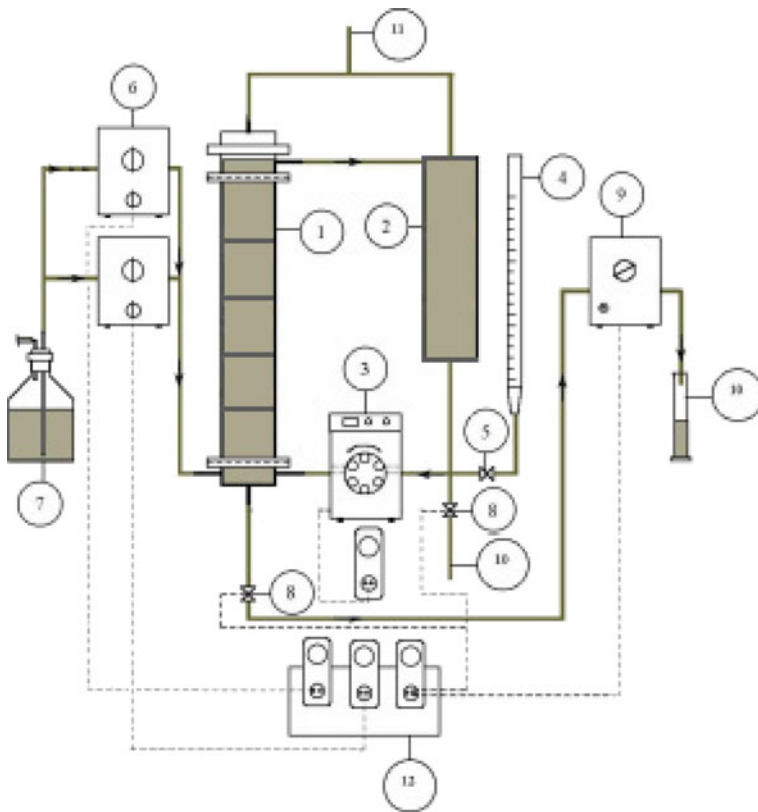
### Industrial Wastewater

The wastewater was prepared by diluting a commercial shampoo and sodium bicarbonate in tap water to simulate wash water from the reactors of a shampoo production unit of a personal care industry. Sodium bicarbonate was added to guarantee buffering of the system. Shampoo concentration varied as a function of applied volumetric organic load in each operational condition, whereas bicarbonate concentration was maintained at 200 mg/L. The amount of organic matter in the shampoo was experimentally estimated at 0.5 gCOD/g shampoo. The major organic constituent present was sodium lauryl ether sulfate (surfactant). Choice of shampoo was based on the brand of highest volume of production. Shampoo name, brand, and manufacturer have been preserved. Moreover, analyses of influent organic matter in filtered and unfiltered samples showed that 20±1% of the COD came from suspended solids.

### Experimental Protocol

The AnSBBR was operated under eight experimental conditions, in batch and fed-batch/batch mode with cycle lengths of 8 h (480 min). The distribution of this time was the following:

1. Feeding step: In the batch operations (B), the reactor was fed with a volume of approximately 0.88 or 2.50 L wastewater in 10 min at the beginning of the cycle, in accordance with the experimental condition. In the fed-batch/batch operations (FB/B), the reactor was fed with 1.62 L in 10 min and 0.88 L in 4 h, or simply 0.88 L in 4 h.



**Fig. 1** Experimental setup of the AnSBBR (adapted from [3]). Notation: 1, reactor containing immobilized biomass; 2, side reservoir; 3, circulation pump; 4, flow rate meter; 5, valve; 6, feed pump; 7, synthetic wastewater reservoir; 8, discharge valves; 9, discharge pump; 10, effluent outlet; 11, biogas outlet; 12, control unit; *solid lines*, hydraulic lines; *broken lines*, power lines

Regardless of operation mode—B or FB/B—under the conditions where volume wastewater fed was just 0.88 L, the volume of 2.50 L liquid medium was obtained by not discharging the 1.62 L of the previous cycle, i.e., there was a residual volume of the previous cycle.

2. Settling step: This step was not necessary since the biomass was immobilized in polyurethane foam (inert support).
3. Decant step: At the end of the cycle, the effluent was discharged in 10 min.
4. Reaction step: The reaction step was 460 min, which corresponds to the cycle time (480 min) minus the feeding batch step (10 min) and decant step (10 min). It should be mentioned that it was assumed that, during the fed-batch period, the reaction occurred.

Influent concentration varied between 300, 500, 850, and 1,425 mgCOD/L, maintaining applied volumetric organic load at 0.9 or 1.5 gCOD/L.d, as shown in Table 1. Total volume of liquid medium maintained in the reactor during each cycle was always 2.50 L. It should be pointed out that condition 1 was used as a stage to optimize alkalinity supplementation to the system.

**Table 1** Overview of the experimental conditions of the AnSBBR.

Condition	Feed strategy	$C_I$	AVOL	Batch		Fed-batch	
				$V_B$	$t_B$	$V_{FB}$	$t_{FB}$
1	B	500	1.5	2.5	10	—	—
2	B	1,425	1.5	0.88	10	—	—
3	B	300	0.9	2.5	10	—	—
4	B	850	0.9	0.88	10	—	—
5	FB/B	500	1.5	1.62	10	0.88	4
6	FB/B	1,425	1.5	—	—	0.88	4
7	FB/B	300	0.9	1.62	10	0.88	4
8	FB/B	850	0.9	—	—	0.88	4

Units:  $[C_I]$ =mg COD/L;  $[AVOL]$ =g COD/L.d;  $[V_B]=[V_{FB}]=L$ ;  $[t_B]=min$ ;  $[t_{FB}]=h$

Reaction took place in 459 min, with a recirculation flow rate of 20 L/h [8], sufficient to make mixing time negligible in relation to cycle length [4]. Considering bed porosity around 40% [8], the adopted recirculation flow rate corresponded to an ascending superficial velocity of 0.20 cm/s. Equation 1 shows the calculation of the velocity, where  $Q$  is the recirculation flow rate,  $\phi$  the bed porosity, and  $A$  the inner cross-sectional area of the reactor.

$$v_s = \frac{Q}{\phi \cdot A} \quad (1)$$

The experimental setup, including the reactor, was maintained in an isothermal chamber at  $30 \pm 1$  °C.

### Physical-Chemical Analysis

Physical-chemical analyses of the influent and effluent of the AnSBBR were performed in accordance with [11], regarding the following variables: influent organic matter concentration ( $C_I$ ) and organic matter concentrations of filtered ( $C_{EF}$ ) and unfiltered ( $C_{ET}$ ) effluent samples, expressed in chemical oxygen demand (COD); pH; total volatile acids concentration (TVA); partial alkalinity (PA), intermediate alkalinity (IA), and bicarbonate alkalinity (BA); concentration of total solids (TS), total volatile solids (TVS), total suspended solids (TSS), and volatile suspended solids (VSS); and volume of liquid medium in the reactor ( $V_{RT}$ ) of the reservoir ( $V_{RS}$ ) and total volume ( $V_U$ ).

Organic matter removal efficiency of the system was defined by the relation between the removed amount and applied amount, considering filtered ( $\varepsilon_F$ ) and unfiltered samples ( $\varepsilon_T$ ) of the effluent, as suggested by Eqs. 2 and 3.

$$\varepsilon_F = \frac{C_I - C_{EF}}{C_I} \quad (2)$$

$$\varepsilon_T = \frac{C_I - C_{ET}}{C_I} \quad (3)$$

The applied volumetric organic load (AVOL) was defined as the amount of organic matter fed to the system per unit time and per unit volume, according to Eq. 4. In this equation,  $V_I$  is the total influent volume and  $t_C$  is the cycle length.

$$AVOL = \frac{C_I \cdot V_I}{V_U \cdot t_C} \quad (4)$$

On the other hand, the removed volumetric organic load was defined as the amount of organic matter removed from the system per unit time and per unit volume, considering filtered (RVOL<sub>F</sub>) and unfiltered (RVOL<sub>T</sub>) effluent samples, as shown in Eqs. 5 and 6.

$$RVOL_F = \frac{(C_I - C_{EF}) \cdot V_I}{V_U \cdot t_C} \quad (5)$$

$$RVOL_T = \frac{(C_I - C_{ET}) \cdot V_I}{V_U \cdot t_C} \quad (6)$$

The removed specific organic load was defined as the amount of organic matter removed from the system per unit time and per unit mass of microorganisms, considering filtered (RSOL<sub>F</sub>) and unfiltered (RSOL<sub>T</sub>) effluent samples, according to Eqs. 7 and 8. In these equations,  $M_{TVS}$  is the mass of microorganisms, expressed as mass of total volatile solids.

$$RSOL_F = \frac{(C_I - C_{EF}) \cdot V_I}{M_{TVS} \cdot t_C} \quad (7)$$

$$RSOL_T = \frac{(C_I - C_{ET}) \cdot V_I}{M_{TVS} \cdot t_C} \quad (8)$$

After stabilization of the system, verified by approximately constant values of the monitored effluent variables, profiles were run over the course of an operational cycle of the following variables: organic matter concentration of filtered samples ( $C_S$ ), total volatile

**Table 2** Monitored variables of AnSBBR at conditions 1 to 8.

Condition	1	2	3	4	5	6	7	8
CI (mg COD/L)	509 (6)	1,382 (18)	306 (18)	860 (14)	507 (17)	1,298 (17)	298 (16)	921 (16)
CET (mg COD/L)	226 (6)	690 (15)	98 (11)	306 (9)	144 (15)	311 (14)	80 (14)	163 (11)
εT (%)	56	50	68	64	72	76	73	82
CEF (mg COD/L)	205 (6)	630 (15)	89 (11)	259 (9)	129 (15)	257 (14)	75 (14)	134 (11)
εF (%)	60	54	71	70	74	80	75	85
RVOLF (g COD/L.d)	0.91	0.79	0.65	0.62	1.13	1.22	0.67	0.8
RSOLF (g COD/g TVS.d)	0.05	0.04	0.03	0.03	0.06	0.06	0.03	0.04

Subscripts: I, influent; E, effluent; F, filtered effluent sample; T, unfiltered effluent sample; ( ) number of samples used for averaging. Monitored variables:  $V_{RT}=(1,707\pm32)$  L;  $V_{RS}=(791\pm38)$  L;  $V_U=(2,498\pm70)$  L;  $pH_I=7.9\pm0.1$ ;  $pH_E=7.6\pm0.1$ ;  $TVA_I=(19\pm2)$  mg HAc/L;  $TVA_E=(19\pm2)$  mg HAc/L;  $BA_I=(211\pm6)$  mg  $CaCO_3$ /L;  $BA_E=(252\pm7)$  mg  $CaCO_3$ /L;  $TSS_I=(76\pm5)$  mg/L;  $TSS_E=(24\pm3)$  mg/L;  $VSS_I=(63\pm4)$  mg/L;  $VSS_E=(19\pm3)$  mg/L.  $C'_X=(0.71\pm0.04)$  gTVS/g-foam;  $X=(50.5\pm2.8)$  g TVS;  $C_X=(20.2\pm1.1)$  g TVS/L

acids concentration (TVA), and methane ( $\text{CH}_4$ ) and carbon gas ( $\text{CO}_2$ ) concentrations. Samples were collected at time intervals of 30 to 180 min, in a way that the total removed volume did not exceed 10% of the volume of liquid medium in the reactor.  $\text{CH}_4$  and  $\text{CO}_2$  profiles were obtained by gas chromatography using an HP® 6890 Series chromatograph equipped with thermal conductivity detector and Porapack Q, 80/100 Mesh, 6 ft×0.125 in column. The reason for running these profiles was to enable better comprehension of the organic matter degradation routes over the course of an operational cycle.

### Microbiological Analysis

The inoculum and biomass of the reactor were submitted to microbiological analysis employing common optical and optical fluorescence phase contrast microscopy, according to the method proposed by [12], utilizing a BX-41 Olympus® equipped with digital Optronics system and image acquisition by Image Pro-Plus version 4.5.0. The analysis revealed the existence of Methanosarcina-like morphologies, confirming the methanogenic character of the sludge used.

## Results and Discussion

The AnSBBR operated 25 days (75 cycles) for each experimental condition and attained stability during the first days of operation. Monitoring results and profiles are shown in Table 2 and Figs. 2, 3, 4, 5, 6, 7, 8, and 9.

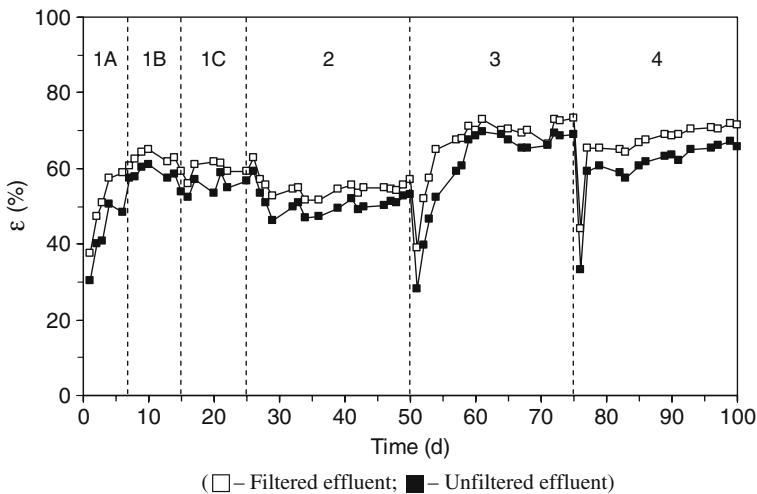
Solids analysis showed concentration of 0.71 gTVS/g foam (solids inside the reactor per mass of inert support) that corresponds to 50.5 gTVS inside the reactor for a foam amount of 71.13 g. The biomass concentration was 20.2 gTVS/L (solids per liquid volume inside the reactor) for a liquid medium volume of 2.5 L inside the reactor in each cycle.

Solids retention time (SRT) was not measured since the presence of inert support prevented loss of biomass during the experimental period. It should be mentioned that solids analysis inside the reactor measured by the biomass attached in the foam presented stable values, i.e., there was no loss of biomass or cell growth during the experimental conditions performed in this work.

### Optimization of Alkalinity Supplementation

To optimize alkalinity supplementation, condition 1 was divided into three stages, namely, conditions 1A, 1B, and 1C, as shown in Fig. 2. Under condition 1A, the system operated for 7 days and treated wastewater with 500 mgCOD/L and sodium bicarbonate concentrations decreasing gradually from 800 to 100 mg/L. Bicarbonate concentration was reduced because of the low TVA, which was less than 20 mgHAc/L, obtained on monitoring the influent and effluent at this stage. On the other hand, average organic matter removal efficiency of filtered effluent samples was 52%, due not only to the variation in sodium bicarbonate concentration, but also to the adaptation of the biomass to the wastewater, as this was the first experimental condition of the AnSBBR. Under condition 1B, the system operated without bicarbonate for 8 days and presented an efficiency of 63%. However, alkalinity started dropping gradually, reaching 46.8 mg $\text{CaCO}_3$ /L, a very low value compared to the average monitored value. So, supplementation was resumed and a concentration of 200 mg/L was set for the other experimental conditions of the project, starting with condition 1C, where the system operated for 10 days. Condition 1C was also the stage chosen for running the profiles of condition 1.



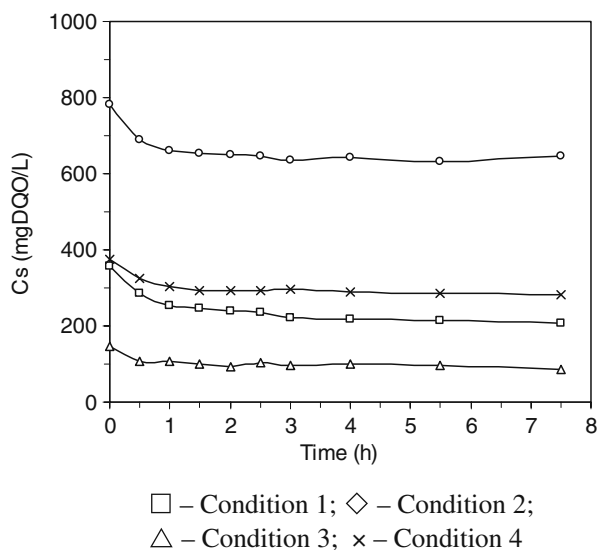


**Fig. 2** Organic matter removal efficiency at conditions 1 to 4 (open squares, filtered effluent; closed squares, unfiltered effluent)

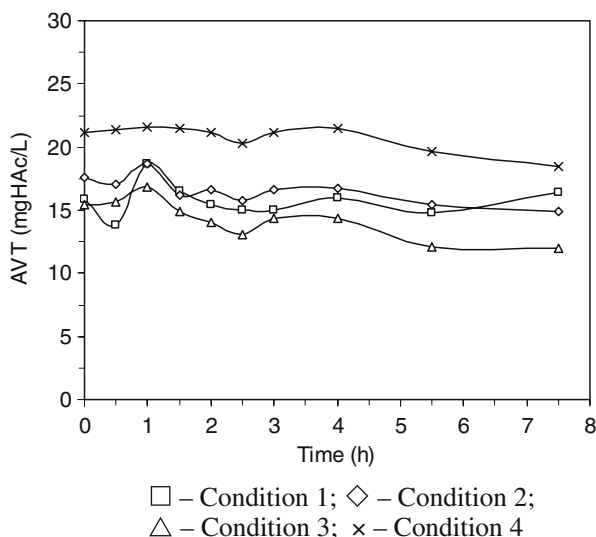
### Batch Operations

Figure 2 contains data on organic matter removal efficiency of filtered and unfiltered effluent samples obtained over the course of conditions 1 to 4. Sudden drops can be seen in the transition from condition 2 to 3, and from 3 to 4, caused not only by the change in AVOL, but also by decompacting the AnSBBR bed, which was done manually always before initiating each experimental condition. This procedure may cause detachment of the biomass for some cycles, increasing momentarily the concentration of organic matter in the effluent. On the other hand, the low efficiency values encountered at the start of condition 1 are rather due to adaptation of the biomass to the wastewater and to the system as a whole.

**Fig. 3**  $C_S$  profile at conditions 1 to 4 (squares, condition 1; diamonds, condition 2; triangles, condition 3; crosses, condition 4)



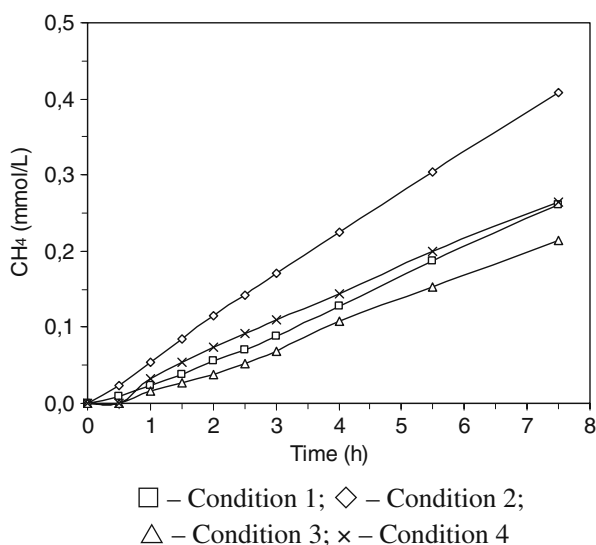
**Fig. 4** TVA profile at conditions 1 to 4 (*squares*, condition 1; *diamonds*, condition 2; *triangles*, condition 3; *crosses*, condition 4)

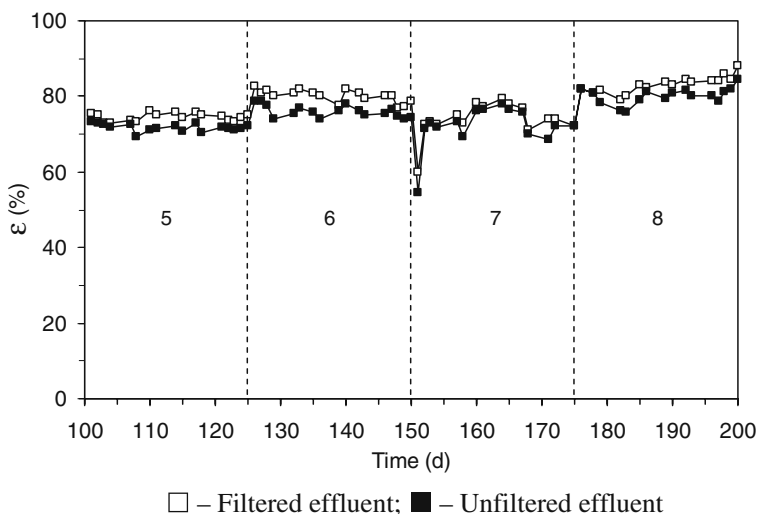


In addition to organic matter removal efficiency, there are other variables that can be assessed and used to corroborate the hypothesis of system stability during a certain experimental condition, such as pH, TVA, and BA. Analysis of the monitoring results for conditions 1 to 4, listed in Table 2, shows that pH remained between 7.5 and 8.0, and TVA did not exceed 30 mgHAc/L. Generation of alkalinity and solids retention were also observed with similar results for conditions 5 to 8.

Comparing condition 1 with 3, and 2 with 4, regarding the monitoring data listed in Table 2, the conditions with higher AVOL, i.e., conditions 1 and 2, also presented higher RVOL and RSOL. This suggests that the same biomass, maintained in the AnSBBR over the course of the experimental conditions, manages to remove larger amounts of organic

**Fig. 5** CH<sub>4</sub> profile at conditions 1 to 4 (*squares*, condition 1; *diamonds*, condition 2; *triangles*, condition 3; *crosses*, condition 4)



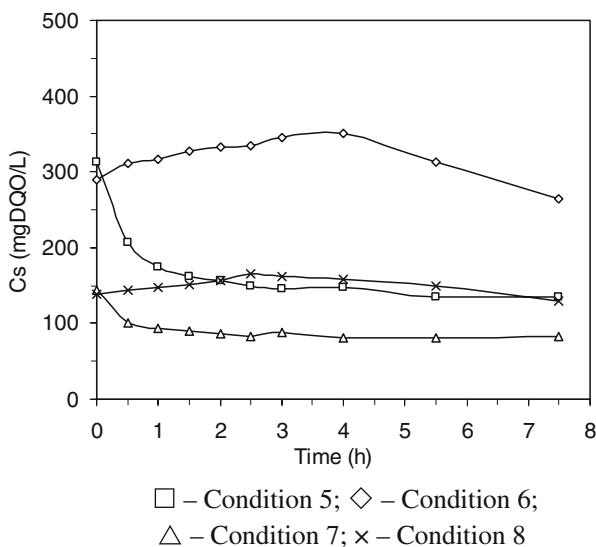


**Fig. 6** Organic matter removal efficiency at conditions 5 to 8 (*open squares*, filtered effluent; *closed squares*, unfiltered effluent)

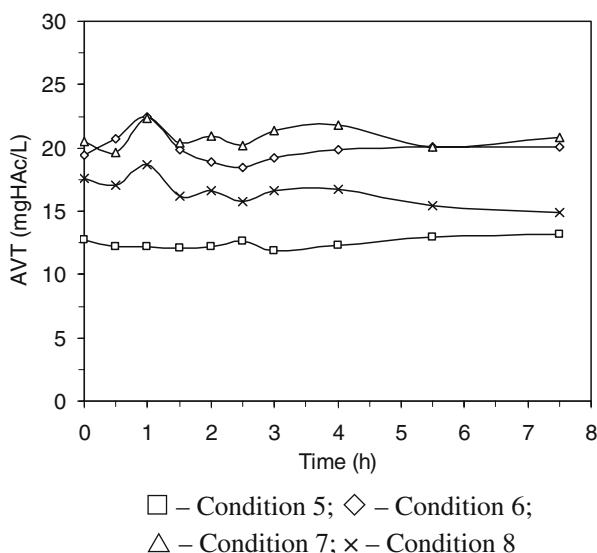
matter as long as substrate is available. However, the highest efficiencies were encountered precisely at conditions 3 and 4, where AVOL values were lower.

The explanation for this is related to the biodegradability of the different constituents of the shampoo formulation used in the preparation of the wastewater. In the presence of these compounds, it is assumed that the biomass consumed the readily degradable more rapidly than the poorly degradable ones. In this way, at the conditions where AVOL was higher, and consequently, the amount of readily degradable compounds was also higher, the biomass managed to remove, in absolute terms, a higher amount of organic matter. The

**Fig. 7**  $C_s$  profile at conditions 5 to 8 (*squares*, condition 5; *diamonds*, condition 6; *triangles*, condition 7; *crosses*, condition 8)



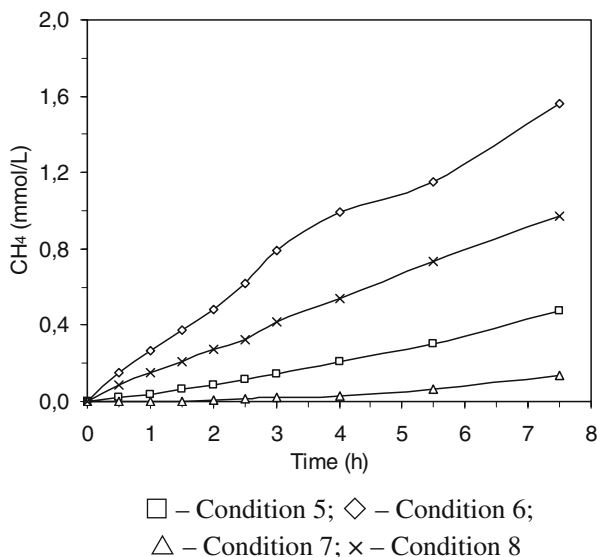
**Fig. 8** TVA profile at conditions 5 to 8 (*squares*, condition 5; *diamonds*, condition 6; *triangles*, condition 7; *crosses*, condition 8)



consumption of poorly degradable compounds occurs concomitantly, but at a lower rate. So, at the end of the cycle, the readily degradable compounds had almost already been fully converted to methane, whereas the poorly degradable ones were mostly partially degraded and distributed throughout the liquid phase.

On the other hand, at the conditions where AVOL was lower, the consumption rates of the easily and poorly degradable compounds should also be lower, due to their low concentration in the reaction medium. Even so, the biomass was able to degrade a proportionally higher amount of these compounds, revealing that the average consumption rate was higher at conditions 3 and 4 compared to conditions 1 and 2. Therefore, although

**Fig. 9** CH<sub>4</sub> profile at conditions 5 to 8 (*squares*, condition 5; *diamonds*, condition 6; *triangles*, condition 7; *crosses*, condition 8)



the average degradation rate of organic matter was higher at the conditions of higher AVOL, when the substrate concentration at the beginning of the cycles was sufficiently high to guarantee high values of RVOL and RSOL, the values were proportionally higher at conditions of lower AVOL, which enabled achieving higher efficiencies. Very likely, the intermediate products from the organic compounds, mainly the poorly degradable ones, like sodium lauryl ether sulfate, interfered directly with the biochemical reactions regarding the conversion of organic matter to methane.

Comparing condition 1 with 2, and 3 with 4, shows that the conditions where a residual volume was used to constitute part of the liquid medium, i.e., conditions 2 and 4, presented lower efficiency, RVOL, and RSOL values. Incorporating part of the organic matter of the previous cycle, deriving from the partial degradation of the substrate, as is the case of the intermediate products, in addition to increasing applied organic load interfered with the average consumption rate of the substrate, reducing RVOL, RSOL, and efficiency values. In practice, the difference between these values is seen to be more significant at the experimental conditions of 1.5 gCOD/L.d than at those of 0.9 gCOD/L.d.

Figure 3 contains the results of the  $C_s$  profiles at conditions 1 to 4. The conversion of organic matter to methane seems to have been interrupted 2 h after the beginning of the cycle at conditions 2, 3, and 4, and 3 h at condition 1. However, this is due to a limitation of the method used to quantify the organic matter in the reaction medium, which does not take into account the biochemical transformation that occurred during the cycle, recording only the fraction converted into methane. Therefore, the variation in organic matter observed at the beginning of the cycles of these conditions is rather related to the consumption of easily biodegradable compounds, which were rapidly converted to methane. The remaining period, where variation was almost imperceptible, may be attributed to the degradation of poorly degradable compounds, which were still processed and converted to methane and to other short carbon chain compounds but at much lower rates.

Figures 4 and 5 contain results of TVA and  $CH_4$  profiles, respectively. The TVA curves showed similar and stable behavior, with no peaks. Values obtained were below 30 mg HAC/L. On the other hand, the continually increasing behavior of the  $CH_4$  curves reinforces the idea that biochemical reactions kept occurring during the operational cycle, as previously discussed. On the other hand, the highest values of methane concentration, which should have been encountered in the profiles of conditions 1, 2, 3, and 4, in this order, turned out to present a difference sequence. This can be justified by the low methane concentrations, which also resulted in low biogas production.

### Fed-Batch Followed by Batch Operations

Figure 6 contains data regarding organic matter removal efficiency of filtered and unfiltered effluent samples during conditions 5 to 8. It can be seen that the system quickly attained stability under all experimental conditions, suggesting that changing feed strategy from batch to fed batch followed by batch improved system response to perturbations caused by alterations in AVOL and by decompaction of the reactor bed. It can be seen, for instance, that the drop in efficiency during transition of conditions 6 and 7, caused by any of the above-mentioned factors, was absorbed.

Comparing condition 5 with 7, and 6 with 8, taking into account the results listed in Table 2, shows that the conditions with higher AVOL, i.e., conditions 5 and 6, presented higher values of RVOL and RSOL, but also lower efficiencies. Again, the explanation for this is related to the biodegradability of the organic matter present in the wastewater.

Under conditions 5 and 6, the consumption of poorly and, mainly, of easily biodegradable compounds was higher, due to their higher concentration in the reaction medium. However, under conditions 7 and 8, where the amount of these compounds was lower, the biomass managed to degrade a proportionally higher fraction, resulting in an increase in process efficiency. Again, the average substrate consumption rate was proportionally higher at the conditions of lower AVOL, evidencing interference with the reaction mechanism of the intermediate products from the organic matter degradation.

Comparison of condition 5 with 6, and 7 with 8, shows that the conditions at which residual volume was used to constitute part of the liquid medium of the subsequent cycle, i.e., conditions 6 and 8, also presented higher efficiency, RVOL, and RSOL values. Dilution of the wastewater by the residual volume, associated with the fed-batch operation, maintained low substrate concentration levels in the reaction medium and, consequently, low degradation rates of organic matter, especially at the beginning of the operational cycles. On the other hand, at conditions 5 and 7, where no residual volume was used, the increase in degradation rate was not very pronounced because the starting volume contained a large amount of substrate, considering that part of the wastewater was fed in 10 min right after the discharge of the previous cycle. However, once more, it was observed that the presence of a large amount of substrate at the beginning of the cycle interferes with the attainment of a higher organic matter degradation rate because of the generation of intermediate products, of which biodegradation is surely complex and slow.

Figure 7 contains the results of the  $C_S$  profiles of conditions 5 to 8. Two types of behavior can be observed from the data, typical of batch and fed-batch operations. At conditions 5 and 7, the profiles decrease over the course of the whole operational cycle, revealing that the substrate consumption rate was higher than the specific flow rate of the substrate when entering the system. Since at these two conditions the reactor had been previously fed with wastewater, the substrate concentration values managed to attain a sufficiently high level to maintain the consumption rate above the inlet specific flow rate, from the beginning of the cycle. On the other hand, the profiles obtained at conditions 6 and 8 show an increase during part of the cycle and a decrease during the other part. In the first part of the cycle, the specific flow rate of the substrate entering the system was higher than the substrate consumption rate because, right at the beginning of the cycle, the organic matter concentration in the reaction medium was very low and corresponded to the residual concentration of the previous cycle. However, the substrate concentration started increasing gradually because the feeding and the consumption rates increased proportionally. The second part of the profile, with decreasing behavior, only started when the substrate consumption rate managed to overcome the specific inlet flow rate, as in condition 8, or simply when feeding was interrupted, as suggested by the profile of condition 6.

Figure 8 contains results of TVA profiles. It can be seen that there was no accumulation of volatile acids during the operational cycles, and TVA always remained below 30 mgHAc/L. This result suggests that the wastewater did not consist only of easily biodegradable compounds; otherwise, the volatile acids would accumulate at some point of the curve. One should also take into account that the shampoo used to prepare the wastewater consisted of several compounds with their respective biodegradation times.

Figure 9 contains the  $CH_4$  profiles of conditions 5 to 8, of which the results were somewhat more expressive than those of conditions 1 to 4. However, the concentrations obtained were also relatively low, confirming the negative interference of the intermediates with the organic matter conversion to methane.

## Comparative Study of the Experimental Conditions

On comparing conditions 1 to 4 with conditions 5 to 8 the last four operated in fed-batch/batch mode are seen to have higher efficiency, RVOL, and RSOL than the first four operated only in the batch mode. Feeding the wastewater in fed-batch mode divided the supply of substrate during the first hours of the cycle, minimizing the interference caused by the accumulation of intermediates in relation to the biochemical transformations that occurred in the reaction medium. It was thus possible to obtain higher average substrate consumption, which resulted in the conversion of a higher fraction of intermediates. The surfactant sodium lauryl ether sulfate is likely a precursor of these compounds, not only because of its chemical properties, but also because it is the major constituent in the shampoo formulation.

Another point that should be clarified is the use of a residual volume which at conditions 1 to 4 caused a decrease in organic matter removal efficiency, in contrast to conditions 5 to 8, where efficiency increased. Indeed, the residual volume alone does not benefit the system because it is just a volume of reaction medium containing partially degraded organic matter. On the other hand, this same volume manages to dilute the fed wastewater, reducing the substrate concentration in the medium and, consequently, minimizing the effects of accumulation of the intermediates that hindered biodegradation of the organic matter. Utilization of the residual volume should, therefore, be interpreted as only part of an adopted feed strategy. Since at conditions 1 to 4 the AnSBBR was operated batchwise, the residual volume contributed to the increase in AVOL, reducing efficiency, RVOL, and RSOL. At conditions 5 to 8, where operation was fed-batch/batch, the dilution caused by the residual volume resulted in the increase in average reaction rate in the system, yielding higher values of RVOL, RSOL, and efficiency.

## Conclusions

The AnSBBR, with recirculation of the liquid phase and containing immobilized biomass on polyurethane foam, attained stability at the investigated operational conditions and showed satisfactory results in the treatment of a wastewater simulating an effluent of a personal care industry. A study to optimize alkalinity supplementation showed that the wastewater would not be able to provide the demand for biomass under the implemented experimental conditions if no other compound were added to this end. Addition of 200 mg/L sodium bicarbonate seemed to be sufficient to guarantee a surplus of alkalinity since, under some conditions, the reactor managed to maintain stability even without correction of the bicarbonate volumetric load.

The AnSBBR presented higher values of efficiency, RVOL, and RSOL when operated under fed-batch/batch feeding compared to the batch operations because of increase in average reaction rate, avoiding accumulation of poorly degradable intermediates in the reaction medium. In this way, the biomass managed to convert a higher fraction of organic matter to methane, including the intermediates and their precursors. In addition, utilization of a residual volume from a previous cycle as part of the reaction medium of the following cycle showed to be advantageous only for the fed-batch/batch conditions since, unlike in the batch operations, the effect of dilution prevails over the effect of organic matter accumulation.

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