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Comparing aerobic granular sludge and flocculent sequencing batch reactor technologies for textile wastewater treatment

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ABSTRACT

Textile wastewaters are rated among the most polluting in all industrial sectors, including high organic loads and dyes that are generally resistant to aerobic biotreatment. Alternative, staged anaerobic/aerobic regimes have been tested, with clear success in the biodecolorization of the widely used azo dyes. The novel aerobic granular sludge (AGS) technology has excellent potential in this context, due to the anoxic/anaerobic zones within granules and their increased tolerance to toxicity. In this study the performance of anaerobic/aerobic, flocculent and AGS sequencing batch reactors (SBR) was compared in the treatment of a simulated textile wastewater. Similar color removal yields (75–80%) of an azo dye were attained in both systems but with higher anaerobic and overall COD removal yields in the AGS SBR, thus demonstrating the latter's good potential. Comparison of the toxicity of samples collected from both SBR using yeast-based assays highlighted the better performance of the AGS SBR as compared with the flocculent SBR, with respect to detoxification potential.

1. Introduction

Textile wastewaters are rated among the most polluting of all industrial sectors, both in terms of discharged volumes and composition, but their most important environmental problems arise from the high organic loads and the presence of color [1]. Azo dyes are the most common synthetic colorants released in textile wastewaters [2] due to their ease of synthesis, stability and variety of colors. Due to the electron-withdrawing nature of azo bonds, azo dyes are easily reduced by bacteria under anaerobic conditions, resulting in color removal with the formation of aromatic amines [3]. Key data on the health hazard associated with the majority of these aromatic amines are limited [4], but some of these azo dye breakdown products have been considered of higher concern than the original dye with respect to their possible (eco) toxicity and/or mutagenicity/carcinogenicity [5,6]. Although these amines are generally not further degraded anaerobically, their potential for aerobic biodegradation has been demonstrated for simple molecular structures [7]. Thus, staged anaerobic/aerobic sequencing batch reactor (SBR) systems using floc-forming biomass have been proposed for complete azo dye biodegradation, including decolorization and further aromatic amine mineralization [3,7,8]. However, the optimization of flocculent sludge technology is hampered by intrinsic operational features, such as poor settling characteristics that require a large footprint and compromise the treatment efficiency [9]. Thus, its replacement is regarded as inevitable in the near future.

In light of the environmental problems raised by textile industry wastewater and of the limitations of the treatment processes currently used, there is an urgent need for effective, environmentally friendly and economically attractive technologies for textile wastewater treatment. In this context, the novel aerobic granular sludge (AGS) technology is a promising bioprocess for textile wastewater treatment. The formation and use of AGS began to be reported in the late 1990s [10] and has recently been applied with success in domestic full-scale wastewater treatment plants (WWTP) with significant reductions in footprint and energy consumption, being pointed as the next generation of wastewater treatment technologies [11,12].

In addition to the general advantages of the staged SBR technology, AGS systems present several unique attributes [13]: excellent settling properties, allowing shorter settling times for good solid–liquid separation and requiring lower construction

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area; good biomass retention, allowing higher concentration in the SBR and consequently lower reaction time and/or reactor volume; ability to withstand toxicity and high organic loading rates, making them attractive for industrial wastewater treatment applications; aerobic and anoxic/anaerobic zones within the granules, allowing organic matter, nitrogen and phosphorus removal in the same system and potentially contributing to the process of azo dye mineralization. Given the particular attributes of aerobic granules, this compact technology has a great potential for the treatment of the highly variable textile wastewaters, including the biodecolorization of textile dyes that are generally resistant to aerobic biodegradation, like azo dyes.

Despite the increasing reported applications of AGS for municipal and industrial wastewater, textile wastewater treatment with this technology has scarcely been reported [14]. Thus, the objective of this work was to assess the applicability of the novel AGS technology in SBR as an alternative to the conventional flocculent activated sludge SBR technology for dye-laden textile wastewater treatment. For that, the performance of two anaerobic/aerobic SBR systems, one with activated sludge flocs and the other with aerobic granules, was evaluated in terms of chemical oxygen demand (COD) and color removal efficiencies in the treatment of a simulated textile wastewater. To our knowledge, this is the first time that such a comparison is performed. In parallel to color and COD removal profiles, the potential detoxification of the simulated textile wastewater during SBR operation was also examined by using yeast-based assays [15,16] with the eukaryotic model *Saccharomyces cerevisiae*. This is a simple, animal-alternative and relatively inexpensive experimental test system that can provide a fast preview of the potential toxicity of chemicals/effluents meaningful for other eukaryotes [15–17].

2. Materials and methods

2.1. Bioreactor setup and operation

2.1.1. Bioreactors and inocula

The experimental system was composed of four anaerobic/aerobic SBR with cycles consisting of five discrete sequential phases, namely fill, reaction, settle, drain and idle. FSBR1 and FSBR2 (1.0-L working volume) were inoculated with activated sludge flocs harvested from a full-scale, conventional municipal WWTP (Chelas, Lisboa, Portugal). GSBR1 and GSBR2 (1.5-L working volume) were inoculated with aerobic granules harvested from the Nereda® demonstration SBR at the Frielas WWTP (Portugal).

2.1.2. Simulated textile wastewater

The synthetic textile wastewater used as feed solution (pH 6.9–7.0) was prepared with a starch-based sizing agent (Emsize E1, Emsland-Stärke GmbH, Germany) as carbon source (1000 mg O_2 L^{-1} as COD), pre-hydrolyzed by a procedure based on the oxidative desizing conditions described by the manufacturer [18], supplemented with phosphorus and nitrogen salts and other macro- and micro-nutrients to the following concentrations: 2310 mg L^{-1} $Na_2HPO_4{\cdot}12H_2O$, 762 mg L^{-1} KH_2PO_4, 143 mg L^{-1} NH_4Cl, 22.5 mg L^{-1} $MgSO_4{\cdot}7H_2O$, 27.5 mg L^{-1} $CaCl_2$, 250 μg L^{-1} $FeCl_3{\cdot}6H_2O$, 40 μg L^{-1} $MnSO_4{\cdot}4H_2O$, 57 μg L^{-1} H_3BO_3, 43 μg L^{-1} $ZnSO_4{\cdot}7H_2O$ and 35 μg L^{-1} $(NH_4)_6Mo_7O_{24}{\cdot}4H_2O$. Acid Red 14 (AR14) was used as a model azo dye and was added directly to FSBR1 and GSBR1, as a concentrated solution in deionized water (3 g L^{-1}), after the end of the fill phase (20 mg L^{-1} at the onset of the reaction phase), FSBR2 and GSBR2 were used as dye-free controls.

2.1.3. SBR cycle operation

FSBR1 and FSBR2 were run in 8-h cycles with a hydraulic retention time (HRT) of 16 h and a sludge retention time (SRT) of 15 days imposed through daily biomass purge. GSBR1 and GSBR2 worked in 6-h cycles with a HRT of 12 h and no imposed SRT (deliberate biomass wastage was limited to the sampling needs). The feed solution was inserted at the bottom of the bioreactors with a volumetric exchange ratio of 50%. At the end of the fill phase, FSBR1 and GSBR1 were additionally supplied with AR14 at the top of the bioreactors. The reaction phase of each SBR cycle included a mixed anaerobic stage followed by an aerated stage. Mixing was provided by magnetic stirring during the non-aerated reaction. During the aerobic reaction, aeration was supplied by air compressors through porous membrane diffusers. The different cycle phase durations tested in the flocculent and granular bioreactors are summarized in Table 1. Pumping, mixing and aeration were automatically controlled via an interface by a computer with dedicated software.

Table 1
Operational cycle conditions of the flocculent (FSBR) and of the granular (GSBR) bioreactors.

	Idle and fill (min)	Mixed reaction (h)	Aerated reaction (h)	Settle and draw
FSBR	60	4.0	1.5	1.5 h
GSBR	54	1.5	3.5	6 min

2.2. Analytical methods

Total suspended solids (TSS) in the bioreactors were measured on mixed liquor samples according to standard procedures [19]. Samples were collected from the four SBR along different treatment cycles and were clarified by centrifugation (15 min at 4400 rpm) prior to dissolved COD and color analyses. Dissolved COD was determined according to standard procedures [19] and color was measured spectrophotometrically against deionized water. Absorbance spectra between 200 and 800 nm were collected from solutions of Acid Red 14 with different concentrations and a calibration curve was established using the absorbance values at the maximum absorbance wavelength in the visible region (515 nm). The concentration of dye in samples collected from the flocculent and the granular SBR systems was determined in terms of color equivalents (dye-color) based on the previously established calibration curve at 515 nm, which was the only absorption peak observed in the visible region for all the collected SBR samples.

Azo dye degradation and metabolite formation were followed along selected SBR cycles by HPLC with spectrophotometric detection at 220 nm using a LiChroCART Purospher STAR RP-18e column (250 mm × 4 mm). The eluent consisted of phosphate buffer (25 mM, pH 5.5) and acetonitrile. A gradient elution (0–50% acetonitrile for 30 min) was used with a flow of 0.7 mL min^{-1}.

2.3. Toxicity assays

Wastewater samples were collected from FSBR1 and GSBR1 for toxicity assessment at three different cycle stages, namely WW_{feed} at the feeding phase (corresponding to the simulated dye-laden textile wastewater prior to treatment), WW_{anaer} at the end of the anaerobic reaction, and WW_{final} at the end of aeration (i.e., at the end of the treatment cycle). A sample corresponding to the feed solution without dye was used as control ($WW_{control}$).

2.3.1. Microplate susceptibility assay

To measure the inhibitory effects of the collected samples on the growth of *S. cerevisiae* BY4741 Δ*erg6* [16], a previously described microplate susceptibility assay was used [15] with adaptations. Briefly, a standardized population of yeast cells grown to mid-exponential phase ($OD_{640\,nm}$ = 0.50 ± 0.05) in liquid YPD growth medium (glucose 20 g L^{-1}, bacto-peptone 10 g L^{-1}, yeast extract