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Research article

# High-rate anaerobic treatment of wastewater from soft drink industry: Methods, performance and experiences

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ABSTRACT

At an Austrian soft drink company, an expanded granular sludge bed reactor for anaerobic wastewater treatment was inoculated with sludge from paper and food industries. Detailed online monitoring and laboratory examinations were carried out during startup and subsequent phases, which included a period of inhibition after ca. 80 days during which reactor degradative performance diminished suddenly, following a period of increased effluent VFA. After dosing iron chloride ($FeCl_2$) and micronutrients and reducing organic loading to startup levels, the reactor eventually reached efficient operation (> 85% COD degradation) after a gradual recovery phase. In this work performance data both at lab and full scale are elaborated along startup, adaptation, pre-inhibition, recovery and stable phases, and correlated between scales. High rate anaerobic treatment of soft drink industry wastewater was successful in terms of COD removal efficiency and final effluent COD ($\sim 300\ mg\ l^{-1}$), with a startup period (including inhibition) of ca. 5 months.

## 1. Introduction

Over the past few decades anaerobic processes have become increasingly important for the treatment of industrial wastewaters. Worldwide nearly 9000 large-scale anaerobic plants are in operation (IEA, 2011); of these roughly half are considered high-rate reactors. A properly functioning anaerobic digestion (AD) system will produce roughly 13.5 MJ of energy from methane per kg of COD removed, resulting in 1.5 kWh electrical energy production (assuming $\eta_{elec}$ = 40%) (Van Lier et al., 2015). High rate anaerobic wastewater treatment processes have been in wide use since the 1970s; among the multiple configurations available (anaerobic filter, fixed film reactor, expanded bed anaerobic reactor) the Upflow Anaerobic Sludge Blanket (UASB) (Lettinga and Van Velsen, 1974) and its derivative technologies have become established globally as the foremost method with roughly 90% of industrial anaerobic treatment systems based on this configuration (Van Lier, 2008). One of the most common modified designs is the expanded granular sludge bed (EGSB) reactor, which includes an internal recirculation of effluent, typically housed in a reactor body with a higher slenderness ratio (H/D) than the standard UASB reactor. This leads to higher upflow velocities (6–10 $m{\cdot}h^{-1}$ in EGSB, 0.5–2 $m{\cdot}h^{-1}$ in UASB) (Gonzalez-Gil et al., 2001; Chernicharo, 2007) and expands the sludge bed upward. High rate anaerobic reactors have been utilized successfully for treatment of carbohydrate-rich wastewaters e.g. from distilleries, wineries and the pulp and paper industry, reaching consistent COD removal efficiencies over 80% at full scale (Warmenhoven and Spanjers, 2011; Ince et al., 2012; Wolmarans and De Villiers, 2002) and well over 90% at reduced scales (Petta et al., 2017; Petropoulos et al., 2016).

The goal of high-rate anaerobic treatment is typically the minimization of effluent COD in order to approach local discharge limits (downstream aerobic processes are typically required to reach direct discharge limits); biogas, if utilized, offers added value through energy recovery. In fact biogas production in terms of both quality and quantity are intrinsically linked to COD removal efficiency. However, due to complex and sensitive metabolic pathways in the AD process, precise control during operation is inherently difficult. Biomass (sludge) is often subjected to shock loads and variable operational conditions, such that even small changes in feedstock characteristics can lead to significant changes in sludge composition, effluent quality and biogas production. In cases of inhibitory organic loading, often the first obvious signal is an increase of volatile fatty acids (VFA) in the effluent. In such cases the conversion of critical intermediate products is no longer thermodynamically feasible and the system may suffer a total collapse

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(Dohanyos et al., 1985; Ma et al., 2009; Petropoulos et al., 2016), which often requires a costly replacement of sludge and an additional startup period lasting weeks.

In addition, granular sludge undergoes population and compositional shifts when exposed to new substrate, which may result in fluctuating performance and periods of reduced activity during the first months of operation. This common occurrence is a primary bottleneck in high-rate anaerobic treatment of industrial wastewater (Weiland and Rozzi, 1991; Baloch et al., 2007). Particularly when the wastewater treated is of industrial origin, the sludge may require additional nutrient supplementation (Khanal, 2008). If nutrient starvation occurs, it is likely that methanogenic activity will diminish, leading to the accumulation of $H_2$ and organic acids and thermodynamics-driven cessation of rapid $H_2$ turnover and VFA conversion to methane.

In addition, the variable nature of industrial substrates, which are heavily influenced by seasonal and production cycles, complicates the study of sludge adaptation independent from loading fluctuations. Thus sludge performance and quality were evaluated during operation under controlled lab conditions, including specific methanogenic activity (SMA) tests using standard substrate (glucose) and actual wastewater during startup. This step is recommended to evaluate kinetic parameters, monitor changes in sludge activity, and to determine optimal organic loading rates in order to accelerate startup and adaptation phases (Chernicharo, 2007).

In this study the performance of a newly-inoculated EGSB and its resident biomass are examined during startup and following phases. Data from full-scale operation are elaborated and correlated with batch results through five phases (after a brief pre-startup phase) which lasted ca. 40 days each: 1) adaptation; 2) pre-inhibition; 3) inhibition; 4) recovery; and 5) stable phases. The reactor of interest is located in Austria and used for high-rate anaerobic treatment of wastewater from the soft drink (i.e. fruit juice, energy drinks) industry.

## 2. Materials and methods

### 2.1. EGSB reactor and operational parameters

Brought into operation in March 2016, the EGSB reactor (Volume = 546 $m^3$, H = 20 m, Ø = 5.9 m) belongs to the R2S type (Rosenwinkel et al., 2011, 2015) and is operated in two stages arranged in vertical series, separated by a gas extraction device. The two stages are filled with granular sludge. Wastewater is distributed along the bottom of the reactor through an inlet system and flows through “high load” and “low load” stages. The flow between the two stages is controlled via internal and external recirculation.

Granular sludge (approx.15,000 kg oDM) was delivered from similar reactors from food and paper industries in December 2015 and January 2016. The reactor inoculum consisted of 56% sludge (by oDM) from an active high-rate reactor treating wastewater from the paper industry, and 44% sludge from the food processing industry, which had been inactive for several months. Temperature in the EGSB was maintained in the mesophilic range (30–35 °C) and pH between 6.5 and 7.5, using NaOH as neutralization agent.

In Table 1 wastewater (i.e. reactor feedstock) composition in terms of COD, VFA and macronutrients N and P is grouped according to operational phase, including minimum, maximum and average daily values:

Operation began with a brief (~2 weeks) pre-startup phase in late March/early April 2016, during which the volumetric organic loading rate (OLR) and sludge loading rate (SLR) were maintained around 1.5 kg COD·($m^3$·d)$^{-1}$ and 0.1 kg COD·kg oDM$^{-1}$ respectively. OLR and SLR were increased gradually until reaching 6.5 kg COD·($m^3$·d)$^{-1}$ and 0.15 kg COD·kg oDM$^{-1}$, respectively around day 82. Likewise total organic loading was increased from approximately 800 kg COD·d$^{-1}$ at the beginning to nearly 3000 kg COD·d$^{-1}$. Hydraulic retention times (HRT) were reduced from high startup values > 50 h to roughly 10 h as the granules adapted to the substrate and operational conditions (adaptation and pre-inhibition phases). These maximum loading rates were followed by a sharp decrease in methane production and COD degradation rate (inhibition phase), as well as an increase of volatile fatty acid (VFA) concentration in the effluent which led eventually to reactor failure.

Iron chloride ($FeCl_2$) was dosed to the reactor on day 106 to prevent binding (and loss) of other micronutrients to hydrogen sulfide ($H_2S$) in the liquid phase, while micronutrients (composition patent-protected) were dosed on day 110. During inhibition both OLR and SLR were reduced to startup levels in order to allow the granules to recover.

### 2.2. Methanogenic activity test and sludge compositional analysis

The procedure for the methanogenic activity test was conducted according to the VDI 4630 protocol (2016). Dry matter (DM) and organic dry matter (oDM) content in the sludge samples were determined according to DIN EN 12880 (2001) and DIN EN 12879 (2001) respectively, and the COD by means of a cuvette (LCK514) test by Hach Lange GmbH. Granular sludge at a reactor height of 3 m was sampled directly after filling the EGSB reactor, as well as in 5 deliveries approximately monthly. In this work sludge (or *inoculum*) samples are designated I1–I6 and ordered chronologically. The inoculum was exhausted at 37 °C for a minimum of 7 days before the test in order to reduce gas production from residual substrates to the greatest extent possible.

The batch tests were carried out with sieved granular sludge and glucose as the reference substrate using the Bioprocess® AMPTS II fermenter (Fig. 1), in which methane is measured semi-continuously after a $CO_2$ removal step in 3 M NaOH. Two additional assays were performed using actual wastewater and inoculum I1 to compare batch performance between simulated and real substrate.

Sludge samples from the EGSB reactor were fed with glucose corresponding to Food/Mass (F/M) ratios of 0.50 and 0.75 g COD·g oDM$^{-1}$ (11 mM and 16.6 mM, respectively). These values correspond with the maximum pre-inhibition concentration and 150% maximum pre-inhibition concentration for batch digestion of glucose (Kalyuzhnyi and Davlyatshina, 1997). Glucose is a readily degradable substrate which is suitable as a control substrate for sludge treating carbohydrate-rich wastewaters. The use of glucose as a standard makes it possible to determine the degradation rate for the overall anaerobic process (excluding hydrolysis) as well as the SMA of the granular sludge. Furthermore, examination of sludge performance independent of SLR, which is not possible using full scale reactor data, is particularly important to distinguish feedstock or operational influences from granule adaptive pressure responses.

Blank samples were used to determine endogenous respiration to be excluded from the substrate-derived gas production calculation. All tests were completed in triplicate. The bottles were flushed with nitrogen gas ($N_2$) for approximately 0.5 min to promote anaerobic conditions and then sealed and incubated at 37 °C in a water bath. Samples were gently mixed using an inbuilt rotating arm at 10 s intervals every six hours in order to promote the outgassing of the produced biogas, increase substrate-biomass contact, and avoid stratification while minimizing shear damage to the granules.

### 2.3. Batch test kinetics

If gas formation is measured continuously, SMA can be determined directly from the ratio of the gas volume difference (ΔV) during the period of observation (Δt). After a short lag phase in the first hours of the experiment, during which glucose is acidified and broken down into compounds which can then be converted by methanogens, a nearly linear increase in gas production occurs which corresponds to the time interval chosen for SMA determination (i.e. slope of the steepest rise). The specific methane activity (normalized per g COD added) is calculated according to Eq. (1):