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# Efficient and automated start-up of a pilot reactor for nitritation of reject water: From batch granulation to high rate continuous operation



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#### HIGHLIGHTS

• Granulation in sequencing batch reactor for nitritation is demonstrated feasible.

• Switching from batch to high rate continuous operation reduced start-up period.

• Sludge recirculation events enhanced stable high granular biomass concentration.

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#### 1. Introduction

## ABSTRACT

An automated sequencing batch reactor operation based on online measurement of the ammonium concentration was investigated as a tool for improving the start-up of a nitrifying granular airlift reactor. The effectiveness of this start-up procedure was verified with the characteristics of the developed granular sludge but also the improvement of the start-up was confirmed when comparing with the results achieved with two continuous-mode start-up strategies. Once a stable granular biomass was obtained, the reactor started to operate in continuous mode during more than 100 days, maintaining the characteristics of the granular biomass and achieving a nitrogen loading rate of 1.75 g N L<sup>-1</sup> d<sup>-1</sup>. The intermittent recirculation of small flocs of nitrifying biomass was explored as an alternative to increase the biomass concentration in the reactor and consequently, to increase the treated loading rate.

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Aerobic granular sludge was developed as a way of obtaining high concentration of active biomass together with good settling properties of the sludge, avoiding in turn, the costly supporting material required for biofilm development [1,2]. One of the key applications of aerobic granular reactors was the achievement of COD, nitrogen and phosphorous removal in the same reactor [3]. Aerobic granules are generally developed from activated sludge in sequencing batch reactors [1–4]. The feast-famine regime, the high shear stress, as well as the short settling time applied are accepted as the main driving forces for an effective granulation [2,4-6]. Studies reported the straightforward development of aerobic granulation for the treatment of high-strength organic wastewaters with relatively high concentration of COD [7], whereas the granulation process of activated sludge was recognized to be more difficult when low COD concentration is present in the wastewater although still feasible [3,8].

Once the granular sludge is formed, its stability is thought to be maintained due to the high shear stress and short settling times steadily applied in the granular reactors during the long-term operation. One of the identified challenges of this type of systems is the eventual destabilization of the granules, which is usually an irreversible process [9,10], eventually requiring a new start-up phase. Once the selection pressure applied with the shear stress and the short settling times is relaxed, heterotrophic bacteria could trigger the development of non-compact biomass which leads to a reduction of the settling velocity, which in turn produces the eventual washout of the biomass. Nevertheless, if the granules are colonized by slow-growing bacteria, like for instance nitrifying bacteria, the possibilities of destabilization of the granular sludge are rather reduced and stable operation is usually reported [11-13]. Therefore the operation mode with such granular reactors might be switched to continuous, just after convenient granule formation under sequencing batch mode operation.

Although nitrifying granules were obtained even before the development of aerobic granulation as new technology itself [14], there are only a reduced number of studies focused on the startup of granular reactors for nitritation of high strength ammonium



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wastewaters [11,15,16]. During the development of a robust ratio control strategy to achieve and maintain nitritation in granular sludge reactors [12], the start-up was identified as a possible drawback for the scaling-up of the process [15]. Alternatives for the start-up of aerobic granular reactors have been described, as for instance the induction of granulation with small particles (activated carbon [12]), or the use of crushed granules that will serve as nuclei for the later development of the aerobic granules (e.g. anaerobic granules [11]).

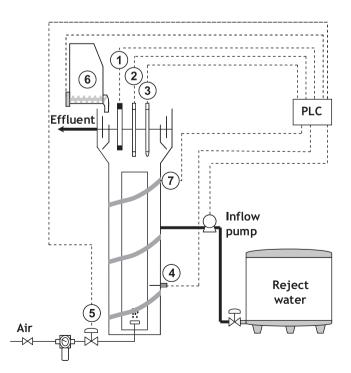
A specific procedure for an efficient start-up of a granular reactor for stable full nitritation operating in continuous mode will be presented in this contribution. An airlift reactor (150 L of capacity) was designed to allow sequencing batch and continuous modes of operation. The reactor was placed on site in a municipal wastewater treatment plant. The reactor treated the side water produced by a set of centrifuges after the anaerobic digestion of sludge for the production of biogas (i.e. reject water).

#### 2. Materials and methods

#### 2.1. Reactor description, location, wastewater and inoculum

A schematic diagram of the granular airlift reactor used in this study is presented in Fig. 1. The reactor capacity was 150 L. Height to diameter ratio is ca. H/D = 8.4. The temperature of the reactor was kept at 30 °C using an electric heating system. The pH was maintained at 7.5 in the reactor bulk liquid through the addition of solid Na<sub>2</sub>CO<sub>3</sub>. The dissolved oxygen (DO) concentration was measured by means of an online DO probe (LDO luminescence sensor, Hach-Lange, Düsseldorf, Germany). The total ammonia nitrogen (TAN = N-NH<sub>4</sub><sup>+</sup> + N-NH<sub>3</sub>) concentration in the bulk liquid was determined with an online probe (NH4Dsc Ammonium sensor with a Cartrical cartridge, Hach Lange, Düsseldorf, Germany).

The reactor was installed in a municipal WWTP in NE Spain with anaerobic digestion, to be fed in situ with the reject water.



**Fig. 1.** Diagram of the granular airlift reactor. (1) TAN probe; (2) DO probe; (3) pH probe; (4) Temperature sensor; (5) Valve for airflow regulation; (6) Na<sub>2</sub>CO<sub>3</sub> dispenser; (7) Electric heating system.

Dewatering of digested sludge is performed by a set of centrifuges which operate discontinuously and usually active during the night to use reduced fees for the electricity. The reject water was stored into two tanks of  $1 \text{ m}^3$  at room temperature and they were connected alternatively to the reactor inflow pump.

Composition of reject water was rather variable with the following concentrations: TAN: 440–758 mg N L<sup>-1</sup>, TOC: 240–696 mg C  $L^{-1}$ , TIC: 358–723 mg C  $L^{-1}$ , total nitrite nitrogen (TNN = N–  $NO_2^-$  + HNO<sub>2</sub>): 2–7 mg N L<sup>-1</sup>, N–NO<sub>3</sub><sup>-</sup>: 0 mg N L<sup>-1</sup>, Total suspended solids (TSS):  $122-239 \text{ mg L}^{-1}$ , Volatile suspended solids (VSSs): 100–206 mg  $L^{-1}$ ; pH: 8.1–8.8. The wide range for TAN and TOC concentrations is due to several intensities applied by the ultrasounds treatment of the sludge prior the anaerobic digestion. During the whole period of operation different types of polyelectrolyte at different concentrations were tested by the WWTP operator to improve the efficiency of the liquid-solid separation in the centrifuges used for the dewatering of the digested sludge. The polyelectrolyte used was cationic polyacrylamide (in solution at 0.4%), commercialized as CH82 by Chemipol; to a minor extent also Actipol C-444K (Brenntag) was utilized during the course of the experiments.

The pilot plant was inoculated with activated sludge from the municipal WWTP operated with a modified Ludzack–Ettinger configuration. The inoculum had a VSS/TSS ratio of 0.70 with a mean particle diameter of 0.1 mm and it was composed by  $97 \pm 2\%$  of heterotrophic biomass,  $2 \pm 0.5\%$  of ammonia–oxidizing bacteria (AOB) and <1\% of nitrite-oxidizing bacteria (NOB).

#### 2.2. Experimental procedure and type of operation

During the start up, the reactor was operated as a SBR to develop a granular sludge. The reactor capacity during SBR operation was reduced to 90 L. Each cycle was divided into a filling phase of 35 min, an aerobic phase (with variable time length), a settling phase of 30 min and a draw phase of 2.5 min. The exchange volume in each cycle was 45 L (50% of the total volume). The on-line TAN measurement was used to automatically manipulate the cycle duration. The aerobic phase was finished when the TAN concentration in the bulk liquid decreased to 50 mg N  $L^{-1}$  (denoted as TANtriggering). This type of operation was based on the previously developed ratio control strategy, which established that the DO/ TAN concentration ratio was governing the nitrite build up in a biofilm reactor [12]. When adapting the strategy for sequencing batch operation aiming to produce granular sludge, a relatively high airflow was applied to assure high shear stress, i.e., superficial air velocity higher than 1.2 cm s<sup>-1</sup> corresponding to a DO was relatively high:  $6-7 \text{ mg } O_2 L^{-1}$ . Due to high TAN concentrations in the bulk, DO/TAN concentrations ratio was maintained at very low values during the whole aerobic phase. In that way, strong oxygen limiting conditions were assured along the total length of the cycle to provide the conditions to outcompete NOB just after inoculation of the reactor and along the period of granular sludge development.

Once the granules were developed, the reactor operation mode was switched to continuous. The control strategy applied was very similar to that used to achieve and maintain stable partial nitrification in the biofilm airlift reactors already described in the literature [12]. A low DO/TAN concentration ratio was imposed, manipulating mainly the inflow rate fed to the reactor and the air flow-rate. To show the performance of the feedback control loop applied to maintain the TAN concentration close to the setpoint, both the measured and manipulated variable were plotted (see Fig. 2). During the continuous operation mode a TAN concentration setpoint was fixed equal to TANtriggering, i.e.  $TAN_{sp} = 50 \text{ mg N L}^{-1}$ . During the whole period of the continuous mode of operation, the total working volume of the reactor was 150 L.

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