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Nitrogen removal from ink-jet textile printing wastewater by autotrophic biological process: first results at lab and pilot scale

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Abstract. Digital textile printing is a rapidly spreading technology in the textile finishing industry, due to the great advantages in making printing much more flexible. On the other hand, wastewater originating from rinsing baths are rich in nitrogen (up to 600 mg/L of ammonium nitrogen), due to the massive use of urea in conditioning the textile before printing. Such high concentration prevents the direct discharge into water bodies or even in public sewers and specific dedicated on-site pretreatment is necessary. PN/anammox processes can offer an economically feasible alternative to conventional nitrogen removal processes, as these require a COD/N ratio of at least 8. The first results of the EU-LIFE DeNTreat project, consisting in the start-up of PN/anammox lab and pilot scale reactors are promising, in spite of the variability of the characteristics of the wastewater originating from rinsing digitally printed textiles.

Keywords: Industrial wastewater, Decentralized treatment, Deammonification, PN/anammox process, Process scale-up.

1 Introduction

Ink-jet (or digital) textile printing is rapidly spreading worldwide, mostly due to the greater versatility with respect to conventional printing techniques [1]. Despite lower wastewater volumes, discharges are rich in nitrogen at concentrations usually ranging from 150 to 600 mgN/L, due to the massive use of urea as additive for pre-treating the fabric. Very often, such a high nitrogen content in the process wastewater results in exceeding discharge limits for nitrogen in the sewer system (100 mgN/L in Italy), so that additional treatments are required, with consequent additional costs.

Over the last 20 years, the autotrophic removal of nitrogen by anaerobic ammonium oxidizing (anammox) bacteria emerged as a disruptive technology. The combined synergic application of anammox with ammonium oxidizing bacteria (AOB) in granular consortia in a single-stage process (partial nitritation (PN)/anammox), showed excellent performance as sustainable alternative to established biological processes in terms of energy requirements [2].

Literature reported several full-scale experiences in which the PN/anammox process was successfully applied to industrial wastewater [3], although the difficulties in achieving a stable process were equally strongly highlighted [4], mostly represented by (i) the need for appropriate COD/N ratio (not exceeding about 3:1), and (ii) the inhibition of the biomass activity due to wastewater toxicity [5].

The EU-LIFE DeNTreat project, whose preliminary results are reported in the present work, aims at demonstrating the feasibility of PN/anammox process as a decentralized treatment for ink-jet textile printing wastewater. While such application has never been reported in literature to the best of authors' knowledge, the main challenging aspects of the project are represented by the sub-optimal application conditions for the PN/anammox process and the urgent need for a competitive and sustainable technological. Results from two PN/anammox reactors are reported, respectively at lab and pilot scale, continuously fed with undiluted wastewater from a textile industry in Como district (Italy).

2 Materials and Methods

Feeding wastewater was taken from the 1200-m^3 equalization tank of textile industry effluents: (i) it was collected about every two weeks for the lab scale reactor, and (ii) it was constantly fed to the pilot scale reactor, placed inside the industrial area.

The lab scale reactor was a 2-L sequencing batch reactor (SBR), while the pilot scale reactor was a 10-m^3 SBR. Both reactors were operated at about 34°C in 6-hour cyclic sequences generally comprising at steady state (i) a 160-min feeding phase, (ii) a 180-min reaction phase, (iii) a 10-min settling phase, (iv) a 9-min discharge phase, and (v) a 1-min idle phase. During feeding and reaction phases, reactors were mixed by recirculating and bubbling the gas in the head space in a closed loop. At steady state, the cyclic exchanged volume was 0.5 L and 2 m^3, respectively for lab and pilot scale reactors. The process was controlled by PLCs equipped with on-line sensors for temperature, conductivity, pH, oxidation reduction potential (ORP) and dissolved oxygen (DO). In the pilot scale reactor, additional sensors for water level, ammonia and nitrite were present. DO was maintained at set-point by submerged aerators, while pH was kept between 7 and 8 by HCl and NaOH dosage. Reactors were inoculated with two different biomass batches from Paques (Balk, The Netherlands).

In both cases, a start-up period was necessary to reach steady state, in which DO set-point values were progressively modified to balance the activities of PN and anammox processes. During the start-up period, the lab scale reactor was fed with synthetic wastewater and then the ratio of textile wastewater in the feeding was progressively increased until undiluted conditions.

3 Results and discussion

The characterization of wastewater from textile industry equalization tank resulted in data reported in **Table 1**. A significant nitrogen content of about 200 mg/L was observed, partially ammonified during the retention time in equalization tank. Respirometric tests showed a bCOD of about half of the total COD. bCOD is at the

upper limit for the application of the PN/anammox treatment. The ratio of the biodegradable COD (bCOD) over total nitrogen (bCOD/N) remained around 2.
At every change of the wastewater feeding the lab scale reactor, we noticed the process needed at least a day to adapt to the new feed. In advantage, the warm temperature of the industrial effluent is beneficial for anammox application.

Table 1. Characteristics of three wastewater samples from textile industry equalization tank.

	Influent 1 (mg/L)	Influent 2 (mg/L)	Influent 3 (mg/L)
COD	628	676	662
NH_4-N	178	168	187
NO_3-N	0	0.95	0.5
NO_2-N	0	0	0
Total Nitrogen	238	216	242

The first start-up of the lab scale reactor confirmed the importance of an acclimation period for reaching steady state conditions. Both inoculums were fed with 100% synthetic influent made of a mixture of ammonium chloride and micronutrients. Initially, NLR was increased up to about 0.7 g/d (**Fig. 1**, left).

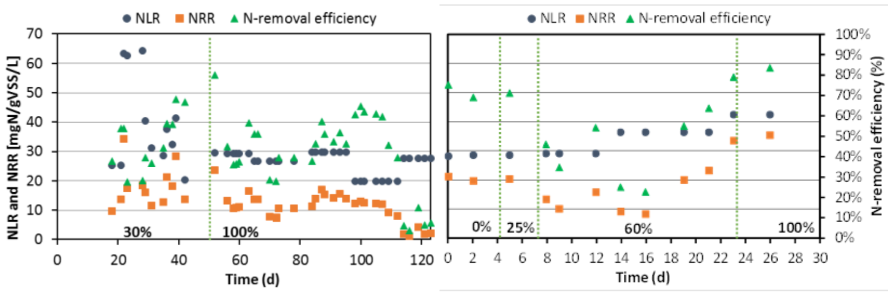

Fig. 1. Lab scale startup with first (left) and second inoculum (right). Nitrogen loading and removal rates (NLR, NRR respectively) on the left axis. N-removal efficiency on the right axis. Green vertical lines indicate the fraction of industrial influent.

After about 30 days of operation with the first inoculum, the feed was changed and 30% industrial effluent was mixed with the synthetic influent (**Fig. 1**, left). This caused a sudden nitrate increase (not shown), probably indicating anammox inhibition and nitrite oxidizing bacteria (NOB) take-over. After a gradual adaptation, at day 52 the feed was entirely made by industrial wastewater. NRR remained around 10-20% until at day 100 a gradual decrease in removal was visible. The gradual adaptation of the biomass to the industrial substrate allowed to reach a peak of 60% nitrogen removal with the first inoculum. However, the variability of the industrial influent due to the colorant treated and the different additives used might be the explanation for the strong variability in nitrogen removal.
The second inoculum, similarly to the first, was started with 100% synthetic influent and showed an initial drop in N-removal reaching 60% of industrial influent (**Fig. 1**, right). However, at day 20, the biomass showed some recovery of the

performance reaching 60% of N-removal even with 100% industrial influent. Operating at lower DO (max 0.1 mg/L) as compared to the first inoculum, seemed to prevent NOB take-over while allowing anammox adaptation.

The pilot installation showed better nitrogen removal over the whole testing period with an average NRR/NLR of 48.6% (±22.7%) (data will be reported in the full paper). For practical reasons, the pilot was fed since the start-up with raw wastewater. This is encouraging further studies and the application of the PN/anammox technology at full scale.

4 Conclusions

Effluents from digital textile printing processes have been treated at lab and pilot scale. First results are promising, in spite of the variability of the characteristics of wastewater originating from rinsing printed textiles. Main lessons learned are:

- Strict DO control is crucial to avoid the development of NOB and the growth of a heterotrophic layer on the granular biomass, which may prevent ammonia oxidation by ammonium oxidizing bacteria;
- Pre-treatment to reduce the bCOD/N ratio to well below 3 may be necessary to avoid excessive growth of heterotrophs;
- Some heterotrophic denitrification has been observed: on one hand, a limited activity may be beneficial, but if it increases it may compete for NO_2-N with the anammox microorganisms, and limit their growth;
- pH control is also essential as the decomposition of urea into ammonium nitrogen releases alkalinity and causes pH increase to above 8.5
- an anammox-rich and heathy inoculum is necessary to counteract initial competition for NO_2-N.

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