

# Continuous anaerobic digestion of highly concentrated secondary sludge and the effect produced by the autohydrolysis pretreatment

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## Abstract

The effect of autohydrolysis pretreatment on the continuous anaerobic digestion of secondary sludge was studied. The pretreatment was evaluated comparing the performance of two anaerobic digesters (of 20L each), for 300 days of operation. One of the reactors was fed with raw sludge and the other with pre-treated sludge. The sludge was previously thickened, providing the anaerobic digestion of high concentrated secondary sludge, around 8% of total solids. As a result, the methane production increased by 20% at 20d of solid retention time, and when the solid retention time was reduced the difference between both digesters decreased, reaching an improvement of 15% and 10% at 17 and 15 days of solid retention time respectively, also at 13d the difference became negligible. The VS and COD<sub>T</sub> removal showed that the organic matter elimination was always higher for the digester fed with pretreated sludge, and the COD<sub>S</sub> was always lower. Additionally, the Alk<sub>T</sub> and NH<sub>4</sub><sup>+</sup> content were higher for the digester operating with pretreated secondary sludge than for the control digester. Therefore, the autohydrolysis pretreatment of highly concentrated secondary sludge increased the methane productivity by 20%, with respect to the untreated sludge, when the anaerobic digestion was carried out at 20 days of solid retention time.

## Keywords

Anaerobic digestion, continuous operation, low-thermal pretreatment, secondary sludge.

## SYMBOLS

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TS (total solids), VS (volatile solids), COD<sub>T</sub> & COD<sub>S</sub> (total and soluble chemical oxygen demand), NH<sub>4</sub><sup>+</sup> (ammonia), TKN (total Kjendal nitrogen), WWTP (waste water treatment plant), AD (anaerobic digestion), D1 & D2 (digesters 1 and 2), SRT (solid retention time), OLR (organic loading rate), ORR (organic removal rate), *P* (productivity) and VPR<sub>b</sub> (volumetric production rate of biogas).

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## INTRODUCTION

Sludge management is responsible for around 50% of the operational costs of the WWTP, therefore its reduction has become an important challenge nowadays (Wei et al. 2003).

There are many studies focused on accelerating the sludge hydrolysis by different strategies in order to cause the disintegration of secondary sludge, some of these techniques are the application of pretreatments such as: mechanical, chemical, sonication, thermal and the utilization of hydrolytic enzymes (Climent et al. 2007; Carrere et al. 2010). All pretreatments in literature have associated benefits and disadvantages, the most common of which are related to the high operation costs and/or the high complexity of the implementation.

The autohydrolysis pretreatment is a biologic-thermal pretreatment that uses the inherent enzymatic activity of the secondary sludge. It involves subjecting the secondary sludge to a temperature of 55°C for nine hours and a limited amount of oxygen in batch operation (Carvajal et al. 2010). However, all the studies performed have studied the pretreatment effect on batch anaerobic digestion.

The main objective of this paper is to evaluate the effect of the autohydrolysis pretreatment of secondary sludge on its continuous anaerobic digestion of secondary sludge with high solid content.

## MATERIAL AND METHODS

### Secondary Sludge

The secondary sludge was delivered once a week from the WWTP of Valladolid, Spain, and stored at 4°C before experimentation. The sludge was thickened in the WWTP by an industrial centrifugal extractor (Pieralisi, Baby) without the addition of polyelectrolyte. The sludge characteristics varied in function of the WWTP operation and are presented in the table 1.

Table 1. Sludge characterization for anaerobic digesters operation [average  $\pm$  standard deviation (maximum-minimum)]

	Raw sludge	Pretreated sludge
TS ( $\text{g}\cdot\text{kg}^{-1}$ )	$65.9 \pm 10.1$ (38.5 - 85.2)	$65.2 \pm 9.7$ (38.5 - 85.0)
VS ( $\text{g}\cdot\text{kg}^{-1}$ )	$50.5 \pm 7.2$ (29.7 - 62.3)	$49.9 \pm 6.9$ (29.7 - 61.9)
COD <sub>T</sub> ( $\text{g}\cdot\text{kg}^{-1}$ )	$78.5 \pm 11.8$ (44.6 - 106.5)	$74.8 \pm 10.8$ (43.8 - 106.5)
COD <sub>S</sub> ( $\text{kg}\cdot\text{m}^{-3}$ )	$1.7 \pm 0.9$ (0.4 - 4.0)	$19.9 \pm 3.0$ (12.1 - 26.4)
TKN ( $\text{g}\cdot\text{kg}^{-1}$ )	$5.1 \pm 0.7$ (3.0 - 6.4)	$5.1 \pm 0.7$ (3.0 - 6.2)
NH <sub>4</sub> <sup>+</sup> ( $\text{kg}\cdot\text{m}^{-3}$ )	$0.2 \pm 0.1$ (0.1 - 0.4)	$0.4 \pm 0.2$ (0.1 - 0.9)

### Autohydrolysis Pretreatment

The pretreatment was carried out in batch conditions using 2-liter bottles loaded with 500g of concentrated sludge and closed with a rubber septum for 10 hours. They were then laid down horizontally in a roller bottle apparatus (Wheaton) inside a thermostatic chamber. The temperature of the chamber was kept constant at  $55\pm 0.5^\circ\text{C}$ .

### Anaerobic Digestion

The continuous anaerobic digestion was performed in two anaerobic digesters with a working volume of 20L each one, which ran at the same time. The digesters' temperature was kept constant at  $35^\circ\text{C}$  ( $\pm 1^\circ\text{C}$ ) with an electric resistor controlled by PID. Each reactor was mixed with solid recirculation provided by a Bredel peristaltic pump, and the feeding was performed automatically with a Watson-Marlow peristaltic pump. The digesters were monitored online: pressure, temperature and biogas production.

The behavior of the two anaerobic digesters continuously feed at different SRT was evaluated, one of them with secondary sludge, and the other one with pretreated sludge. Previous to this study, both digesters were started-up with thickened raw sludge, and the 206<sup>th</sup> day of operation the feed of one of the digester, (D2), was switched to pretreated sludge. The results that are presented in this paper correspond to the 320<sup>th</sup> to 500<sup>th</sup> days of operation, corresponding to four SRT conditions: 17, 15, 13 and 20 days.

### Chemical and Statistical Analysis

Samples of the feed and effluent were taken and analyzed two times per week. TS, VS, COD<sub>T</sub> & COD<sub>S</sub>, NH<sub>4</sub><sup>+</sup> and TKN were determined by Standard Methods (APHA et al. 2005). Biogas composition was measured once a day by gas chromatography using a Varian CP-3800, and helium was a carrier gas.

All data presented has been analyzed using statistic analysis software (STATGRAFICS Centurion ®), with a confidence level of 95%. The comparison of the digesters' behavior was performed by a means comparison using a t-test.

## RESULTS AND DISCUSSION

Figure 1 present the operational conditions of D2 during each stage of operation (D1 data not showed due to space requirements). As a consequence of the SRT reduction from 17 to 13d of SRT, the OLR increased from 2.5 to 4 ( $\text{kg}\cdot\text{m}^{-3}\cdot\text{d}^{-1}$ ), corresponding to the upper limit of recommended values for anaerobic digestion of sludge treatment (Metcalf et al. 2003). The high OLR reached during the experimentation, showed the feasibility of operating at a stable condition, when concentrated secondary sludge was used as the only feed in the anaerobic digestion process. Other studies have also shown the feasibility of operating at high OLR, with a feed thickened as much as

possible, thus reducing the water content of the sludge (Duan et al. 2012). The application of a dewatering process with the aid of a high molecular flocculants, reaching a sludge content between 8 and 12% of TS, and as result the anaerobic digesters were operated at 15d of SRT with an OLR of 4 ( $\text{kg}\cdot\text{m}^{-3}\cdot\text{d}^{-1}$ ) (Nges et al. 2010). These experimental values were not much higher than those presented in this work, considering that no polyelectrolyte was added, and only secondary sludge was used, which contains a lower solid content than the primary sludge.

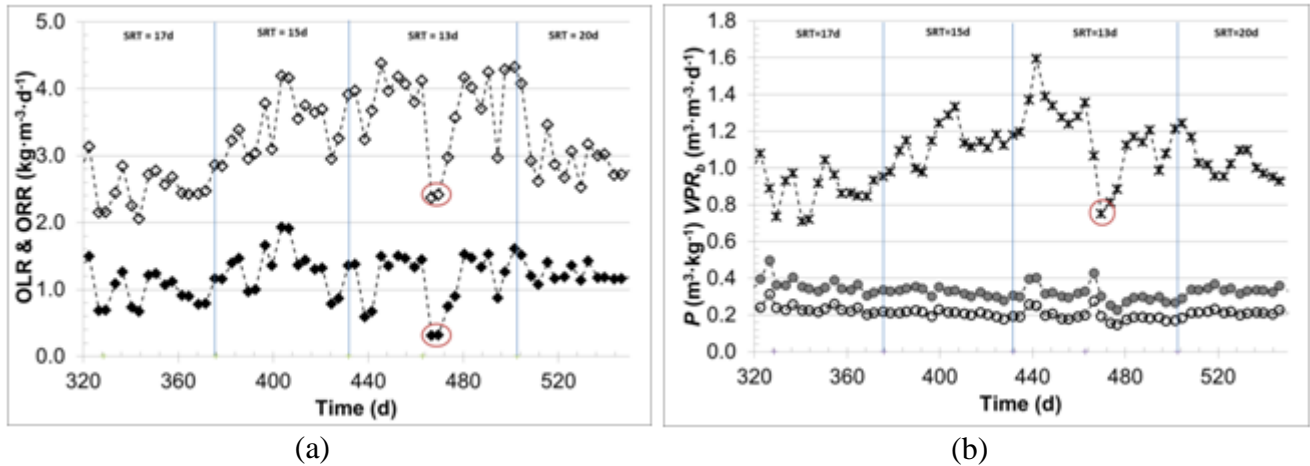


Figure 1. Operational conditions of D2 working with pretreated sludge: (a)  $\diamond$  OLR and  $\blacklozenge$  ORR, and (b) P of  $\bullet$  biogas and  $\circ$  methane, and  $\ast$  VPR<sub>b</sub>.

Table 2 shows the average and standard deviation values for the steady state period obtained for both digesters for each SRT studied. The performance of both digesters showed similar behavior, but with different intensities; digester D2 always showed higher values of removal and biogas production than digester D1. The maximum ORR reached was 1.36 and 1.50 ( $\text{kg}\cdot\text{m}^{-3}\cdot\text{d}^{-1}$ ) for D1 and D2 respectively at 15d of SRT, corresponding to a difference of 10% between them. When the SRT was reduced from 15 to 13d, the VPR<sub>b</sub> increased, as was described above, but the ORR decreased for both digesters, showing a reduction of the removal capacity, and therefore a probable destabilization of the anaerobic digestion process; however, the difference in the ORR between them was maintained. Previous studies of the effect of SRT on anaerobic digestion showed no proportional increase of the OLR and ORR, when the OLR was higher than 4 ( $\text{kg}\cdot\text{m}^{-3}\cdot\text{d}^{-1}$ ) (Lee et al. 2011).

On the 467<sup>th</sup> day, the feed solid content decreased to 30 ( $\text{kg}\cdot\text{m}^{-3}$ ) of VS, due to a malfunction of the thickening operation in the WWTP. This operational problem was used to evaluate the effect produced in the anaerobic digester process, when a stress dilution condition was produced. The diluted feed was maintained during one week, and afterwards the regular operation was restored in the next step. After the dilution step, both digesters apparently recovered their previous operational conditions, however some differences were observed at steady state: the ORR increased but the biogas production decreased; the new operational conditions reached were similar to the representative values obtained at 15 days of SRT. Even though, the removal capacity was increased, the biogas production decreased, probably due to the wash-out of some active population. Also, the difference observed between both digesters was reduced, reaching non significant difference between the two.

Previous studies of this research group showed the autohydrolysis pretreatment gave an improvement on the anaerobic digestion of secondary sludge, on batch assays. The increase in methane productivity was around 20% respect the non pretreated sludge (Carvajal 2012). The comparison between both digesters' operation, showed that the methane productivity was always higher for the digester operating with pretreated secondary sludge than the non-pretreated sludge. This difference becomes smaller by reducing the SRT. The methane productivity decreased for both digesters when the SRT decreased. The difference in methane productivity between both

digesters was 20% at 20d of SRT, 14% at 17d of SRT, 10% at 15d of SRT, and finally become negligible at 13d of SRT. On the other hand, when the SRT returned to 20d, with an OLR of 3 ( $\text{kg}\cdot\text{m}^{-3}\cdot\text{d}^{-1}$ ), the methane productivity increased for both digesters, obtaining a difference of 20% between them.

Table 2 presents the methane yield, i.e. the methane produced in terms of COD removed. All values obtained were similar to the stoichiometric ratio ( $0.35\text{m}^3\cdot\text{kg}^{-1}$ ), with the exception of the last value for D1 (20d of SRT), which is probably an indicator of a reduced methanogenic activity.

The VS and COD removal showed that the organic matter elimination was always higher for the digester fed with pretreated sludge. However, the difference between both digesters' elimination was small. The average levels for  $\text{Alk}_T$  and  $\text{NH}_4^+$  content, at the steady state, were higher for the digester operating with pretreated secondary sludge than for the control digester. The  $\text{COD}_S$  content was always lower for the D2 than D1, showing a higher removal capacity in the soluble phase from the pretreated sludge.

Table 2. Operational conditions at steady-state of both digesters

$\Delta t$ (d)	SRT (d)		OLR ( $\text{kg}\cdot\text{m}^{-3}\cdot\text{d}^{-1}$ )		VPR <sub>b</sub> ( $\text{m}^3\cdot\text{m}^{-3}$ )		P <sub>CH<sub>4</sub></sub> ( $\text{m}^3\cdot\text{kg}^{-1}$ )		Y <sub>CH<sub>4</sub></sub> ( $\text{m}^3\cdot\text{kg}^{-1}$ )	
	D1	D2	D1	D2	D1	D2	D1	D2	D1	D2
30	17±1	17±0	2.6±0.2	2.7±0.2	0.81±0.09	0.92±0.09	0.20±0.02	0.23±0.02	0.36±0.06	0.36±0.06
41	15±1	15±1	3.6±0.4	3.7±0.3	1.08±0.10	1.17±0.09	0.19±0.02	0.21±0.03	0.33±0.07	0.36±0.07
18	13±1	13±1	4.1±0.2	3.9±0.3	1.24±0.05	1.30±0.04	0.19±0.02	0.19±0.02	0.35±0.03	0.35±0.03
20	13±1	13±1	4.0±0.5	3.9±0.5	1.11±0.11	1.16±0.09	0.17±0.02	0.18±0.02	0.34±0.06	0.33±0.05
23	20±3	19±2	2.9±0.2	3.0±0.3	0.87±0.08	1.00±0.07	0.18±0.02	0.22±0.02	0.29±0.05	0.34±0.05

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## REFERENCES

- APHA, AWWA, et al. (2005). Standard Methods for the examination of water and wastewater. Washington DC.
- Carrere, H., C. Dumas, et al. 2010. Pretreatment methods to improve sludge anaerobic degradability: A review. *Journal of Hazardous Materials* **183**(1-3): 1-15.
- Carvajal, A. (2012). Study of the autohydrolysis pretreatment of secondary sludge and its influence on the anaerobic digestion. Department of Chemical Engineering and Environmental Technology, University of Valladolid. **PhD**.
- Carvajal, A. R., M. Peña, et al. (2010). Evaluation of autohydrolysis pretreatment over high concentrated secondary sludge. IWA-12th World Congress on Anaerobic Digestion, Guadalajara, México.
- Climent, M., I. Ferrer, et al. 2007. Effects of thermal and mechanical pretreatments of secondary sludge on biogas production under thermophilic conditions. *Chemical Engineering Journal* **133**(1-3): 335-342.
- Duan, N., B. Dong, et al. 2012. High-solid anaerobic digestion of sewage sludge under mesophilic conditions: Feasibility study. *Bioresource Technology* **104**(0): 150-156.
- Lee, I.-S., P. Parameswaran, et al. 2011. Effects of solids retention time on methanogenesis in anaerobic digestion of thickened mixed sludge. *Bioresource Technology* **102**(22): 10266-10272.
- Metcalf, Eddy, et al. (2003). Wastewater Engineering: Treatment and Reuse, McGraw-Hill.
- Nges, I. A. and J. Liu. 2010. Effects of solid retention time on anaerobic digestion of dewatered-sewage sludge in mesophilic and thermophilic conditions. *Renewable Energy* **35**(10): 2200-2206.
- Wei, Y., R. T. Van Houten, et al. 2003. Minimization of excess sludge production for biological wastewater treatment. *Water Research* **37**(18): 4453-4467.