

SEPARATE DIGESTION OF LIQUID AND SOLID FRACTIONS OF THERMALLY PRETREATED SECONDARY SLUDGE. ASSESSMENT AND GLOBAL EVALUATION

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Abstract - The fractioning into separate liquid and solid fractions obtained by centrifugation of thickened fresh and thermally pretreated (170 °C, 50 minutes) secondary sludge showed that 30% of the particulate organic matter was released during the pretreatment, correspondingly increasing the methane production of the particulate matter by 30% (from 259 to 329 mL CH₄/g VS_{fed}). The responsible of this enhancement was the liquid fraction, as the biodegradability of the solid fraction remained constant after the pretreatment. Mass balances showed that 34% of the VS were released to the liquid fraction, generating nearly 50% of the total methane produced, with much faster kinetics compared to the solid fraction. These results support the hypothesis of a separate liquid-solid digestion of thermally pretreated sludge, which would result in decreasing the digestion volume to half while duplicating the methane productivity per kilogram of sludge fed to digestion.

Keywords: Anaerobic digestion; BMP; Liquid/solid fractions; Secondary sludge; Thermal hydrolysis.

INTRODUCTION

Owing to environmental, economic, social and legal factors, there is a great interest in the reduction of sludge production volume, as well as resource recovery options. Anaerobic digestion is a well-proven route, combining sludge removal with energy production. However, waste activated sludge (WAS) is biological (Neyens and Baeyens, 2003) and, therefore, anaerobic digestion is unable to remove a high fraction of the organic matter and unable to reduce significantly its final volume.

Hydrolysis being the rate limiting step for the biological degradation of WAS (Li and Noike, 1992; Shimizu *et al.*, 1993), the introduction of a thermal hydrolysis pretreatment has proven to be a very interesting option (Kepp *et al.*, 1999; Bougrier *et al.*,

2008; Carrère *et al.*, 2010), currently implemented full-scale (mainly Cambi® and Biothelys®).

On the other hand, taking into account the different kinetics of hydrolytic, acidogenic and methanogenic stages, and the solubilisation reported for the organic matter after a thermal pretreatment (Carrère *et al.*, 2008), it is reasonable to consider the possibility of a separate digestion of solid and liquid fractions. This scenario has been proposed and studied for slurries coming from pig and dairy installation (Nozhevnikova *et al.*, 1999; Rico *et al.*, 2007; Sutaryo *et al.*, 2013), but is still unexplored in the case of sludge.

The hypothesis that supports this research is that a thermal hydrolysis pre-treatment improves the biodegradability and kinetics of sludge, but the resulting solid and liquid phases are probably radically

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different and can be considered separately in order to conceive a novel and optimized global digestion approach (higher methane productivity and smaller digestion volume).

The aim of this study is to identify the contributions to the final methane potential of the liquid and solid fractions obtained by centrifugation of thickened and thermally pretreated waste activated sludge, by performing biochemical methane production tests (BMP) on the different fractions, and a subsequent balance which integrates and compares the mass fractioning and methane production of the different phases in a global digestion scheme.

MATERIALS AND METHODS

Sludge Sampling, Fractioning and Pretreatment

A single sample of waste activated sludge (WAS) was taken from the municipal wastewater treatment plant of Valladolid (Spain) and thickened without polyelectrolyte to perform the research with concentrated sludge, that is the real feeding to pre-treatment units, as justified in Pérez-Elvira *et al.* (2008).

A fraction of the fresh WAS was treated in a thermal hydrolysis batch unit, where it was heated with live steam at 170 °C (8 bar) during 50 minutes, and then suddenly decompressed to atmospheric pressure. The effect of the pretreatment on the sludge solubilisation was expressed as the ratio of soluble organic matter with respect to the total (SCOD/TCOD).

Solid and liquid fractions of both fresh ("CONTROL") and pre-treated ("TH") sludge were separated by centrifugation at 5000 rpm for 10 minutes. Table 1 presents the results of the fractioning and phase characterization.

Biochemical Methane Potential Tests (BMP)

The evaluation of the anaerobic digestion of the different samples (total, solid and liquid fractions) was performed in BMP assays at 35 °C, conducted in

triplicate in 160 mL serum bottles filled with 50 mL of a mixture of anaerobic inoculum (from the digester of a municipal WWTP, 15 days SRT) and the corresponding substrate (fresh WAS, hydrolyzed WAS, or solid and liquid fractions of both) at a substrate to inoculum ratio (SIR) of 0.5 g/g (on VS basis). During the test, the bottles were incubated in a thermostated chamber in an orbital shaker. Methane production in the BMP assays was recorded by periodic measurements of pressure and biogas composition in the headspace of the bottles. The specific methane production was expressed with respect to the volatile solids of substrate fed to each test (mL CH₄/g VS). The endogenous methane production of the inoculum was subtracted from the total methane production in the BMP tests to obtain the real methane production of each substrate.

Analytical Methods

Total solids (TS), volatile solids (VS), total chemical oxygen demand (TCOD) and soluble chemical oxygen demand (SCOD) concentrations were determined according to Standard Methods (21st edition, 2005). The soluble phase for SCOD was obtained by centrifugation at 5000 rpm for 10 minutes. The pressure in the headspace of the BMP bottles was measured with a pressure sensor PN 5007 (IFM, Germany), and biogas composition was determined using a gas chromatograph coupled with a thermal conductivity detector (Varian CP-3800, USA).

RESULTS AND DISCUSSION

Sludge Characterization and Thermal Hydrolysis Performance

Table 1 presents the fractioning balance obtained in the centrifugation, the characterization of the different samples in terms of solids and COD, and the ratio SCOD/TCOD in each sample (no SCOD can be measured in solid fractions).

Table 1: Sludge fractioning and characterization of the different fractions (total, solid and liquid) before (CONTROL) and after (TH) the pretreatment, and soluble COD ratio.

	TS	VS	TCOD	SCOD	SCOD/TCOD
	g/kg	g/kg	mg/L	mg/L	%
CONTROL	132.1	104.6	164,000	19,120	12
CONTROL-LIQUID	15.4	10.9	24,651	19,120	78
CONTROL-SOLID	137.6	108.9	245,110	--	--
TH	78.3	59.5	100,063	36,947	37
TH-LIQUID	31.7	27.6	37,500	36,947	99
TH-SOLID	208.4	149.2	344,825	--	--

Comparing the values of TCOD and TS of “CONTROL” and “TH” samples, it can be observed that sludge is diluted by steam condensation (1.7 dilution factor). The corresponding SCOD/TCOD ratios of both samples exhibit that 30% of the particulate matter was solubilized during the thermal pretreatment.

Methane Potential of Control and Hydrolyzed Fractions

Figure 1 presents the digestion curves obtained in the BMP tests. It can be observed that, in the untreated sludge, the methane production of the solid and liquid fractions is similar, although the liquid phase presented an unexpected 5 days lag-phase, probably due to the fact that this phase is the result of the centrifugation of a thickened sludge, and therefore not representative of the real soluble easily

degradable phase. As expected, the methane production of the hydrolyzed sludge was higher compared to the untreated (329 vs. 259 mL CH₄/g VS_{fed}), representing a 30% increase in the methane production with respect to the particulate phase, which is consistent with the 30% solubilisation of the particulate phase obtained. The fractioning of the hydrolyzed sludge showed that the contribution of the enhancement in methane production and kinetics after the hydrolysis is due to the liquid fraction, remaining the solid fraction difficult to degrade.

Performance of Thermal Hydrolysis in Solid and Liquid Fractions

As announced in the previous discussion, it is interesting to compare the separate performance of liquid and solid fractions. Figure 2 compares the results obtained in the BMP for each phase.

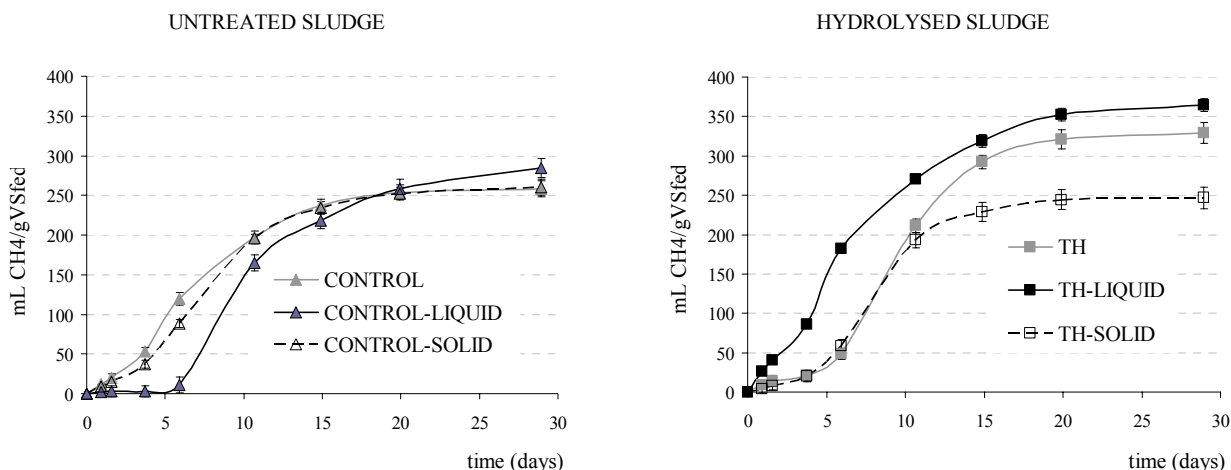


Figure 1: Methane production curves in the BMP test performed for the different fractions of WAS (total, liquid and solid) before (CONTROL) and after (TH).

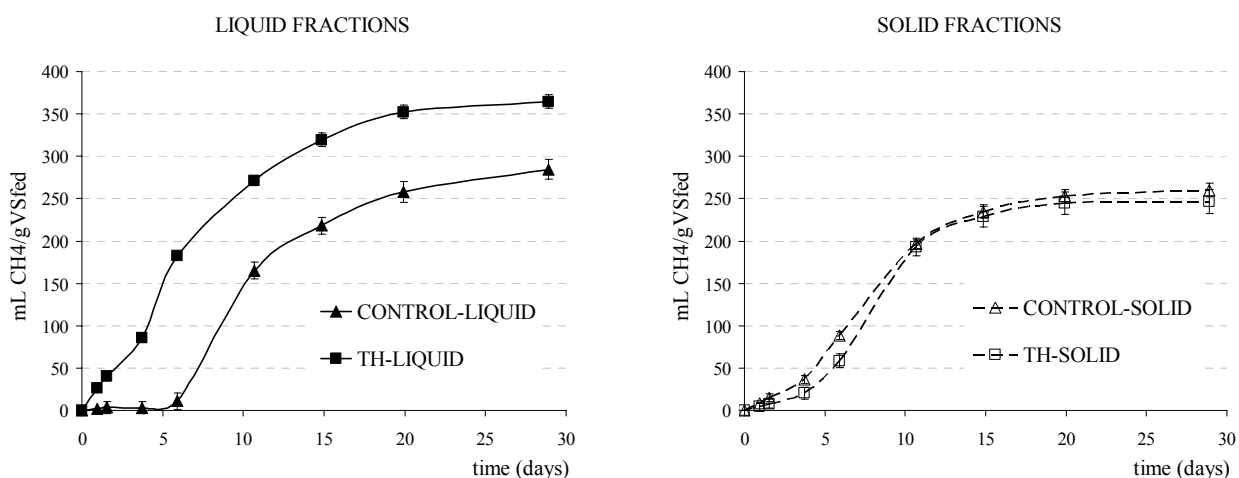


Figure 2: Comparison of the methane production curves for untreated (control) and pre-treated (TH) in the solid and liquid fractions.

The thermal hydrolysis pre-treatment increased the methane production due to the solubilisation of solids, which moved to the liquid phase, obtaining a value of 370 mL CH₄/g VS_{fed} in this liquid phase. However, the methane production of the solids remained similar to the original value for the solid phase, around 260 mL CH₄/g VS_{fed}.

Global Evaluation from Mass Balances

Although the previous results exhibit very useful information to understand the performance of the thermal pretreatment on the sludge. This is useless if the corresponding mass balances are not performed, as the contribution of solid and liquid fractions is key to an overall evaluation. From the experimental data obtained, mass balances were performed in order to quantify the influence of each fraction (liquid and solid) on the total methane production.

A calculation basis of 1 kg of sludge to be digested was considered, and the experimental data were translated into a methane production parameter, expressed as the CH₄ obtained per kilogram of sludge fed to digestion.

Table 2 presents the experimental data extracted

from the previous discussion, together with the results of the balances performed. Figure 3 compares the methane production per kilogram of sludge treated in the different fractions.

From this analysis, it is evident that the advanced digestion that combines thermal hydrolysis and anaerobic digestion presents a very noticeable difference with respect to the untreated sludge (control) in terms of the contribution of phases to the total methane production.

In the untreated sludge, the solid fraction is responsible for all the methane production, because the mass balance shows that the solid fraction accounts for 96% of the total mass (representing nearly 100% of the total VS). To complete a more realistic global balance, the liquid stream separated by centrifugation before the thickening of the WAS should be considered, although it is expected that this contribution would still be negligible.

In the case of the thermally pretreated sludge, the solid fraction is only 26% of the total mass, although it contains 66% of the VS to be degraded. The remaining 34% of VS released to the liquid phase are responsible for the production of 49% of the methane, with very different kinetics (higher rate, no lag-phase).

Table 2: Mass balances for VS feeding contribution and methane production for the total and the liquid/solid fractions of untreated (CONTROL) and thermally pretreated (TH) sludge.

	EXPERIMENTAL DATA			MASS BALANCES		
	Mass values	Phases fractioning	Specific methane production	Contribution to VS feeding	Methane production	Contribution to methane production
	(g)	%	mL CH ₄ /g VS _{fed}	%	mL CH ₄ /kg of sludge digested	%
CONTROL	1000	100%	259		27.2	
CONTROL-LIQUID	43	4%	285	0.5%	0.1	0.5%
CONTROL-SOLID	957	96%	260	99.5%	27.1	99.5%
TH	1758	100%	329		34.4	
TH-LIQUID	1297	74%	370	34.2%	16.7	48.6%
TH-SOLID	461	26%	257	65.8%	17.7	51.4%

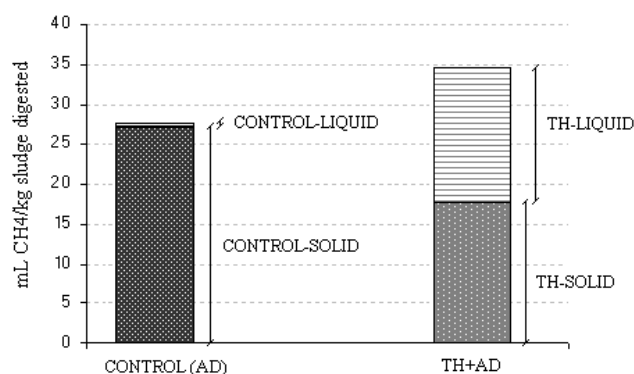


Figure 3: Methane production calculated per kilogram of sludge digested for the liquid and solid fractions of untreated (CONTROL) and thermally pretreated (TH) sludge.

These results suggest the possibility of a new scenario for the anaerobic digestion of sludge, combining a thermal hydrolysis pre-treatment with the separate digestion of the liquid and solid fractions in digesters operated at different hydraulic retention times (HRT), thus decreasing the total digestion volume. The idea from the experimental results is that the organic content of the liquid phase presents faster degradation kinetics, corresponding to the compounds solubilised during the hydrolysis pre-treatment, while the solid phase still needs high retention time, similar to a conventional solids digester. Therefore, the separate digestion proposed operates at different HRT, with different digester rheologies and control.

Proposal of a Separate Digestion Scenario

Two scenarios were compared: Scenario A corresponds to the conventional digestion of sludge, at 3% VS concentration and a HRT of 30 days.

Scenario B (presented in Figure 4) corresponds to the digestion scheme evaluated in this paper, consisting of a thermal hydrolysis pre-treatment followed by phase separation and digestion of solid and liquid phases in two different digesters, operated at different HRT.

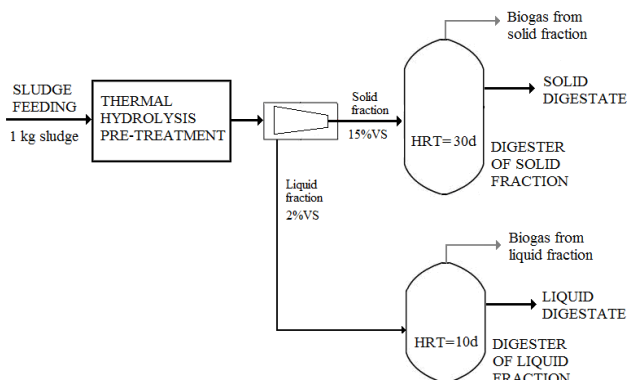


Figure 4: Proposed scenario for the digestion of separate solid and liquid phases resulting from a thermal hydrolysis pre-treatment.

Mass balances were performed for both scenarios according to the previously described calculation basis and methodology. However, as the HRT is a key parameter from the point of view of digester volume, the methane production obtained per kilogram of sludge feeding was expressed as methane productivity per day ($\text{mL CH}_4/\text{kg sludge}\cdot\text{day}$) dividing by the HRT. This parameter really represents the productivity of the digester.

The results obtained are presented in Figure 5 for both scenarios A and B (separating liquid and solid digestion in this second case).

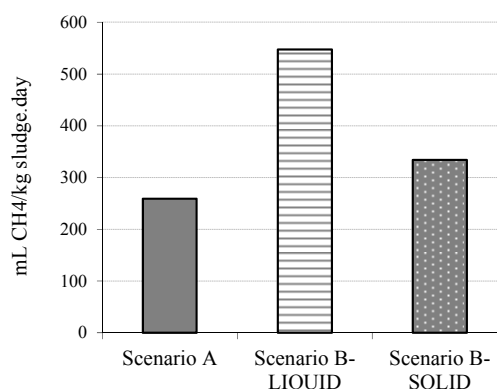


Figure 5: Methane productivity, calculated per kilogram of sludge digested and day, for the conventional sludge digestion (Scenario A) and the proposed advanced separate digestion scheme (Scenario B).

These values clearly exhibit that the digestion of the liquid fraction alone after the thermal hydrolysis of the sludge accounts for more than twice compared to the conventional digestion of the sludge. Furthermore, this “liquid-fraction digester”, while operated at a similar sludge concentration compared to the conventional, is four times smaller.

Regarding the solid fraction, the methane productivity is slightly higher than the conventional digester, although this “solid-fraction digester” is again four times smaller compared to the conventional, while operated at a high concentration.

CONCLUSIONS

The study performed in this paper is based on the hypothesis that the combination of sludge pre-treatment (170 °C, 50 minutes) and the subsequent separate digestion of the solid and liquid fractions can result in a global better methane productivity digester and smaller digestion volume.

The results obtained for the methane potential of fresh and pre-treated solid and liquid fractions showed that the 30% increase in the methane production obtained after the pre-treatment (from 259 to 329 $\text{mL CH}_4/\text{g VS}_{\text{fed}}$) corresponds to the enhancement in the methane production of the liquid fraction, due to the release of the particulate organic matter to this phase, while the biodegradability of the solid fraction remained constant after the pretreatment.

The mass balances performed based on these experimental data showed that 34% of the VS were released to the liquid fraction, generating nearly 50% of the total methane produced per kilogram of sludge digested, with much faster kinetics compared to the solid fraction.

These results support the initial hypothesis of a separate digestion of the liquid and solid fractions resulting from thermal pre-treatment of sludge. This approach would result in decreasing the digestion volume to half while duplicating the methane productivity per kilogram of sludge fed to digestion.

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