

Grease waste and sewage sludge co-digestion enhancement by thermal hydrolysis: batch and fed-batch assays

R. Cano, A. Nielfa, A. Pérez, L. Bouchy and M. Fdz-Polanco

ABSTRACT

Grease waste (GW) is an adequate substrate for sewage sludge co-digestion since, coming from a waste water treatment plant, it has a high methane potential (489 NmLCH₄/gVSin); however, no synergistic effect takes place when co-digesting with 52%VS grease. Conversely, thermal hydrolysis (TH) improves the anaerobic digestion of GW (43% higher kinetics) and biological sludge (29% more methane potential). Therefore, the application of TH to a co-digestion process was further studied. First, biochemical methane potential tests showed that the best configuration to implement the TH to the co-digestion process is pretreating the biological sludge alone, providing a 7.5% higher methane production (398 NmLCH₄/gVSin), 20% faster kinetics and no lag-phase. Its implementation in a fed-batch operation resulted in considerable methane production (363 NmLCH₄/gVSin) and TH improved the rheology and dewaterability properties of the digestate. This leads to important economical savings when combined with co-digestion, reducing final waste management costs and showing interesting potential for full-scale application.

Key words | co-digestion, fed-batch reactor, grease waste, pretreatment, sewage sludge, thermal hydrolysis

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INTRODUCTION

Anaerobic digestion of sewage sludge has been applied at wastewater treatment plants (WWTP) for decades. It is a well-known, efficient and environmentally sustainable technology which enables green energy production, as well as stabilization of sludge. The co-digestion of organic wastes with sludge offers several benefits over conventional digestion, such as increasing cost efficiency and improving the degradation of the substrates due to possible synergistic effects (Luostarinen *et al.* 2009). The use of an intermediate waste generated in the WWTP, such as the grease trapped in the dissolved air flotation (DAF) unit, would lead to optimization of the entire plant because of its availability on site. The cost of managing the grease waste (GW) to landfill will be avoided and its high fat content will increase the biogas yield since lipid-rich materials are known to have high methane potential (Silvestre *et al.* 2011). However, its degradation products, long-chain fatty acids (LCFA), may be severely inhibitive to methanogenesis and a lag-phase is usually observed. Accordingly, co-digestion of fats can be more profitable than when used as a single substrate,

which is also uneconomical considering the low amounts of GW produced in WWTP (7.3 kg/person/year according to Noutsopoulos *et al.* (2013)). Previous works have shown interesting results using this waste for sludge co-digestion at laboratory-scale (Davidsson *et al.* 2008; Luostarinen *et al.* 2009; Silvestre *et al.* 2011) showing high synergistic effects. However, the production of GW in WWTP could be reinforced by the addition of fat, oil and grease waste (FOG) collected in grease traps from different sources (food industry, restaurants, etc.) with a production rate of 7.1 L/person/year, with which co-digestion with sewage sludge has also been studied (Kabouris *et al.* 2009; Long *et al.* 2012).

When dealing with solid wastes, the degradation rate of the overall digestion process is limited by the first hydrolytic step. In order to accelerate it, thermal hydrolysis (TH) pretreatment is one of the most efficient techniques, leading to high organic matter solubilisation, pathogen reduction, dewaterability and rheology improvement, and an increase in biogas production. TH has been widely tested with sewage sludge and even applied in full-scale processes in

several WWTP (Carrère *et al.* 2010) in a fully energy integrated self-sufficient design. Nevertheless, pretreating co-digestion mixtures with grease has hardly been studied and needs further research for its implementation in a full-scale plant: Li *et al.* (2013) pretreated FOG and sewage sludge applying ultrasound and thermo-chemical techniques; Donoso-Bravo & Fdz-Polanco (2013) studied enzyme (lipase) addition to grease trapped from WWTP and sewage sludge co-digestion.

In this study, the implementation of TH pretreatment in a co-digestion process of GW and sludge, both from WWTP, is tested in progressive laboratory scales, from initial batch tests with raw substrates to a fed-batch co-digestion assay, with the aim of checking the potential for a full-scale application.

MATERIALS AND METHODS

Substrates

Thickened primary (SS1) and biological sludge (SS2) samples were taken from a municipal WWTP (Spain). GW comes from the DAF unit of another WWTP located in Spain. Characterization values are summarized in Table 1.

First, SS1 and SS2 were mixed in 1:1 weight ratio (according to the common ratio in the WWTP) to obtain mixed sludge, and then GW was added to it according to a specific ratio in the final co-digestion mixture: 48%COD, over 50%VS or 15% weight basis (COD: chemical oxygen demand; VS: volatile solids). This ratio was set in accordance to a previous study (Bouchy *et al.* 2012): for this ratio, there was no increase in the methane production

(no synergy by co-digestion) when compared with lower GW addition, being the final objective in overcoming the identified limits.

TH plant

The hydrolysis plant is made up of a 2 L reactor, fed with a substrate and heated with steam until the desired temperature is reached, and a flash tank where the steam explosion takes place after the hydrolysis reaction time has elapsed. TH was only applied to biological sludge rather than to the primary because it is mainly composed of biomass, hardly degradable carbohydrates and easily degradable proteins only available with a break of the cellular wall; hydrolysis breaks these cells and helps the biodegradation of biological sludge (Perez-Elvira *et al.* 2010). The operational conditions for these tests were 170 °C and 30 min, which were the optimized conditions for biological sludge obtained by Fdz-Polanco *et al.* (2008). Different conditions could be tested for grease in order to optimise its hydrolysis, but the aim of this study is to integrate grease and sludge TH in an already operating sludge TH plant. In fact, many full-scale plants have implemented TH technology at 170 °C, leading to considerable benefits (Carrère *et al.* 2010).

Fed-batch digesters

The fed-batch experiments were carried out in two cylindrical reactors of 20 L of useful capacity with a 10 L gas chamber. Both reactors were operated at mesophilic temperature (35 °C ± 1 °C). The biogas production was continuously measured by a pulse electrical system and analyzed by gas chromatography (Varian CP-3800). The biogas internal recycle ensured correct mixing. Feeding was carried out once per day.

Biochemical methane potential (BMP) tests

The BMP assays were performed by triplicates following an internal protocol. The reactor volume was 300 mL and a substrate-inoculum ratio of 1:1, in terms of VS, was applied. The incubation temperature was 35 °C. The inoculum was WWTP mesophilic digested sludge. Periodical monitoring analyses of biogas production by pressure meter and biogas composition by gas chromatography (Varian CP-3800) were performed during the tests. Methane potentials were expressed as average values of the net volume of methane per gram of initial substrate VS. Removal

Table 1 | Substrates characterization and mixture ratio for co-digestion

Parameter	Units	GW	Sludge	
			SS1	SS2
COD _t	g/kg	648.3	174.2	77.1
COD _s	g/kg	–	3.62	1.20
TS	g/kg	505.2	198.3	69.9
VS	g/kg	468.2	99.1	52.8
TKN	N-g/kg	3.27	4.69	5.75
NH ₄ ⁺	N-g/kg	0.24	0.29	0.24
Grease	g/kg	128.0	15.5	1.2
MIXTURE RATIO	Weight %	15	42.5	42.5
(GW + SS1 + SS2)	COD %	47.6	36.3	16.1
	TS %	39.9	44.4	15.7
	VS %	52.1	31.2	16.7

efficiencies have been determined by the %VS reduction from the substrate after BMP tests.

Modelling

The Modified Gompertz Equation (1) was considered in order to fine-tune the experimental data from BMP tests to a theoretical equation

$$B = P \times \exp \left\{ - \exp \left[\frac{R_m \cdot e}{P} (\lambda - t) + 1 \right] \right\} \quad (1)$$

The model has three parameters: the methane yield rate (R_m), which indicates the initial slope of the curve (mLCH₄/gVS/d), the maximum biogas production (P) expressed as mLCH₄/gVSin and the lag-phase (λ) in days. B is the calculated methane production (mLCH₄/gVSin) for time t . The model's fine-tuning to the experimental data was achieved by least squares methodology, by minimising the next objective function (2)

$$OF(\varphi) = \min \sum_{t=1}^N (B_{\text{exp}}(t) - B_m(t, \varphi))^2 \quad (2)$$

where B_{exp} is the consumption velocity obtained from measurements (plotted in BMP results graphs as points), B_m is the corresponding velocity calculated by the model (plotted with continuous curves), N is the number of measurements, t is time and φ represents the Gompertz parameters. The correlation factor (R^2) was then calculated to assess the accuracy of each model with respect to the experimental data.

Hydrodynamic and dewaterability tests

To assess the dewaterability and hydrodynamic properties of the fed-batch reactors, filterability, centrifugability and rheology tests were performed following an internal method for sludge characterization of the University of Valladolid. These tests were very relevant in terms of assessing the impact on mixing requirements, digestate dewaterability and handling properties. 'Filterability' was measured by forcing the sludge to pass through a 1.2 μm filter under a 1 bar g pressure and then the filtration constant (FC) was calculated. Capillary suction time (CST) was determined using a Triton Electronics Ltd and Whatman 17 filter paper. 'Centrifugability' assessed the liquid and solid phase separation after 5 min centrifugation at 5,000 rpm by % separated

liquid, % solid recovery in cake and solid concentration in cake. 'Rheology' was evaluated by viscosity curves obtained with a 'Brookfield Digital Viscometer DV-1'.

Experimental procedures

The experimental setup in this study was composed of three consecutive stages:

- **First BMP trials** to study the effect of TH pretreatment in raw substrates (GW and SS2) by BMP tests.
- **Co-digestion BMP tests** of GW and mixed sludge and then studying the implementation of TH to co-digestion.
- **Fed-batch operation** – co-digestion of GW and sludge with and without pretreatment in two identical reactors to study the effect of the pretreatment in a fed-batch operation. Hydrodynamic tests were applied to digestates to study their dewaterability and the rheology of the reactors.

Analytical methods

Internal protocols for solid substrates characterization based on *Standard Methods* (APHA 2005) were applied to determine the next parameters: total solids (TS) and VS, total and soluble chemical oxygen demand (CODt/s), volatile fatty acids (VFA), total Kjeldahl nitrogen (TKN), ammonium (NH₄⁺) and grease content.

Statistical analysis

All BMP tests were carried out in triplicate. The experimental methane production is always referred to average values and standard deviations were calculated and represented in BMP curves with vertical lines. For the hydrodynamic tests, duplicates were measured and the results were averaged.

RESULTS AND DISCUSSION

First BMP trials: effect of TH to substrates

As a first approach, BMP tests to raw substrates with and without pretreatment were carried out. As can be observed in Figures 1(a) and 1(b) and Table 2, the behaviour of the raw substrates biodegradation is completely different: while SS2 has a fast start-up ($\lambda = 0$) and a low final methane potential (215 mLCH₄/gVS), the GW presents a long

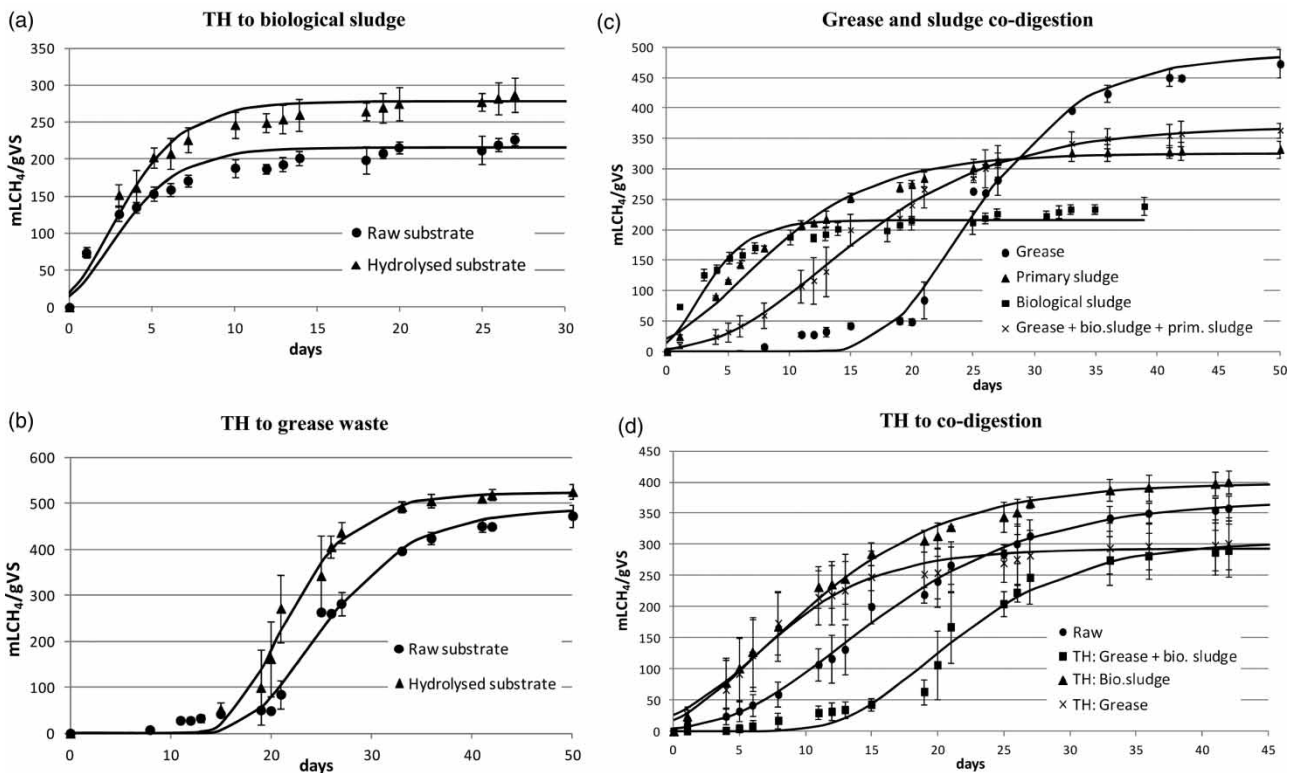


Figure 1 | BMP tests results: (a) biological sludge (raw and thermally hydrolysed); (b) GW (raw and thermally hydrolysed); (c) grease and sludge co-digestion: raw substrates; (d) different thermally hydrolysed co-digestion configurations.

Table 2 | BMP results: Gompertz parameters, TH improvements, VS removal efficiencies and co-digestion factors

Substrates		Gompertz parameters				% Increase				
		P mLCH ₄ /gVS	R_m mLCH ₄ /gVS/d	λ d	R^2 -	P %	R_m %	Lag-phase reduction days	Co-digestion factors (α) -	VS removal %
GW	Raw	488.6	30.3	17.6	0.990	7.2	43.3	-1.8	-	41.5
	TH	524.0	43.5	15.8	0.999	-	-	-	-	53.0
Biological sludge (SS2)	Raw	215.0	32.8	0.0	0.917	29.2	25.4	0.0	-	33.5
	TH	277.7	41.1	0.0	0.956	-	-	-	-	42.8
Primary sludge (SS1)	Raw	324.8	20.1	0.0	0.984	-	-	-	-	44.3
Co-digestion mixture (GW + SS1 + SS2)	Raw	370.3	16.3	4.3	0.996	-	-	-	0.94	52.0
	TH to GW	293.4	20.1	0.2	0.985	-20.8	22.6	4.2	0.75	34.7
	TH to SS2	398.1	19.6	0.0	0.991	7.5	19.7	4.3	1.01	50.6
	TH to GW + SS2	305.7	17.5	12.8	0.986	-17.4	7.0	-8.5	0.78	32.9

lag-phase (almost 18 days) but a high methane potential (488.6 mLCH₄/gVS), quite similar to the one reported by [Silvestre *et al.* \(2011\)](#) in batch assays (432–529 mLCH₄/gVS). However, TH leads in both cases to an improvement of those limitations: SS2 and GW methane potentials are increased by 29.2 and 7.2% and their methane yield rates become 43.3 and 25.4% higher, respectively. The application of TH to SS2 leads to great improvements of its

biodegradation parameters, because of the liberation of easily degradable material during cell disruption, as has already been tested by [Perez-Elvira *et al.* \(2010\)](#). However, TH to GW presents slight improvements but its long lag-phase (over 15 days) is still an important drawback for its biodegradation, which could be caused by the inactivation of methanogens because of the increase of LCFA, as was widely reviewed by [Long *et al.* \(2012\)](#), but this was not

experimentally proved. Its high lipid content and slow degradable materials could not be subjected to significant alterations during the pretreatment.

Co-digestion BMP tests

Raw substrate co-digestion

First, the effect of raw substrate co-digestion has been studied. GW, SS2 and SS1 have been biodegraded separately and together (Figure 1(c) and Table 2). Furthermore, the co-digestion factor (α) has been calculated, which indicates the ratio between the experimental methane potential of the co-digested mixture (P_{exp}) and the theoretical value (P_{theo}) calculated according to the mixture ratio from the individual co-substrate (i) methane potentials according to Equation (3):

$$\alpha = \frac{P_{\text{exp}}}{P_{\text{theo}}} = \frac{P_{\text{exp}}}{\sum_i \text{ratio}_i(\text{VS basis}) \cdot P_i(\text{mLCH}_4/\text{gVS})} \quad (3)$$

The final methane potential of the co-digested mixture is 6% lower than the theoretical value (Table 2), so that the mixture does not offer any synergistic effect in terms of methane production. This is what was expected from previous work but it is not in accordance with the literature: Silvestre *et al.* (2011) reported an increase of 138% for GW addition of 37%VS and Davidsson *et al.* (2008) between 9–27% for 10–30%VS addition. This could be explained by the higher GW input in this study (52%VS), which could cause an overload by LCFA. However, the lag-phase of the raw GW (18 days) decreases until values are below 5 days when the three co-substrates are degraded together, which is interesting in view of a continuous process. It is also remarkable that the SS1 methane yield rate is lower than that of SS2, which could be due to the high content of lipids, fibres and solids in SS1. However, its methane production is 50% higher than SS2, which justifies the application of the pretreatment to the latter.

Implementation of TH to GW and sludge co-digestion

In order to overcome the co-digestion limitation when adding too much GW to the mixture, TH is applied. Three different configurations to carry out the pretreatment to the co-digestion mixture were tested: TH applied to the GW, to the SS2 alone or to the GW and SS2 mixture. Then, the mixtures were subjected to the same ratios

explained in Table 1 by adding the non-pretreated substrates prior to the BMP tests. The results are presented in Table 2 and Figure 1(d).

In view of the results, TH only improves the raw mixture biodegradation when SS2 alone is pretreated. This sample leads to 7.5% higher methane potential, 20% faster kinetics and a null lag-phase. The increase of methane production in this case increases the co-digestion factor slightly over 1, making the co-digestion process more profitable. The results concerning the two other configurations of pretreatment do not show any improvement with respect to the non-pretreated sample in terms of methane production. This fact supports the high efficiency effect that TH has on SS2 rather than on GW, as was already observed while pretreating raw substrates. However, the efficiency of TH on SS2 is partially overshadowed when co-digesting since SS2 VS content in the mixture scarcely increases until it reaches 17% (Table 1). Then, if the GW content in the co-digestion mixture were lower, the effect of TH would be greater and synergies could be more favourable. Therefore, applying TH to just SS2 seems to be the most appropriate configuration to be implemented in the next step.

Organic matter removal in BMP tests are quantified as VS removal and are shown in Table 2. Applying TH to single substrates has led to higher VS removal efficiencies, which are directly proportional to final methane production. The values oscillate between 30 and 50%, which are acceptable values and correlate with the obtained experimental methane production.

Fed-batch operation: co-digestion of grease and thermally hydrolysed sludge

The study of a fed-batch process aims to simulate a real operation and foresee the impact that TH would have in a full-scale co-digester. The simultaneous operation of two identical reactors of 20 L capacity during five months enables comparison of the traditional co-digestion process without pretreatment (R1) with a co-digestion of thermally hydrolysed SS2, raw GW and SS1 (R2). All the operational variables were maintained at the same values for both reactors during the study: 20 days sludge retention time (SRT), 3.4 kg VS/m³/d organic loading rate (OLR), same mixture ratios (Table 1) and similar feed characterization. Table 3 summarizes all the parameters that were monitored during an operation time of 70 days (because of fluctuations during this period, parameter values were averaged and standard deviations are included) after an adaptation period equivalent to 3 SRT (60 days), as well as the main

Table 3 | Average values of the main parameters in both reactors during the fed-batch operation and dewaterability properties of both digestates (R2 thermally hydrolysed)

	Parameter	Units	R1	R2	% Increase R2 vs R1	
DESIGN DATA	Volume	L	20	20		
	SRT	d	20	20		
	OLR	kg VS/m ³ /d	3.4	3.4		
FEED CHARACTERIZATION	TS	g/L	82.2 ± 4.4	84.4 ± 6.9		
	VS	g/L	65.6 ± 3.2	67.8 ± 6.3		
	CODt	g/L	103.3 ± 13.2	106.2 ± 14.0		
REACTOR MONITORING	pH	–	7.5 ± 0.2	7.6 ± 0.1		
	Alkalinity	gCaCO ₃ /L	3.68 ± 0.12	4.09 ± 0.16		
	Alkalinity ratio	–	0.20 ± 0.02	0.19 ± 0.02		
	VFA	mgAcH/L	859 ± 170	951 ± 209		
	VFA/Alkalinity	–	0.22 ± 0.08	0.22 ± 0.07		
	TS	g/L	54.0 ± 4.7	44.3 ± 4.3		
	VS	g/L	34.8 ± 4.3	27.2 ± 2.5		
	CODt	g/L	43.5 ± 4.1	41.2 ± 8.1		
	CODs	g/L	2.07 ± 0.37	1.94 ± 0.22		
	TKN	N-g/L	2.47 ± 0.06	2.39 ± 0.11		
	NH ₄ ⁺	N-mg/L	682 ± 38	831 ± 41		
SUBSTRATE REMOVAL	TS	%	34.3 ± 5.5	46.5 ± 6.1	35.4	
	VS	%	46.9 ± 6.9	58.8 ± 5.3	25.5	
	CODt	%	56.7 ± 5.7	59.1 ± 10.1	4.2	
PRODUCTION EFFICIENCY	BIOGAS	NLbiogas/d	36.7 ± 4.5	38.7 ± 6.6	5.5	
		NLCH ₄ /gVSin	0.35 ± 0.04	0.36 ± 0.06	2.3	
	% CH ₄	%	64.9 ± 1.9	63.3 ± 4.1	– 2.4	
Digestates	Filterability	FC	m ² /s	48	67	40.0
		CST	s	545	471	– 13.5
	Centrifugability	Separated liquid	%	52	57	4.5
		Solid recovery in cake	%	98	99	1.6
		Solid concentration in cake	%	11	13	2.4

results of biogas production, substrate removal and digestate dewaterability properties. In Figure 2 the methane production during the entire assay (130 days in total) is plotted, and the dewaterability properties and rheology of digestates (viscosity curves) are represented.

Monitoring parameters that were analyzed twice a week (Table 3) show a similar and stable operation for both reactors: correct pH level (between 7 and 8), enough alkalinity content to ensure buffer capacity (over 1,000 mgCaCO₃/L), low VFA level and correct ammonia level (below 1,000 mg/L). Substrate removal efficiency is higher in R2, especially in terms of solids removal (35%TS and 25%VS higher than R1), but does not lead to a corresponding increase of methane production; trends are shown in Figure 2. Then, TH results in a significant increase of VS and TS destruction but the improvement in biogas production is not as high. This discrepancy does not occur in the case of COD, for which the removals are very similar in both reactors (57 and 59%) and coincide with the theoretical value estimated with the methane production

(considering the conversion factor 0.35 NLCH₄/gCOD). Average methane productivity in both reactors scarcely rise over 350 NmLCH₄/gVSin (in 20 days SRT), which is somewhat lower than the methane potential obtained in BMP tests (370 and 398 mLCH₄/gVS, respectively after 50 days). Even so, it is acceptable to carry out a continuous co-digestion process considering the high stability of the process. Moreover, although kinetics have not been evaluated in the fed-batch process, Table 2 shows an increase of 20% in the methane yield rate after TH. This is interesting in view of reaching a lower SRT in a continuous reactor, which will lead to an increase in biogas production per unit of reactor volume.

Considering the dewaterability and rheological properties from the digestates (Figure 2), it is remarkable the high influence that TH presents: FC is 40% higher; CST decreases 13.5%; and centrifugation is also improved, separating 4.5% more liquid and recovering 1.6% more solids in a 2.4% thicker cake. All these figures result directly in a more energetically efficient dewaterability process with its

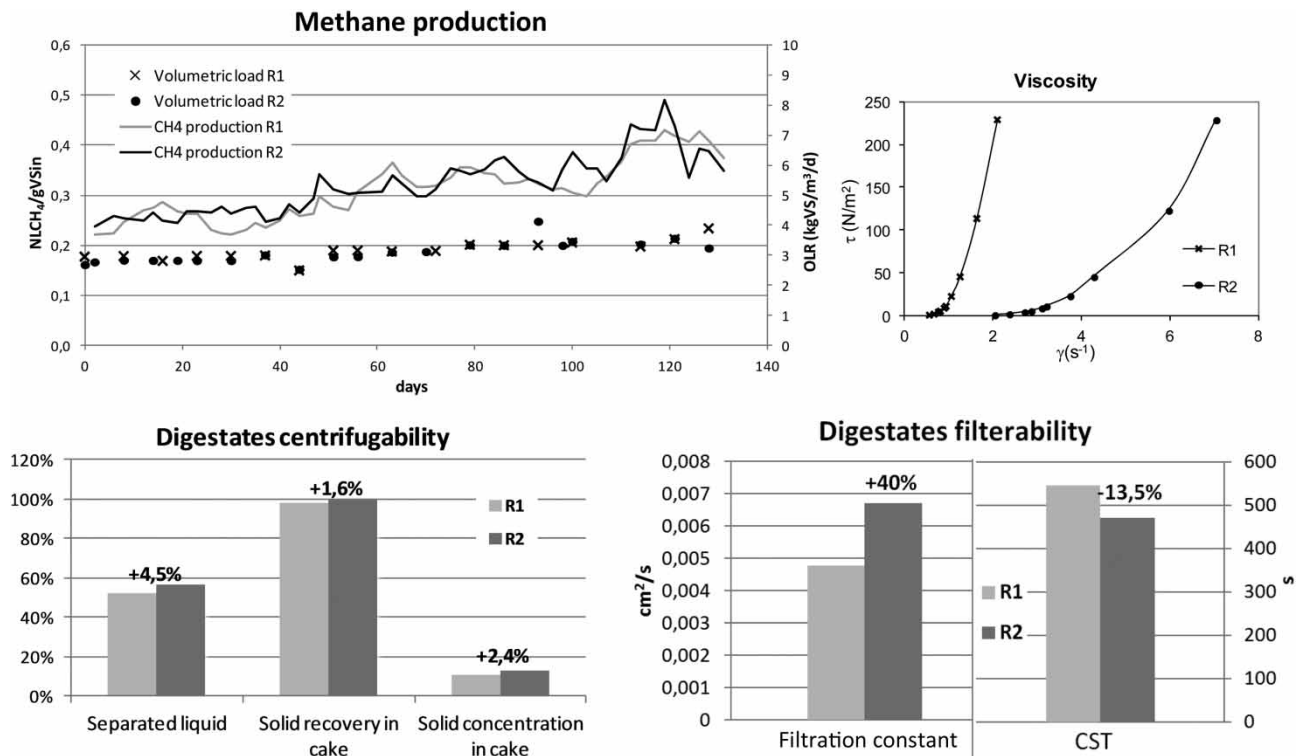


Figure 2 | Fed-batch operation results: methane production, OLR. Hydrodynamic parameter results.

consequent economic savings: a reduction of $50 \text{ kg}_{\text{biowaste}}/\text{t}_{\text{digestate}}$ will be avoided with TH, which supposes savings of $1.25 \text{ €/t}_{\text{digestate}}$ (considering the average landfill tax in Spain: 25 €/t). Furthermore, viscosity curves (viscosity corresponds to the slope, which does not have a constant value because of the non-Newtonian behaviour of these fluids) show that R2 clearly presents a lower viscosity than R1 (approximately 3 times lower). This, in view of a full-scale continuous operation, is an important consideration to facilitate pumping, mixing and for avoiding operational problems, such as blocking or settling. It also indicates energy savings since mixing and feeding are the main electricity consumers in a digester (Carrère et al. 2010).

According to other reported values from laboratory-scale semi-continuous reactors, very similar results have been obtained when co-digesting GW and sewage sludge without pretreatment: Silvestre et al. (2011) reached $331 \text{ Nm}^3\text{CH}_4/\text{tVSin}$ for the same SRT but lower GW addition; Davidsson et al. (2008) found a similar production ($344 \text{ Nm}^3\text{CH}_4/\text{tVSin}$) in just 13 days of SRT but with a lower GW input; Luostarinen et al. (2009) obtained $463 \text{ Nm}^3\text{CH}_4/\text{tVSin}$ when adding 46%VS grease trap sludge from a meat processing plant. It is noteworthy that a more recent study (Noutsopoulos et al. 2013) has doubled these methane

yields, reaching $700 \text{ Nm}^3\text{CH}_4/\text{tVSin}$, in 15 days of SRT when adding 60%VS of GW.

Despite TH not presenting an improvement in terms of methane production, it leads to important advantages related to dewaterability efficiency (saving $1.25 \text{ €/t}_{\text{digestate}}$) and rheology properties (important energy savings). Moreover, the co-digestion of GW and sewage sludge in the same facility avoids the landfill taxes associated with GW management. Considering a 500,000 equivalent inhabitant WWTP, these savings ascend to over 60,000 €/year (considering a GW production of $7.3 \text{ kg/person/year}$ in the WWTP and without counting FOG wastes from external sources). Therefore, the application of this pretreatment in a co-digestion reactor of GW and sewage sludge is an interesting alternative to be considered for a full-scale process.

CONCLUSIONS

- TH leads to a 29% increase of the methane potential of biological sludge and 43% higher kinetics of GW, which also shows a high methane production ($489 \text{ NmLCH}_4/\text{gVSin}$) but a long lag-phase (16 days).

- No synergistic effect of grease and mixed sludge co-digestion was found at the studied mixture ratio (52%VS grease), but the lag-phase was reduced to 4 days.
- The best configuration for applying TH to the co-digestion process is pretreating the biological sludge alone, providing 7.5% higher methane production, 20% faster kinetics and no lag-phase. This could be improved if the grease content of the mixture were lower, since TH showed higher efficiency on the biological sludge.
- The implementation of this assay in fed-batch reactors resulted in considerable methane production (363 NmLCH₄/gVSin) and TH improved the rheology and dewaterability properties of the digestate. This leads to important economical savings when combining with co-digestion, reducing final waste management costs and showing interesting potential for full-scale application.

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