Evaluation of thermal steam-explosion key operation factors to optimize biogas production from biological sludge

S. I. Pérez-Elvira, I. Sapkaite and F. Fdz-Polanco

ABSTRACT

Thermal steam-explosion is the most extended hydrolysis pretreatment to enhance anaerobic digestion of sludge. Thermal hydrolysis key parameters are temperature (*T*) and time (*t*), and the generally accepted values reported from full-scale information are: 150–230 °C and 20–60 min. This study assesses the influence of different temperature–time–flash combinations (110–180 °C, 5–60 min, 1–3 re-flashing) on the anaerobic degradation of secondary sludge through biochemical methane potential (BMP) tests. All the conditions tested presented higher methane production compared to the untreated sludge, and both solubilization (after the hydrolysis) and degradation (by anaerobic digestion) increased linearly when increasing the severity (*T*–*t*) of the pretreatment, reaching 40% solubilization and degradation of the particulate matter at 180° C–60 min. However, for the 180 °C temperature, the treatment time impacted negatively on the lag phase. No influence of re-flashing the pretreated matter was observed. In conclusion, thermal steam-explosion at short operation times (5 min) and moderate temperatures (145 °C) seems to be very attractive from a degradation point of view thus presenting a methane production enhancement similar to the one obtained at 180 °C and without negative influence of the lag phase.

Key words | anaerobic digestion, BMP, secondary sludge, severity, thermal hydrolysis

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INTRODUCTION

Sludge production minimization and resource recovery are nowadays priority issues in modern wastewater treatment plants (WWTPs) due to stringent environmental laws on wastewater treatment and sludge disposal routes. In spite of the suitability of anaerobic digestion as the key management and valorization option, the biological nature of sludge, especially secondary, limits both digestion and dewatering. Thermal hydrolysis has proven to optimize the anaerobic digestion of biological sludge by accelerating the rate-limiting hydrolysis step, improving biogas production, volatile solids removal and sludge dewatering, preventing foam formation, and removing pathogens.

However, the reported results on performance are difficult to summarize and compare, varying depending on the sludge source and treatment conditions (Carrère *et al.* 2010).

According to the reviews by Carrère *et al.* (2010) and Hii *et al.* (2014) most of the laboratory- or pilot-scale studies report optimum treatment temperature in the range of 160–180 °C, 180 °C being the generally accepted temperature limit to avoid inhibition by formation of refractory compounds at high temperatures (Dwyer *et al.* 2008; Ariunbaatar *et al.* 2014).

With regard to the treatment time, the range 30–60 min is mostly accepted, but recent studies (Donoso-Bravo *et al.* 2010) found that for the pretreatment at 170 °C temperature, the time (ranging from 5 to 30 min) did not influence sludge digestion, suggesting that long operation carried out at full scale could be unnecessary.

Regarding the effect of the flash, none of the bibliographic information analyses whether the re-flashing (repetition of sudden decompression without extra heat consumption) of sludge can enhance even further the solubilization of organic matter, thereby increasing the methane production.

From a full-scale implementation point of view, the available information on the operation conditions of the different thermal hydrolysis commercial processes (Cambi®, Biothelys®, Exelys®, TPH®, Lysotherm®, Turbotec®) shows that the operation conditions are in the generally accepted range

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150–230 °C, 20–60 min and one single sudden decompression (flash). As key factors for process design and economics, the revision of these values is a matter of major interest.

This study aims at evaluating different temperature–time-flash combinations (ranging from 110 to 180 °C and 5 to 60 min) on the biochemical methane potential (BMP) of steam-exploded secondary sludge, compared to untreated samples. The results are analyzed in terms of methane yield, kinetic parameters and severity factor.

MATERIALS AND METHODS

Sludge sampling

According to Pérez-Elvira *et al.* (2008), the study was performed for waste activated sludge (WAS) from the municipal WWTP of Valladolid (Spain), operated at 13 day solids retention time. A single sample of sludge was thickened without polyelectrolyte to 14%TS (79%VS) before being fed to the thermal hydrolysis unit in all the batch experiments performed.

The anaerobic inoculum for the BMP tests was sampled from the anaerobic digester in the WWTP treating mixed sludge, and pre-incubated for 2 days at 35 °C in a thermostated chamber prior to use in order to activate the microorganisms and to deplete most of the residual organic matter.

Steam explosion pretreatment, operation variables and experimental set-up

The thermal hydrolysis pilot plant operated (Figure 1) consists of a 20 L hydrolysis reactor heated with live steam (12 bar) from a boiler, and connected to an atmospheric flash tank (100 L) by a decompression valve that opens in a steam-explosion effect (sudden decompression). The operation is batch, and automatically controlled by fixing both temperature and hydrolysis time.

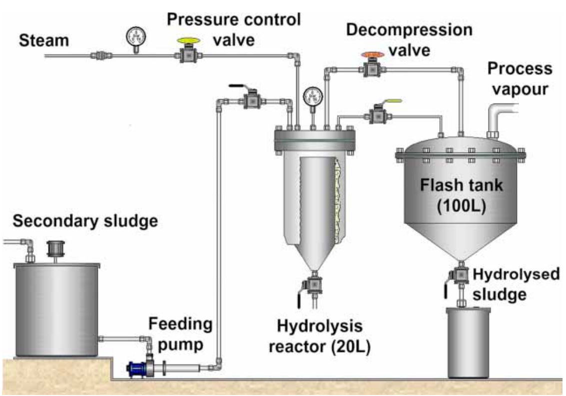

Figure 1 | Thermal pretreatment system (Fernández-Polanco *et al.* 2008).

To obtain the experimental plan (Table 1), a response surface methodology with Box–Behnken experimental design was used in this work. Three factors at two levels were considered: temperature (110° and 180 °C), time (5 and 50 min) and number of flashes (1 and 3). This last variable was evaluated by re-flashing the treated sludge twice or three times. The experimental plan consisted of 15 runs, including three repetitions at the center point of the experimental design.

Anaerobic digestion tests

BMP assays at 35 °C were conducted in triplicate in 160 mL serum bottles filled with 50 mL of a mixture of anaerobic inoculum and the corresponding substrate (untreated or treated secondary sludge) at a substrate to inoculum ratio of 0.5 g/g (on volatile solids (VS) basis). In this test, micronutrients and macronutrients were used for optimal function of anaerobic micro-organisms. Moreover, $NaHCO_3$ and Na_2S were added to provide a buffer capacity and avoid aerobic conditions, respectively. The methodology used was the one suggested by Angelidaki *et al.* (2009).

The bottles were closed with butyl septa, sealed with aluminum caps, purged with helium for 5 min and incubated in a

Table 1 | The Box–Behnken experimental design for thermal hydrolysis with three independent variables

	T (°C)	*t* (min)	log R_0	Number of flashes
CONTROL	–	–	0	0
TH-1	110	5	1.0	2
TH-2	110	30	1.8	1
TH-3	110	30	1.8	3
TH-4	110	50	2.0	2
TH-5	145	5	2.0	1
TH-6	145	5	2.0	3
TH-7	145	30	2.8	2
TH-8	145	30	2.8	2
TH-9	145	30	2.8	2
TH-10	145	50	3.0	1
TH-11	145	50	3.0	3
TH-12	180	5	3.1	2
TH-13	180	30	3.8	1
TH-14	180	30	3.8	3
TH-15	180	50	4.1	2

thermostated chamber at 35 °C in an orbital shaker at 150 rpm/min. Methane production in the BMP assays was determined by periodic measurements of pressure and biogas composition in the headspace of the bottles. Reference tests containing only anaerobic inoculum were prepared to determine the endogenous methane production of the inoculum, which was subtracted from the total methane production in the BMP tests to obtain the real methane production of the substrate. The experimental values obtained are always referred to average values, with the corresponding standard deviation.

Performance parameters

Table 2 summarizes the target parameters calculated.

It is worth mentioning that both solubilization and degradation factors (SF and DF) are calculated with respect to the particulate fraction of the chemical oxygen demand (COD), in contrast to most of the references that express these parameters with respect to the total COD. These proposed expressions are more accurate as the particulate matter is the potentially hydrolyzable fraction during the pretreatment.

And sludge biodegradability (BD) was calculated as the ratio of the experimental (mL CH_4/gCOD) to the theoretical methane production (350 mLCH_4/$gCOD_{removed}$).

Modelling

Four models were considered to fine-tune the experimental data from BMP tests to theoretical equations in order to estimate kinetic parameters with a certain degree of confidence.

Based on similar studies with solid wastes (Cano Herranz 2014), the models considered were (see Table 3): first order equation (FO), Modified Gompertz (MG) equation, transference function (TF), and logistic function (LF).

Despite differing mathematically from each other, the four models have common features: a kinetic parameter (R_m or μ_{max}) which indicates the maximum slope of the curve (mL CH_4/gVS/d), a maximum biogas production parameter (P) expressed as mL CH_4/gVS, and a lag-phase parameter (λ), in days. B is the calculated methane production (mL CH_4/gVS) for each time *t*. The correlation factor (R^2) was also calculated to assess the accuracy of each model with respect to the experimental data.

Analytical methods

Total solids (TS), VS, total chemical oxygen demand (TCOD) and soluble chemical oxygen demand (SCOD) concentrations were determined according to *Standard Methods* (Eaton *et al.* 2005). The soluble phase for SCOD was obtained by centrifugation at 5,000 rpm for 10 min. 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

Evaluation of thermal hydrolysis operation parameters through BMP test curves

Figure 2 presents the digestion curves obtained in the BMP tests: Figure 2(a), (b) and (c) the influence of temperature and time, whereas Figure 2(d) the influence of re-flashing.

Table 2 | Prediction parameters for the evaluation of TH

Parameter	Symbol	Units	Equation
Severity factor	$\log R_0$	–	$\log R_0 = \log\left(t.\exp\left(\frac{T-100}{14.75}\right)\right)$
Solubilization factor	SF	%	$\%SF = \frac{(SCOD/TCOD)_{TH} - (SCOD/TCOD)_0}{((TCOD - SCOD)/TCOD)_0} \times 100$
Methane potential	CH_4	mL CH_4/g VS_{fed}	$CH_4 = \frac{mLCH_4}{gVS_{fed}}$ at standard conditions$(0^\circ C, 1\ atm)$
Biodegradability	BD	%	$\%BD = \frac{mLCH_4/gVS_{fed}}{(350mLCH_4/gTCOD_{rem}) \cdot (gTCOD/gVS)} \times 100$
Degradation factor	DF	%	$\%DF = \frac{(mLCH_4/gTCOD)_{TH} - (mLCH_4/gTCOD)_0}{(mLCH_4/gTCOD)_0 \cdot ((TCOD - SCOD)/TCOD)_0} \times 100$

Table 3 | Model equations

Model	Equation	References
FO	$B = P \times [1 - \exp(-\mu_{\max} \cdot t)]$	Pavlostathis & Giraldo-Gomez (1991)
MG	$B = P \times \exp\left\{-\exp\left[\frac{R_m \times e}{P}(\lambda - t) + 1\right]\right\}$	Lay et al. (1997); Nopharatana et al. (2007)
TF	$B = P \times \left[1 - \exp\left(\frac{-R_m(t-\lambda)}{P}\right)\right]$	Donoso-Bravo et al. (2011); Redzwan & Banks (2004)
LF	$B = \frac{P}{1 + \exp\left[\frac{4R_m(\lambda - t)}{P} + 2\right]}$	Donoso-Bravo et al. (2010); Altas (2009)

The first evidence of these experimental results obtained is that all the conditions tested presented higher methane production in the final values for day 30 compared to the control (untreated sample), from 20% improvement (at 110 °C) to 40% (at 180 °C). Surprisingly, time was not a relevant parameter, the results for the different temperatures tested being rather similar for pretreatments at 110 and 145 °C (Figure 1(a) and 1(b)). Only at 180 °C did the pretreatment time influence negatively the lag phase (see kinetic parameters in Table 2).

Therefore, thermal steam-explosion at short operation times (5 min) and moderate temperatures (145 °C) seems to be very attractive from a degradation point of view, thus presenting a methane production enhancement similar to the one obtained at 180 °C and without negative influence of the lag phase.

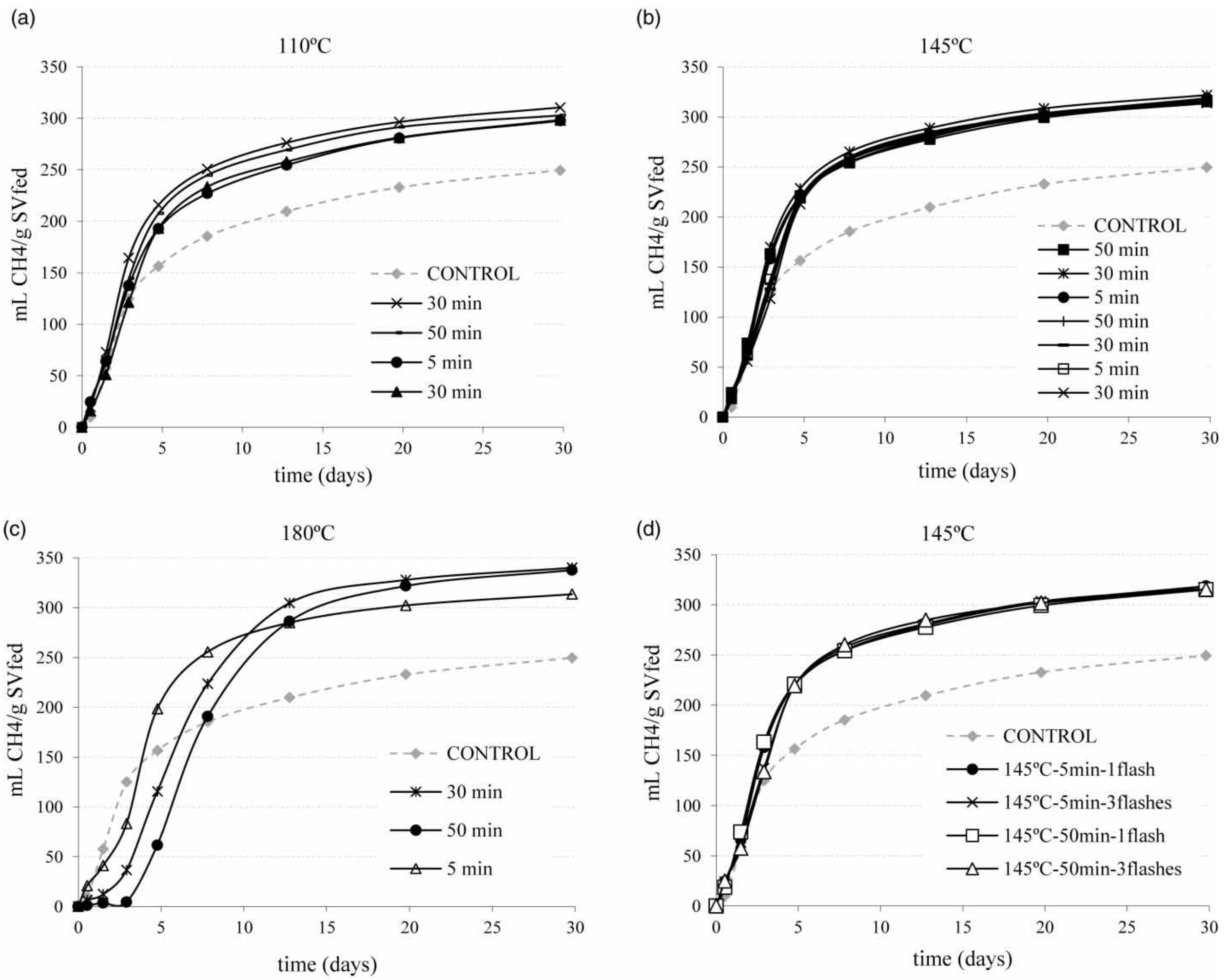

Figure 2 | Methane production curves from BMP test of the pretreated samples at 110 °C (a), 145 °C (b) and 180 °C (c), and after re-flashing (d).

Figure 2(d)) clearly exhibits that neither time nor re-flashing influenced the methane production.

Evaluation of modeling accuracy

The modeling approach was performed for all the samples, showing generally a good accuracy except for the tests with lag-phase. To summarize and focus the discussion, the results presented below correspond only to three samples: the untreated (CONTROL), TH-1 (pretreated, with no lag-phase) and TH-15 (pretreated, with lag-phase).

Table 4 summarizes the results of the estimated parameters obtained with the four models, and Figure 2 presents the model fit to the experimental BMP curves.

Table 4 shows that in most cases the estimated parameters were determined with a high degree of confidence ($R^2 = 0.98$). Regarding the maximum methane production, the four models estimate similar values in all the tests. However, some differences can be observed among the different models. FO estimates the micro-organisms' growth velocity (μ_{max}), which cannot be compared to the maximum methane production rate (R_m), which is estimated by the other models. While TF tends to overestimate this parameter, LF and MG estimate more similar values. Donoso-Bravo *et al.* (2010) also found this coincidence between LF and MG models.

Regarding the lag-phase, it is only determined by all the tri-parametrical models (MG, TF and LF), but only MG and LF fine-tune correctly this kind of kinetic. Table 4 shows that in the test with 3–4 day lag-phase (TH-15), TF and FO exhibited a poor correlation ($R^2 = 0.948$ and 0.943, respectively), showing that these models do not fit lag-phase kinetics. This experimental evidence is very clear in Figure 3, where only MG and LF follow accurately the experimental points in TH-15, and is consistent with the experimental results of Cano Herranz (2014) performed with grease waste.

Comparing the accuracy of the four models, the final conclusion is that the MG equation results to be in general the most appropriate to fine-tune thermal hydrolyzed secondary sludge kinetics, showing an average regression coefficient R^2 of 0.989. Similar accuracy values were obtained by Donoso-Bravo *et al.* (2010) and Cano Herranz (2014) with solid substrates and thermally pretreated sludge, respectively.

Table 4 | Estimated parameters by the four models for control, TH-1 and TH-15 samples

Sample	Estimated parameters	Model: MG	TF	LF	FO
CONTROL	P (mL CH_4/gS$_{fed}$)	226	238	221	239
	R_m (mL CH_4/gSV$_{fed}$/d)	37.4	52.0	39.9	–
	λ (d)	0.105	0.109	0.387	–
	μ max (d^{-1})	–	–	–	0.209
	R^2	0.971	0.988	0.955	0.988
TH-1	P (mL CH_4/gS$_{fed}$)	274	287	269	288
	R_m (mL CH_4/gSV$_{fed}$/d)	43.3	60.8	46.7	–
	λ (d)	0.077	0.066	0.403	–
	μ max (d^{-1})	–	–	–	0.206
	R^2	0.982	0.994	0.969	0.993
TH-15	P (mL CH_4/gS$_{fed}$)	288	348	282	374
	R_m (mL CH_4/gSV$_{fed}$/d)	41.7	30.4	48.8	–
	λ (d)	3.30	0.990	3.91	–
	μ max (d^{-1})	–	–	–	0.067
	R^2	0.995	0.948	0.991	0.943
	Average R^2 all samples	0.989	0.986	0.981	0.985

Relationship between pretreatment severity and performance parameters

Most of the studies on thermal hydrolysis of secondary sludge report that it is an effective pretreatment method to improve anaerobic digestion kinetics and methane production from sludge (Wilson & Novak 2009; Oosterhuis *et al.* 2014; Zhang *et al.* 2014). However, the quantification of this improvement is difficult to measure by the sole observation of BMP curves and the information that could be extracted can be inaccurate.

Therefore, Table 5 summarizes for the different operation conditions the prediction parameters calculated from the experimental curves (SF, methane, BD and DF), together with the parameters obtained with the application of the MG (which was the most accurate).

As previously stated, thermal pretreatment improved the anaerobic digestion of the sewage sludge evaluated ('control': 47% biodegradable, 250 mL CH_4/g VS$_{fed}$) by increasing its BD and methane potential for all the conditions tested.

When correlating the main parameters with respect to the severity factor (Figure 4), some interesting behaviors can be observed.

First, the SF increased linearly with the severity as a direct consequence of the cell disruption that takes place during the thermal pretreatment (Dwyer *et al.* 2008), obtaining a solubilization of 40% of the particulate matter at the highest severity factor evaluated (log $R_0 = 4.1$ in TH-14&15). The same results were obtained for the DF (maximum 40% degradation of the particulate matter in TH-14&15), thus meaning that all the organic matter

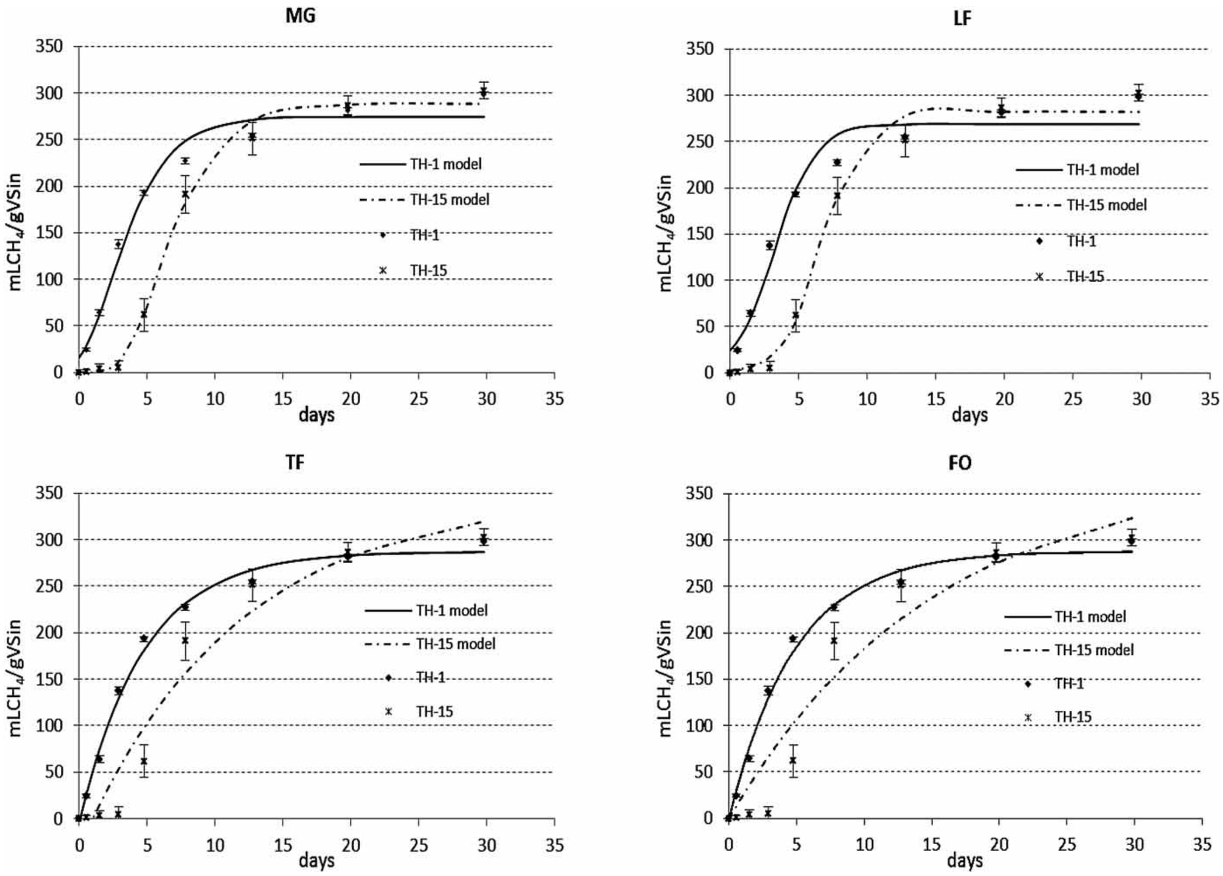

Figure 3 | Experimental (points) and estimated (lines) methane production curves.

Table 5 | Experimental set-up and corresponding results

	Pretreatment				**Anaerobic digestion**			**Kinetics**			
	T °C	***t* min**	**Number of flashes**	**SF %**	**CH_4 mL/gVS**	**BD %**	**DF %**	**P mL CH_4/gVS**	**Rm mL CH_4/gVS/d**	**λ d**	**R^2–**
CONTROL	–	–	0	0	250	47	0	226	37.4	0.10	0.971
TH-1	110	5	2	9	298	56	22	274	43.3	0.08	0.982
TH-2	110	30	1	15	311	59	27	288	55.7	0.24	0.985
TH-3	110	30	3	15	298	57	22	277	45.4	0.41	0.989
TH-4	110	50	2	21	303	57	24	284	49.4	0.21	0.989
TH-5	145	5	1	19	319	60	31	295	54.4	0.27	0.986
TH-6	145	5	3	18	316	60	30	298	51.6	0.32	0.991
TH-7	145	30	2	23	322	61	33	300	60.7	0.35	0.988
TH-8	145	30	2	18	316	60	30	299	51.4	0.44	0.993
TH-9	145	30	2	25	314	59	29	298	50.3	0.51	0.994
TH-10	145	50	1	27	315	60	30	291	57.0	0.28	0.986
TH-11	145	50	3	27	315	60	30	299	52.6	0.42	0.993
TH-12	180	5	2	28	313	59	29	301	48.2	0.89	0.993
TH-13	180	30	1	34	344	65	42	297	59.7	0.69	0.996
TH-14	180	30	3	41	340	64	41	302	41.4	2.05	0.998
TH-15	180	50	2	39	338	64	40	288	41.7	3.30	0.995

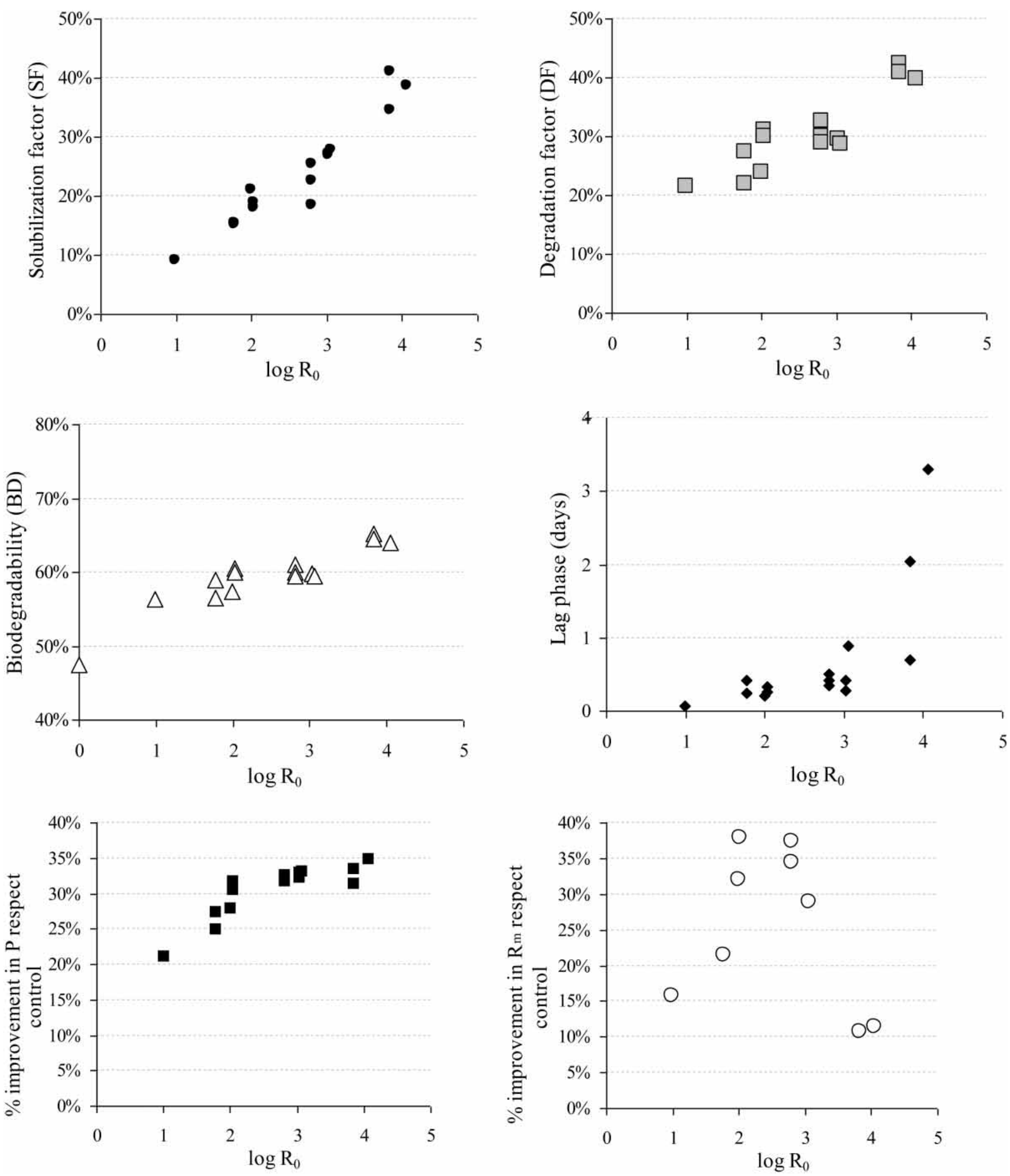

Figure 4 | Relationship between the different performance parameters and the severity factor (log R_0).

solubilized was degraded to methane. Correspondingly, the BD increased linearly, from 47 to 64% at 180 °C–50 min pretreatment (TH-14). This linear link between COD solubilization and methane production is consistent with the results obtained by Carrère *et al.* (2008).

The lag phase increased dramatically from $\lambda = 0$ in the fresh control up to 3.5 days for severity log $R_0 > 3$ (180 °C, $t > 30$ min), as previously presented in Figure 1(c)). It is generally considered that the extreme thermal hydrolysis conditions could lead to producing some slowly biodegradable or non-biodegradable recalcitrant compounds (in most TH cases melanoidins), making slower the multiplication of necessary bacteria and decreasing soluble phase consumption (Dwyer *et al.* 2008; Ariunbaatar *et al.* 2014).

When comparing the improvement in the methane production (mL CH_4/g VSfed) and in the production rate (mL CH_4/gVSfed.d), it can be observed that although the methane production increased with the severity (as previously commented), the methane production rate did not show the same trend, and exhibited an optimum at a severity factor of 3, then decreasing sharply, again pointing to the fact that the most severe pretreatment could lead to the formation of recalcitrant compounds.

An analysis of variance (ANOVA) was done for methane production in order to test the model significance and suitability. The significance of each coefficient was determined using the *F*-value test, at a 95% confidence level.

The results of the variance analysis are presented in Table 6. From there it can be concluded that coefficient for the linear effect of the temperature (A) on methane production is a statistically significant model term at 95% confidence level ($P < 0.05$), thus confirming that only temperature affected the increment of methane production.

Although neither linear effects of the time (B) nor of the flash (C) on the methane production are statistically significant, the linear effect of the time (B) on methane production is more valid than the linear effect of the flash (C).

Based on the model, a maximum methane production 345 mL CH_4/g VS (40% increase) was predicted at conditions: 180 °C, 45 min and 1 flash, with a desirability of 0.882.

CONCLUSIONS

This paper assesses through BMP tests of the influence of different temperature–time–flash thermal hydrolysis pretreatment conditions and combinations on the anaerobic degradation of secondary sludge. All the conditions tested (110–180 °C, 10–50 min, 1–3 flashes) presented higher methane production, exhibiting a maximum improvement of 40% solubilization and subsequent degradation of the particulate matter. Generally, the correlation between the severity of the pretreatment and the performance of the subsequent digestion was linear. However, only temperature showed a positive influence on the methane production, although at extreme thermal hydrolysis conditions, the lag phase increased dramatically, probably due to the formation of recalcitrant compounds. Time and re-flashing exhibited no significant influence.

Table 6 | ANOVA table for the methane production of WAS using TH

Source	Sum of squares	DF	Mean square	*F*-value	*P*-value
A: Temperature	1,953.13	1	1,953.13	48.91	0.0001
B: Time	78.125	1	78.125	1.96	0.1995
C: Flash	50.0	1	50.0	1.25	0.2956
AB	100.0	1	100.0	2.50	0.1522
BB	123.626	1	123.626	3.10	0.1165
CC	74.5728	1	74.5728	1.87	0.2089
Error total	319.481	8	39.9351		
Total (corr.)	2,713.73	14			

Critical value (F_0) for the *F*-test: 5.32 ($F_{0.05,\ 1,8}$).

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