



Research article

Optimization of a thermal hydrolysis process for sludge pre-treatment

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ABSTRACT

At industrial scale, thermal hydrolysis is the most used process to enhance biodegradability of the sludge produced in wastewater treatment plants. Through statistically guided Box-Behnken experimental design, the present study analyses the effect of TH as pre-treatment applied to activated sludge. The selected process variables were temperature (130–180 °C), time (5–50 min) and decompression mode (slow or steam-explosion effect), and the parameters evaluated were sludge solubilisation and methane production by anaerobic digestion. A quadratic polynomial model was generated to compare the process performance for the 15 different combinations of operation conditions by modifying the process variables evaluated. The statistical analysis performed exhibited that methane production and solubility were significantly affected by pre-treatment time and temperature. During high intensity pre-treatment (high temperature and long times), the solubility increased sharply while the methane production exhibited the opposite behaviour, indicating the formation of some soluble but non-biodegradable materials. Therefore, solubilisation is not a reliable parameter to quantify the efficiency of a thermal hydrolysis pre-treatment, since it is not directly related to methane production. Based on the operational parameters optimization, the estimated optimal thermal hydrolysis conditions to enhance of sewage sludge digestion were: 140–170 °C heating temperature, 5–35min residence time, and one sudden decompression.

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1. Introduction

In a conventional wastewater treatment plant, up to 60% of chemical oxygen demand (COD) initially present in the influent is recovered as a mixture of primary and activated sludge (Garrido et al., 2013). A new perspective is being generated due to the increase of the amount of sewage sludge and its disposal limitation by reason of strict environmental regulations, effective methods of making full use of sludge's rich organic components are on continuing interest (Zhang et al., 2014). Anaerobic digestion still appears as the most suitable method to treat the sludge due to its limited environmental impact, high potential for energy recovery as biogas, and reduction in the amount of biosolids to be disposed (Ariunbaatar et al., 2014). It is well known that for solid wastes the hydrolysis (liquefaction or solubilisation) step is the main rate limiting factor for digestion. The introduction of a physical, chemical or biological pre-treatment step before digestion has

demonstrated to improve the global kinetics and performance of the process.

On an industrial scale, the thermal hydrolysis represents the most profitable and reliable alternative (Cano et al., 2015). The main drivers for the use of thermal hydrolysis is the better energy balance due to the increase on biogas production and on better quality of the biosolids produced after digestion, both factors positively affect the operational cost of the treatment plant. From a disposal point of view, it is important to note that the hydrolysed sludge is sterilised at elevated temperatures and complies with the Environmental Protection Agency (EPA) requirements for Class A sludge.

Although nowadays developed at industrial scale (Cambio[®], Biothelys[®], Exelys[®], TPH[®], Lysotherm[®], Turbotec[®]), there is no general agreement on the optimum operation conditions.

Two different mechanisms can be applied to hydrolyse the sludge: i) thermal effect based on the single action of elevated temperatures and ii) steam explosion generated by a sudden decompression (Pérez-Elvira et al., 2008).

According to some researches, heating temperature and duration of the thermal pre-treatment depend on the nature of the

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sludge (Ariunbaatar et al., 2014; Appels et al., 2008). The generally accepted operation conditions vary between 150 and 230 °C temperature, with time ranging from 20 to 60 min. Regarding the steam explosion option, some commercial processes flash the sludge, while other commercial technologies do not use this steam explosion mechanism. There is no bibliographical reference evaluating if flashing or re-flashing (several sudden decompressions) can further enhance the solubilisation of organic matter thereby increasing the methane production. However, the recovery of steam when flashing the sludge can be a clear economic advantage by steam recovery.

The objective of this study was therefore to determine the optimum conditions for thermal hydrolysis of waste activated sludge after studying the combined effect of pre-treatment temperature, time and flash by Response Surface Method (RSM) using the Box-Behnken experimental design (BBD). An optimal combination of factors that maximize methane production is proposed and analyzed.

2. Materials and methods

2.1. Sludge sampling

The study was performed with a single sample of waste activated sludge (WAS), provided by the municipal waste water treatment plant of Valladolid (Spain). According to Pérez-Elvira et al. (2008), the sludge was thickened without polyelectrolyte to 14% TS (73% VS) to perform the study with concentrated sludge, which is the real operation in pre-treatment units.

The anaerobic inoculum for the biochemical methane potential (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 to activate the microorganisms and to deplete most residual organic matter.

2.2. Thermal pre-treatment procedure

The thermal hydrolysis pilot plant operated (Fig. 1) consisted of a 20 L hydrolysis reactor heated with live steam (12 bars) from a boiler, and connected to an atmospheric flash vessel (100 L) by a decompression valve. The operation is batch, controlling heating temperature and time. The decompression is also controlled by an automatic decompression valve, that relieves the reactor pressure slowly (no flashing) or suddenly (in a steam-explosion effect). When the flash takes place, the hydrolysed solids are collected in the flash vessel at atmospheric pressure.

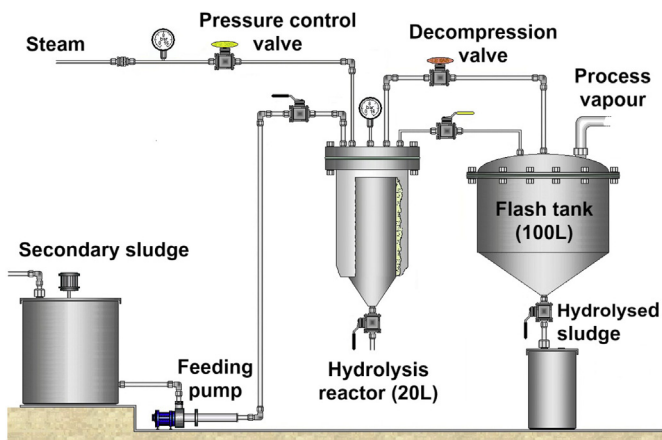


Fig. 1. Thermal pre-treatment system (Fernández-Polanco et al., 2008).

2.3. Anaerobic digestion tests

Biochemical methane potential (BMP) tests were performed in triplicate assays using 300 ml serum bottles, filled with 100 ml of a mixture of anaerobic inoculum and the corresponding substrate at a substrate to inoculum ratio (SIR) of 0.5 g/g (on volatile solids (VS) basis). In this test, micronutrients and macronutrients were added ensuring no nutritional limitation for optimal function of anaerobic microorganisms. Moreover, NaHCO₃ and Na₂S were added to provide buffer capacity and avoid aerobic conditions respectively. The methodology used was based on the one suggested by Angelidaki et al. (2009).

The bottles were incubated in a thermostated chamber at 35 °C in an orbital shaker at 150 rpm/min. Methane production in the BMP tests was determined by periodic measurements of pressure and biogas composition.

2.4. Analytical methods and performance parameters

Total solids (TS) and volatile solids (VS) concentrations were determined according to Standard Methods (21st edition, 2005). The soluble phase for chemical oxygen demand (SCOD) was obtained by centrifugation at 5000 rpm for 10 min and filtration using a 47 mm hydrophilic Glass Fiber filter with a 0.7 μm pore size (AP40). The total COD was obtained by a direct COD analysis.

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 to a thermal conductivity detector (Varian CP-3800, USA).

Two performance parameters were calculated: solubilisation and methane production increase.

The solubilisation factor (SB) (Equation (1)) was calculated with respect to the particulate fraction of the chemical oxygen demand (TCOD-SCOD), in contrast to the most of the references (Jung et al., 2015; Kim et al., 2015; Appels et al., 2010) that express this parameter with respect to the total TCOD. This proposed expression is more accurate as the particulate matter is the potentially hydrolysable fraction during the pre-treatment.

$$\%SB = \frac{(SCOD/TCOD)_{TH} - (SCOD/TCOD)_0}{((TCOD - SCOD)/TCOD)_0} \times 100 \quad (1)$$

The specific methane production was evaluated according to Equation (2):

$$CH_4 = \frac{mLCH_4}{gVS_{fed}}, \text{ at standard conditions (0 °C, 1 atm)} \quad (2)$$

And the performance of the digestion was calculated by comparing the CH₄ values obtained for the treated samples with respect to the untreated WAS, calculating the increase in methane production (Equation (3)):

$$\%CH_4 = \frac{(CH_4)_{TH} - (CH_4)_0}{(CH_4)_0} \times 100 \quad (3)$$

2.5. Experimental design

In order to statistically and mathematically determine the optimal conditions of the key operational conditions and the effects of their interactions on the global efficiency of the process, RSM with Box-Behnken experimental Design BBD (Benito-Roman et al., 2013; Jung et al., 2015; Sarat Chandra et al., 2014) was used in this work.

Three factors at two levels were considered: temperature (130 and 180 °C), time (5 and 50 min) and type of decompression (0 and 2). The value 0 means that the reactor pressure was relieved slowly, and the hydrolysed sludge was cooled directly without leaving the reactor. Value 2 indicates that the treated sludge was re-flashed twice (just decompressed, not heated twice).

The resulting experimental plan consisted of 15 runs (see Table 1), including three repetitions at the center point of the experimental design.

In order to develop the regression equation, the relationship between the coded values and actual values are described according to the following Equation (4):

$$x = \frac{x_j - x_0}{\Delta x} \quad (4)$$

where x is the coded value, x_j is the corresponding actual value, x_0 is the actual value in the center of the domain, and Δx is the increment of x_j corresponding to a variation of one unit of x .

The relationship between the variables and responses was correlated with a quadratic polynomial Equation (5), that was fitted as follows:

$$Y = \beta_0 + \sum_{j=1}^k \beta_j X_j + \sum_{j=1}^k \beta_{jj} X_j^2 + \sum_{i=1}^k \sum_{j=1}^k \beta_{ij} X_i X_j \quad (5)$$

where Y is the estimated response variable to be optimized, X_j are the variables evaluated, β_0 is the constant, β_j is the linear coefficient, β_{jj} is a quadratic coefficient and β_{ij} is the interactive coefficient.

An analysis of variance (ANOVA) with 95% confidence level was done for response variable 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.

3. Results and discussion

3.1. Experimental set-up and results

Table 1 summarizes the experimental set-up together with the results obtained for the response variables evaluated (solubilisation and methane production). The CONTROL corresponds to the untreated WAS.

From these values, the effect of heating temperature, hydrolysis time and type of decompression (number of flashes) is analyzed. It

Table 1
Box-Behnken experimental design for the thermal hydrolysis and experimental results.

Trials	Pre-treatment			Results		
	T (°C)	t (min)	No. of flashes	SB (%)	CH ₄ (mL/gVS)	ΔCH ₄ (%)
CONTROL	–	–	–	0	220	–
TH-1	150	50	0	40%	318	45%
TH-2	130	50	1	34%	320	45%
TH-3	130	5	1	30%	316	44%
TH-4	180	30	2	39%	339	54%
TH-5	150	5	2	31%	312	42%
TH-6	180	5	1	39%	325	48%
TH-7	180	50	1	39%	340	55%
TH-8	150	30	1	36%	336	53%
TH-9	150	30	1	35%	361	64%
TH-10	150	30	1	37%	378	72%
TH-11	180	30	0	41%	312	42%
TH-12	150	50	2	38%	335	52%
TH-13	150	5	0	31%	300	36%
TH-14	130	30	0	33%	276	25%
TH-15	130	30	2	33%	296	35%

can be noticed that all the pre-treatments evaluated increase the solubilisation and the methane production with respect to the untreated WAS. The results range between 30 and 41% solubilisation increase and 25–72% increase in methane production. However, and as will be analyzed, no direct correlation was observed between both responses.

3.2. Effect of TH variables on the WAS solubilisation

Fig. 2 represents graphically the influence of the three variables on the waste activated sludge solubilisation. The results clearly exhibit that the increase in sludge solubility is mainly affected by temperature and time, in a rather linear trend: solubilisation is enhanced as the pre-treatment temperature and time increase. The maximum increase in soluble COD is therefore obtained in the most extreme operating conditions (180 °C and 50 min heating).

On the other hand, the decompression effect can be considered negligible.

From the analysis of variance evaluation, presented in Table 2, the obtained coefficients for the linear effect of the temperature (A) and time (B) on the WAS solubility are statistically significant model terms at 95% confidence level ($p < 0.05$). These results confirm that the temperature and time have a major effect on the sludge solubilisation during the thermal hydrolysis process.

Based on the ANOVA results, Equation (6) was obtained using Equation (5) to fit the experimental data of the sludge solubilisation, being X_1 the heating temperature, X_2 the hydrolysis time and X_3 the decompression mode.

$$SB(\%) = 2.29409 + 0.188889 * X_1 + 0.521411 * X_2 + 0.111111 * X_3 - 0.00177778 * X_1 * X_2 - 0.00204586 * X_2^2 - 0.0222222 * X_2 * X_3 \quad (6)$$

The model was considered valid because the criteria $r^2 > 0.8$ was satisfied ($r^2 = 0.878$) (Bup Nde et al., 2012). Fig. 3 presents the three-dimensional response surface plot and two dimensional contour plot for the solubilisation factor (%SB) obtained from Equation (6) for the interaction between temperature and time (sudden decompression in 1 flash was maintained constant).

The graphs reveal that for the evaluated operation ranges of heating temperature and time, the more severe intensity of the thermal pre-treatment (higher temperature and longer times), the higher the solubilisation achieved. These results are in agreement with those obtained by other authors confirming that solubilisation efficiency increase proportional to the temperature rise. Higher pre-treatment temperatures enhance extracellular polymeric substances (EPS) solubilisation and cell lysis (Zhang et al., 2015;

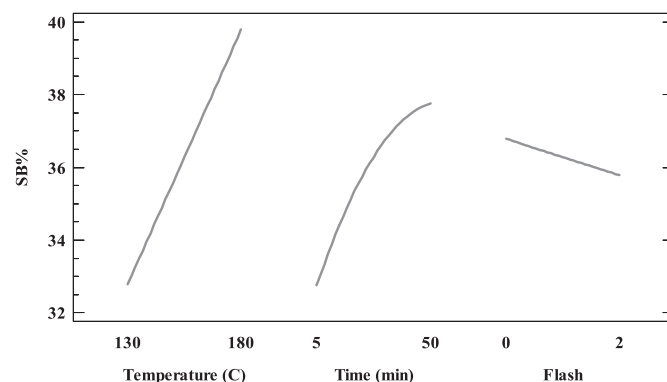


Fig. 2. Influence of the factors evaluated on WAS solubilisation.

Table 2
ANOVA table for WAS solubility after thermal pre-treatment.

Source	Sum of squares	DF	Mean square	F-value	P-value
A:Temperature	98.0	1	98.0	35.75	0.0003
B:Time	50.0	1	50.0	18.24	0.0027
C:Flash	2.0	1	2.0	0.73	0.4178
AB	4.0	1	4.0	1.46	0.2615
BB	4.0	1	4.0	1.46	0.2613
BC	1.0	1	1.0	0.36	0.5626
Error total	21.9	8	2.7		
Total (corr.)	180.9	14			

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

Seongyeob et al., 2014; Xue et al., 2015).

It is also interesting to observe that same solubilisation is obtained for the thermal treatment at high temperature – short time, and lower temperature – higher time (i.e.: 36% SB at 170 °C-10 min and 140 °C-50 min).

3.3. Effect of TH variables on the methane production

The methane production was compared with the production obtained for the control of untreated sludge (220 ml CH_4/VS_{fed}) following the same evaluation methodology followed for the solubilisation: separate influence of variables, analysis of variance and response plots.

Fig. 4 shows that methane production is affected by all the factors experimentally quantified. In contrast to the results previously obtained for solubilisation, the trend is not linear for the ranges evaluated, and an optimum is obtained for each factor analyzed. First, when temperature increased from 130 °C to 160 °C, the methane production increased deeper and later started to decrease. The same trend was obtained for the treatment time, exhibiting a decrease in the methane enhancement for values higher than 30 min. Finally, a single sudden flash is the optimum decompression alternative.

Results of the variance analysis are presented in Table 3, exhibiting that coefficients for the linear effect of the temperature (A) and for the squared effects of the temperature (AA) and flash (CC) on methane production are statistically significant model terms at 95% confidence level ($p < 0.05$). Although not statistically significant, the linear effect of the flash (C) on methane production appears to be more valid than the linear effect of the time (B).

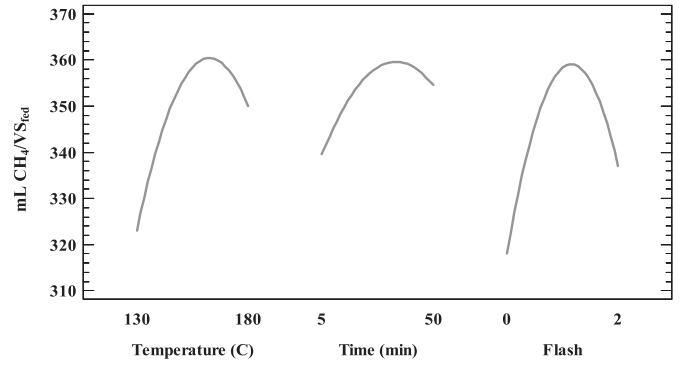


Fig. 4. Influence of the factors evaluated on methane production.

Based on the ANOVA results, Equation (7) was obtained using Equation (5) to fit the experimental data of the methane production, being X_1 the heating temperature, X_2 the hydrolysis time and X_3 the decompression mode.

$$CH_4 \left(mL/VS_{fed} \right) = -629.364 + 11.3487 * X_1 + 1.56008 * X_2 + 71.0833 * X_3 - 0.0348667 * X_1^2 - 0.0223045 * X_2^2 - 30.7917 * X_3^2 \quad (7)$$

The model was considered valid with the same criteria as the previous model for WAS solubilisation, being $r^2 = 0.851$ in this case. The three dimensional response surface plot and two dimensional contour plot shown in Fig. 5 describe the interaction between

Table 3
ANOVA table for the methane production of WAS after thermal pre-treatment.

Source	Sum of squares	DF	Mean square	F-value	P-value
A:Temperature	1458	1	1458	8.61	0.0189
B:Time	450	1	450	2.66	0.1417
C:Flash	722	1	722	4.26	0.0728
AA	1753	1	1753	10.35	0.0123
BB	470	1	470	2.78	0.1340
CC	3500	1	3500	20.67	0.0019
Error total	1354	8	169		
Total (corr.)	9098	14			

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

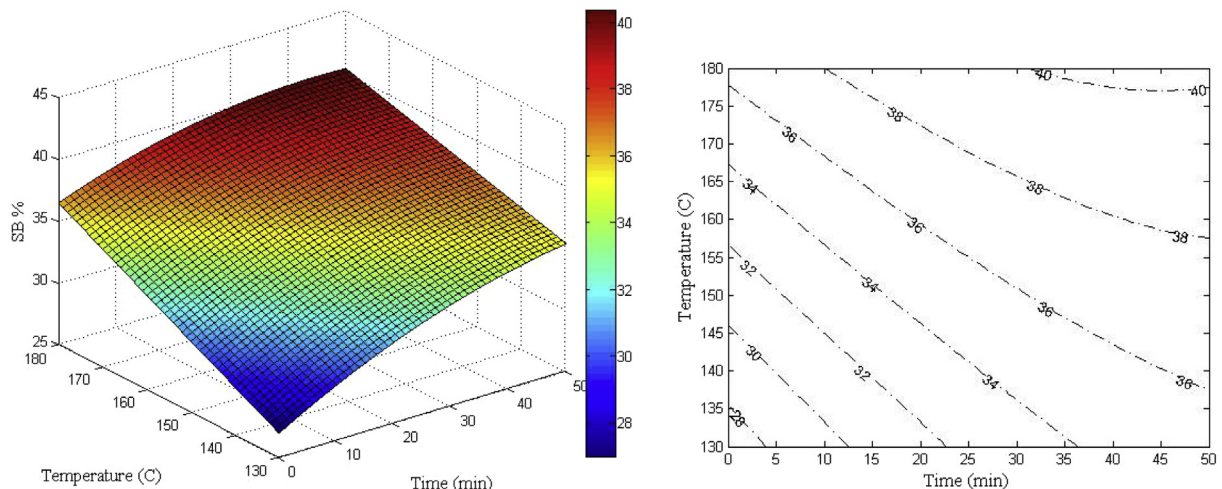


Fig. 3. Response surface plot and contour plot showing the effects of temperature and time on WAS solubilisation.

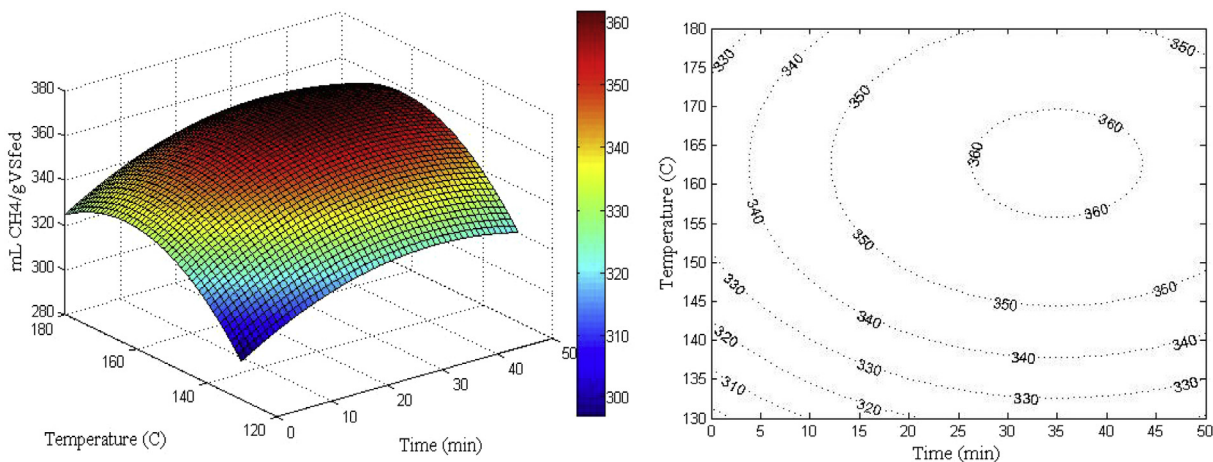


Fig. 5. Response surface plot and contour plot showing the effects of temperature and time on methane production.

thermal pre-treatment temperature and time (again, sudden decompression in 1 flash was maintained constant). These graphs can be used to predict the optimum level of each factor to obtain the maximum expected value for methane production (Sarat Chandra et al., 2014).

Both plots exhibit a peak value for methane production, not corresponding to the maximum values of pre-treatment temperature and time, in contrast to the results obtained for solubilisation. In fact, the negative quadratic effect of temperature and time obtained in Equation (7) implies that, at high severity of the pre-treatment, the methane production decreases (Bup Nde et al., 2012). These results agree with those studies reporting inhibition by formation of refractory compounds at high temperatures, generally stated over 170 °C (Bougrier et al., 2008; Dwyer et al., 2008). This formation of complex substances can also occur at lower temperatures for longer pre-treatment time (Elliot and Mahmood, 2012) as also obtained in Fig. 5.

Fig. 5 also reveals that thermal hydrolysis allows operating in a wide range of conditions reaching similar methane production. In our experiment the highest specific production of 360 mLCH₄/gVS_{fed} can be obtained in the range 155–175 °C and 25–45 min. This wide range clearly demonstrates that TH is a robust process which does not require a very strict and sophisticated operation control system. Expanding the working field until a specific methane productivity 5% lower than the maximum (340 mLCH₄/

gVS_{fed}), the operational range can be extended to 145–170 °C and 5–45 min, with corresponding savings in energy consumption.

Finally, other relevant technical aspect to be mentioned is that at higher pre-treatment temperature, shorter reaction time is required. The contour plot (Fig. 5) shows that it is possible to achieve identical specific productivity (340–350 mLCH₄/gVS_{fed}) operating at 140–145 °C during 20–35 min or at 160–170 °C for 5–10 min. These results are consistent with those obtained by Zhang et al. (2014) and Hii et al. (2014).

3.4. Optimization of the TH process

The previous discussion of results for solubilisation and methane production show that the evaluated parameters (heating temperature, time and decompression mode) do not influence both responses in the same way, and the trend followed is not the same.

First, the experimental results presented in Table 1 show that solubilisation and methane enhancement do not present a link. For example, a solubilisation around 30% can lead to a methane increase in the range of 25–44%, and 40% solubilisation is responsible for 42–55% methane increase.

Second, based on the models, the optimal values for solubilisation and methane production can be compared. A maximum methane production of 362 mLCH₄/gVS_{fed} was predicted at the optimum conditions (163 °C, 35 min and 1 flash) while a maximum

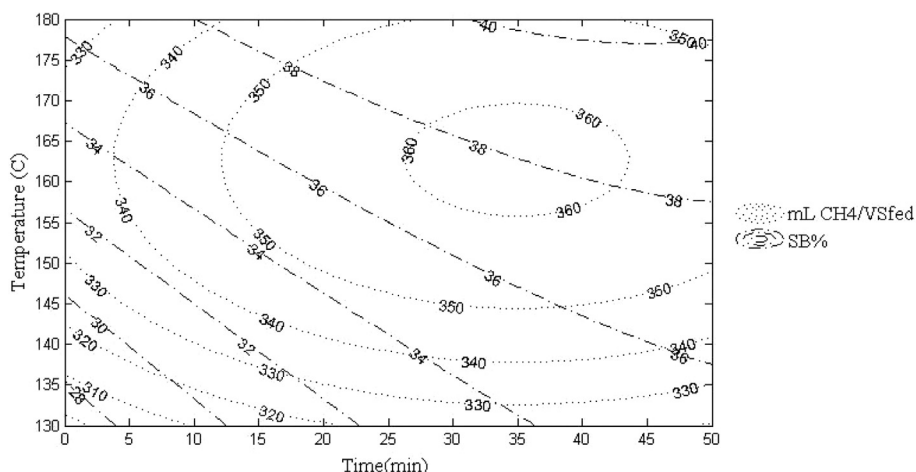


Fig. 6. Superimposed contour plots for methane production and solubilisation.

solubilisation of 41% was achieved at higher operation conditions (180 °C, 49 min and 1 flash). The increase of WAS solubilisation and parallel decrease on methane production at high pre-treatment temperature and long times, indicate that some soluble but non-biodegradable compounds were produced during severe thermal pre-treatment. This is probably because of the production of melanoidins, as reported by many studies (Ariunbaatar et al., 2014; Xue et al., 2015).

Third, similar thermal hydrolysis efficiency (more than 50%) could be obtained working at a wider process conditions: at 140–170 °C for 5–35 min respectively. These results are very interesting in order to minimize energy requirement of thermal hydrolysis process.

Finally, surface plots and contour plots for sludge solubilisation (Fig. 3) and methane production (Fig. 5) can be equated in order to compare the trend and optimal values for temperature and time. The contour plot curves of solubilisation and methane production were superimposed (Fig. 6) to exhibit that there is no joint optimum thermal hydrolysis conditions for both variables.

From Fig. 6 it can be clearly concluded that there is no common peak for both variables, meaning that thermal hydrolysis conditions for solubilisation do not correspond to maximum methane production. This conclusion approves that solubilisation is not a trustworthy parameter for thermal hydrolysis productivity.

The translation of these results from a practical point of view is that the solubilisation measurement is not a suitable prediction parameter to quantify thermal hydrolysis efficiency. The increase in solubilisation obtained at high intensity pre-treatment is not always associated with increase in methane production.

4. Conclusions

Thermal hydrolysis of waste activated sludge is a robust pre-treatment, leading to significant improvements in anaerobic digestion (up to 70% increases in methane production) operating in a wide range of experimental conditions. The key mechanisms and variables of the process are: heating (temperature and time) and decompression mechanism (slow or steam explosion). The statistical analysis performed exhibited that efficiency of the pre-treatment is mainly influenced by temperature, while the reaction time has a lower incidence. And there is no direct correlation between the solubilisation achieved and the methane production increase. While solubility increases sharply at high intensity pre-treatment (high temperature and long times), the methane production presents an optimum, that when exceeded, recalcitrant compounds may appear. Consequently, it is not recommended the use of solubilisation as a prediction parameter to quantify pre-treatment efficiency, since it is not related to methane production.

An increase in methane production exceeding 50% was obtained by a steam explosion pre-treatment performed in the range 140–170 °C heating and 5–35 min. Therefore, an exhaustive control of thermal pre-treatment conditions appears to be not necessary, while the selection of operation conditions play a key role on the process economics.

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