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Increased temperature in the thermophilic stage in temperature phased anaerobic digestion (TPAD) improves degradability of waste activated sludge

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ABSTRACT

Two-stage temperature phased anaerobic digestion (TPAD) is an increasingly popular method to improve stabilisation of sewage waste activated sludge, which normally has inherently poor and slow degradation. However, there has been limited systematic analysis of the impact of the initial thermophilic stage (temperature, pH and retention time) on performance in the main mesophilic stage. In this study, we demonstrate a novel two-stage batch test method for TPAD processes, and use it to optimize operating conditions of the thermophilic stage in terms of degradation extent and methane production. The method determines overall degradability and apparent hydrolysis coefficient in both stages. The overall process was more effective with short pre-treatment retention times (1–2 days) and neutral pH compared to longer retention time (4 days) and low pH (4–5). Degradabilities and apparent hydrolysis coefficients were 0.3–0.5 (fraction degradable) and 0.1–0.4 d^{-1}, respectively, with a margin of error in each measurement of approximately 20% relative (95% confidence). Pre-treatment temperature had a strong impact on the whole process, increasing overall degradability from 0.3 to 0.5 as temperature increased from 50 to 65 °C, with apparent hydrolysis coefficient increasing from 0.1 to 0.4 d^{-1}.

1. Introduction

Modern wastewater treatment plants focus on biological nutrient removal. To achieve enhanced nitrification and denitrification, primary sedimentation is commonly removed, and sludge age extended. This has resulted in producing greater volumes of waste activated sludge, with poorer degradability compared to activated sludge treatment for carbon removal only. Anaerobic digestion is widely used for biological treatment of activated sludge. However, long sludge age activated sludge is inherently non-degradable due to accumulation of inerts and decay byproducts [1], and results in poor solids destruction and low methane production during sludge digestion. Pre-treatment methods enhance sludge degradation rate or extent, and can facilitate application of anaerobic digestion. A leading option is thermophilic (50–70 °C) pre-treatment prior to mesophilic anaerobic digestion (temperature phased anaerobic digestion, or TPAD), which has a number of advantages over alternatives (e.g. ultrasonication, thermal hydrolysis, and alkaline or acidic pre-treatment), including low capital expense, low operating expenses and the use of low quality thermal energy instead of electrical energy or industrial chemicals [2].

Given the changing nature of sludge stabilisation, substantial effort is going into investigation of TPAD processes. Ge et al. [3] investigated TPAD for primary sludge (50 °C, 2 days hydraulic retention time (HRT) and 35 °C, 14 days HRT) and found TPAD could achieve 54% VS destruction compared to a mesophilic–mesophilic control which could only achieve 44% (35 °C, 2 days HRT and 35 °C, 14 days HRT). TPAD processes have successfully been used to treat activated sludge where pre-treatment at 60 °C for 4 days [4] or 2–4 days [5] resulted in improvements in biogas production of 100% or 26–50%, respectively.

Nges and Liu [6] tested the effects of pre-treatment temperatures (25–70 °C) on overall degradability in TPAD. The sewage sludge solubilisation in the pre-treatment stage (2 days HRT) was greatest at 50 °C (22.5%), higher than that at 25 °C (11.6%) and 70 °C (21.7%). The peak performance in the subsequent methanogenic stage was 42% VS destruction and 284 $\mathrm{mL\,gVS_{added}}^{-1}$ achieved by feeding sludge pre-treated at 50 °C and 2 days HRT.

The effects of pre-treatment HRT were also tested in the study of Nges and Liu [6], which showed the sludge solubilisation was enhanced from 13% to 21% when increasing HRT from 0.5 to 3 days, respectively (50 °C). A similar experiment was conducted by Bolzonella et al. [7] to optimise the thermophilic stage with 1–5 days HRT at 70 °C using activated sludge. The maximum solubili-

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Table 1
Characteristics of the substrate used in this study.

Characteristic	Substrate
TS ($g\,L^{-1}$)	26 (0.2)
VS ($g\,L^{-1}$)	18 (0.1)
pH	7–7.5
COD ($g\,L^{-1}$)	28 (4)
VFA ($g\,L^{-1}$)	0.2 (0.1)

Standard deviation across 4 different activated sludge samples collected over a 10-month period shown in parenthesis.

sation was obtained at 5 days HRT (43%). Subsequent batch tests showed around 55% increase in methane production over the base mesophilic level (37 °C).

These studies highlight the potential of TPAD, with reasonable gains available in terms of both solids destruction (32–54%) and gas production. However, the focus of previous work has been on the effect of thermophilic temperature and HRT on the solubilisation during the thermophilic stage, and systematic analysis of both the pre-treatment stage and the methanogenic stage has been limited. There is little systematic analysis to evaluate another experimental variable in the pre-treatment stage, pH, which may have an impact on the sludge solubilisation. Most studies have also been focused on performance (e.g., solubilisation and methane yield), rather than inherent changes in sludge material properties. There are two key measures of sludge properties that are relevant – degradability extent (f_d) and apparent first order degradation rate coefficient (k_{hyd}), which indicate the extent and speed of sludge conversion under ideal conditions [8]. Improved performance alone does not indicate whether either, or both of these have been improved. In addition, most studies have been conducted in continuous reactors, which are highly relevant, but require long operational periods, have inherently limited parameter identifiability, and are expensive to run. Batch testing would provide a low cost method to assess individual materials [9].

In this paper, a novel two-stage batch test is presented involving independent thermophilic pre-treatment and mesophilic methanogenic stages. This is then used in to systematically analyse the impact of pre-treatment conditions (temperature, pH and retention time) on both solubilisation, and performance during subsequent anaerobic digestion.

2. Materials and methods

Experiments in this study consisted of two stage batch tests, with the temperature and duration of each stage being different in order to represent a TPAD process. Inocula for each stage were harvested from continuous parent digesters enriched at the corresponding test temperature and approximate HRT. For example, a two-stage 60–37 °C test used two different inocula from parent reactors enriched at those respective temperatures. The two-stage test consisted of a batch thermophilic pre-treatment (Stage 1), conducted at different conditions (50, 60, 65, 70 °C), pH (4, 5, 6, 7) and retention time (1, 2, 4 days); and a subsequent mesophilic digestion (Stage 2), conducted uniformly at 37 °C.

2.1. Substrate

The substrate used in this study was biological nutrient removal (BNR) sludge, collected from an activated sludge BNR plant with 10 days sludge age and water temperature of approximately 20 °C, located at Gold Coast, Australia. Sludge was collected at intervals of 1–2 months, and was settled using a centrifuge to increase solids concentration and stored at below 4 °C. The average characteristics of the substrate are shown in Table 1.

2.2. Inoculum

2.2.1. Thermophilic inoculum (Stage 1)

Thermophilic inoculum was harvested from a continuous 2 L lab-scale reactor. The thermophilic parent reactor was originally inoculated from a lab-scale mesophilic anaerobic digester (approx. 14 days HRT, 35 °C), which was operated as the second stage in a TPAD process treating activated sludge for over 12 months. The thermophilic parent reactor was operated at a HRT of 2 days with a feed of 1 L of activated sludge per day and fixed volume. Feed intervals were 4 h (6 times daily). The temperature in the reactor was maintained using a temperature controlled water jacket. The reactor was continually mixed using a magnetic stirring bar. The volume and quality (H_2, CH_4, CO_2) of biogas produced were recorded. Liquid samples were periodically collected and analysed to monitor reactor performance.

The thermophilic parent reactor was operated over 10 months. During this time there were 4 operating temperatures 50 °C (103 days), 60 °C (100 days), 65 °C (67 days) and 70 °C (34 days). At each operating temperature, thermophilic inoculum was collected after the reactor performance stabilised, which was exhibited by stable VS destruction of 16% over 103 days, 24% over 100 days, 22% over 67 days and 20% over 34 days achieved for each period (based on the mass balance and standard measurement method described below). The average characteristics of thermophilic inoculum at each temperature are summarised in Table i (Supplementary data).

2.2.2. Mesophilic inoculum (Stage 2)

Mesophilic inoculum was harvested from a continuous 4 L lab-scale reactor, which was operated as the second stage in a TPAD process (approx. 14 days HRT, 35 °C). The lab-scale TPAD process was operated for over 15 months treating BNR sludge, collected from the same plant where provided the substrate for thermophilic parent reactor stated above. During the operating period, the first stage of the TPAD configuration was varied (50–70 °C, 2 days HRT), while the second stage was operated consistently (35 °C, 14 days HRT). Mesophilic inoculum was harvested only when the overall TPAD process was achieving stable operation, which was also reflected by stable VS destruction at 34% over 103 days, 41% over 100 days, 48% over 67 days and 47% over 34 days during each different pre-treatment period (measured as described in thermophilic inoculums above). The average characteristics of mesophilic inoculum at each operating period are summarised in Table ii (Supplementary data).

2.3. Set two-stage batch test

2.3.1. Stage 1 (thermophilic pre-treatment)

Stage 1 of batch tests were performed in 160 mL non-stirred glass serum vials (80 mL working volume). Each test contained 40 mL inoculum and 40 mL substrate. Bottles were flushed with high purity N_2 gas for 3 min ($1\,L\,min^{-1}$), sealed with a butyl rubber stopper retained with an aluminum crimp-cap and stored in temperature controlled incubators (±1 °C) at 50, 60, 65, and 70 °C, respectively. Blanks contained inoculum and MilliQ water without substrate. Stage 1 batch tests were conducted at 4 thermophilic temperatures, at each temperature there were 7 test conditions, varying Stage 1 (pre-treatment) pH, and retention time. Thus in total, two-stage batch tests were conducted at 28 test conditions, consisting of individual pre-treatment and methanogenic batch tests at temperatures of 50–70 °C and 37 °C. When varying pre-treatment pH, retention time in Stage 1 was maintained at 2 days, while the initial pH in Stage 1 was not controlled (around pH 7) while varying pre-treatment retention time. A summary of test conditions is shown in Table iii (Supplementary data). The pre-treatment pH was adjusted to the initial set point using 1 M HCl.

Three independent batch tests were conducted for each test conditions, and error bars shown are based on 95% confidence in the triplicate values.

2.3.2. Stage 2 (mesophilic digestion)

Stage 2 of batch tests were effectively biological methane production (BMP) tests to determine biogas production, degradability extent and degradability rate of sludge pre-treated in Stage 1. A 20 mL sample was withdrawn from the bottles at the end of the Stage 1, and directly transferred to a 160 mL serum bottle with 60 mL mesophilic inoculum described previously. Bottles were flushed and sealed using the same procedure described for Stage 1, and placed in a temperature controlled incubator at 37 °C (±1 °C). Triplicate blanks were carried out containing mesophilic inoculum and MilliQ water.

2.4. Chemical analysis

Biogas volume was measured by manometer at the start of each sampling event. Accumulated volumetric gas production was calculated from the pressure increase in the headspace volume (80 mL) and expressed under standard conditions (25 °C, 1 atm) [10]. At each sample event, the biogas quality (CH_4, CO_2, H_2) was determined using a Perkin Elmer autosystem gas chromatograph equipped with thermal conductivity detector (GC-TCD). In Stage 1, where pH was varied, gas production was monitored twice daily. Where retention time was varied, gas production was monitored five times for retention time of 1 day, and twice daily for retention time of 2 and 4 days. In Stage 2, gas production was monitored daily until production stopped. The net gas production was obtained by subtracting gas production of the blanks, which was between 20% and 26% of the total methane production from the combined two-stage test.

At the start and end of each stage, the substrate, inoculum and combined slurry samples were analysed for total chemical oxygen demand (COD(T)), soluble COD (COD(S)), total solids (TS), VS, ammonia (NH_4^+) and volatile fatty acid (VFA). All analytical methods were performed according to Standard Methods [11], and as previously described in Ge et al. [3].

2.5. Extent of solubilisation calculation

Sludge solubilisation in Stage 1 was calculated as the ratio of total solubilised products and the particulate feed solids concentration [12]. It can be expressed as

$$\text{Solubilisation}\ (\%) = \frac{\text{COD}_{\text{CH}_4} + \text{COD(S)}_\text{o} - \text{COD(S)}_\text{i}}{\text{COD(T)}_\text{i} - \text{COD(S)}_\text{i}} \times 100 \tag{1}$$

where COD_{CH_4} is methane production as COD equivalents, COD(S)_i and COD(S)_o are COD(S) concentration in the influent and effluent of Stage 1, and COD(T)_i is COD(T) concentration in the influent of Stage 1.

2.6. Mathematic analysis

2.6.1. Model implementation and inputs

A two-stage anaerobic model was implemented in Aquasim 2.1d [13]. This model is available in supplementary information as an Aquasim 2.1d model (.aqu). The processes involved in this model included the pre-treatment stage (Stage 1), transfer of a portion of substrate to the mesophilic stage (Stage 2), and removal of the remaining substrate for analysis. The model incorporated both stages to facilitate continuity between the stages representing a TPAD process and allow consistent determination of process kinetics across both stages. The variables were defined according to the states of liquid and gas in the batch tests (e.g. volume, pressure, concentration, time, etc.). The initial condition used for the model estimation and simulation was COD(T) of substrate. The main output was methane flow on a COD basis.

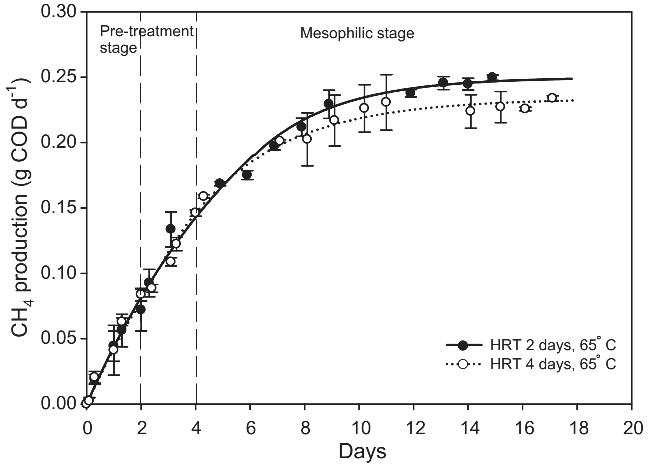

Fig. 1. Cumulative methane production from two-stage batch tests using thermophilic pre-treatment at retention times of 2 and 4 days, 65 °C. Error bars are 95% confidence intervals based on triplicate analyses. Lines are model output.

2.6.2. Parameter estimation and analysis

The degradability extent (f_d) and apparent first order hydrolysis rate coefficient (k_{hyd}) were the key parameters used to assess and optimise the batch TPAD process [14]. In each case, apparent hydrolysis rates in Stage 1 (k_{hyd1}) and Stage 2 (k_{hyd2}) and f_d were simultaneously estimated to achieve the optimal values. The two parameter surface for k_{hyd1} and f_d was determined by first setting the optimal value for k_{hyd1} and using the method of Batstone et al. [14,15] for a two-parameter system. Similarly, a two parameter surface for k_{hyd2} and f_d was determined by fixing k_{hyd2} at the optimal value. Parameter uncertainty was expressed by a 95% confidence region in both parameters, fully accounting for parameter-objective non-linearity and correlation. Appropriate F values were used for two parameters and the number of degrees of freedom. A modified version of Aquasim 2.1d was used to determine the parameter surfaces, and simulate the methane flow over both stages of the two-stage test. Methane flow (COD basis) was used as the fit objective, with residual sum of squares (RSS) as the objective function (J).

3. Results

3.1. Effect of pre-treatment retention time and temperature

Characteristic methane production curves are shown in Fig. 1. This indicates methane production over time from the batches, with a vertical line indicating transfer from Stage 1 to Stage 2. As indicated in Fig. 1, methane production is continuous from the first stage to the second. These two methane production curves overlap, and as would be expected, the parameter confidence regions also overlap (Fig. 2). Fig. 2 shows the confidence regions of k_{hyd1}, k_{hyd2} and f_d for the range of temperatures at 50, 60, 65, and 70 °C at each retention time. The optimal values of k_{hyd2} and f_d in the mesophilic stage are summarized in Table iv (Supplementary data). Increased temperature in the pre-treatment stage consistently resulted in better degradability across both stages. However, increased temperature produced a slower process in the pre-treatment stage, and a faster process in the second mesophilic stage (based on gas flow and model estimations). Pre-treatment retention time had a much lower impact

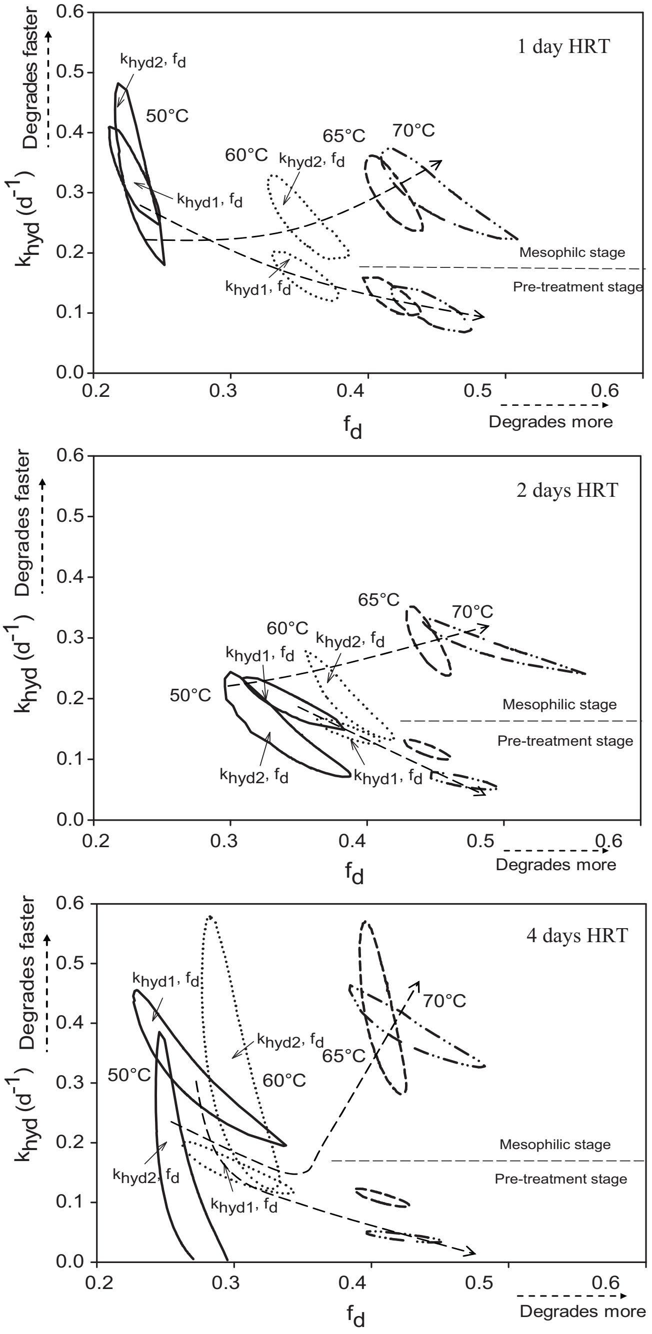

Fig. 2. Confidence regions of k_{hyd1} and f_d (pre-treatment stage) and k_{hyd2} and f_d (mesophilic stage) for thermophilic pre-treatment at different retention times at 50 °C, 60 °C, 65 °C, and 70 °C.

than pre-treatment temperature, with no real changes in confidence region locations from 1 day to 4 days retention time. There was an increase in confidence region area at longer pre-treatment retention time, caused by more methane production in the pre-treatment stage.

The extent of solubilisation and product composition during the pre-treatment stage using different retention times and temperatures are shown in Fig. 3. The extent of solubilisation during pre-treatment did not appear to be influenced by extending the retention time from 1 to 4 days. However, the profile of solubilisation products was influenced by retention time, in tests using shorter retention times (1 or 2 days), there was a greater accumulation of intermediate products (e.g. VFAs) and lower methane production than the tests with longer pre-treatment retention times (4 days).

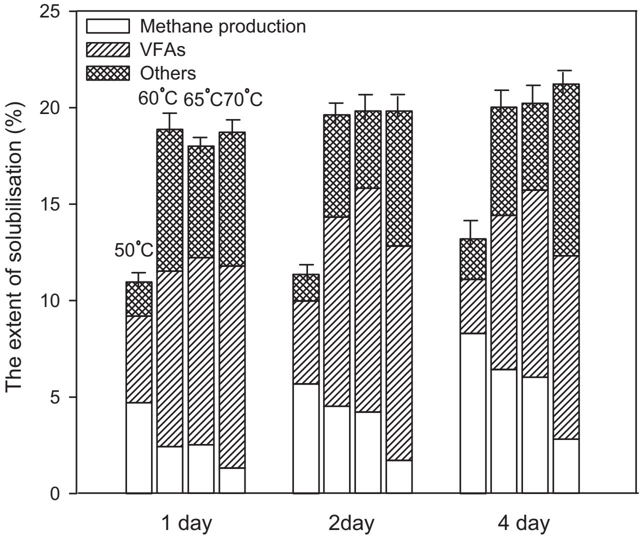

Fig. 3. The extent of solubilisation in the pre-treatment stage after retention times of 1, 2 and 4 days at 50, 60, 65 and 70 °C. Error bars are 95% confidence in mean methane production and VFA concentrations.

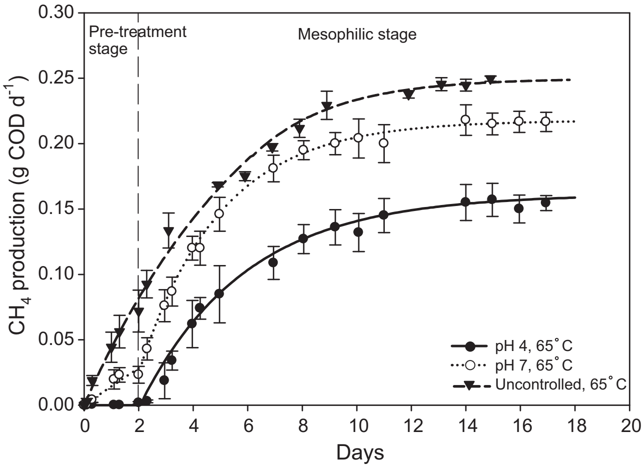

Fig. 4. Cumulative methane production from two-stage batch tests using thermophilic pre-treatment at pH 4, 7 and uncontrolled, 65 °C. Error bars are 95% confidence intervals based on triplicate analyses. Lines are model output.

3.2. Effect of pre-treatment pH and temperature

Fig. 4 shows an example of methane production across the two stages at pH 4 and 7 for 65 °C. It indicated that the pre-treatment stage at pH 4 was less active compared to other pHs, and demonstrated the increase in degradability at higher pre-treatment pH.

Confidence regions of k_{hyd1}, k_{hyd2} and f_d, and the optimal values for k_{hyd2} and f_d are shown in Fig. 5 and Table v (Supplementary data). These indicated poor hydrolytic activity for pH 4 and 5 (with unbounded lower intervals of k_{hyd1}). The results again indicated the strong impact of temperature on f_d, but also indicated that pre-treatment pH also had an impact, with performance in the mesophilic stage improving with higher pre-treatment pH. The reduced hydrolytic activity was further reflected by relatively poor solubilisation achieved during pre-treatment stage at acidic pH, and the gradual increase in solubilisation as pH increased (Fig. 6). Overall, temperature has a far higher impact than pH, and results are consistent with the variable retention time tests.

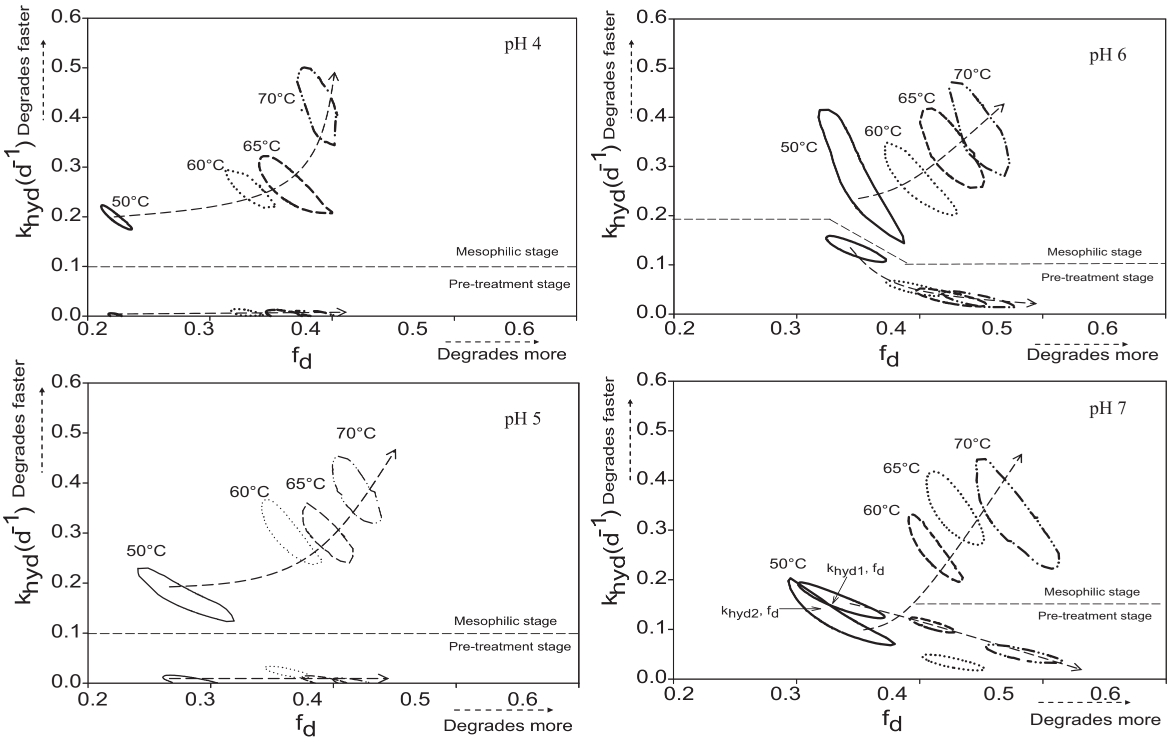

Fig. 5. Confidence regions of k_{hyd1} and f_d (pre-treatment stage) and k_{hyd2} and f_d (mesophilic stage) for thermophilic pre-treatment at different pHs at 50 °C, 60 °C, 65 °C, and 70 °C.

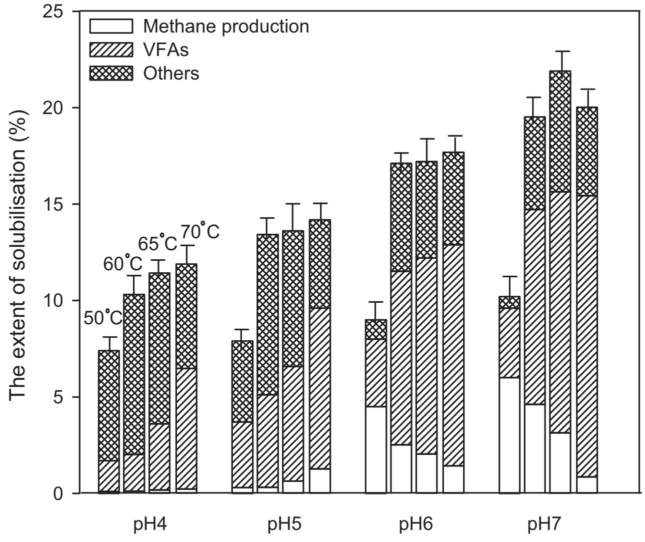

Fig. 6. The extent of solubilisation in the pre-treatment stage at pH 4, 5, 6, 7 and 50, 60, 65 and 70 °C. Error bars are 95% confidence in mean methane production and VFA concentrations.

4. Discussion

4.1. Identification and variation in sludge degradability

While degradability varied with the tested pre-treatment conditions, estimates of f_d were always statistically the same between the pre-treatment and mesophilic stages in a single set of process conditions, as demonstrated by overlap of confidence regions in the *x*-domain in Figs. 2 and 5. The consistency in the degradability fits between both stages indicates that degradability is measurable across two stages; sometimes quite accurately even with a very short retention time and limited data points.

Ultimate degradability is often considered as an inherent characteristic of a particular substrate, however, values of degradability in this study varied as pre-treatment temperature increased, indicating that degradability is at least partially dependent on the configuration of the TPAD process. Furthermore, as the operating condition of the second mesophilic stage was constant throughout the study, it is reasonable to determine that degradability is influenced by the pre-treatment conditions. Increased temperature in the pre-treatment stage enhanced sludge solubilisation and was consistent with previous reported studies [6,16]. Activated sludge contains a complex polymer matrix and it is likely that the increased temperature was required to degrade specific components of this matrix. The solubilisation products were then converted to methane or passed to the mesophilic stage as readily degradable materials and subsequently converted to methane.

4.2. Optimisation of pre-treatment conditions

Pre-treatment temperature had a very clear affect on sludge degradability with an increase from 21 ± 2% degradability at 50 °C to 48 ± 3% degradability at 65 °C. Accordingly, the methane yield increased from approx. 160 mL gVS_{added}^{-1} at 50 °C to approx. 300 mL gVS_{added}^{-1} at 65 °C (all at standard temperature and pressure). Methane yield per VS destroyed decreased from approx. 760 mL $gVS_{destroyed}^{-1}$ at 50 °C to approx. 625 mL $gVS_{destroyed}^{-1}$ at 65 °C. This is possibly due to an increase in destruction of complex organics which may have lower carbon oxidation state (e.g. waxes fats, etc.), and thus generate a higher proportion of methane. Enhanced degradability of sludge will reduce the mass of solids requiring disposal or reuse. Enhanced degradability may also improve sludge dewaterability, due to a higher destruction of the organic fraction [17,18] to further reduce the mass of sludge produced, and to reduce the cost of disposal and transportation.

Thermophilic pre-treatment at 70 °C has been commonly studied [6,7], however results from our study indicate degradability (f_d)

is not further improved over 65 °C using a pre-treatment temperature of 70 °C regardless of pre-treatment retention time and pH. Pre-treatment at 70 °C requires a greater heat energy input than pre-treatment at 65 °C, and the increased energy demand is not justified by the process performance.

The optimal pre-treatment temperature was 65 °C in this study, which was based on the comprehensive testing in the thermophilic temperature range (50–70 °C). Thermophilic pre-treatment at 65 °C has been poorly assessed during previous studies where temperatures were limited to mildly thermophilic (e.g. below 60 °C) [19], or temperatures were assessed with the straight increase from mildly (50 °C) to extremely thermophilic (above 70 °C) [6]. Therefore, discovery of a pre-treatment optimum at 65 °C is interesting and suggests that the optimal range is narrow.

Variations in the extent of solubilisation achieved during pre-treatment at retention times of 1, 2 and 4 days, were minimal at each thermophilic temperature; and resulted in similar performance in the subsequent methanogenic stage. This was represented by statistically overlapped regions of f_d and indicates a short retention time (1 day), is as effective to solubilise activated sludge as longer retention times (2–4 days). The optimal process configuration would depend on the design objectives of a specific application, shorter retention times would require lower capital and operating expenditure, while longer retention times may increase operational flexibility. Oles et al. [19] and Watts et al. [20] also report that shorter pre-treatment retention times (1–2 days) are sufficient to achieve the benefits of TPAD.

The TPAD process is clearly less effective at low pH levels (4–5), and this is the first study that systematically evaluates the impact of pH on substrate degradability in the TPAD process. No attempts were made during this study to adapt the inoculum to acidic pH, however significant adaptation of the inoculum to acidic pH was not expected. Series analyses done by Ge et al. [3], also indicated TPAD performance was reduced when pre-treatment was at acidic pH (4.5), and reported no significant improvement through adaptation of the pre-treatment community. This is supported by Ponsá et al. [21] who found no improvement in performance at low pH. Rapid hydrolysis could be achieved under extremely acidic conditions, however this is due to a chemical hydrolysis, not the activity of the microbial community. Overall, there is no benefit in acidifying sludge for biological pre-treatments at thermophilic temperature, and low pH operation is not justified.

4.3. Batch test quality and utility

The quality and utility of the two-stage batch test method may be assessed from the parameter surface and the repeatability of the tests under similar conditions. Good repeatability was exhibited by similar f_d values identified in the tests using a similar TPAD configuration (pre-treatment retention time of 2 days and pre-treatment pH 7).

Kinetic parameter estimations showed significant overlap in confidence regions when compared to model based analysis of a lab-scale continuous TPAD system used to generate mesophilic inoculum. The estimates of f_d based on the Van Kleeck VS destruction for the continuous system showed the overall degradability ranged from 0.28 to 0.57, and was not influenced by increased thermophilic pre-treatment temperature [22]. While similarly, estimates of f_d from the batch tests were conservative compared to continuous system (0.22–0.48). However, in the batch tests f_d was influenced by thermophilic pre-treatment temperature.

Batch tests are often conservative against continuous performance [13], and especially, hydrolysis coefficient (k_{hyd}) is often lower. This case is also similar, with a best k_{hyd} of $0.5\,d^{-1}$ in the batch tests presented compared to a best k_{hyd} on the same material of $1.1\,d^{-1}$ in the continuous system [22]. Lower values were similar at $0.2–0.3\,d^{-1}$. This is likely caused by the basic differences between batch and continuous processes. In a batch test, the rate is determined as the substrate concentration in the test vessel goes from a relatively high initial concentration to a low final concentration, while in a continuous process, the substrate concentration in the vessel is reasonably constant, and relatively low.

Overall, the two-stage batch test appears to be slightly conservative when estimating degradability for TPAD process. However, it offers a conservative estimate of kinetic parameters, and a good estimate of stoichiometry (possibly against an existing conventional process), which can be expected to be exceeded in a real plant. Benefits of the batch test include high accuracy, minimal time requirements, and lower cost as compared to bench scale continuous reactor testing.

5. Conclusion

A novel two-stage batch strategy was developed in this study and has been successfully applied to assess degradability rate and extent during TPAD of waste activated sludge. The method was further applied to assess the configuration of the thermophilic pre-treatment stage of TPAD. Generally, pre-treatment for shorter retention times (1 and 2 days) could achieve similar or better degradability as a longer retention time (4 days). The combined TPAD process was also more effective at pre-treatment of pH 6–7 with 33–48% degradability, compared to low pH (4–5) with 21–42% degradability. Thermophilic temperature had stronger impact on degradability, which was increased from 21% to 49% with temperature increased from 50 to 65 °C.

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Appendix A. Supplementary data

Supplementary data associated with this article can be found, in the online version, at doi:10.1016/j.jhazmat.2011.01.032.

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