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# Anaerobic digestion of liquid products following hydrothermal carbonisation of faecal sludge at different reaction conditions

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**ABSTRACT**

The hydrothermal carbonisation (HTC) conversion of wet wastes, such as sewage sludge, generates a carbon-rich material (called 'hydrochar'), and an aqueous fraction with a small release of gas. The liquid fraction is high in soluble chemical oxygen demand, from 10 to 50 g/L, and could not be discharged to the natural environment without treatment. This study investigates the anaerobic digestibility of this HTC liquid stream from different HTC temperatures and retention times (140°C–200°C for 30–240 min). It is focused on biogas production in order to improve the energy input of the HTC process and to improve process sustainability. The results demonstrated that liquid products from the lower HTC temperatures gave better biogas production. The biogas yield from the 140°C HTC filtrate digestion was 0.45–0.86 L/L reactor/d, while 0.33 L/L reactor/d was obtained from 170°C and 0.31–0.45 L/L reactor/d from 180°C HTC filtrates. The lowest anaerobic digestion (AD) efficiency was recorded for the treatment from 200°C with biogas yield of 0.07 L/L reactor/d. The data also show that low AD hydraulic retention time (HRT), typical of high rate fixed biomass digesters can be used to treat the HTC filtrate. Halving the AD HRT to 0.9 d resulted in 1.8–6.8 times greater biogas yield.

*Keywords:* Anaerobic digestion; Hydrothermal carbonisation; biogas; Sewage sludge; Wastewater treatment

## 1. Introduction

There has been renewed interest in thermal sewage sludge treatment due to the commercialisation of hydrothermal carbonisation (HTC), both to increase biogas yields in anaerobic digestion (AD) but also to generate a carbon-enriched char at temperatures above 160°C [1–3]. The higher temperature applied in HTC, compared with non-thermal traditional sludge and sewage treatment, sanitised and stabilised the products. Most studies on sewage sludge HTC have focused on the energy value of the hydrochar that is the main product [4,5]. The potential of its use for soil improvement [6,7] or as an adsorbent for soil remediation has been also reported [8,9]. However, to produce hydrochar with improved energy characteristics (higher heating value), a higher temperature range is required [2]. This generates a high concentration of soluble organic compounds as leachate (a by-product), which requires further treatment (chemical oxygen demand [COD] >20 g/L) and limited work has been carried out on this. Water plays an important role in HTC reaction as a solvent and depending on the reaction temperature, about 13%–66% of the carbon remains in the liquid fraction, which increases with increasing temperature [2,10].

Recent work on thermal pre-treatment of sewage sludge followed by AD confirms that dewaterability and soluble organic matter increase as the HTC temperature increases (above 150°C) [11]. The increase in solubilisation of organic solids, carbohydrates, proteins [12] and COD [13] leads to the

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increase in methane production during AD. Thus, hydrothermal treatment has been implemented successfully as a pre-step to sewage treatment (Cambi, Biothelys™) [14,15] as well as municipal solid waste treatments (Suzhou Food Waste Treatment Plant, Changchun Food Waste Treatment Plant, and Shenzhen Municipal Organic Waste Treatment Plant) [16]. Some literature, also suggests concurrent formation of refractory COD [17] at higher temperatures, mainly through Maillard reactions, reducing digestibility. Compounds such as furans, phenols, acetic acid, levulinic acid, and other persistent coloured soluble organic compounds have been reported in previous works [2,6,18–20].

The liquid fraction is completely sterilised, but application of this organic-rich by-product has received comparatively little attention. AD of the HTC wastewater for methane production seems to be an ideal process to reduce the organic load, which could contribute to the energy sustainability of the HTC process for sewage sludge treatment [1,10]. However, studies on AD of the liquid product following HTC of sewage sludge for biogas or methane production are limited. Danso-Boateng et al. [2] reported potential methane yields from the liquid phase resulting from sewage sludge HTC, and suggested methane yield to decrease at higher HTC temperatures. However, in their study the liquid phase was not subjected to AD; instead, the total organic carbon (TOC) and COD concentrations of the liquid were used to estimate the theoretical yields of methane. Wirth et al. [3] conducted AD of the liquid product (called 'liquor') from sewage sludge HTC at mesophilic and thermophilic conditions in two identical continuously fed-batch reactors for 20 weeks. They found no significant difference between biogas production from mesophilic (37°C) and thermophilic (55°C) digestion of the HTC liquor. However, in their study the HTC conditions were constant (220°C for 6 h), therefore, the effect of HTC temperature and reaction time on AD performance and methane yields were not investigated.

This study investigates the anaerobic treatment of the liquid phase generated from faecal sludge HTC at different temperatures ranging from 140°C to 200°C and retention times between 30 and 240 min. The effect of HTC reaction conditions on biodegradability of the HTC liquid products and biogas yields after AD are studied. In addition, the effect of different AD hydraulic retention times (HRTs) on the performance was investigated.

## 2. Materials and methods

### 2.1. Faecal sludge

The faecal sludge used in this study was formulated according to the recipe of Wignarajah et al. [21], which comprised of 37.5% cellulose, 37.5% yeast, 20% peanut oil, 4% KCl and 1% $Ca(H_2PO_4)$ in a suspension of 90% moisture (i.e., 10% solids).

### 2.2. HTC process

A workshop designed and built rig was used for the HTC process. Approximately 4.5 L of faecal simulant was pumped into the reactor (5 L total reactor volume) and heated to temperatures between 140°C and 200°C (via an oil heater unit) under self-increasing pressure and maintained for residence times ranging from 30 to 240 min. Following the hydrothermal treatment, the slurry was transferred into a flash tank where the pressure was reduced and the material was allowed to cool to room temperature. The carbonised material was then filtered using a 60-μm slotted pore, stainless steel, filter under a slight residual pressure. This filtrate was used for the AD experiments. The operation cycles were pre-programmed using Lab View®.

### 2.3. Anaerobic digestion

Two identical anaerobic fixed-film digesters (9 L working volume) were operated continuously at standard temperature (37°C). An initial experiment with direct feeding of the raw waste led to digester instability, low conversion of COD to gas and finally acidic inhibition. Dilution by recycling was successfully used as a method to cope with the strong HTC filtrate. It was shown that a dilution ratio of (10:1) provided optimum buffering and recycling of nutrients. This was achieved by recycling part of the effluent from each digester back into the feed tank (as shown in Fig. 1). Recycle ratios of between 2 and 10:1 are widely used in higher rate digesters to buffer strong feeds and recycle trace nutrients. Bulk batches of feedstock including the recycle were prepared every 3 d (to include week-end running).

### 2.4. Analytical methods

The influent and effluent of the anaerobic reactors were analysed according to the APHA standard methods [22]. COD, total and soluble, was measured using a COD Analyser (Palintest 8000, Palintest Ltd, UK) at a wavelength of 570 nm, following the procedure of Standard Methods 5220 D – closed reflux colorimetric method. TOC was determined using a TOC Analyser (DC-190, Rosemount Dohrmann, USA), in line with Standard Methods 5310 B – high temperature combustion. Total solids were determined according to Standard Methods 2540 B – total solids dried at 103°C–105°C. According to Standard Methods 2540 E, volatile solids were determined by igniting the residue from the dry weight test to constant weight at 550°C. Biochemical oxygen demand ($BOD_5$) was measured respirometrically using a BOD analyser (BODTrak II, HACH, USA) by the HACH Standard Method procedure.

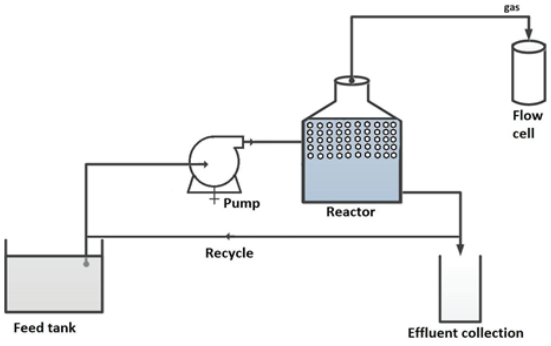


Fig. 1. Schematic of the anaerobic digestion process operated in a warm room (37°C).

Volatile fatty acids (VFA) were analysed using a spectrophotometer (DR3900, HACH LANGE, Germany) at a wavelength of 497 nm, by following the HACH Method 10240 (LCK 365). pH was measured using a pH meter (METTLER DELTA 340, Mettler Toledo, The Netherlands). Ripley's ratio was determined using the partial to alkalinity ratio method [23]. The volume and composition of the biogas produced were monitored using a FLO CELL™ flow meter and a GMF 400 series infrared, respectively.

## 3. Results and discussion

### 3.1. Analytical characteristics of the HTC filtrates

The filtrate characteristics following HTC at different reaction temperatures and times are presented in Table 1. In order to investigate the increase of the biodegradability at lower temperatures, 140°C was chosen as the lowest temperature to ensure sterilised hydrochar and the filtrate (>130°C recommended autoclave temperature).

### 3.2. Effect of HTC reaction conditions on AD performance

Fig. 2 illustrates the COD removal and biogas production for filtrates processed at 140°C and 180°C for 30–240 min, as averages from 40 d of continuous AD operation. Ripley's ratio was measured daily and during the whole duration of the experiments and it was monitored below 0.3, which indicates a stable AD operation. Increasing the HTC reaction time, increased the solubilisation of COD, increased the AD load, and gave better conversions of COD to biogas from HTC filtrates for up to 120 min at 140°C and up to 90 min at 180°C. Further increased in the reaction time reduced the AD performance. TOC on the other hand was less affected by HTC conditions (Fig. 4).

The data in Fig. 2 indicate a lower performance at the higher temperature and additional experiments were conducted over a wider temperature range of 140°C–200°C and reaction times between 30 and 240 min. These results are shown in Table 2 and Fig. 3. Fig. 3 suggests that the severity of conditions (temperature and reactor retention) affects biodegradability. Charing or blackening of the HTC solids was not observed at 140°C at any retention time tested but occurred at 180°C and above, after 30 min, confirming the previous study that concluded the extent of carbonisation influenced treatability [2].

Methane percentage in the biogas yield was stable throughout the experiment ranging from 63.5% to 77.7%. Typical percentage of methane after AD of organic solids and liquid waste is around 60%. Stoichiometrically, 1 kg of COD releases about 15,625 mol of methane gas. Thus, 1 kg of COD produces 0.35 $m^3$ or 0.25 kg of methane at standard

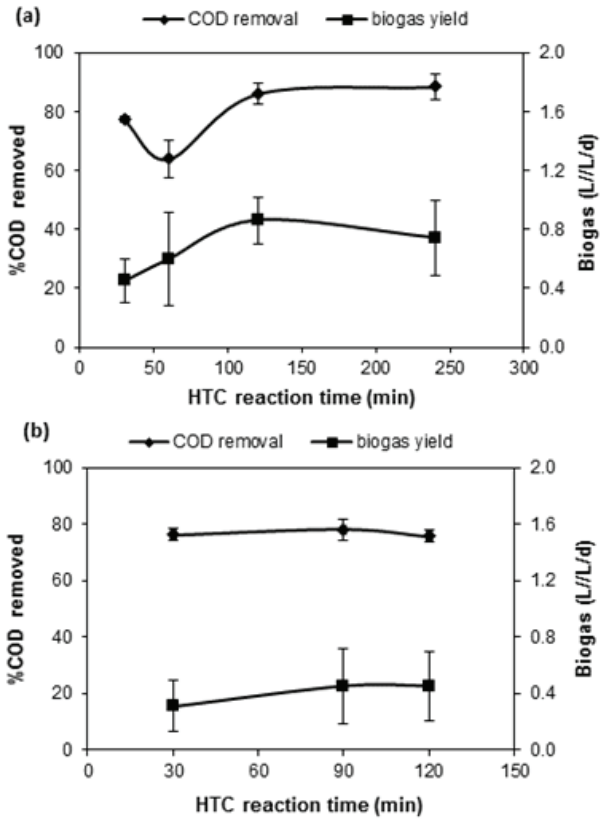


Fig. 2. Percentage of COD removed and the biogas produced for digestion of (a) 140°C and (b) 180°C HTC filtrate.

Table 1
Overview of the analytical characteristics of the HTC filtrate (<60 μm) processed at 140°C–200°C for 30–240 min

| HTC operating conditions | Filtrate characteristics | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| | COD soluble (g/L) | COD total (g/L) | TOC (g/L) | TS (g/L) | TS (%) | VS (g/L) | VS (%) | pH | VFA (g/L) |
| 200°C, 30 min | 30.19 | 54.52 | 10.38 | 44.07 | 4.06 | 34.83 | 80.21 | 4.41 | 4.55 |
| 180°C, 30 min | 27.19 | 47.53 | 8.70 | 18.65 | 1.88 | 15.11 | 20.98 | 5.06 | 2.14 |
| 180°C, 90 min | 25.90 | 41.52 | 7.98 | 22.53 | 2.64 | 17.93 | 80.36 | 4.58 | 3.61 |
| 180°C, 120 min | 30.07 | 43.27 | 10.89 | 24.52 | 1.94 | 20.10 | 82.01 | 4.59 | 3.89 |
| 170°C, 60 min | 20.07 | 36.45 | 5.90 | 25.04 | 2.47 | 18.75 | 74.88 | 4.92 | 2.78 |
| 140°C, 30 min | 19.95 | 33.67 | 6.78 | 26.60 | 2.66 | 20.89 | 78.28 | 6.91 | 3.23 |
| 140°C, 60 min | 24.57 | 26.6 | 9.11 | 27.03 | 2.70 | 19.66 | 72.80 | 7.79 | 3.38 |
| 140°C, 120 mi | 30.99 | 97.63 | 8.92 | 24.85 | 2.49 | 18.78 | 75.60 | 5.06 | 2.96 |
| 140°C, 240 min | 38.39 | 85.02 | 10.18 | 26.77 | 2.68 | 20.73 | 77.44 | 3.83 | 2.39 |

Table 2
Overview of AD performance for HTC filtrates processed at different temperatures and reaction times

| HTC temperature | 140°C | | | | 170°C | | 180°C | | 200°C |
|---|---|---|---|---|---|---|---|---|---|
| HTC time (min) | 30 | 60 | 120 | 240 | 60 | 30 | 90 | 120 | 30 |
| OLR (g COD/L/d) | 2.217 | 2.730 | 3.674 | 4.755 | 1.115 | 3.223 | 3.646 | 1.842 | 1.813 |
| COD removal (%) | 77.3 (±0.979) | 64.0 (±6.383) | 85.9 (±3.517) | 88.4 (±4.115) | 58.2 (±12.942) | 76.3 (±2.315) | 78.0 (±3.844) | 75.8 (±2.377) | 62.0 (±6.066) |
| Biogas yield (L gas/L reactor/d) | 0.453 (±0.148) | 0.599 (±0.319) | 0.861 (±0.155) | 0.744 (±0.256) | 0.326 (±0.231) | 0.311 (±0.183) | 0.449 (±0.264) | 0.451 (±0.246) | 0.07 (±0.079) |
| $CH_4$ (L/g COD removed) | 0.197 (±0.015) | 0.269 (±0.015) | 0.203 (±0.016) | 0.132 (±0.012) | 0.376 (±0.0.17) | 0.094 (±0.008) | 0.118 (±0.007) | 0.118 (±0.015) | 0.045 (±0.001) |
| COD effluent (g/L) | 4.52 | 9.60 | 4.56 | 4.89 | 8.41 | 6.88 | 7.20 | 7.27 | 11.83 |

Note: Numbers in parenthesis represent the standard deviations.

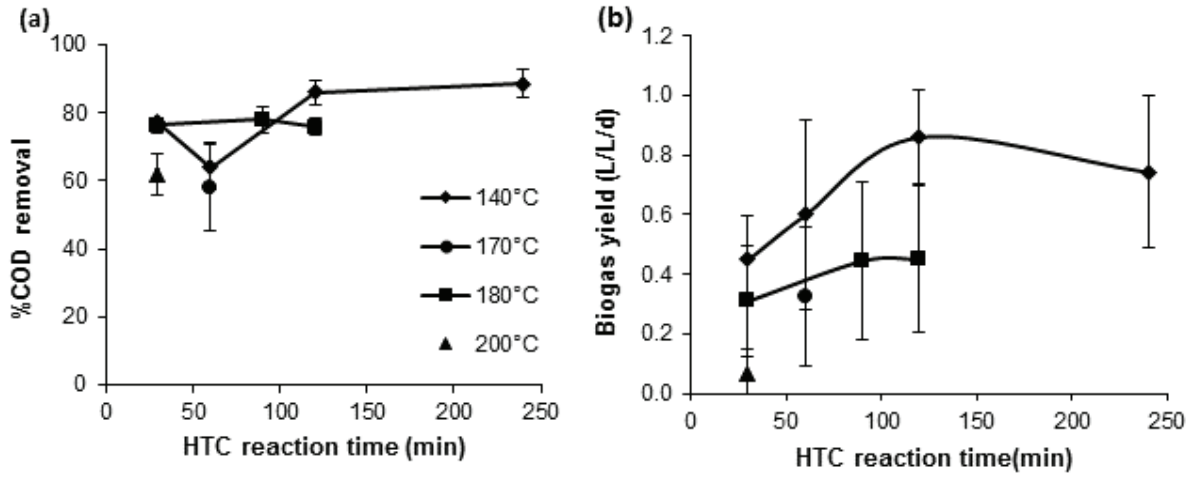


Fig. 3. Results of (a) COD removal and (b) biogas yield, during AD treatment of HTC effluent carbonised at 140°C–200°C for 30–240 min.

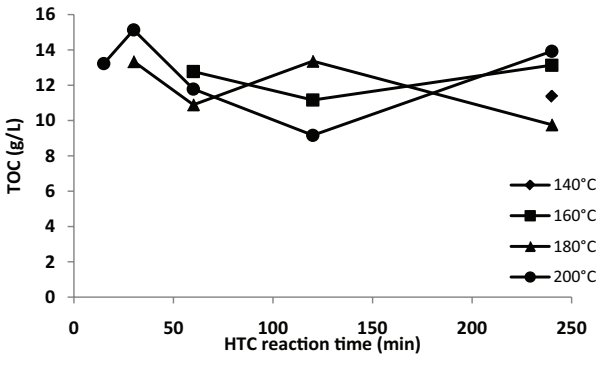


Fig. 4. Total organic carbon (TOC) of liquid filtrates after 140°C–200°C and 30–240 min HTC treatment.

temperature and pressure (STP) regarding an ideal substrate as acetate [24]. STP corrected yields from the 140°C HTC filtrate digestion were between 0.132 and 0.269 L $CH_4$/g COD removed and 0.094–0.118 L $CH_4$/g COD removed for the filtrate at 180°C. The lowest AD efficiency was recorded from HTC treatment at 200°C with the biogas yield of 0.045 L $CH_4$/g COD removed. Thus, it can be suggested that HTC treatment at lower temperatures affects positively the production of biogas during the AD of the liquor. The lower biogas yields obtained at higher temperatures could be attributed to the formation of refractory COD [17] and less degradable organic compounds through Maillard reaction product in the liquid fraction [2] at these conditions.

Wirth et al. [3] conducted AD experiments of liquid product (liquor) produced from HTC sewage sludge treated at 200°C for 6 h and reported methane yields in the range of 0.144–0.178 L $CH_4$/g COD for different organic loading rates (OLRs) between 1 and 5 g/L. However, in their study the HTC conditions did not vary. Also, the feedstock in this study reported was a simulant; hence, the performance differences are expected.

The reduction in biodegradability with increased HTC temperature was supported by both BOD and respiration rate measurements shown in Figs. 5(a) and (b). These two indicators show the same tendency as biogas yields. With the increase in HTC temperature a decreased in the respiration

rate and the BOD occurs. Standard biological methane potential tests were also used but gave poor reproducibility compared with respiration rates and were abandoned.

Methanogenesis is the rate limiting step of AD of soluble substrates by the previous research using similar kind of feedstock [3]. The Chapman model [25] was used to determine the kinetic constants for the methane production (Eq. (1), Table 3).

$$Y\,CH_4 = Y\,CH_4\,max \times (1 - exp^{1-km \times t})^c \quad (1)$$

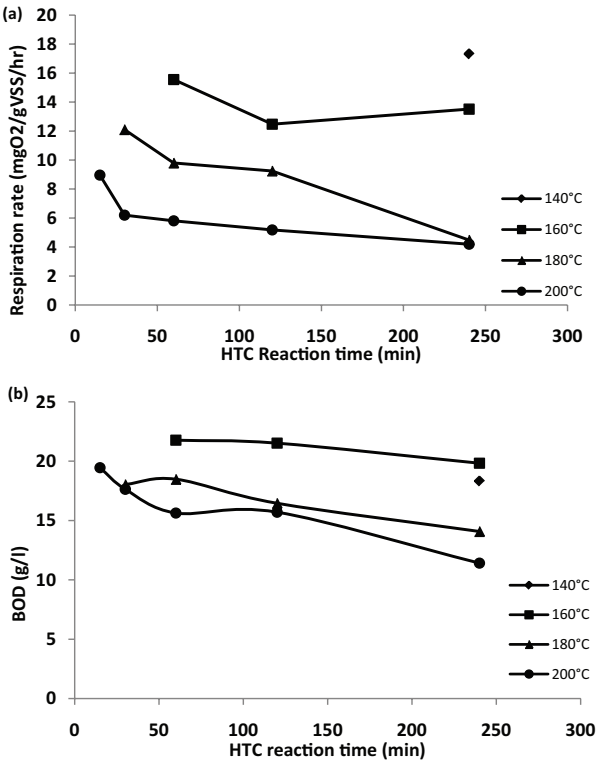


Fig. 5. (a) Respiration rate and (b) BOD of liquid filtrates after HTC treatment at 140°C–200°C and 30–240 min.

Table 3
Methanogenesis constants for each HTC filtrate

| HTC temperature (°C) | HTC time (min) | Methane production constant ($k_m$) |
|---|---|---|
| 140 | 30 | $6.96 \times 10^{-4}$ |
| | 60 | $1.01 \times 10^{-2}$ |
| | 120 | $9.46 \times 10^{-3}$ |
| | 240 | $6.70 \times 10^{-2}$ |
| 170 | 60 | $1.59 \times 10^{-4}$ |
| 180 | 30 | $3.27 \times 10^{-2}$ |
| | 90 | $2.35 \times 10^{-7}$ |
| | 120 | $1.37 \times 10^{-7}$ |
| 200 | 30 | $8.92 \times 10^{-8}$ |

As the treating HTC temperature rises the methanogenesis became slower except for the filtrate from HTC at 180°C for 30 min which showed a much higher constant compared with that of 170°C filtrate and those from longer HTC treatment at the same temperature. Writh et al. [3] reported a kinetic constant of 0.044 whereas an ideal substrate as the acetate is expected to produce methanogenesis constants in the range of 0.2–0.7 [26]. Moestedt et al. [27], however, reported methanogenesis constants between 0.02 and 0.09 during AD of food, slaughterhouse waste and glycerin considered as high biodegradable substrate.

The COD concentrations in the effluent of the digesters range between 4.52 and 11.83 g COD/L according to the different filtrates. The percentage of COD removed was typical of AD and stable throughout the operation as judged by the standard deviations (Table 2). However, the organic load in the final effluent was still high and further treatment would be needed if disposal to the environment is to be considered. Recycling the effluent back to the head of the treatment works has been commonly used but may cause problems if the refractory COD was to accumulate.

The COD removal equivalent to the methane production was calculated using the theoretical relation (for a perfect substrate) mentioned previously (1 g COD consumed produces 0.35 L of methane) and this was compared with the percentage of COD removed calculated based on the soluble effluent in Table 2. Only 19.5%–47.2% of the COD was converted to methane (Fig. 6) although the COD conversion based on the soluble effluent was a lot higher (58.2%–88.4%). This can be referred to the microbial growth and the accumulation of solids in the AF digesters. It can be suggested that some COD removal may be from coagulation with the biofilms but this need more extensive data. The low biogas measurements reported (most in the range of 1–3 L) were at the accuracy limits of the instrumentation, but other non-analysed components, e.g., hydrogen and VOC could have contributed to the overall mass balance.

### *3.3. Effect of the hydraulic retention time on the efficiency of the anaerobic digestion*

Two AD HRTs of 1.8 and 0.9 d were tested. The main difference was observed for the biogas production (Table 4). Decreasing the HRT by increasing the flow rate and OLR

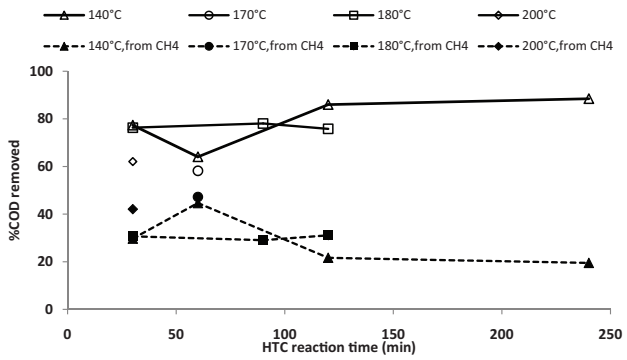


Fig. 6. Comparison of measured COD removal calculated based on dissolved COD in the effluent and theory, based on the methane production.

Table 4
Biogas production after AD of different HTC conditions filtrates under two HRT times

| HTC temperature (°C) | HTC time (min) | Biogas yield (L/L reactor/d) | | COD removal (%) | |
|---|---|---|---|---|---|
| | | HRT = 1.8 d | HRT = 0.9 d | HRT = 1.8 d | HRT = 0.9 d |
| 140 | 30 | 0.250 | 0.453 | 73.9 | 77.3 |
| | 60 | 0.089 | 0.599 | 55.3 | 64.0 |
| | 120 | 0.215 | 0.861 | 90.3 | 85.9 |
| | 240 | 0.197 | 0.744 | 92.2 | 88.4 |
| 180 | 30 | 0.089 | 0.310 | 75.1 | 76.3 |

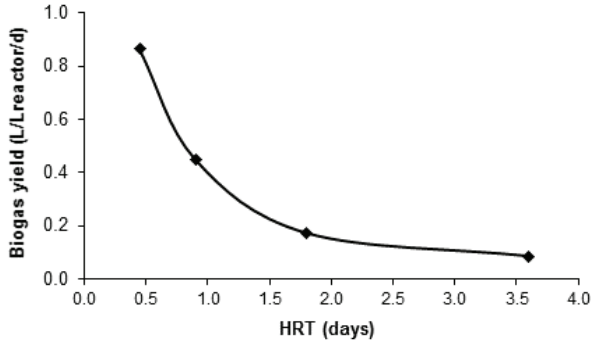


Fig. 7. Biogas yield during AD treatment of HTC effluent at 180°C for 90 min with AD hydraulic retention times of 0.45–3.6 d.

improves the performance. The yield was more than double for the lower retention time (0.9 d) in all the experiments. Specifically, when the lower HRT was applied the biogas produced was 1.8–6.8 times greater. This is supported by the literature on biomass retaining reactors which have a typical retention time of 5–10 h and loading rates up to 40 kg COD/m$^3$/d for the highly treatable wastes [28]. Further work is needed to identify the maximum load.

Additional AD HRT experiments were carried out on filtrate from the HTC run at 180°C for 90 min, since obvious char was produced at this temperature but not at 140°C. The data are shown in Fig. 7. Gas yields at 0.45 d are similar to those achieved at 140°C and suggest that char production without prejudicing the treatability of the filtrate could be possible.

## 4. Conclusions

The liquid products following HTC of sewage sludge can be digested anaerobically to produce biogas. The results demonstrated that liquids generated from HTC at lower temperatures produced higher biogas yields, with HTC at 140°C and a shorter HRT of 0.9 d resulting in maximum yields. Liquids obtained from HTC at 180°C for 30 min also resulted in high biogas yield when the HRT was shorter (0.9 d), which could be considered as the optimal conditions as dewatering of the products was easier at these HTC conditions than lower temperatures. For liquids from HTC at 140°C up 120 min residence time and 180°C for 90 min, increasing the HTC reaction time resulted in an increased in COD solubilisation, increased in AD load and a better conversion of COD to biogas. The results show that low AD HRT, typical of high rate fixed biomass digesters can be used to treat the HTC liquid from sewage sludge. Levels of COD in the digester effluent were still high, indicating further treatment is required if the option is to dispose it to the environment.

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