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Volatile fatty acids platform from thermally hydrolysed secondary sewage sludge enhanced through recovered micronutrients from digested sludge

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ABSTRACT

The extracellular polymeric substances and microbial cytoplasmic contents seem to hold inorganic ions and organic products, such as proteins and carbohydrates that are of critical importance for the metabolism of hydrolytic and acidogenic anaerobic microorganisms. The addition of soluble microbially recovered nutrients from thermally treated digestate sludge, for the fermentation of thermally hydrolysed waste activated sludge, resulted in higher volatile fatty acids yields (VFAs). The yield of VFAs obtained from the recovered microbial nutrients was 27% higher than the no micronutrients control, and comparable to the yield obtained using a micronutrients commercial recipe. In addition, the use of a low pH resulting from a high sucrose dose to select spore forming acidogenic bacteria was effective for VFA production, and yielded 20% higher VFAs than without the pH shock and this associated with the addition of recovered microbial nutrients would overcome the need to thermally pre-treat the inoculum.

1. Introduction

A significant focus of research into anaerobic digestion process (AD) in recent times, has been the enhancement of the acidogenic process, for the production of volatile fatty acids (VFAs), as essential precursors for the second-stage biomethanation process (Massanet-Nicolau et al., 2013), or as substrate for polyhydroxyalkanoate (PHA) production (Kedia et al., 2014), or for bioelectrochemical systems (BES) (Guwy et al., 2011).

A variety of feedstocks, including, foodwaste, organic fractions of municipal solid waste (OFMSW), dairy wastewater and olive oil mill waste, have been evaluated for the production of VFAs (Lee et al., 2014). Some of the process conditions that have critically been evaluated in the optimisation of the production of VFA include: organic loading rate (OLR) (Kyazze et al., 2006), temperature (Yuan et al., 2009), pH (Massanet-Nicolau et al., 2008), hydraulic retention time (HRT) (Massanet-Nicolau et al., 2009) and additives such as surfactants and enzymes (Lee et al., 2014). It is important to note however that most of the previous research related to VFA production was conducted for optimisation of biohydrogen production, and not necessarily to investigate or optimise VFA production. In particular for sewage sludge, studies related to VFA production have been limited to a couple of lab-scale (semi) continuous fermentation studies (Morgan-Sagastume et al., 2011; Maharaj and Elefsiniotis, 2001) and very few full scale applications (Shana et al., 2003).

The selection of inoculum source, such as activated sludge, aerobic compost, thermally treated anaerobically digested sludge and soil, have also been identified as an important step in the optimisation of the hydrolytic-acidogenic fermentation process for VFA production (Hawkes et al., 2002). Thermally treated digested sewage sludge is often selected as inoculum for VFA and biohydrogen production, in that, the thermal treatment helps to eliminate the presence of VFA and hydrogen utilising microorganisms, leaving spore-forming bacteria species (Hawkes et al., 2002;

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Wang et al., 2003). As explained by Hawkes et al. (2002) bacterial species, such as *Bacillus* and *Clostridium*, undergo sporulation under unfavourable or stressful environmental conditions such as rising temperatures, low or high pH and depletion of nutrients. Non-spore forming microorganisms on the other hand, undergo cell lyses under unfavourable environmental conditions. As observed by Massanet-Nicolau et al. (2008), spore formers effectively undergo sporulation by heating mixed microflora at 110 °C for 20 min whilst non-spore formers undergo cell lyses. The cytoplasmic content of non-spore forming microbes is therefore expected to be released to solution after thermal treatment. According to Feeherry et al. (1986), even spore-forming bacteria undergo injury and eventually to cell lyses at ≥124 °C within 1 min; 62 min is however required to achieve same results at 112.8 °C.

Thermal treatment for the selection of acidogenic microbes is also expected to cause the dissolution of extracellular polymeric substances (EPS) (a complex high-molecular-weight mixture of polymers) produced by the microorganisms that surrounds the cell wall, which also contain inorganic ions. Soluble microbial products such as nucleic acids, uronic acids, proteins, lipids and carbohydrates, also become available after thermal treatment of digested sludge (Sheng et al., 2010). Several studies have investigated the influence of various micronutrients on AD (Feng et al., 2010). There is however little knowledge about the influence of micronutrients on hydrolytic-acidogenic fermentation.

This study aims to evaluate the importance of selection of inoculum and the use of micronutrients in VFA production from thermally hydrolysed secondary sewage sludge or waste activated sludge (WAS). The study also explores the use of soluble microbial nutrients (potentially in the EPS and soluble microbial products) after thermal treatment of digested sludge as micronutrients source to enhance VFA production.

2. Materials and methods

The acidogenic fermentation was carried out in 2.5 L reactors with working volume of 2-L. The reactors were kept in a shaking incubator equipped with temperature controllers, which maintained the operation temperature at 37 ± 1 °C. The incubator provided a constant agitation at 110 rpm. The inoculum was obtained from Welsh Water at the Cog Moor Wastewater plant; a conventional AD plant treating largely secondary sewage sludge without any pre-treatment. The feedstock for the fermentation process was thermally hydrolysed waste activated sludge (TH-WAS), obtained from Welsh Water treatment plant in Cardiff. The thermal hydrolysis pre-treatment is carried out at 165 °C and 6 bars for 30 min. The TH-WAS feedstock was collected at 2 weeks interval and stored in a refrigerator at 4 °C. All the reactors were operated at semi-continuous mode (once a day feeding during the week days), at an OLR of 25.54 gVS L^{-1} d^{-1}. The reactors were operated at HRT of 2.8 days. The pH of the reactor content during the fermentation process was not controlled in the study. Samples were taken periodically for VFA measurement. The TH-WAS feedstocks for some of the reactors were supplemented with a tailor-made commercial micronutrients (CM) (Table 5 shows the concentration trace minerals in the CM added). The CM was added to the TH-WAS substrate and stored at 4 °C for later use. Two runs of the same experiments were performed with the repeat taking place one month apart.

2.1. Inoculum preparation and micronutrients recovery from digested sludge

The digested sludge was sieved through a 2 mm mesh to remove particulate matter. The sieved sludge was either used directly or heated at 110 °C for 20 min.

A portion of the thermally treated sludge was then centrifuged at 3000 rpm for 20 min, after being allowed to cool to ambient temperature. The supernatant was decanted, and then filtered through a 0.2 μm pore size hollow-fibre membrane module (Tianjin Motian Membrane Eng. and Tech. Co. Ltd.), to remove any particulate matter as well as bacterial cells. The filtrate was heat-treated at 110 °C, and then stored at 4 °C for later use as micronutrient source (Recovered microbial nutrients, RMN).

2.2. Experimental set-up

The study included six experimental set-ups involving inoculum pre-treatments and micronutrients addition, and their controls, as shown in Table 1.

The start-up of all the NI-reactors (NI, NI-CM and NI-RMN) involved a high loading of sucrose (a concentration of 30 g/l) causing a pH reduction and therefore making the environmental conditions more favourable for the acidogens, the important microbial population for the synthesis of VFAs. After the initial feeding with sucrose, the NI reactors were left for 56 h without feed, at which time the pH of the reactor content reduced from an initial 7.35 to 4.05. The reactors were then fed with the TH-WAS substrate. The TH-WAS substrate was fed to the reactors in a semi-continuous mode by withdrawing (for analysis) and feeding the same amount daily. The N0 reactor used as control for the NI-reactors involved the use of untreated inoculum with no sucrose dosing. A schematic illustrating the experimental desiagn is shown in Fig. 1.

2.3. Analytical methods

The VFA concentrations in the samples were determined as described by Cruwys et al. (2002) using a head space gas chromatograph equipped with a flame ionisation detector (Perkin Elmer, UK). Soluble COD was measured using the Hach Lanch COD kit. Soluble carbohydrate was determined in using the phenol-sulphuric assay (Dubois et al., 1956). The type and quantity of elements in the soluble fraction of the sludge, CM and RMN solutions were determined using inductively coupled plasma optical emission spectrometry (ICP-OES) as described by Fassel and Knlseley (1974).

3. Results and discussions

The different conditions of the acidogenic fermentation were repeated twice at one month interval. As shown in Table 2, the VFA yield obtained from Run 2 was generally higher than the yield obtained in Run 1. The difference in the two runs was in the concentration of the feedstocks, due to full scale variation in sludge concentration, which after preparation in the lab were 7.28 ± 0.43 g L^{-1} TS (59.03± 1.27% VS) in Run 1, compared to 8.23 ± 0.17 g L^{-1} TS (62.07 ± 0.35) in Run 2. The OLR in Run 2 was also higher than in Run 1, and that potentially resulted in the difference in the yield of VFAs.

As shown in Table 2, the patterns of increase of VFA with the different conditions in the two runs were very similar. The major difference in the two runs was the VFA yield from the N0 reactor, which was nearly 72% higher in Run 2, as compared to Run 1. The lack of selecting specific microbial strains for acidogenic fermentation makes the digester more susceptible to process instability during start-up of the fermentation process. It can be deduced that acidogenic fermentation in the N0 reactor, was more active in Run 2 compared to Run 1 and that could have resulted from the higher loading in Run 2, which potentially contributed to an improved acidogenic promoting condition with a reduced pH. The discussion in this study was based on data analysis from Run 2. The