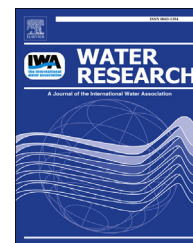


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Enhancing methane production from waste activated sludge using combined free nitrous acid and heat pre-treatment

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ABSTRACT

Methane production from anaerobic digestion of waste activated sludge (WAS) is often limited by the slow degradation and poor substrate availability of WAS. Our previous study revealed that WAS pre-treatment using free nitrous acid (FNA, i.e. HNO_2) is an economically feasible and environmentally friendly method for promoting methane production. In order to further improve methane production from WAS, this study presents a novel strategy based on combined FNA and heat pre-treatment. WAS from a full-scale plant was treated for 24 h with FNA alone (0.52–1.43 mg N/L at 25 °C), heat alone (35, 55 and 70 °C), and FNA (0.52–1.11 mg N/L) combined with heat (35, 55 and 70 °C). The pre-treated WAS was then used for biochemical methane potential tests. Compared to the control (no FNA or heat pre-treatment of WAS), biochemical methane potential of the pre-treated WAS was increased by 12–16%, 0–6%, 17–26%, respectively; hydrolysis rate was improved by 15–25%, 10–25%, 20–25%, respectively, for the three types of pre-treatment. Heat pre-treatment at 55 and 70 °C, independent of the presence or absence of FNA, achieved approximately 4.5 log inactivation of pathogens (in comparison to ~1 log inactivation with FNA treatment alone), thus capable of producing Class A biosolids. The combined FNA and heat pre-treatment is an economically and environmentally attractive technology for the pre-treatment of WAS prior to anaerobic digestion, particularly considering that both FNA and heat can be produced as by-products of anaerobic sludge digestion.

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1. Introduction

In a typical wastewater treatment plant (WWTP), roughly 1/3 of the organic carbon from wastewater is converted to waste activated sludge (WAS) (Metcalf and Eddy, 2003). Anaerobic digestion has been widely applied to the WAS treatment since it transforms organic carbon in WAS into methane and reduces the amount of biosolids to be disposed of (Feng et al.,

2014; Appels et al., 2008; Eskicioglu et al., 2008; Tiehm et al., 2001). However, methane production via anaerobic digestion is often limited by the slow hydrolysis rate and poor biochemical methane potential of the WAS (Appels et al., 2008; Shimizu et al., 1993). Therefore, a number of pre-treatment strategies including mechanical, heat and chemical pre-treatment have been developed to improve methane production by enhancing hydrolysis rate and/or biochemical methane potential (Zhang et al., 2011; Zhang et al., 2010;

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Carrere et al., 2010; Foladori et al., 2010; Appels et al., 2008; Eskicioglu et al., 2008; Lissens et al., 2004; Tiehm et al., 2001; Stuckey and Mc Carty, 1978). These methods destroy cells and/or extracellular polymeric substances (EPS) with subsequent release of intracellular and extracellular constituents (Carrere et al., 2010; Foladori et al., 2010; Appels et al., 2008). These released constituents can be biodegraded more easily via digestion, thereby promoting methane production. For instance, Stuckey and Mc Carty (1978) found that methane production increased by 42% at a digestion time of 25 days after pre-treating WAS at 175 °C for 60 min. However, it has been reported that most of the above mentioned approaches are cost intensive due to high energy and/or chemical requirements, and have negative environmental consequences (e.g. higher net CO₂ emissions compared with the case without pre-treatment) (Carballa et al., 2011; Foladori et al., 2010).

Our recent studies showed that free nitrous acid (FNA, i.e. HNO₂), at parts per million (mg N/L) levels, is a strong biocidal agent for microbes residing in anaerobic wastewater biofilms and in WAS (Pijuan et al., 2012; Jiang et al., 2011). It was reported that the fraction of viable cells in WAS decreased by 50–80% after FNA treatment at 1–2 mg N/L for 24–48 h (Pijuan et al., 2012). The FNA itself and its derivatives (e.g. NO, N₂O₃ and NO₂) were hypothesized to be the biocidal agents, which damage lipids, proteins, carbohydrates and deoxyribonucleic acid (DNA) in cells/EPS by reacting with them (Yoon et al., 2006; Halliwell et al., 1992; Rowe et al., 1979; Horton and Philips, 1973; Lewis and Updegraff, 1923). More recently, we demonstrated that methane production from a full-scale WAS, with FNA pre-treatment at 0.4–2.1 mg N/L for 24 h, was improved by 10–30% at a digestion time of 20 d in comparison with the WAS without FNA pre-treatment (Wang et al., 2013). It was found that FNA pre-treatment improved both the hydrolysis rate and the biochemical methane potential of the WAS. Also, the FNA-based pre-treatment method was shown to be both economically feasible and environmentally favourable (Wang et al., 2013).

It is well known that a high temperature can stimulate chemical reactions in general according to thermodynamics and Arrhenius equation (Moore et al., 2010; Metcalf and Eddy, 2003). We therefore hypothesized that combined FNA and heat pre-treatment could facilitate more destruction of lipids, proteins, carbohydrates and DNA by FNA and its derivatives, thereby achieving even higher methane production during anaerobic digestion compared with FNA pre-treatment alone. Thermal energy is readily available in a WWTP with anaerobic sludge digestion and cogeneration. The combined FNA and heat pre-treatment, if proven effective, could easily be implemented.

The aim of this study is to assess the effect of combined FNA and heat pre-treatment on methane production from WAS. A full-scale WAS was subject to FNA + heat pre-treatment (0.52–1.11 mg N/L at 35, 55 and 70 °C) for 24 h, with FNA pre-treatment alone (0.52–1.43 mg N/L at 25 °C) and with heat pre-treatment alone (35, 55 and 70 °C) as comparisons. WAS solubilisation, pathogen destruction and biochemical methane production were then assessed and compared. The enhancement of methane production was interpreted using model-based analysis to determine

both the hydrolysis rate and the biochemical methane potential.

2. Materials and methods

2.1. Sludge sources

WAS was collected from the dissolved air flotation thickener of a local (Brisbane, Australia) biological nutrient removal wastewater treatment plant (WWTP) with a sludge retention time (SRT) of 15 days. Its main characteristics (with standard errors obtained from triplicate measurements) were: total solids (TS) 48.7 ± 0.2 g/L, volatile solids (VS) 39.4 ± 0.2 g/L, total chemical oxygen demand (TCOD) 59.9 ± 1.4 g/L, soluble chemical oxygen demand (SCOD) 2.34 ± 0.04 g/L, pH = 6.64 ± 0.00.

For the biochemical methane potential (BMP) tests to be further described in Section 2.3, the inoculum was harvested from a mesophilic anaerobic digester treating mixed primary sludge and WAS in the WWTP from which WAS was collected. Its main characteristics (with standard errors obtained through triplicate measurements) were: TS 31.3 ± 0.1 g/L, VS 23.3 ± 0.1 g/L, TCOD 34.8 ± 0.2 g/L, SCOD 1.94 ± 0.01 g/L, pH = 7.52 ± 0.00.

2.2. Pre-treating waste activated sludge using FNA, heat and their combinations

Batch tests were performed to assess and compare the effect of FNA, heat and FNA + heat pre-treatment on the characteristics of WAS. 3.3 L of WAS was evenly distributed into eleven batch reactors. Each test lasted for 24 h. For FNA pre-treatment, a nitrite stock solution (4.0 M) was added to four batch reactors in different volumes to achieve the designated nitrite concentrations varying between 75 and 200 mg N/L, as summarized in Table 1 pH was controlled at 5.5 ± 0.1 via a programmable logic controller using 1.0 M HCl solution. The nitrite concentrations and pH levels applied gave rise to FNA concentrations ranging from 0.52 to 1.42 mg N/L (see Table 1), which were calculated using the formula $S_{NO_2^- - N} / (K_a \times 10^{pH})$

Table 1 – Pre-treatment conditions applied in this study.

Pre-treatment	FNA (mg N/L)	Temperature (°C)	NO ₂ ⁻ -N (mg N/L)	pH
Control	0	~ 25 (ambient temperature)	0	6.6
FNA pre-treatment				
F1	0.52	~ 25	75	5.5
F2	0.70	~ 25	100	5.5
F3	1.11	~ 25	160	5.5
F4	1.43	~ 25	200	5.5
Heat pre-treatment				
H1	0	35	0	6.6
H2	0	55	0	6.6
H3	0	70	0	6.6
Combined FNA and heat pre-treatment				
FH1	1.11	35	200	5.5
FH2	0.70	55	200	5.5
FH3	0.52	70	200	5.5

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