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Effect of free nitrous acid pre-treatment on primary sludge biodegradability and its implications

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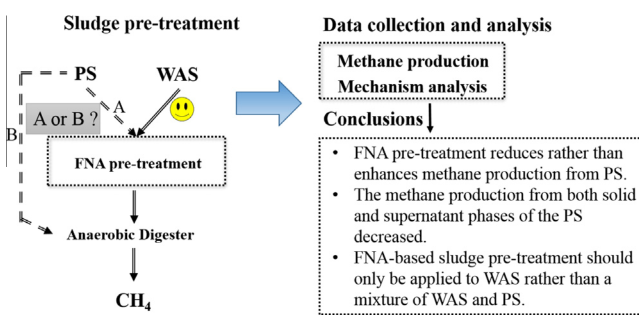
HIGHLIGHTS

- FNA pre-treatment reduces rather than enhances methane production from PS.
- The methane production from both solid and supernatant phases of the PS decreased.
- The decrease was ascribed presumably to the inherent sludge properties of PS.
- FNA treatment should only be applied to WAS rather than a mixture of WAS and PS.

GRAPHICAL ABSTRACT



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ABSTRACT

Free nitrous acid (FNA i.e. HNO_2) pre-treatment has been demonstrated to be effective in enhancing methane production from waste activated sludge (WAS). In some wastewater treatment plants (WWTPs), primary sludge (PS) and WAS are commonly mixed and digested simultaneously in the anaerobic digester. In order to reveal whether and how the PS and WAS should be jointly treated by FNA in WWTPs, this study presents the effects and mechanisms of FNA pre-treatment on methane production from PS. Full-scale derived PS was pre-treated with FNA at concentrations of 0–3.85 mg N/L followed by biochemical methane potential (BMP) tests. FNA treated PS was centrifuged to separate the supernatant from the solid phase for BMP tests on both fractions. FNA pre-treatment resulted in the methane potential reduction of 1–7%. The methane production from both supernatant and solid phases also decreased. PS solubilisation in combination with the molecular weight distribution and chemical structure analysis of the soluble phase showed very limited release of readily biodegradable substances from PS with FNA pre-treatment. The fact that FNA pre-treatment compromised the methane production from PS indicates that FNA-based sludge pre-treatment technology should implemented solely on WAS to maximise the methane production from the two sludge streams.

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1. Introduction

Anaerobic digestion (AD) has been widely used in waste activated sludge (WAS) treatment for sludge reduction and methane production [1,2]. However, the poor biodegradability of the WAS often seriously limits methane production. Many WAS pre-treatment methods including mechanical, thermal, and chemical treatment have been widely applied prior to AD to improve WAS degradability.

Abbreviations: FNA or HNO_2, free nitrous acid; WAS, waste activated sludge; WWTP, wastewater treatment plant; PS, primary sludge; BMP, biochemical methane potential; AD, anaerobic digestion; MW, molecular weight; IS, inoculum sludge; TS, total solids; VS, volatile solids; TCOD, total chemical oxygen demand; SCOD, soluble chemical oxygen demand; GPC, gel permeation chromatography; FTIR, Fourier transform infrared spectroscopy; GC, gas chromatography.

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Among them, a novel pre-treatment based on free nitrous acid (FNA i.e. HNO_2), a renewable chemical that can be produced on site by nitritation of the anaerobic digestion liquor [3], was recently proven effective in promoting methane production from WAS [4–6]. The method was shown to be both economically attractive.

However, in many domestic wastewater treatment plants (WWTPs), two major sludge streams, primary sludge (PS) and WAS, are mixed and digested together. The PS has distinctive characteristics in comparison to WAS, with a much lower level of microbial cells, lower levels of protein but higher fatty acids content [7–9]. It has been demonstrated that the macromolecular constituents of the sludge has an inherent impact on the effectiveness of the sludge pre-treatment [9]. For example, the thermal pre-treatment is more effective in enhancing methane production from WAS than from PS [8,10]. In addition, the optimal temperature and duration of thermal pre-treatment were also affected by the mixing ratio of PS to WAS [8].

Due to the differences in the characteristics between PS and WAS, it is important to unravel the effects of FNA pre-treatment on PS biodegradability. This will reveal whether the PS and WAS should be treated simultaneously with FNA prior to anaerobic digestion and consequently affect the design of FNA-based sludge treatment unit in WWTPs.

This study aims to investigate the effects of FNA pre-treatment on methane production from PS and the associated mechanisms. Full-scale PS was pre-treated with FNA at a series of concentrations (0.77, 1.54, 2.31, 3.08, 3.85 mg HNO_2-N/L) with the sludge without any pre-treatment as control. PS solubilisation and biochemical methane potential (BMP) were then assessed and compared. Afterwards, FNA treated PS was centrifuged to separate the supernatant from the solid phase, followed by BMP tests on both fractions to reveal their contributions to methane production. Model-based interpretation, in combination with the molecular weight (MW) distribution and chemical structure analysis of the substances in the supernatant with and without pre-treatment, was used to uncover the mechanisms responsible for the reduced methane production from PS with FNA pre-treatment.

2. Materials and methods

2.1. Sludge source

Two batches of primary sludge (PS1 and PS2) were collected from the primary clarifier in a local biological nutrient removal wastewater treatment plant (Brisbane, Australia). The main characteristics of PS1 (with standard errors obtained from triplicate measurements) were: total solids (TS) 44.5 ± 0.3 g/L, volatile solids (VS) 38.5 ± 0.2 g/L, total chemical oxygen demand (TCOD) 70.2 ± 0.2 g/L, soluble chemical oxygen demand (SCOD) 3.7 ± 0.03 g/L, pH = 5.6 ± 0.1. The main characteristics of PS2 (with standard errors obtained from triplicate measurements) were: TS 47.9 ± 1.2 g/L, VS 39.6 ± 0.1 g/L, TCOD 75.6 ± 0.5 g/L, SCOD 4.0 ± 0.04 g/L, pH = 5.7 ± 0.1.

Two batches of inoculum sludge (IS1 and IS2) used for biochemical methane potential (BMP) tests were collected from the mesophilic anaerobic digester of the same WWTP, which treats mixed primary sludge and WAS. The main characteristics of IS1 (with standard errors obtained from triplicate measurements) were: TS 20.7 ± 0.4 g/L, VS 15.3 ± 0.3 g/L, TCOD 22.8 ± 0.4 g/L, SCOD 0.61 ± 0.05 g/L, pH = 7.3 ± 0.0. The main characteristics of IS2 (with standard errors obtained from triplicate measurements) were: TS 26.0 ± 0.5 g/L, VS 18.6 ± 0.7 g/L, TCOD 27.1 ± 0.6 g/L, SCOD 0.72 ± 0.03 g/L, pH = 7.5 ± 0.0.

2.2. Pre-treatment of PS with FNA

Batch test I: PS1 was used in this set of tests. Six 300 mL batch reactors, including one control, were set up as shown in Table 1. The pH in the reactors for FNA treatment was adjusted to 5.5 and controlled at 5.5 ± 0.2 via a programmable logic controller (PLC) using 1 M HCl, whereas in the control reactor, pH was not controlled, but measured during the pre-treatment. After pH adjustment, a nitrite stock solution (40 g N/L) was added to the experimental reactors to achieve the designed FNA concentrations (0.77, 1.54, 2.31, 3.08, and 3.85 mg HNO_2-N/L) with FNA concentration calculated based on the acid-base equilibrium $S_{NO_2^- -N}/(K_a \times 10^{pH})$ with the K_a value determined as a function of temperature T (°C) by $K_a = e^{-2300/(273+T)}$ (22 °C in this study) [11], while no nitrite was added to the control reactor. All the tests lasted for 24 h. The SCOD, NH_4^+-N and NO_2^--N in PS were measured both before and after the pre-treatment.

Batch test II: PS2 was used in this set of tests. In order to investigate the effects of FNA on primary sludge at the bio-molecular level and its subsequent influences on methane production from the soluble phase and the solid phase of the primary sludge, another six 300 mL batch reactors, including one control, were set up under the same conditions as in Batch test I (Table 1). After 24 h treatment, the sludge with and without FNA treatment was centrifuged to separate the supernatant from the solid phase (i.e. sludge pellet). The sludge pellet was then dissolved into the same volume using the secondary effluent (re-dissolved PS2). The secondary effluent was collected from the same WWTP with a TCOD level of 43 ± 3 mg/L. The SCOD, NH_4^+-N and NO_2^--N in the supernatant phase of PS were measured both before and after the treatment. The supernatant was then freeze-dried for the analyses of the molecular weight distribution and chemical structure of the macromolecules in the supernatant.

2.3. Anaerobic biochemical methane potential (BMP) batch tests

Methane production from PS1, the solid and soluble fractions of PS2 with and without FNA treatment was assessed using BMP tests, using the protocols described in Jensen et al. [12]. The BMP tests were performed in 160 mL serum bottles with 100 mL working volume. The volume of primary sludge and inoculum in the BMP bottles for the three batches of BMP tests were as follows: 20 mL PS1 and 80 mL IS1; 25 mL PS2 re-dissolved solids and 75 mL IS2; and 70 mL PS2 supernatant and 30 mL IS2, to assure the inoculum to substrate ratio between 1.5 and 2.0 (VS basis for sludge samples and TCOD basis for supernatant samples). The bottles were sealed with butyl rubber stoppers and aluminium crimp-caps after being flushed with nitrogen gas. The sludge samples

Table 1
Pre-treatment conditions applied in this study.

Reactor No.	Pre-treatment	FNA (mg N/L)	NO_2^--N (mg N/L)	pH
Batch test I: with PS1				
1	Control	0	0	5.6–5.8
2	FNA 1	0.77	100	5.5 ± 0.2
3	FNA 2	1.54	200	5.5 ± 0.2
4	FNA 3	2.31	300	5.5 ± 0.2
5	FNA 4	3.08	400	5.5 ± 0.2
6	FNA 5	3.85	500	5.5 ± 0.2
Batch test II: with PS2				
1	Control	0	0	5.7–5.9
2	FNA 1	0.77	100	5.5 ± 0.2
3	FNA 2	1.54	200	5.5 ± 0.2
4	FNA 3	2.31	300	5.5 ± 0.2
5	FNA 4	3.08	400	5.5 ± 0.2
6	FNA 5	3.85	500	5.5 ± 0.2

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