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## Effects of thermal pre-treatment and recuperative thickening on the fate of trace organic contaminants during anaerobic digestion of sewage sludge



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### ABSTRACT

This study examined the effects of thermal pre-treatment and recuperative thickening on anaerobic digestion of sewage sludge on biogas production and removal of trace organic contaminants (TrOCs). Thermal pre-treatment and recuperative thickening resulted in approximately 15% increase in biogas production. However, the effects of thermal pretreatment and recuperative thickening on anaerobic digestion performance in respect to the removal of TrOCs were less obvious and varied widely depending on the molecular properties of each compound. Of the 40 TrOCs monitored in this study, 16 TrOCs were detected in all primary sludge samples. Removal from the aqueous phase was negligible for most of these 16 TrOCs. Caffeine and paracetamol were the only two TrOCs with a high removal from the aqueous phase. In comparison to the aqueous phase, TrOC removal from the solid phase was considerably higher. Through a mass balance calculation, it was shown that thermal pre-treatment or a combination of thermal pre-treatment and recuperative thickening could enhance the biodegradation of five persistent TrOCs, namely TCEP, verapamil, clozapine, triclosan, and triclocarban by 17–50%.

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### 1. Introduction

Urbanization and continuous population growth have imposed an increasing demand on wastewater treatment plants (WWTPs) particularly in regard to the management of sewage sludge. In Australia, sewage sludge production (as dried solids) has increased from 0.30 to 0.33 million tonnes between 2010 and 2013 (Semblante et al., 2014). Sewage sludge contains biodegradable organics and an array of pathogens. Thus, sewage sludge treatment is necessary before any beneficial use or land disposal. Anaerobic digestion is currently the most widely used technique for sewage sludge treatment. Anaerobic digestion is a biological process in which microorganisms convert biodegradable materials in the absence of oxygen to biogas and more stable organics. It is well

established that anaerobic digestion can efficiently stabilise organic materials and remove pathogenic agents in sewage sludge while simultaneously producing valuable biogas (Sawatdeenarunat et al., 2016; Sihuang et al., 2016; Tuyet et al., 2016). Biogas is a form of renewable fuel, which can be used to generate electricity and heat (Nghiem et al., 2017). The remaining and more stable solids are rich in nutrient and organics, thus, can be used for soil amendment (Nghiem et al., 2017).

Anaerobic digestion consists of four stages with hydrolysis being the first during which organic materials are transformed to fatty acids and other soluble organic compounds (Habiba et al., 2009). Since hydrolysis is the rate limiting step during anaerobic digestion, several pre-treatment methods, including thermal hydrolysis, biological treatment, ultrasonication, and ozonation, have been suggested to increase the digestion rate or improve the inherent degradability of sewage sludge (Carrère et al., 2010; Dhar et al., 2012). Thermal hydrolysis is a promising pre-treatment method to improve methane production during anaerobic processing

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(Supplementary data Table S1) since complex organic molecules can be transformed into short-chain fragments better suited for biological digestion (Liao et al., 2016; Mottet et al., 2009; Schieder et al., 2000). The effects of thermal pre-treatment at temperature of up to 180 °C and duration up to 2 h on anaerobic digestion performance have been evaluated by several recent studies (Bougrier et al., 2008; Kim et al., 2003; Pérez-Elvira and Fdz-Polanco, 2012; Phothilangka et al., 2008; Valo et al., 2004). The optimal temperature of thermal hydrolysis was reported to be 150–180 °C by Bougrier et al. (2008) for a pre-treatment duration of 30–60 min. Thermal hydrolysis has been successfully used at a full scale wastewater treatment plant (Kepp et al., 2000). The energy balance calculation showed the net electricity production due to enhanced biogas production increased by over 20%, which is more than the energy input for thermal hydrolysis.

In addition to thermal pre-treatment, recuperative thickening has also been identified as a cost-effective and readily implementable method to improve anaerobic digester performance without the need to increase the size of the digester (Cobbledick et al., 2016). Recuperative thickening can increase the solids retention time (SRT) independently of the hydraulic retention time (HRT) by thickening a proportion of the digestate to remove water and then returning the thickened sludge back to the digester (Reynolds et al., 2001; Torpey and Melbinger, 1967; Yang et al., 2015). The increase in SRT helps to improve the conversion of organics to methane and increase the volatile solid (VS) reduction (Sieger et al., 2004; Yang et al., 2015). Recuperative thickening has been successfully applied in a few WWTPs in North America and Australia. Full scale monitoring data suggest that recuperative thickening can improve both biogas production and VS reduction by 15–30% (Greer, 2011; Reynolds et al., 2001).

A major issue associated with beneficial reuse of reclaimed water and biosolids from sewage treatment is the ubiquitous occurrence of trace organic contaminants (TrOCs) in municipal wastewater. These TrOCs include several groups of widely used compounds including pharmaceuticals and personal care products, steroid hormones, industrial chemicals, pesticides, phytoestrogens, and UV filters. Their toxicological effects on human and other biota even at a very low concentration (less than 1 µg/L) remain largely unknown but are generally suspected (Luo et al., 2014). Some TrOCs can partition from the aqueous phase in wastewater to the solid phase in sludge during wastewater treatment (Citulski and Farahbakhsh, 2010; Semblante et al., 2015). When applied to farm land, these TrOCs may accumulate in soil, presenting a potential risk to human health and the ecosystem (Citulski and Farahbakhsh, 2010). However, to date, there have been only a few investigations on the removal of TrOCs from sewage sludge by anaerobic treatment.

Of a particular note, little is known about the impact of pre-treatment on the removal of TrOCs from sewage sludge by anaerobic digestion. In a systematic lab-scale study, McNamara et al. (2012) observed no discernible impact of thermal hydrolysis on the degradation of nonylphenol ethoxylates by anaerobic digestion. Similarly, Carballa et al. (2006) reported that thermal pre-treatment of sewage sludge had no observable impact on the removal of several pharmaceuticals, musks, and steroid hormones. By contrast, Hamid and Eskicioglu (2013) observed a notable increase in the removal of estrone and estradiol by anaerobic treatment following microwave-assisted pre-treatment (80–160 °C, 2.45 GHz, 1200 W). Given the paucity of information on this important issue, the present study aims to evaluate the influence of thermal hydrolysis and recuperative thickening on the fate of TrOCs in sewage sludge during anaerobic digestion. The influence of thermal hydrolysis and recuperative thickening on anaerobic digestion performance in terms of biogas production and organics

removal is also investigated.

## 2. Materials and methods

### 2.1. Lab-scale anaerobic digester and sludge

Three lab-scale anaerobic digesters previously described by Yang et al. (2016) were used in this study (Fig. 1). Briefly, each digester consisted of a 28 L stainless steel reactor (Core Brewing Concepts, Victoria, Australia), a peristaltic hose pump (DULCO®flex from ProMinent Fluid Controls, Australia), a temperature control unit (Neslab RTE 7), a thermal couple with temperature gauge, a biogas counter, and a gas trap for biogas sampling. One digester (denoted as D1) was operated as the control system without thermal pre-treatment and recuperative thickening. One digester (denoted as D2) was operated with thermal pre-treatment. The last reactor (denoted as D3) was operated with both thermal pre-treatment and recuperative thickening. All three reactors were operated in parallel and were each seeded with 20 L anaerobically digested sludge. The digested sludge and primary sludge were all sampled from a full scale wastewater treatment plant in New South Wales, Australia, with average total solid (TS) content of  $29.0 \pm 1.0$  g/L and  $22.2 \pm 2.2$  g/L, respectively. All sludge samples were stored at 4 °C until use or else discarded within two weeks.

All anaerobic digesters were operated under the same HRT of 20 d by wasting 1 L of digestate and the feeding with 1 L of primary sludge each day. Primary sludge, thermally pre-treated sludge, and digested sludge were collected weekly for characterisation. Parameters that were regularly measured include TS, volatile solid (VS), chemical oxygen demand (COD), alkalinity and pH.

### 2.2. Thermal pre-treatment

The feed sludge to digester D2 and D3 was thermally pretreated at 150 °C and 500 kPa for 30 min using a New Tek Machinery pressure vessel (Changzhou, China) with a heating jacket. At the conclusion of the process, the pressure inside the vessel was released and the sludge was allowed to cool to room temperature (ca. 25 °C) before feeding to the digester.

### 2.3. Recuperative thickening

Digester D3 was operated with recuperative thickening to achieve an SRT of 30 d with the HRT at 20 d i.e., same as the other digesters. A thickening ratio of 1.33 (which is the ratio of the total TS from primary sludge feed and return thickened sludge over the TS from primary sludge feed) was used. Each day, 2 L of the digestate was withdrawn from digester D3 and dosed with thickening polymer (Zetag 8169, BASF) at 7.5 g/Kg dry sludge. The sludge was gently mixed and allowed to settle by gravity for at least 10 min. 1 L of thickened sludge was then mixed with the thermally pre-treated (150 °C, 30 min) primary sludge (1 L) to form 2 L of feed to return to the digester. The excess thickened sludge and supernatant were discarded.

### 2.4. Analytical methods

#### 2.4.1. Anaerobic digestion performance

Biogas production rate was monitored daily by a custom-made gas counter (Yang et al., 2016). The biogas composition was detected weekly by a portable gas analyser (GA5000 gas analyser, Geotechnical Instruments Ltd, UK) (Nghiem et al., 2014). Additionally, samples from primary sludge (before and after thermal treatment) and digested sludge were taken weekly to analyse sludge characters such as TS, VS, total COD (tCOD), soluble COD

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