Recuperative thickening (RT) has been used to improve anaerobic digestion performance of the Bondi wastewater treatment plant (WWTP). This paper describes a laboratory scale study to fill the knowledge gaps identified during recent assessment of the Bondi WWTP. The results indicate that RT leads to a notable increase in biogas production. This effect is attributed to the increase in solids residence time and reduction in short circuiting achieved by RT. There is also evidence that polymer addition during sludge thickening would lead to the sequestration of biodegradable colloidal and soluble macromolecules resulting in improved soluble COD removal. RT could also reduce the concentration of total volatile sulphur compounds in the biosolids, resulting in less odorous biosolids.

**Introduction**

In conventional anaerobic digestion, both the hydraulic residence time (HRT) and solids residence time (SRT) are the same. In the late 1960s, Torpey and Melbinger (1967) introduced the recuperative thickening (RT) concept, in which a portion of digested sludge is thickened and returned to the digester for further digestion. RT allows for the decoupling of SRT and HRT. Thus, the SRT value and solids concentration in the digester can be increased independently of the HRT.

Sydney Water and CH2M HILL have recently completed a collaborative research project at the Bondi wastewater treatment plant (WWTP) to review RT performance and its current status as a process for biosolids reduction and quality improvement. This collaborative project identified several knowledge gaps that needed research to establish the operational envelop and better understanding of the factors controlling RT performance. The results can be used to facilitate the installation of RT at other WWTPs. Thus, the study reported in this paper has been formulated to address the knowledge gaps previously identified by (Bharambe et al., 2015).

Based on the literature and also on the assessment at Bondi WWTP by Bharambe et al., (2015) whose paper is also presented at Ozwater 2015, five main interlinked mechanisms may occur during the RT process. These are called 5-S hypotheses whose elucidation would lead to better anaerobic digestion performance than by conventional anaerobic digestion. These are:

1. The increase in SRT independent of HRT;
2. Reduction in Short-circuiting;
3. Microbial Selection or gradual shift to the methanogen community toward a more resilient population;
4. Sequestration of biodegradable and soluble macromolecules during thickening as a result of polymer addition; and
5. The impact of Shearing during thickening that reduces particle size with the consequent release of biodegradable substances to the aqueous phase.

It is well established that extended SRT could lead to further organic conversion to methane and an incremental increase in volatile solid reduction (Sieger et al. 2004; 2008). The increased volatile solid (VS) reduction may also limit volatile organic sulphur compound generation (directly associated with biosolids malodour) in downstream digested sludge processing such as dewatering and disposal of the biosolids cake. A few WWTPs in North America have successfully applied the RT process into their anaerobic digesters. Some positive effects on the biogas production have been observed at these plants (Reynolds et al. 2001; Greer 2011). However, previous data were obtained mostly from full scale operation where there could be many factors in play that could also influence anaerobic digestion performance. None of these previous studies have attempted to understand the underlying mechanisms of RT to enhance anaerobic digestion performance and particularly to assess and verify the main factors (5-S hypotheses) described above.

This study aims to experimentally assess the five S hypotheses that would influence RT. In particular, this study evaluates the use of RT to increase treatment capacity and efficiency with respect to a range of performance parameters including biogas production, VS and COD reduction, process stability, and biosolids odour.
MATERIALS AND METHODS

Wastewater sludge
Raw primary sludge and anaerobically digested sludge from the Wollongong Wastewater Treatment Plant, NSW, Australia, were used as the feed and inoculum for the lab scale anaerobic digesters, respectively. Raw primary sludge was collected fortnightly and was stored at 4°C in a sealed container.

Laboratory scale anaerobic digesters
Three identical lab scale anaerobic digesters were used for this study (Figure 1). Each digester consists of a 28L cone shape 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 digital biogas counter and a gas trap (1L) for biogas sampling, and a S shape air lock as gas trap. The digester was heated by hot water from the temperature control unit flowing inside plastic tube wrapping around the reactor. The entire reactor was also covered with insulation foam to minimise heat loss. The thermal couple probe was used to monitor the reactor temperature. A gas line was used to connect the digester to the gas trap, the S-shape air lock, and the digital gas meter which was used to record the gas production rate. When required, the gas trap could be used to collect 1L of biogas for composition analysis. Otherwise, the gas trap could be by-passed.

Experimental protocol
All three anaerobic digesters were seeded with 20L digested sludge individually at the beginning of the experiment. One digester was operated without RT as the control for baseline comparison. The peristaltic hose pump was operated continuously at a flow rate of 60L/h to ensure adequate circulation and mixing within the digester. The temperature of all three anaerobic digesters was maintained at 35±1°C throughout the experiment. The anaerobic digesters were fed once a day. Prior to each feed, 2L of digested sludge was withdrawn from the digester. Part of the withdrawn sludge volume was then thickened. Thickening polymer (Zetag 8169, BASF) was added to the digested sludge to obtain 4g/Kg dry sludge. The sludge was stirred at 400 rpm for 2 min and thickened by gravity to about 5% dry solids content. A pre-determined volume of the supernatant was wasted. The remaining thickened sludge and supernatant was mixed and fed back into the digester together with a pre-determined volume of raw feed (primary sludge). Mass balance calculation was used to determine the volume of wasted supernatant and feed. Using the above mentioned RT protocol, three experimental campaigns were conducted to compare AD performance at different SRTs (Table 1). Each Campaign ran for approximately 2 months.

Table 1: Hydraulic and solids residence times of the three parallel digesters (operated for 2 months).

<table>
<thead>
<tr>
<th>Experiment</th>
<th>SRT (d)</th>
<th>D1 (no RT-Blank)</th>
<th>D2 (RT)</th>
<th>D3 (RT)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Camp. 1: HRT=20d</td>
<td></td>
<td>20</td>
<td>25</td>
<td>30</td>
</tr>
<tr>
<td>Camp. 2: HRT=20d</td>
<td></td>
<td>20</td>
<td>30</td>
<td>60</td>
</tr>
<tr>
<td>Camp. 3: HRT=10d</td>
<td></td>
<td>10</td>
<td>15</td>
<td>20</td>
</tr>
</tbody>
</table>

Monitoring and analysis
Sludge samples were collected on a weekly basis for analysis. The measured sludge characteristics included total solid (TS), volatile solid (VS), total COD (CODt), soluble COD (CODs), pH, alkalinity, as well as NH4+ and PO43- concentration of the sludge centrate. All other parameters were measured according to the standard methods (Eaton et al. 2005). Biogas composition was also monitored on a fortnightly basis using a portable gas analyser (GA5000 gas analyser, Geotechnical Instruments, England) (Nghiem et al. 2014).

Volatile sulphur organic compounds analysis
At the end of Campaign 3, the digested sludge was dewatered using the modified centrifuge technique previously reported by Higgins et al. (2006) to produce biosolids cake. The dry solid content of the biosolids cake was 20±1%. The biosolids cake was
immediately characterised by its content of total volatile sulphur organic compounds (TVSOC). This was conducted using method developed by WERF. It is based on an incubation technique followed by Gas Chromatography – Mass Spectrometry (GC-MS analysis). In brief, 30g of biosolids was collected into a 500mL PET bottle. The bottle was sealed using a rubber cap and incubated at 28±1°C. The head space was extracted using a syringe at a specific time interval for GC-MS analysis. The results are reported in ppmv in the incubation bottle head space.

RESULTS AND DISCUSSION

Wastewater sludge characterization
The VS and TS values of the raw primary sludge are weather dependent and thus can vary. In this study, TS of the raw primary sludge was 23.7±5.7g/L. Similar temporal variation could also be observed with the CODT of the raw primary sludge. Despite this variation in the TS content, VS/TS ratio of the raw primary sludge remained relatively constant at 0.90±0.02.

Biogas composition and system stability
Variation in the TS of the raw primary sludge did not significantly influence the stability (i.e. abnormal decrease in biogas production or digester alkalinity) of the anaerobic digestion process in all experiments conducted with RT.

A major concern associated with RT is the exposure of the returned sludge to air during the thickening and sludge recycling process. However, no deleterious effects due to possible air exposure were observed in the digesters using RT. In all experiments, the biogas composition was stable and consistent with values reported in the literature (Brisolara and Qi 2013; Nghiem et al. 2014). Biogas composition obtained from Campaign 1 is presented in Table 2 as example. In all experiments, alkalinity of the digested sludge was consistently higher than 2000 mg CaCO3/L and pH was stable at around 7, with only one exception, as described above, at 10d SRT. The results confirm that exposure of the thickened sludge to air did not negatively affect anaerobic performance. This is
consistent with a previous study by Conklin et al. (2007) who studied the effect of short-term oxygen exposure to anaerobic digester sludge in batch mode to simulate RT condition.

**Table 2:** Composition of biogas from the three digesters operated at HRT of 20 day (Campaign 1). Data show mean ± standard deviations of 6-8 measurements over 2 months.

<table>
<thead>
<tr>
<th></th>
<th>CH₄ (%)</th>
<th>CO₂ (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>D1 (SRT=20 d)</td>
<td>56.3±3.1</td>
<td>39.0±4.0</td>
</tr>
<tr>
<td>D2 (SRT=25 d)</td>
<td>60.2±1.1</td>
<td>37.2±1.4</td>
</tr>
<tr>
<td>D3 (SRT=30 d)</td>
<td>59.2±0.8</td>
<td>37.0±1.0</td>
</tr>
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**Biogas production**

Decoupling of SRT from HRT and the increase in SRT, resulted in a notable increase in biogas production as observed in all three Campaigns (Figure 2). It is noteworthy that the improvement in biogas production was most significant when the SRT was increased from 15 to 20 d (Campaign 3). By contrast, doubling the SRT from 30 to 60 d resulted in a considerably smaller increase in biogas production (Campaign 2). RT also resulted in a notable increase in the methane production activity which is defined as methane production per gram of VS removed (Figure 3). In agreement with Hypothesis 3 (see Introduction), it is possible that RT could enrich methanogenic bacteria in the digester, allowing for the digestion of more slowly degradable VS fraction such as fats. It is noted that detailed microbial characterisation is still required to conclusively verify Hypothesis 3. A concurrent study is being conducted to further elucidate the role of microbial selection due to RT on anaerobic performance.

**TS, VS, and CODₜ removal**

Considerable variation in the removal of VS (Figure 4) and CODₜ (Figure 5) over time was observed. The standard deviation of VS and CODₜ removal over time was up to 12%. This variation is attributed to the temporal variation in the raw primary sludge as discussed above. RT resulted in a notable increase in biogas production and specific methane production activity from 0.4 to 0.7 and 1.4 L CH₄/g VS removed as the SRT increased from 20 d (no RT) to 25 and 30 d (with RT) in Campaign 1.

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**Figure 4:** VS removal at different SRT values during the three Campaigns.

**Figure 5:** CODₜ removal at different SRT values during the three Campaigns.
respectively. However, the improvement in biogas production occurred with minimal increases of both VS destruction and COD removal in Campaigns 1 and 2. This occurred for 20 d HRT and the SRT was in the range of 20 to 60 d. On the other hand, the increase in both VS and COD removal was only conclusively observed in Campaign 3 when the HRT was 10 d and the SRT was in the range of 10 to 20 d.

Our results provide additional experimental evidence to previous findings by Reynolds et al. (2001) and Ostapczuk et al. (2011). These authors reported that RT would be ineffective to remove VS in full-scale digesters if adequate SRT value had been achieved. The finding reported in our study and those by Reynolds et al. (2001) and Ostapczuk et al. (2011) can be attributed to several factors. For instance, RT resulted in an increase in the organic loading rate. Such an increase in organic loading rate could decrease the COD removal in the digester (Rincón et al. 2007; Li 2011). In addition, the improvement in system stability could be masked by the variation in the characteristics of the raw primary sludge and the build-up of less biodegradable volatile solids in the digester. It is known that the organic content of raw primary sludge is heterogeneous. In other words, primary sludge contains organic fractions with different degrees of biodegradability. At a sufficiently high baseline SRT value, the returned thickened sludge due to RT would contain mostly organic matter that is rather recalcitrant to further biodegradation. Our results (Figures 4 and 5) suggest that VS and COD destruction was not significantly improved because the solids returned to the anaerobic digester have a smaller proportion of readily biodegradable materials at SRT greater than 20 d.

To further elucidate the build-up of less

![Figure 6: COD distribution in the digester, effluent, and conversion into biogas during the three Campaigns.](image-url)
biodegradable organic matter in the digester at high SRT and the associated influence on COD removal, a mass balance with respect to COD was calculated for all experiments (Figure 6). The anaerobic digestion process consumes COD$_T$ of raw sludge, which was converted to biogas. The remaining COD$_T$ is discharged via the digested sludge withdrawn and would built-up in the digester. Thus mass balance was calculated using the following equation:

\[
\text{COD}_T (\text{raw sludge}) = \text{COD} (\text{gas}) + \text{COD}_T (\text{effluent}) + \text{COD}_T (\text{accumulated})
\]

Each bar in Figure 6 presents the total COD$_T$ input of the sampling date. The COD accumulated in the digested sludge is calculated as the balance of the COD input and output. Mass balance analysis showed that RT resulted in higher conversion of COD to biogas. However, both Digesters 2 and 3 (compared with Digester 1) had more COD accumulated throughout the experimental period (Figure 6). This explains the insignificant improvement in COD removal due to RT when SRT of the Digester 1 (Blank, ie, no RT) was sufficiently high (20 d). Thus, RT resulted in an increase in not only the COD consumed for biogas production but also COD accumulation in the digester.

COD$_S$ removal was improved in all RT experiments in comparison to the blank (data not shown). This can also explain the higher biogas production without discernible improvement in the removal of COD$_T$ with RT. This can be attributed to the sequestration of soluble and biodegradable macromolecules and colloidal particles from the aqueous solution into the solid phase caused by polymer addition during thickening. In other words, the addition of polymeric to the sludge prior to thickening would allow for some soluble macromolecules and colloidal materials to be captured and returned to the digester. This is consistent to Hypothesis 4 described above. However, further investigation is required to conclusively confirm Hypothesis 4. Although COD$_S$ (approximately 1000 mg/L) only contributed to less than 5% of the total COD content of raw primary sludge, the improvement in COD$_S$ removal could also be a factor attributing to the higher biogas production due to RT.

Our results demonstrate that RT could be used for plants with inadequate HRT (i.e. < 15d) to improve the removal of VS and COD$_T$. Our results highlight the role of RT in decoupling SRT from HRT thus allowing for the increase in SRT (Hypothesis 1) and improve system stability through the reduction of short circuiting (Hypothesis 2) and alleviating the impact of feed variation.

Figure 7: Concentration of specific odour compounds from biosolids of Campaign 3 as a function of incubation time.

**Hydrogen sulphide and total volatile organic sulphur compounds concentration in the biosolids**

The reduction in the concentration of total volatile organic sulphur compound indicated that RT reduces malodour generation associated with the biosolids cake. Hydrogen sulfide was prevalent in all biosolids cake samples. In addition, methyl mercaptan (which is one of the more important compounds for malodour reception associated with biosolids) and dimethyl sulfide could also be detected from biosolids cake samples. Figure 7 shows the concentration of odour compounds from
Campaign 3 as a function of incubation time. Biosolids cake from digester 1 (which was operated at SRT of 10d without RT) exhibited significant higher concentration of all three odour compounds compared to both digesters 2 and 3 which were operated with RT at SRT of 15 and 20d, respectively. Results reported in this study show, for the first time, that RT can contribute to a notable reduction in volatile organic sulphur compounds, and hence, in biosolids cake odour.

CONCLUSIONS

The increase in biogas production and system stability due to recuperative thickening (RT) reported are due to the increase in sludge residence time (SRT) and a reduction in short circuiting. There is also evidence that the improved performance associated with RT would be due to sequestration of biodegradable and soluble macromolecules and colloidal particles promoted by polymer addition used to thicken the sludge as well as possible enrichment of methanogens in the digesters. RT led to a reduction in total volatile organic sulphur compounds. This would result in the production of less odorous biosolids. The improvement in VS and COD removal associated with RT could only be clearly observed when the reference digester (operated without RT) was operated at a low SRT (i.e. 10d). Results reported in this study indicate that RT would be a viable technique to improve the performance of anaerobic digesters with inadequate SRT or issues with system stability. The results obtained in this study need validation by a full scale investigation.

REFERENCES

Central Kitsap County Wastewater Treatment Plant Alternatives Development Workshop, Brown and Caldwell.


