Optimisation of primary sludge acidogenic fermentation for biological nutrient removal

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Abstract

Laboratory batch experiments were conducted, at room temperature, to investigate ways of optimising the performance of primary sludge acidogenic fermentation systems, used at four major Johannesburg waste-water treatment plants. Retention time, seeding, solids concentration and mixing were found to be important variables governing volatile fatty acid (VFA) production. At least 75% of the potential VFA yield was reached within 6 d fermentation time. The addition of partially fermented sludge to fresh primary sludge improved the VFA yields after 6 d from between 48 to 170%, using a one-to-three-day-old fermented seed sludge, of 10 to 20% (v/v) the total volume of sludge in the fermenter reactor. A threefold dilution of the primary sludge with primary settled tank effluent, and the absence of mixing in the batch reactor, each showed at least a 70% improvement in the VFA yields. Furthermore, dewatering of fermented sludges with a cationic polyelectrolyte can be applied as a final solid-liquid separation technique with elutriation of the desired adsorbed organics, generated during the digestion process, to the supernatant liquor.

Introduction

Background

Municipal raw sewage represents a potential feedstock of readily biodegradable carbon including VFAs (Eastman, 1977). Sufficient levels of VFAs in the influent can provide the key to efficient biological nutrient removal (BNR) in waste-water treatment (Venter et al., 1978; Barnard, 1984; Rabinowitz and Oldham, 1985a and b; Osborn et al., 1989). The VFAs pass readily through the cytoplasmic membrane of heterotrophic organisms present in the sewage flora of the BNR process, to be metabolised internally as a carbon or energy source (Lilley et al., 1990).

Acid-phase anaerobic digestion of primary settled sludge is used in South Africa and other parts of the world, to generate VFAs, and so to boost the fraction of readily biodegradable carbon in the feed waste water (Pitman, 1995). This includes both in-line and side-stream fermentation facilities, the latter allowing for more operational flexibility and control. In-line systems increase the solids retention time in the primary clarifier to promote fermentation (Barnard, 1984; Frese et al., 1993; Pitman, 1995), while side-stream systems capture and retain the solids in a separate fermenter and feed a VFA-rich ferment to the BNR process or back to the primary clarifier (Comeau et al., 1987; Rabinowitz and Oldham, 1985a and b). At four of the major Johannesburg waste-water treatment plants, separate side-stream thickeners and elutriation tanks have been introduced, for fermentation and extraction of the VFAs to the supernatant liquor, to combat erratic biological P removal as a result of low VFA concentrations in the waste-water feed (Osborn et al., 1986) and reduce the need for chemical P removal (Pitman et al., 1991).

Research, undertaken over the years to increase the understanding of VFA production, has indicated that these "acid-phase" anaerobic digestion products are greatly influenced by parameters such as retention time, pH, temperature, waste-water characteristics, reactor configuration, trace minerals and oxidation-reduction potential (Ghosh et al., 1975; Elefsiniotis and Oldham, 1991 and 1994). Eastman (1977) identified hydrolysis of particulate organic matter to soluble substrate as the rate-limiting step during the acid generation phase of anaerobic digestion. Lilley et al. (1990), reported that VFA formation is a first-order reaction with a maximum potential conversion of influent COD to VFA of 17%, at 20°, at retention times of less than 10 d. They also reported that VFA yields could be improved with seeding of partially fermented sludge. However, the effect of seed age and size of seed inoculum, on the conversion rates of particulate matter, still needed further investigation. This forms part of the focus of this research paper. Furthermore, Skalsky and Daigler (1995) reported higher VFA yields at solids concentrations of less than 1% (m/v) compared to 2.6% (m/v) solids at 2 d retention time. This may have been linked to better mixing or a reduction in inhibitory substance effects in the dilute reactor. This solids inhibitory effect, however, was not observed by Lilley et al. (1990) at 11 000 to 42 000 mg VSS/l (VSS = volatile suspended solids).

In the four Johannesburg systems, relatively long solid retention times (±6 d) are needed, to obtain suitable VFA levels for nutrient removal (Pitman, 1997), and therefore relatively large fermentation tankage volumes are required. Furthermore, methane production is more likely to occur at longer retention times (6 to 10 d) and reduce yields of VFA (Osborn et al., 1986; Lilley et al., 1990). Supplementary chemical addition with iron salts to the BNR process is needed, particularly when the influent ratio of phosphorus to total carbon load (i.e. TP/COD) is high (e.g. approximately 0.02), to achieve the regulatory effluent limits on phosphate (Pitman, 1995). This is costly and it was the objective of this research paper to investigate ways to optimise the VFA yields, which will result in a concomitant decrease in retention time, stability of operation and improvement in BNR performance. Therefore, once the acid generation characteristics of the primary sludges from four major Johannesburg waste-water treatment plants had been established, the effects of seeding, solids concentration and mixing were evaluated, and better
process design criteria outlined. Moreover, the high solids concentration of fermented sludge generated in the thickeners at the Johannesburg plants had proved difficult to deal with, having poor settling characteristics and contributing to plant odours. The poor settleability caused an increase in solids carryover and unwanted additional loading of the BNR process. The use of a more sophisticated sludge handling technique (viz. dewatering as this would be compatible with existing waste sludge handling facilities on some of the Johannesburg plants) was also investigated as a means of solid-liquid separation and extraction of the adsorbed VFAs from the fermented primary sludges.

**Experimental**

The primary sludge used in this study was obtained from four major BNR plants in Johannesburg, namely Northern Works (NRAW), Goudkoppies (GRAW), Olfantsvele (ORAW) and Bushkoppie (BRAW). The sludge was collected from the underflow lines of the primary clarifiers. Settled tank effluent (STE), clarified from the overflow lines of the primary clarifiers. Anaerobic batch reactors, with a liquid volume of 5 l., were used, each equipped with a central stirring shaft connected to a 90 W output, 36 r·min⁻¹ motor. The reactors were filled with fresh primary raw sludge and the experiments were run at room temperature. Daily maximum temperatures ranged from 18 to 28°C in these batch tests.

**Acid generation characteristics of different sludges**

Two separate batch tests (Batches 1 and 2), were run to evaluate the acid fermentation characteristics of the four Johannesburg primary sludges. Four reactors were run concurrently for each batch test i.e. the reactors were filled with NRAW, ORAW, GRAW and BRAW respectively, with total solids (TS) concentrations ranging between 0.5 to 5.5% (m/v), and then allowed to ferment for a period of 8 d. The effects of acid fermentation, specifically VFA levels, soluble and total chemical oxygen demand (COD), nitrogen (TKN and NH₃-N) and phosphate (TP and ortho-PO₄), were monitored for each sludge type, on a daily basis. Volatile (VS) and volatile (VS) solids concentrations were determined every third day. All the analytical tests, were conducted in accordance with Standard Methods (1995) (i.e. method numbers 5220C; 4500-N org, NH₃, P; 2540B and E respectively), except the VFA analysis.

The VFA content was measured using a headspace-gas chromatography system (Perkin Elmer HS40 - Hewlett Packard 5890), equipped with a flame ionization detector (FID). A 25 m 0.5% phenyl-methylsilicon column was used.

- **Headspace parameters**: Sample temperature, 75°C; Needles and transfer temperature, 130°C; Thermostat time, 31 min; GC Cycle time, 22 min; Pressurisation time, 5 min; Injection time, 0.1 min; Withdrawal time, 0.2 min.
- **Gas chromatography (GC) parameters**: Initial temperature, 35°C. Initial time, 1 min; Rate 1, 10 degree/min; Final temp 1, 95°C; Rate 2, 50 degree/min; Final temp 2, 230°C; Final time, 5 min. Helium 10 psi. Detector and injector temperature, 250°C.
- **Esterification method**: 10 ml of sample was pipetted into a headspace vial. 0.5 ml concentrated sulphuric acid and 1 ml ethanol was added to the vial and capped. The vial was boiled in water for 30 min and placed in the headspace sampler. Samples were prepared in duplicate. The GC column was calibrated by injecting from a standard VFA solution (prepared as for sample) in a headspace vial.

**Effect of seeding**

Two batch tests (i.e. Batches 3 and 4) were conducted, using NRAW sludge, to study the effects of the age and size of seed inoculum on the rate of VFA production. For each batch test, fresh primary sludge was collected and allowed to ferment in one reactor with mixing. This provided seed bacteria as well as serving as the control experiment.

In Batch 3, three reactors, namely one control (NRAW at 0.98% m/v TS) and two seeded reactors were used. Initially, the control reactor was allowed to ferment for a total of 6 d. On Day 3, seed aliquots were removed from the control reactor and added to fresh feed stock, at 10% and 20% (seed volume/total volume), in the second and third reactors, respectively. These seeded reactors were also allowed to ferment for a total of 6 d.

Batch 4 was set up identically to Batch 3, except that a fresh sludge sample (NRAW at 1.2% TS) was used, and on Day 1, a seed aliquot was removed from the control reactor and added to fresh sludge, in a fourth reactor, at 10% (v/v).

Feed stock was taken from primary sludge stored at 4°C. The cold sludge was allowed to equilibrate to room temperature first so as to avoid cold shock and possible inhibition of the acidogenic bacteria.

**Effect of dilution and mixing**

Batch 5 used ORAW (4% TS) sludge. Five reactors were run in parallel for a period of 6 d. Reactor 1 (control) was allowed to ferment with mixing. In the next three reactors, ORAW was diluted with STE to a final concentration of 3, 2 and 1% TS, respectively. The last reactor, was filled with ORAW (4% TS) and allowed to ferment without mixing. Prior to sampling, however, this reactor was mixed for 15 min to ensure that a representative aliquot was taken.

In Batch 6, three reactors were run concurrently for a period of 6 d. NRAW (1.5% TS) with mixing, served as the control experiment. In the second reactor, NRAW was diluted with STE to 0.5% TS and allowed to ferment with mixing. The third reactor was filled with NRAW (1.5% TS) and allowed to ferment without mixing.

**Dewatering of fermented sludge**

In order to evaluate the recovery of VFAs after dewatering a fermented sludge sample, 8 d old fermented sludge, from the four treatment plants, was dosed with a cationic polyelectrolyte (Zetag 57, Chemserv Colloids). On the basis of visual floc formation viz. size of floc, a dose of 10 ml or 15 ml, 0.1% (m/v) Zetag 57 was added to a 100 ml aliquot sample with vigorous stirring for a few seconds. The sample was then filtered through Whatman GF-C filters and the levels of VFA, ortho-PO₄ and NH₃-N in the liquid phase, measured.

**Results and discussion**

**Acid fermentation characteristics of different sludges**

**Volatile fatty acids**

Batch studies of acid fermentation, with the four primary sludges tested, show that VFA production begins immediately in each case, increases at a rate that continually declines, and approaches a maximum potential VFA concentration after 8 d. VFA produc-
tion therefore appears to conform to a first order reaction as reported by Lilley et al. (1990). Similar patterns of VFA production were followed by each of the four sludges in batch test 1 and 2. Normalised time profiles for acid generation in Batch 1, calculated as a percentage of the total VFA production at the end of the 8 d fermentation period, are presented in Fig. 1.

Three of the sludges viz. NRAW, GRAW and ORAW follow similar VFA production trends, while BRAW shows a higher VFA production rate during the first 3 d retention time. This is most likely due to differences in sludge composition. Variable waste-water solids degradability makes the design of fermentation systems more difficult, and emphasises the need to run preliminary evaluation studies for each primary sludge type before design criteria are specified.

In these batch experiments (Batch 1 and 2) approximately 9% conversion of the initial reactor sludge COD to VFA was achieved by the sixth day of fermentation (see Table 1). This is at least 77% of the potential VFA yield after 8 d (Table 1). This figure is similar to Rabinowitz and Oldham’s (1985 b) pilot-plant studies, at approximately 10% mg VFA as COD/mg primary sludge COD after 3.5 to 5 d, 21°C. In general, conversion of initial COD to VFA is relatively low. It contributes only a small percentage of the readily biodegradable COD requirement for BNR removal and improvement would be beneficial to process performance (Rabinowitz and Oldham, 1985b; Pitman et al., 1991).

The COD conversion figure is, however, higher than the maximum potential figure quoted by Lilley et al. (1990) in laboratory batch studies, of 0.065 mg VFA as COD/mg influent sludge COD at 6 d, 20°C. The difference may be attributed to sludge composition, or to the fact that temperature was not controlled in our experiments, and VFA generation kinetics would be improved when maximum temperatures reached 28°C (Eastman, 1977; Gupta et al., 1985; Elefsiniotis and Oldham, 1991). The temperatures of these batch tests were typical of the warmer summer climate in Johannesburg. Average temperatures during the winter months can drop to 12 to 16°C, causing a decrease in VFA yields (Eastman, 1977). In a separate batch test, Banister (1996), found that VFA yields decreased by 45% at 12°C compared to yields observed at ambient temperatures similar to those reported here viz. daily maximum of 22 to 28°C, in NRAW (1% TS) after 6 d retention time. Skalsky and Daigger (1995), reported that VFA production at 14°C was approximately 42% less than that observed at 21°C.

Furthermore, since little more is gained, in VFA yield, by extending the retention time over 6 d (Table 1), as well as the increased possibility of methanogenesis commencing (Lilley et al., 1990), it was decided to limit the retention time to 6 d in subsequent experiments. Solids retention times at full scale exceeding 6 d require large tankage volumes and this becomes very costly (Skalsky and Daigger, 1995).

Table 1 also gives the net average VFA yields for each of the sludge types tested in Batches 1 and 2. Yields ranged between 0.07 and 0.18 mg VFA (expressed as COD)/influent mg VS at a retention time (SRT) of 6 d, 0.47 to 5.6% solids. VFA yields reported here would again seem to depend on the sludge composition. Skalsky and Daigger (1995) reported similar yields of 0.06 to 0.26 mg VFA/mg VSS at an SRT of 2 to 6 d at temperatures of 14 to 23°C, and 0.43 to 2.6% solids. It is significant that relatively lower VFA production yields were observed, in each set of experiments, for the ORAW sludge (Table 1) which may also be linked to high solids concentration (Skalsky and Daigger, 1995) or high levels of VFAs in the sludge itself (Pavlostathis and Giraldo-Gomez, 1991; Dinopoulou et al., 1988). ORAW had a higher total solids concentration in the order of 5%, compared to concentrations of 0.4 to 1.7% for the other primary sludge types (Table 1). The effect of solids concentration on VFA yields, is discussed later. Another possibility could be linked to the visible high fat and grease content in ORAW, which according to Eastman (1977) is degraded at a much slower rate during acid fermentation.
phase digestion.

Composition changes and trends in these batch tests, with regard to increases in soluble COD, ammonia and phosphate, and decreases in pH, in the four primary sludges, concurred with previous batch studies on primary sludge, at laboratory scale (Lilley et al., 1990; Pitman et al., 1991; Skalsky and Daigger, 1995). VFA as COD accounted for at least half of the total soluble COD, while pH levels dropped to between 4.8 and 5.2 after 6 d retention time. Release of phosphorus averaged 0.03 mg P/mg VFA as acetic acid, while ammonia release averaged at 0.07 mg N/mg VFA as acetic acid. The high concentrations of phosphorus in anaerobically digested sludges, and the chemical treatment of these sludge liquors, to reduce the additional nutrient load, which is then fed to the BNR process, is discussed in a second paper (to be published).

Effect of seeding

Increasing the seed population of facultative micro-organisms, capable of acid fermentation, with addition of partially fermented sludge to fresh primary sludge, was found to significantly boost VFA yields. This allows for reduction in retention time and consequently the tankage volume required for fermentation. Gross yields for NRAW were more than doubled with a 3-d old seed (Table 2). However, increasing the size of the inoculum, of the 3-d old fermented sludge, from 10% to 20% of the total volume, allowed for only very little or no improvement in VFA yields (Table 2). Table 2 also shows that the sludge age of the seed bacteria has an effect on the acid phase kinetics. A 1-d old seed appeared to improve the VFA concentration levels by a further 18% compared to the 3-d old seed, after 6 d retention time, using the same percentage seed volume. While the differences between a 1- and 3-d old seed inoculum need to be investigated further, the importance of organism constitution in the seed inoculum is highlighted. Acidogens must predominate in the inoculum and initial mix of organisms for optimum VFA production in primary sludge (Lilley et al. 1990).

The net VFA yields in Batch 4 (Table 2) are lower than those of Batch 3, and this may be attributed to the sludge composition, since the solids concentrations were similar. However, there is also the possibility that methanogenesis was occurring, since the maximum ambient temperatures were higher in Batch 4 (Table 2), thus increasing the rate of formation of VFAs (Gupta et al., 1985). and prevalence of substrate for the methanogens (Pavlostathis and Giraldo-Gomez, 1991). While the acidogens grow faster than the methanogens, which convert the VFAs to methane, CO₂, and water (Pavlostathis and Giraldo-Gomez, 1991), methanogens may be also be present in the seed sludge in sufficient quantities to reduce VFA yields.

At full scale, especially in semi-batch systems, one could allow partially fermented sludge (Pitman, 1995) (<3 d old) to remain in the fermenter tank to seed the fresh incoming sludge and optimise process performance.

Effect of dilution

Figure 4 also indicates that there may be an optimum point of dilution of solids concentration, below which the drop in solids concentration will start to limit the actual VFA levels attainable, although this needs to examined further. High solid concentrations (>2%) in the fermenter/thicker systems should therefore be minimised. This will also alleviate some of the sludge handling difficulties experienced at full scale with thickened sludge (Osborn et al., 1989).

Full-scale operation at lower solids concentration, will require proportionally larger reactor volumes for a given sludge retention time (Skalsky and Daigger, 1995). The economic benefits of higher yields at lower solids concentration, will therefore need to be weighed up against the costs of providing higher yields with longer retention times.

Effect of mixing

Figure 4 shows the VFA yields of a reactor that was left unmixed, against a mixed reactor, following speculation (Skalsky and Daigger, 1995) that mixing may play a contributing role in the inhibition effect. VFA yields increased from 0.04 to 0.07 in ORAW (3.8% TS) and from 0.09 to 0.15 mg VFA (as COD)/mg

<table>
<thead>
<tr>
<th>Sludge type</th>
<th>Total solids %</th>
<th>Seed age Days</th>
<th>Seed volume/total volume %</th>
<th>Net VFA/Initial COD mg COD/mg sludge COD (initial)</th>
</tr>
</thead>
<tbody>
<tr>
<td>NRAW Unseeded</td>
<td>0.98</td>
<td>-</td>
<td>0</td>
<td>0.081</td>
</tr>
<tr>
<td>NRAW Seeded</td>
<td>3</td>
<td>10</td>
<td>0.189</td>
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<tr>
<td>NRAW Seeded</td>
<td>3</td>
<td>20</td>
<td>0.216</td>
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<td>-</td>
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<td>0.091</td>
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<tr>
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<tr>
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<td>3</td>
<td>10</td>
<td>0.134</td>
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<tr>
<td>NRAW Seeded</td>
<td>3</td>
<td>20</td>
<td>0.135</td>
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</tr>
</tbody>
</table>

Daily maximum temperatures ranged from 19 to 20°C (Batch 3) and 22 to 28°C (Batch 4).

**TABLE 2**

**EFFECTS OF SEEDING ON VFA PRODUCTION YIELDS FOR NRAW PRIMARY SLUDGE, AFTER 6 D FERMENTATION TIME (BATCH 3 AND 4 RESPECTIVELY)**
enzyme at the lipid water interface (Heukelekian and Mueller, 1958) is increased as is found in more vigorous mixing conditions. Since carbohydrates and proteins are the primary sources of organic substrate in domestic raw sludge (Eastman, 1977) it would appear that the absence of mixing or slow/intermittent mixing regimes supported hydrolysis and higher VFA production yields in primary sludge. It should also be noted that this increase in VFA production efficiency may be reduced at lower temperatures or higher solids concentrations (discussed in previous section).

Dewatering of fermented sludge

Dewatering of fermented primary sludge with a cationic polyelectrolyte (Zetag 57), the flocculant routinely used at the Johannesburg sludge handling treatment plants viz. Northern Works, Goudkoppies and Olifantsvlei, allowed for good recovery of the adsorbed soluble organics (as VFA) to the liquid phase (Table 3).

Table 3 also shows that dosages of 3 to 27 g polyelectrolyte/kg sludge were needed for visual floc formation (size of floc) and capture of the solids. Polyelectrolyte dosages were not optimised for each sludge type in these experiments, but solids concentrations of the filtrate were negligible (<0.01% (m/v)). Solids capture is important when considering the potential for further P release from suspended solids in the filtrate (Pitman et al., 1991). Relatively high levels of phosphate and ammonia are already present in the sludge liquor of fermented sludge (Table 3). This may cause additional nutrient loading on the BNR plant when VFA-rich liquors are recycled. Further treatment e.g. chemical precipitation or biological removal, of these fermented sludge liquors may be needed to ensure compliance with effluent discharge standards in nutrient sensitive areas (Pitman et al., 1991; Banister and Pitman, submitted to Water SA).
Future design of primary sludge acid fermentation systems could therefore include a solid-liquid separation step for the fermented sludge, using mechanical dewatering systems. This could replace gravity separation systems, which have been shown to be problematic in terms of solids carryover and odour (Pitman et al., 1991). It would, however, dictate that a sludge-cake handling process such as composting, would follow. At some of the Johannesburg waste-water treatment plants, use of existing dewatering facilities may be more economical. Further investigation is needed to assess the full impact and cost benefits of dewatering sludge, and treatment of fermented sludge liquors, if necessary, on a BNR plant.

Conclusions

Four primary sludge types from different waste-water treatment plants in Johannesburg were investigated. The results suggest that the efficiency of VFA production is effected by a number of factors, including seeding, solids concentration, agitation and the source of the sludge.

Acid fermentation proceeded rapidly at retention times of less than 6 d, with VFA yields of approximately 10% of the total influent COD concentration after 6 d. Variable sludge solids degradability in the four sludges, however, would seem to dictate acid production kinetics.

The rate of VFA production was found to increase with the addition of partially fermented sludge. Preliminary tests show that the use of a seed inoculum taken from sludge that had been allowed to ferment for a period of 1 to 3 d, at 10 to 20% (v/v) of the total reactor volume, is effective in improving performance.

Furthermore, lack of agitation in the batch reactor or a three-fold dilution of the fermenter solids concentration improved VFA yields significantly. The results suggest that it would be advantageous to limit influent solids concentration of the primary sludge to approximately 0.5 to 2% TS and allow these solids to settle in the fermenter.

Mechanical separation with dewatering of the fermenter solids, on a plant equipped with sludge handling facilities viz. composting, may provide a useful and simple alternative to gravity separation and recycle of fermented solids for elutriation of VFA. This will extract the adsorbed VFAs and minimise solids loading to the BNR plant.

New process design considerations should therefore include back-seeding with partially fermented sludges to optimise growth and performance of the acid-producing micro-organism population and reduce retention times or tankage volumes needed. Moreover, primary sludge digestion without mixing and at solids concentrations not exceeding 2 to 3% (m/v) is recommended for maximum VFA yields. Further batch-, pilot- and full-scale plant testing of these new acid fermentation criteria will be needed in the future, to better define the final operation and control strategies with respect to semi-batch processes using side-stream fermenters and elutriation/dewatering systems.

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