Laboratory Scale Investigations of Potential Odour Reduction Strategies in Biosolids Phase II

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Summary Report

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Executive Summary

This report is the first summary report for the Phase II project, produced for the Water Corporation of Western Australia (WCWA) and the Australian and New Zealand Biosolids Partnership (ANZBP). The outcomes of the Phase I project are briefly summarised and the objectives of the Phase II study are described. The wastewater treatment plants selected for study in this project are described and the results of experiments conducted during the period of October 2012 to December 2012 are presented.

The objectives of the Phase II study are to: (a) expand biosolids and sludge sources to provide information on the odorous compounds present in biosolids produced at other Western Australian WWTPs, in addition to those studied in Phase I; (b) determine if the best odour reduction strategy identified in the Phase I project (i.e. alum addition to digested/treated sludge prior to dewatering) is applicable to wastewater sludge and biosolids produced from different treatment processes; (c) conduct dilution olfactometry measurements and correlate/compare the results with measurements obtained using HS SPME/GC-MS; and (d) depending on the results of the Phase II trials, determine if laboratory trials can be expanded to site trials.

In addition to Woodman Point WWTP, three additional WWTPs have been identified for study in the Phase II project: Beenyup WWTP, Gordon Rd WWTP and Kwinana WWTP. Fresh sludge and biosolids samples obtained from these WWTPs as well as two aged biosolids samples from Woodman Point WWTP were analysed for volatile organosulphur compounds (methanethiol (MT), ethanethiol (ET), dimethyl disulphide (DMS), dimethyl disulphide (DMDS), dimethyl trisulphide (DMTS), ethyl methyl sulphide (EMS) and diethyl disulphide (DEDS)) as well as other volatile organic compounds (toluene, ethylbenzene, styrene, *p*-cresol, indole and skatole) using HP SPME-GC-MS methods developed in Phase I of this study. The biosolids samples (fresh and aged) were also assessed by an odour panel.

Based on the Phase II results obtained to date, the following conclusions have been made:

- The main odour compounds identified in fresh biosolids cakes from Woodman Point, Beenyup, Gordon Rd and Kwinana WWTPs included: DMS, DMDS and DMTS. Significant quantities of indole and skatole were detected after approximately one month of incubation but were mostly not detectable in fresh samples, with the exception of biosolids cakes from Gordon Rd and Kwinana, which showed the presence of indole and skatole as early as day 1 and 2 of incubation, respectively. The bulk of the compounds tentatively identified in the aged biosolids samples consisted of long chain hydrocarbons, various alkyl benzenes, terpenes, cyclic hydrocarbons and other aromatic compounds.
- There were no significant differences between the sludge and the dewatered biosolids cakes in terms of the types of compounds present however, the dewatered biosolids cakes contained much greater quantities of the odorous sulphur compounds (DMS, DMDS and DMTS) than the corresponding sludge samples.
- Dewatered biosolids cakes obtained from Gordon Rd and Kwinana WWTPs, which use oxidation ditches to process the sludge, contained the highest concentrations of the volatile sulphur compounds (DMS, DMDS and DMTS) as well as the highest concentrations of the OVACs indole and skatole, compared to the anaerobically digested cakes from Woodman Point and Beenyup WWTPs.
- Results from odour panel assessments of fresh biosolids cakes from Woodman Point, Beenyup and Gordon Rd WWTPs as well as two aged biosolids samples showed that the aged biosolids sample ABS1, sampled from Woodman Point WWTP in January 2011, was ranked as the least odorous with an earthy/musty/mouldy odour by the majority of panellists. Biosolids cake from Gordon Rd WWTP was ranked as the most odorous with a faecal/septic

odour by 100% of the panellists. The odour panel results correlated well with analysis of the biosolids by HS SPME-GC-MS.

- A follow up analysis of the same biosolids cakes by the same odour panel approximately one month after the initial assessment showed an increase in the percentage of panellists detecting a strong faecal odour in biosolids cakes from both Woodman Point and Beenyup, which was not detected in the initial assessment. These observations were consistent with the detection of significant concentrations of indole and skatole by HS SPME-GC-MS and are supported by literature reports that indole and skatole begin to form after approximately 30 – 40 days of incubation.

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Acronyms and Abbreviations

ABS	Aged biosolids sample
ANZBP	Australian and New Zealand Biosolids Partnership
DAFT	Dissolve air floatation thickener
DEDS	Diethyl disulphide
DMDS	Dimethyl Disulphide
DMS	Dimethyl Sulphide
DMTS	Dimethyl Trisulphide
DS	Dry solids
EAS	Excess activated sludge
EMS	Ethyl methyl sulphide
ESD	Egg-shaped digester
ET	Ethanethiol
HS	Headspace
GC-MS	Gas Chromatography-Mass Spectrometry
MLE	Modified Ludzack-Ettinger process
MT	Methanethiol (also known as methyl mercaptan)
OD	Oxidation ditch
OVACs	Odorous Volatile Aromatic Compounds
RST	Rotary screw thickener
SBR	Sequencing batch reactor
SIM	Selective Ion Monitoring
SPME	Solid-Phase Microextraction
SRT	Sludge retention time
TVOSCs	Total Volatile Organic Sulphur Compounds
WCWA	Water Corporation of Western Australia
WWTP	Wastewater Treatment Plant

1.0 Introduction

In Phase I of this study we investigated the potential sources of odours from biosolids produced from Woodman Point Wastewater Treatment Plant (WWTP) in the Perth metropolitan area and examined potential odour reduction strategies on a laboratory scale. The odour reduction methods that were trialled were chemical additions and reduction of centrifuge speed. The chemical addition trials were conducted by adding alum, polyaluminium chloride or ferric chloride to digested sludge that had been sampled prior to the dewatering stage. Trials of chemical addition (alum) to plant dewatered cake were also undertaken on a laboratory scale. The impact of reducing the centrifuge speed on biosolids odour was also investigated using a laboratory scale centrifuge that had been calibrated to operate such that the shear forces on the sample would be representative, as closely as possible, to those on the plant. To assess the effectiveness of the odour reduction measures trialled in this study, analytical methods were developed to target the most potent odour compounds likely to be present in the samples. The methods were based on headspace solid-phase microextraction coupled with gas chromatography-mass spectrometry (HS SPME-GC-MS) and target odour compounds included volatile organic sulphur compounds (methanethiol (MT), ethanethiol (ET), dimethyl disulphide (DMS), dimethyl disulphide (DMDS), dimethyl trisulphide (DMTS), ethyl methyl sulphide (EMS) and diethyl disulphide (DEDS)) and other volatile organic compounds (toluene, ethylbenzene, styrene, *p*-cresol, indole and skatole). Under our method conditions, methanethiol was oxidised to DMDS (major peak) and DMTS (minor peak); and ethanethiol (ET) was oxidised to DEDS (major) with only a very minor peak visible for ET. Thus, it was assumed that any MT present in the biosolids would be transformed to DMDS and DMTS. Similarly, any ET present in the biosolids would be converted to DEDS.

Based on the results obtained from the Phase I study the following conclusions were made:

- The main odour compounds identified in fresh biosolids samples from Woodman Point WWTP included: DMS, DMDS and DMTS. No ET, EMS or DEDS were observed in the biosolids samples, suggesting that neither of these ethylated sulphur compounds were major contributors to the odours. Indole and skatole were identified in older biosolids samples that had been stored at room temperature for a few months and exhibited a strong faecal/nauseating odour. Other types of compounds which were tentatively identified based on their mass spectra and/or library matches, but not confirmed with authentic analytical standards, included various long chain aliphatic hydrocarbons, terpenes, alkyl benzenes and other aromatic compounds.
- Aluminium sulphate addition (4% dose based on aluminium) to digested sludge prior to dewatering offered the best odour reduction strategy amongst the options that were investigated, resulting in approximately 40% reduction in peak TVOSC concentration, relative to a control sample.
- Reduction of centrifuge speed would not be a viable option for Woodman Point WWTP as it resulted in a significant reduction in the solids content of the resulting biosolids cake.
- Addition of 4% alum to plant dewatered cake did result in significant odour reduction, however the pH of the resulting cake was also reduced (pH 4.2) to levels which would not be suitable for land application. To overcome the problem of low pH, lime would need to be incorporated into the biosolids prior to land application to raise the pH (6 – 8) to a suitable level for land application, resulting in additional costs for the utility.

1.1 Objectives of the Phase II study

The objectives of the Phase II study are to:

- Expand biosolids and sludge sources to provide information on the odorous compounds present in biosolids produced at additional Western Australian WWTPs.

- Determine if the best odour reduction strategy identified in the Phase I project (i.e. alum addition to digested/treated sludge prior to dewatering) is applicable to wastewater sludge and biosolids produced from different treatment processes.
- Conduct dilution olfactometry measurements and correlate/compare the results with measurements obtained using HS SPME-GC-MS.
- Depending on the results of the Phase II trials, determine if laboratory trials can be expanded to site trials.

1.2 Test sites identified for Phase II study

In addition to Woodman Point WWTP, three other WWTPs have been identified for study in the Phase II project. The WWTPs and their sludge handling processes are summarised in Table 1.

Table 1. Summary of processes at wastewater treatment plants selected for this study (adapted from Agarwalla, 2012).

Process	Woodman Point WWTP	Beenyup WWTP	Gordon Rd WWTP	Kwinana WWTP
Screening	step screen	step screen	step screen	band screen
Grit removal	yes	yes	yes	yes
Primary settling	yes	yes	none	none
Primary sludge thickening	RST	none	none	none
Secondary treatment	SBR	MLE	OD	OD
EAS Thickening	DAFT	DAFT	DAFT	DAFT
Sludge digestion	anaerobic digestion (ESD)	Conventional anaerobic digestion	none	none
Sludge dewatering	centrifuge	centrifuge	centrifuge	centrifuge
Sludge conditioning	none	none	none	none
SRT aeration tanks (days)	16 – 22	10 – 12	20 – 25	20 – 25
SRT digester (days)	20 +	20	N/A	N/A

RST = rotary screw thickener. SBR = sequencing batch reactor. MLE = modified Ludzak-Ettinger process. OD = oxidation ditch. EAS = excess activated sludge. DAFT = dissolved air floatation thickener. ESD = egg-shaped digesters. SRT = sludge retention time. N/A = not applicable.

This report is the first summary report for the Phase II project, produced for the Water Corporation of Western Australia (WCWA) and the Australian and New Zealand Biosolids Partnership (ANZBP). The report describes the work conducted during the period of October 2012 to December 2012.

2.0 Methodology

Fresh samples of anaerobically digested sludge and dewatered biosolids cake were obtained from Woodman Point and Beenyup WWTPs. Samples of partially treated sludge (oxidation ditch) and dewatered biosolids cake were obtained from Gordon Rd and Kwinana WWTPs. Aged biosolids samples were obtained from Woodman Point WWTP. Two aged biosolids samples were obtained: aged biosolids sample 1 (ABS1) sampled in January 2011 and aged biosolids sample 2 (ABS2) sampled in September 2012. The aged biosolids samples, ABS1 and ABS2 were stored for several months either in the fridge or at room temperature. The sludge and biosolids properties of the samples collected are summarised in Table 2. The sludge and biosolids samples were analysed for the presence of volatile sulphur compounds (MT, ET, EMS, DMS, DMDS, DMTS and DEDS) and other volatile organic compounds (toluene, ethylbenzene, styrene, *p*-cresol, indole and skatole) using the HS SPME-GC-MS methods developed in Phase I of this project (Gruchlik *et al.*, 2012a; 2012b). In an initial screen, small quantities (50 – 80 mg) of the sludge and biosolids samples were analysed as is without calibration and without an internal standard. The amounts of the VOSCs present in these samples are represented by the peak areas, corrected for the amount of moist sample used.

Table 2. Summary of sludge and biosolids properties of the samples collected.

Sample	Sampling Date	SRT (days)	% Dry solids (DS) in digested or treated sludge (before polymer)	% DS in the resulting dewatered biosolids cake
ABS1	21/01/2011	35	3.0	19.4
ABS2	09/2012	This sample is a mixture of different batches of biosolids cake, so specific SRT and % DS data are unavailable for this sample.		
Woodman Point	03/10/2012	28.5	3.0	18.0
Beenyup	03/10/2012	22	1.3	19.9
Gordon Rd	03/10/2012	21.3	3.3	16.7
Kwinana	17/10/2012	23	3.8	15.3

The fresh biosolids cake samples from Woodman Point, Beenyup and Gordon Rd WWTPs and the aged biosolids samples, ABS1 and ABS2, were also assessed by an odour panel of six panellists. The panellists were asked to describe the odour of each sample and to rank the samples from the least to the most odorous. The odour panel measurements were conducted according to standard protocols.

3.0 Results and discussion

3.1 Analysis of sludge and biosolids samples by HS SPME-GC-MS

Semi-quantitative comparison of the sludge samples and the corresponding biosolids samples from each of the WWTPs showed that there were no significant differences between the sludge and the dewatered biosolids cakes in terms of the types of compounds present (see Appendix for examples of the GC chromatograms obtained for each sample). However, the dewatered biosolids cakes did contain much greater quantities of the VOSCs (DMS, DMDS and DMTS) compared to the corresponding sludge samples (Figures 1 – 4). This finding is consistent with previous reports that odours produced from dewatered biosolids were generally greater than the odours produced from other locations samples throughout the treatment plants (Higgins, *et al*., 2008). However, it should be noted that the quantities of the VOSCs reported in this report are based on the weight of moist samples and the moisture content would have an effect on the amount of VOSCs present.

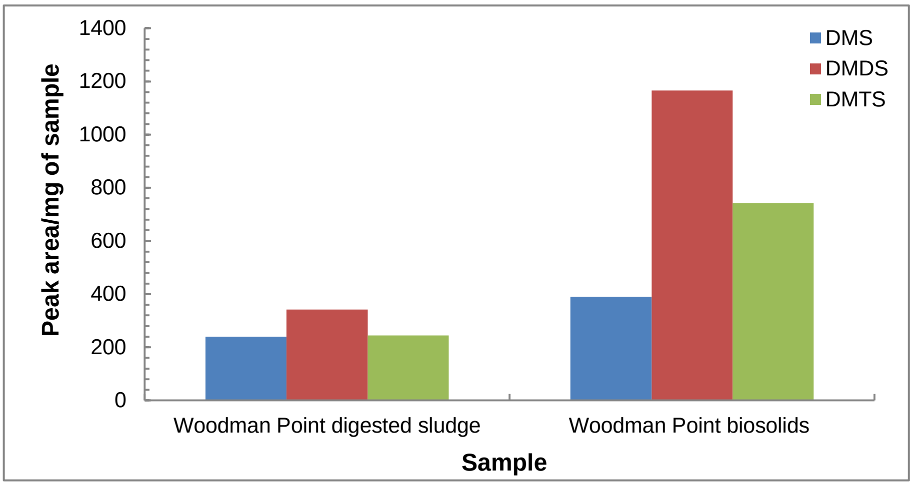

Figure 1. Semi-quantitative assessment of odorous sulphur compounds present in Woodman Point anaerobically digested sludge and biosolids samples. Sampled and analysed on 3 October 2012.

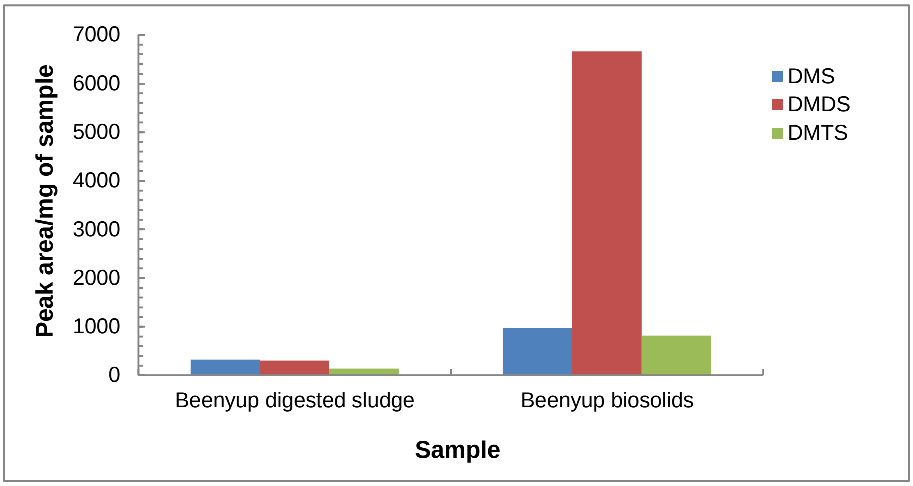

Figure 2. Semi-quantitative assessment of odorous sulphur compounds present in Beenyup anaerobically digested sludge and biosolids samples. Sampled and analysed on 3 October 2012.

Figure 3. Semi-quantitative assessment of odorous sulphur compounds present in Gordon Rd partially treated (oxidation ditch) sludge and biosolids samples. Sampled and analysed on 3 October 2012.

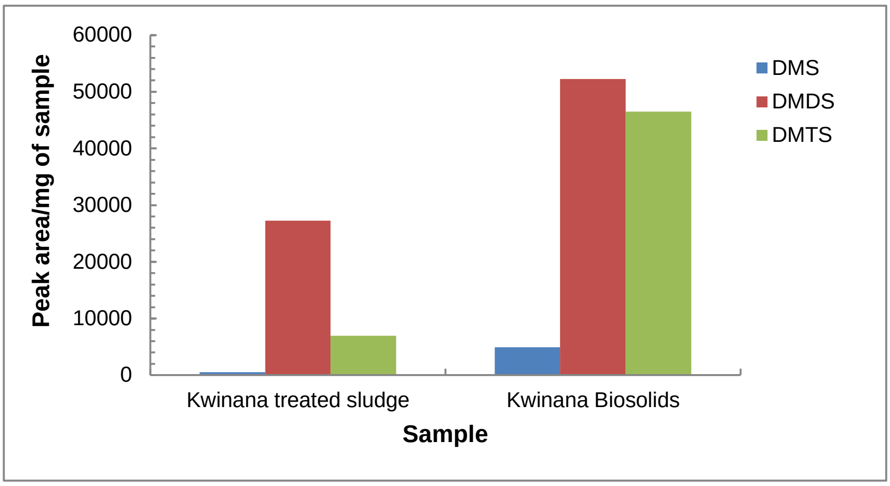

Figure 4. Semi-quantitative assessment of odorous sulphur compounds present in Kwinana partially treated (oxidation ditch) sludge and biosolids samples. Sampled on 17 October 2012 and analysed on 18 October 2012.

Semi-quantitative assessment of the initial concentrations of VOSCs present in fresh biosolids samples showed that those from Gordon Rd and Kwinana WWTPs contained the highest concentrations of DMS, DMDS and DMTS, compared to cakes from Woodman Point and Beenyup (Figure 5).

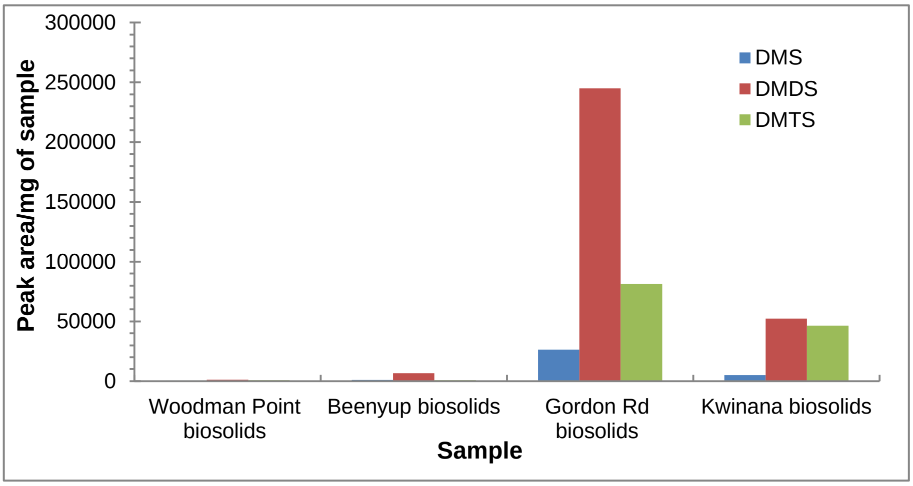

Figure 5. Semi-quantitative assessment of the initial concentrations of sulphur compounds present in fresh biosolids samples from the different WWTPs. Biosolids samples from Woodman Point, Beenyup and Gordon Rd WWTPs were sampled and analysed on 3 October 2012. Biosolids sample from Kwinana WWTP was sampled on 17 October 2012 and analysed on 18 October 2012.

The TVOSC profiles of the dewatered biosolids cakes from Woodman Point, Beenyup and Gordon Rd were monitored over a period of 38 days and are shown in Figures 6 and 7. As expected, the TVOCS concentrations peaked within the first 1 to 2 weeks and then decreased for all three cakes.

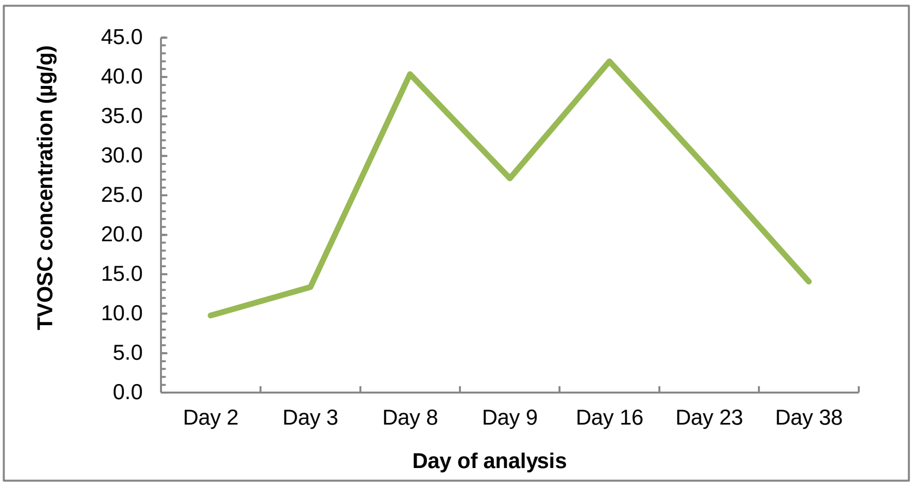

Figure 6. TVOSC profile for dewatered biosolids cake from Gordon Rd WWTP (TVOSC = total volatile organic sulphur compounds, measured as the sum of the DMS, DMDS and DMTS concentrations per gram of moist biosolids).

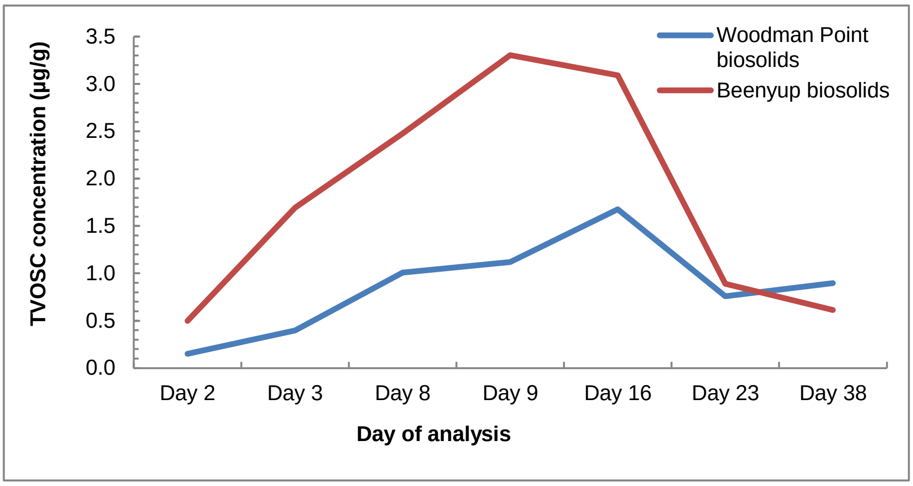

Figure 7. TVOSC profiles for dewatered biosolids cakes from Woodman Point and Beenyup WWTP (TVOSC = total volatile organic sulphur compounds, measured as the sum of the DMS, DMDS and DMTS concentrations per gram of moist biosolids).

The dewatered biosolids cakes were also analysed for the presence of the odorous volatile aromatic compounds (OVACs), such as indole and skatole, after several weeks of incubation. Significant quantities of indole and skatole were found in biosolids samples from all of the four study sites although overall concentrations were greater in samples from Gordon Rd and Kwinana than in those from Woodman Point and Beenyup (Figures 8 and 9). In the cakes from Woodman Point, skatole occurred in greater concentrations than indole, whereas in cakes from Beenyup the reverse was true.

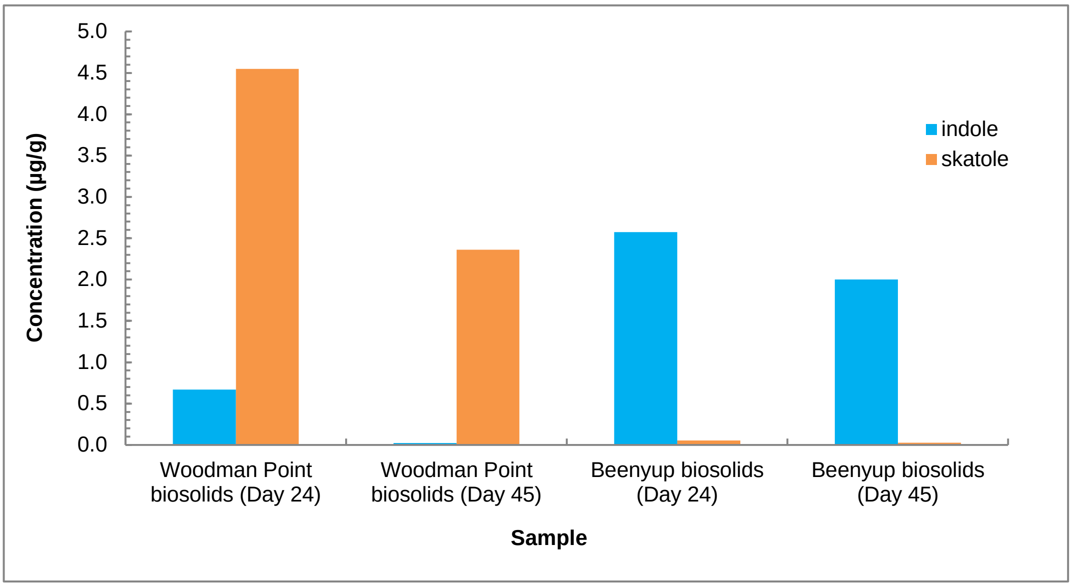

Figure 8. Concentrations of indole and skatole in dewatered biosolids cakes from Woodman Point and Beenyup WWTPs after 24 and 45 days of incubation, as measured by HS SPME-GC-MS method for the analysis of OVACs. Concentration is per gram of moist biosolids sample.

It should be noted that indole and skatole were detected in GC chromatograms of cakes from Gordon Rd and Kwinana as early as day 1 and 2 of incubation (see Figure 19 in the Appendix) but were not detected in cakes from Woodman Point and Beenyup until much later.

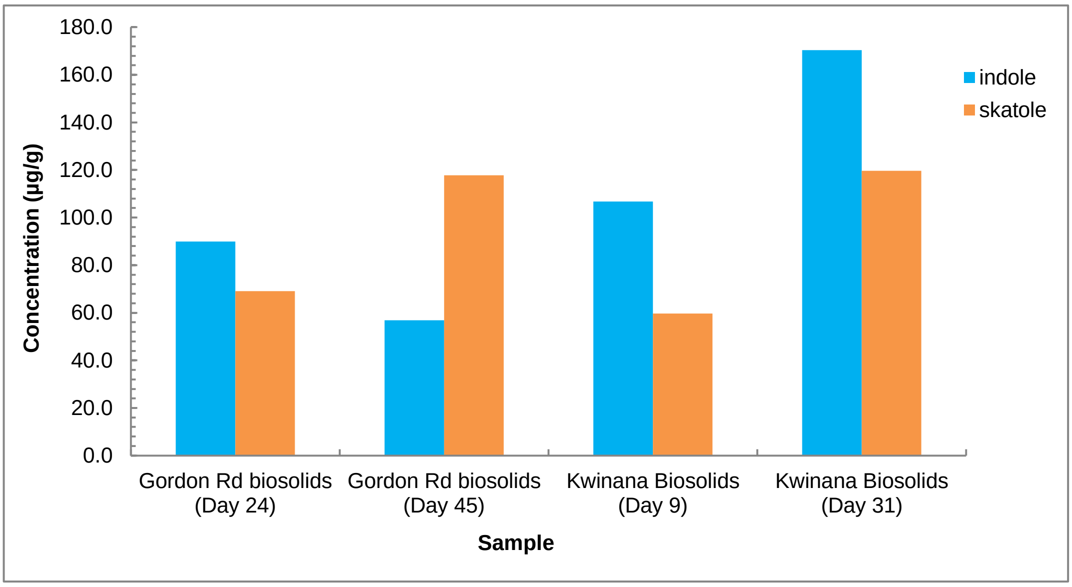

Figure 9. Concentrations of indole and skatole in dewatered biosolids cakes from Gordon Rd WWTP (after 24 and 45 days of incubation) and Kwinana WWTPs (after 9 and 31 days of incubation), as measured by HS SPME-GC-MS method for the analysis of OVACs. Concentration is per gram of moist biosolids sample.

Indole and skatole were not detected in the aged biosolids sample ABS1 (sampled from Woodman Point in January 2011), but the aged biosolids sample ABS2 (sampled from Woodman Point in September 2012) still contained minor traces of indole and skatole. The bulk of the compounds tentatively identified, based on their mass spectra and/or library matches, in the aged biosolids samples ABS1 and ABS2 consisted of long chain hydrocarbons, various alkyl benzenes, terpenes, cyclic hydrocarbons and other aromatic compounds.

3.2 Odour panel assessment of fresh and aged biosolids cakes

Biosolids samples from Woodman Point (fresh and aged (ABS1 and ABS2)), Beenyup and Gordon Rd WWTPs were assessed by an odour panel (6 panellists) on day 3 of incubation (with respect to the fresh samples). The panellists were asked to describe the odour of each sample and to rank the samples from the least to the most odorous. The aged biosolids sample ABS1, sampled from Woodman Point WWTP in January 2011, was described as having an earthy/musty/mouldy odour by 100% of the panellists (Figure 10) and was ranked as the least odorous by 83% of the panellists (Figure 11). Biosolids cake from Gordon Rd WWTP was ranked as the most odorous with a faecal/septic odour by 100% of the panellists (Figures 10 and 11). The odour panel results were consistent with chemical analyses of the biosolids (HS SPME-GC-MS analyses carried out on the same day, i.e. the sample that was designated as the most odorous by the panellists also contained the highest concentrations of TVOSCs; the sample that was designated as the least odorous contained the lowest concentrations of TVOSCs (Figure 12)). A sample from Kwinana WWTP was not available for odour panel testing on this occasion.

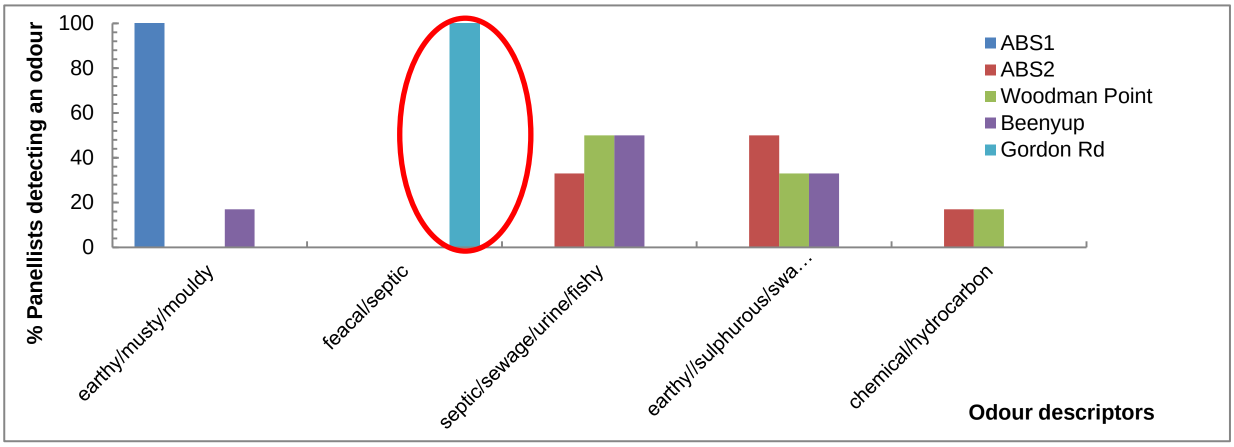

Figure 10. Description of the odours detected in aged (ABS 1, ABS 2) and fresh biosolids cakes by an odour panel on 5 October 2012 (i.e. day 3 of incubation with respect to the fresh biosolids samples).

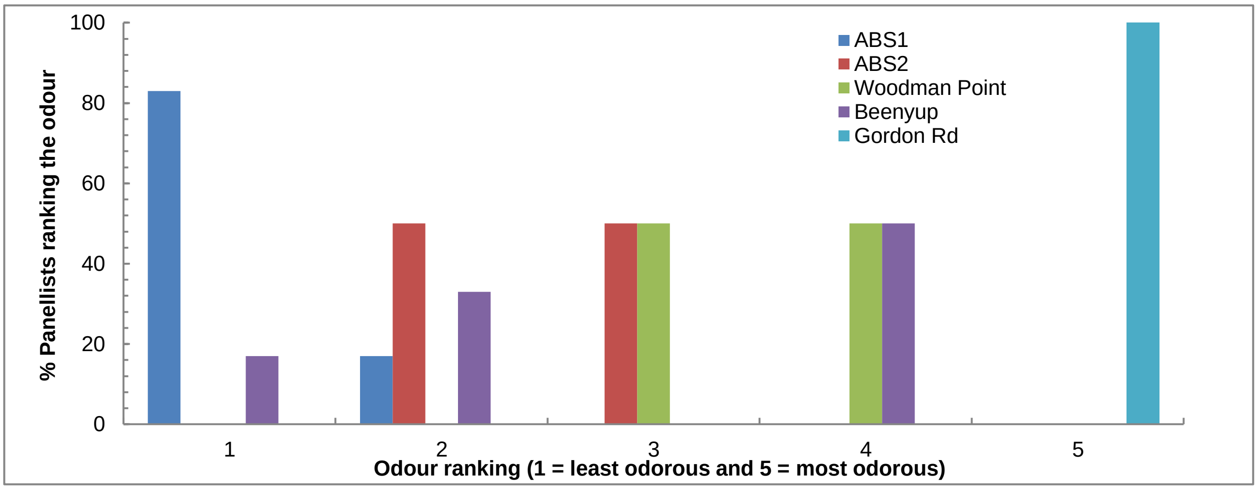

Figure 11. Odour ranking of aged and fresh biosolids cakes, as ranked by an odour panel on 5 October 2012 (i.e. day 3 of incubation with respect to the fresh biosolids samples).

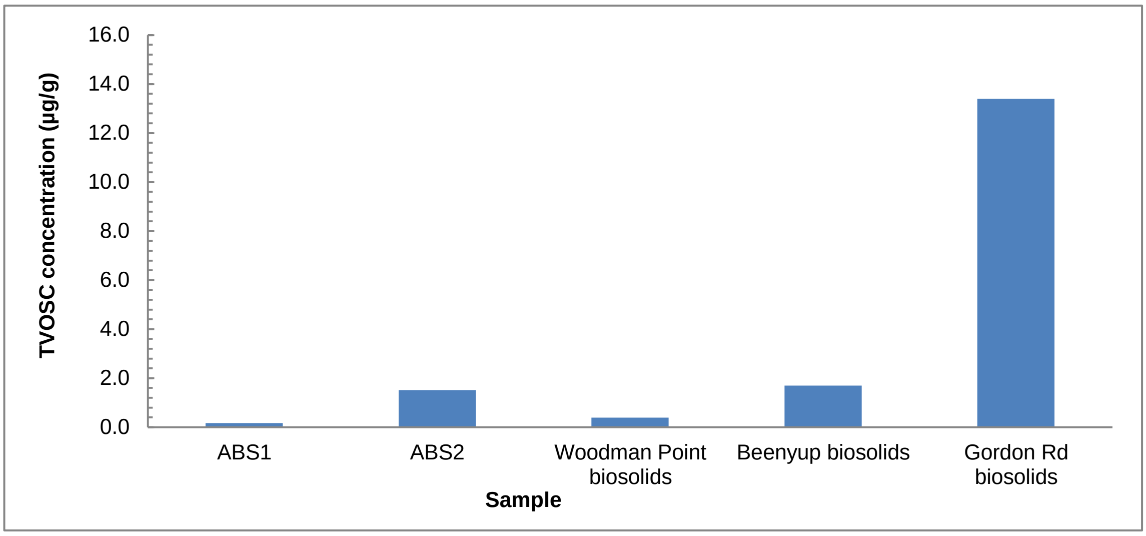

Figure 12. TVOSC concentrations in aged and fresh biosolids samples as measured by HS SPME-GC-MS method for the analysis of sulphur compounds on 5 October 2012 (i.e. day 3 of incubation with respect to the fresh biosolids samples). Note: Kwinana sample was not available at this time of testing. (TVOSC = total volatile organic sulphur compounds, measured as the sum of the DMS, DMDS and DMTS concentrations per gram of moist biosolids).

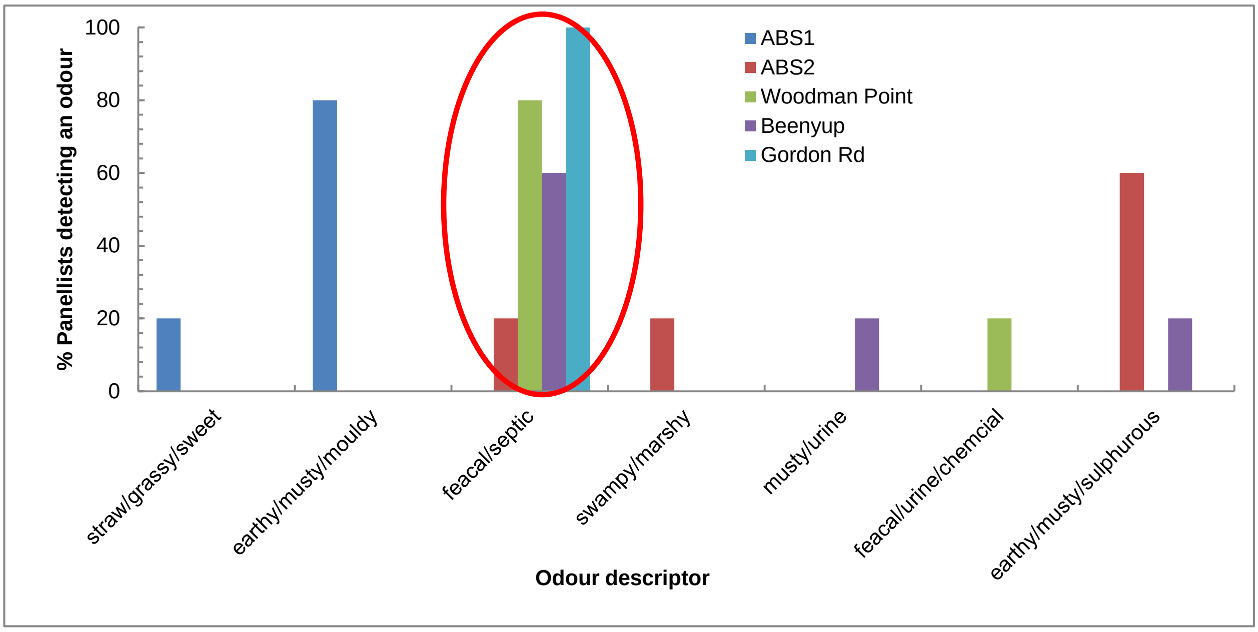

Figure 13. Description of the odours detected in aged and fresh biosolids cakes by an odour panel on 9 November 2012 (i.e. day 38 of incubation with respect to the fresh biosolids samples).

A follow up assessment of the fresh and aged biosolids cakes was undertaken by an odour panel (5 panellists) approximately one month after the initial odour panel assessment to determine if there were any changes in the odours detected. The panellists (same as those used in the initial assessment except for one panellist) were again asked to describe the odours and rank the samples from the least to the most odorous. The results are shown in Figures 13 and 14. The most significant difference between the two assessments was the increase in the percentage of panellists detecting a faecal odour in biosolids cakes from Woodman Point and Beenyup WWTPs in the second assessment (Figure 13), compared to the initial assessment (Figure 10). The detection of the faecal odour by panellists in cakes from Woodman Point and Beenyup during the second assessment was consistent with the detection of significant concentrations of indole and skatole by HS SPME-GC-MS (Figure 8). No faecal odour was detected by the panellists in cakes from Woodman Point and Beenyup during the initial assessment. The biosolids cakes from Woodman Point and Beenyup were not analysed for OVACs by HS SPME-GC-MS at the time of the initial odour panel assessment because, based on previous observations (Gruchlik *et al*., 2012a; 2012b) and literature reports (Chen, *et al*., 2004 and 2006; Novak, 2012), one of the major sources of odours during the first 1-2 weeks of biosolids storage is due to the production of VOSCs, while the OVACs start to accumulate only after VOSCs have been mostly depleted (Chen, *et al*., 2004 and 2006). For example, indole and skatole begin to form at approximately 30 – 40 days, peak at approximately 100 days and begin to disappear at about 125 – 135 days of incubation (Novak, 2012).

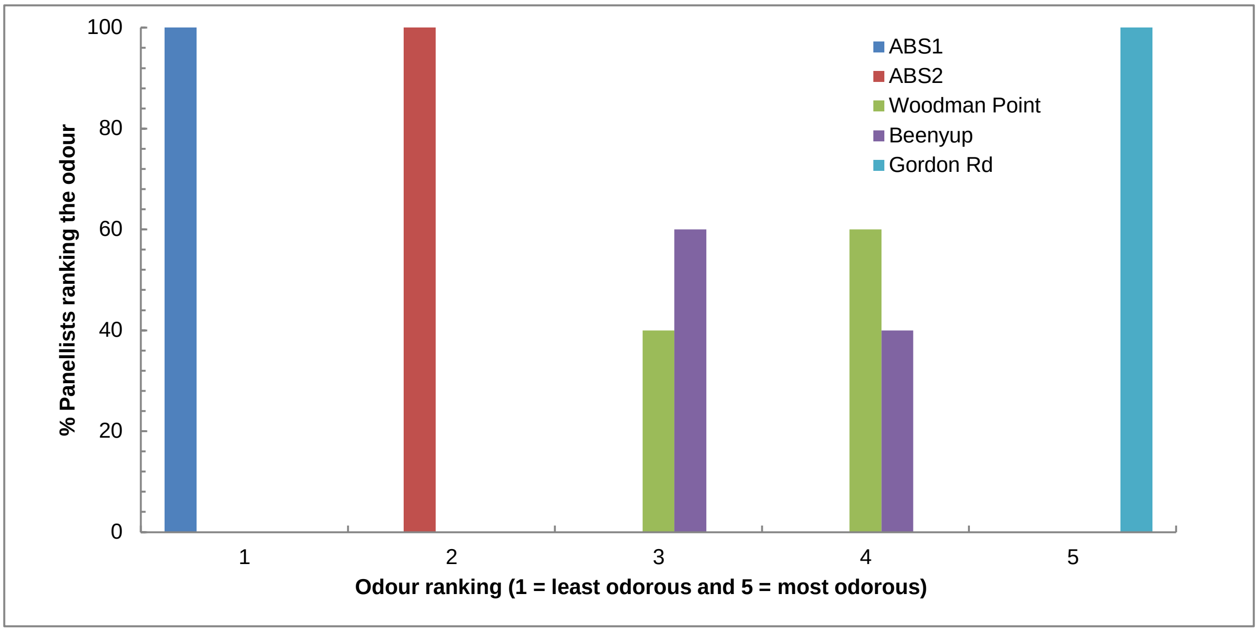

Figure 14. Odour ranking of aged and fresh biosolids cakes, as ranked by an odour panel on 9 November 2012 (i.e. day 38 of incubation with respect to the fresh biosolids samples).

The aged biosolids sample ABS1, sampled from Woodman Point WWTP in January 2011, was again described as having an earthy/musty/mouldy odour by 80% of the panellists (Figure 13) and was ranked as the least odorous by 100% of the panellists (Figure 14). Biosolids cake from Gordon Rd WWTP was again ranked as the most odorous with a faecal/septic odour by 100% of the panellists (Figures 13 and 14).

4.0 Conclusions and future work

In the reporting period from October to December 2012, four sites were selected for study in this Phase II project: Woodman Point, Beenyup, Gordon Rd and Kwinana WWTPs. Fresh samples of sludge and biosolids cake were collected from each of these WWTPs and analysed for volatile sulphur compounds and OVACs by HS SPME-GC-MS. Two aged biosolids samples were also obtained from Woodman Point WWTP. The fresh and aged biosolids samples were assessed by an odour panel. The following conclusions are based on this limited set of experimental results:

- The main odour compounds identified in fresh biosolids cakes from Woodman Point, Beenyup, Gordon Rd and Kwinana WWTPs included: DMS, DMDS and DMTS. Significant quantities of indole and skatole were detected after approximately one month of incubation but were mostly not detectable in fresh samples, with the exception of biosolids cakes from Kwinana and Gordon Rd which showed the presence of indole and skatole as early as day 1 and 2 of incubation. The bulk of the compounds tentatively identified in the aged biosolids samples consisted of long chain hydrocarbons, various alkyl benzenes, terpenes, cyclic hydrocarbons and other aromatic compounds.

- There were no significant differences between the sludge and the dewatered biosolids cakes in terms of the types of compounds present however, the dewatered biosolids cakes contained much greater quantities of the odorous sulphur compounds (DMS, DMDS and DMTS) than the corresponding sludge samples.
- Dewatered biosolids cakes obtained from Gordon Rd and Kwinana WWTPs, which use oxidation ditches to process the sludge, contained the highest concentrations of the volatile sulphur compounds (DMS, DMDS and DMTS) as well as the highest concentrations of the OVACs indole and skatole, compared to the anaerobically digested cakes from Woodman Point and Beenyup WWTPs.
- Results from odour panel assessments of fresh biosolids cakes from Woodman Point, Beenyup and Gordon Rd WWTPs as well as two aged biosolids samples showed that the aged biosolids sample ABS1, sampled from Woodman Point WWTP in January 2011, was ranked as the least odorous with an earthy/musty/mouldy odour by the majority of panellists. Biosolids cake from Gordon Rd WWTP was ranked as the most odorous with a faecal/septic odour by 100% of the panellists. The odour panel results correlated well with chemical analysis of the biosolids by HS SPME-GC-MS.
- A follow up analysis of the same biosolids cakes by the same odour panel approximately one month after the initial assessment showed an increase in the percentage of panellists detecting a strong faecal odour in biosolids cakes from Woodman Point and Beenyup, which was not detected in the initial assessment. These observations were consistent with the detection of significant concentrations of indole and skatole by HS SPME-GC-MS and are supported by literature reports that indole and skatole begin to form after approximately 30 – 40 days of incubation.

Chemical addition trials of alum addition to digested sludge prior to dewatering will be conducted in April 2013, commencing with sludge from Woodman Point. The work from Phase I study will be presented at the 5th IWA Odours and Air Emissions Conference in San Francisco in early March 2013 (4 – 7 March 2013).

5.0 References

Agarwalla, B. (2012). Water treatment processes. Power point presentation at Water Corporation of Western Australia, 13 June 2012.

Chen, Y., Higgins, M., Murthy, S., Maas, N., Covert, K. and Toffey, W. (2006) Production of Odorous Indole, Skatole, *p*-Cresol, Toluene, Styrene and Ethylbenzene in Biosolids. Journal of Residuals Science & Technology, 3, No. 4, 193-202.

Chen, Y., Higgins, M., Murthy, S., Maas, N., Covert, K., Weaver, J., Toffey, W., Rupke, M. and Ross, D. (2004). Mechanisms for the Production of Odorous Volatile Aromatic Compounds in Wastewater Biosolids. In *Proceedings of Water Environment Federation and AWWA Annual Biosolids and Residuals Conference*, Salt Lake City, Utah, USA, 540-553.

Gruchlik, Y., Heitz, A., Joll, C., Driessen, H., Fouché L., Charrois, J. and Penney, N. (2012a) Laboratory scale investigations of potential odour reduction measures in biosolids – Phase 1. *CWQRC Report No: CWQRC-2012-006*.

Gruchlik, Y., Heitz, A., Joll, C., Driessen, H., Fouché L., Penney, N. and Charrois, J. (2012b) Laboratory scale investigations of potential odour reduction strategies in biosolids. *Water*, **39**, No. 7, 58-64.

Higgins, M.J., Chen, Y., Novak, J.T., Glindemann, D., Forbes, R., Erdal, Z., Witherspoon, J., McEwen, D., Murthy, S., Hargreaves, J.R. and Adams, G. (2008) A Multi-Plant Study to Understand the Chemicals and Process Parameters Associated with Biosolids Odours. *Environmental Engineer: Applied Research and Practice*, **5**, 1-13. http://www.aaee.net/Downloads/EEJournalV5P1.pdf

Novak, J. T. (2012). WERF ROSI Project: Regrowth, Odours and Sudden Increase (ROSI): Odours. WERF 2012 Webinar Series, 5 September 2012.

APPENDIX

GC chromatograms of sludge and biosolids samples.

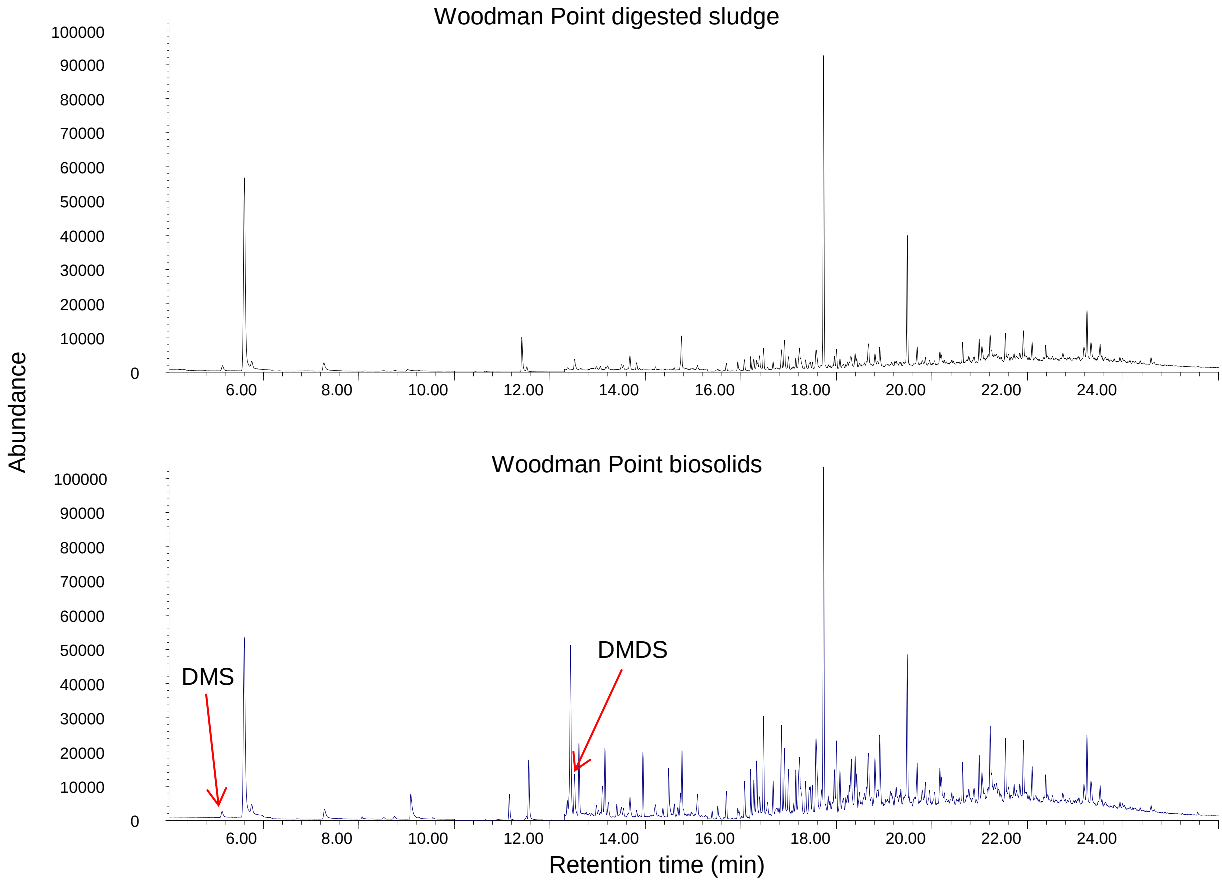

Figure 15. GC chromatograms of digested sludge and biosolids samples from Woodman Point WWTP. Samples were collected and analysed on 3 October 2012. Samples were analysed using the HS SPME-GC-MS method for the analysis of sulphur compounds in selected ion monitoring mode (SIM) using a ZB5-MS capillary column.

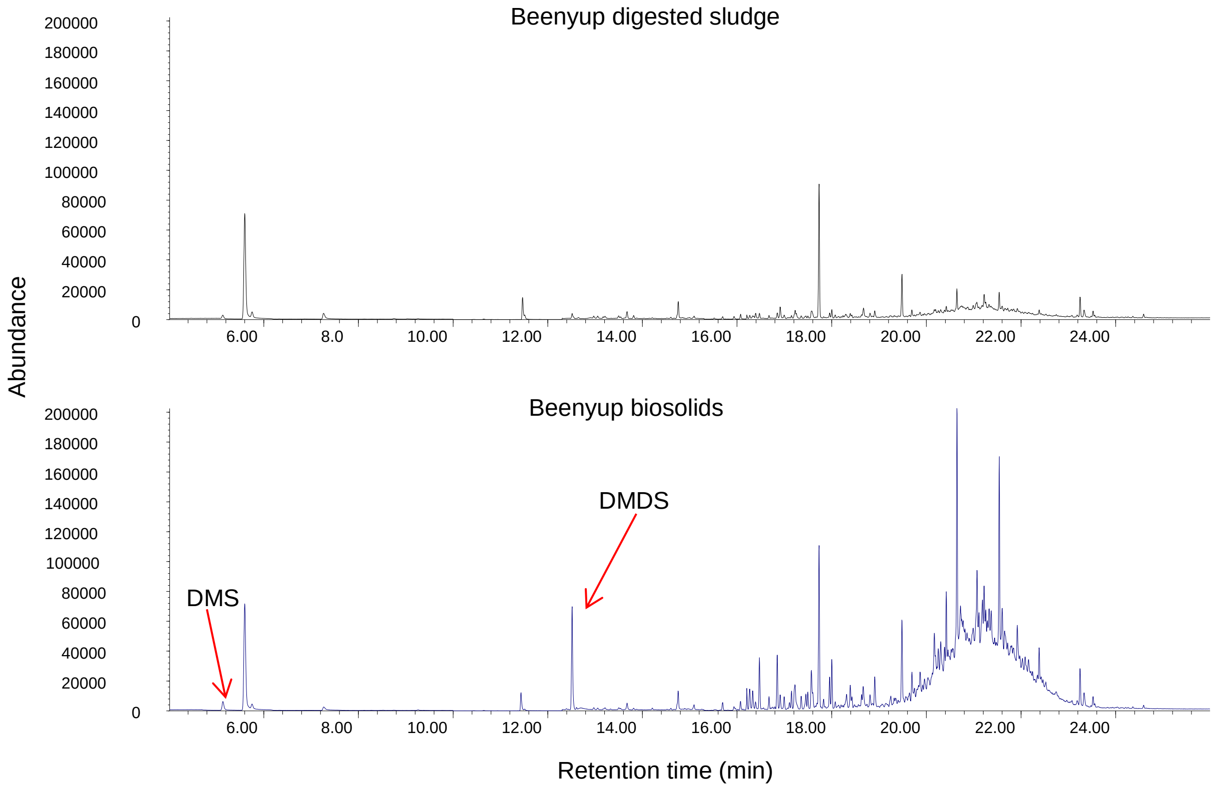

Figure 16. GC chromatograms of digested sludge and biosolids samples from Beenyup WWTP. Samples were collected and analysed on 3 October 2012. Samples were analysed using the HS SPME-GC-MS method for the analysis of sulphur compounds in selected ion monitoring mode (SIM) using a ZB5-MS capillary column.

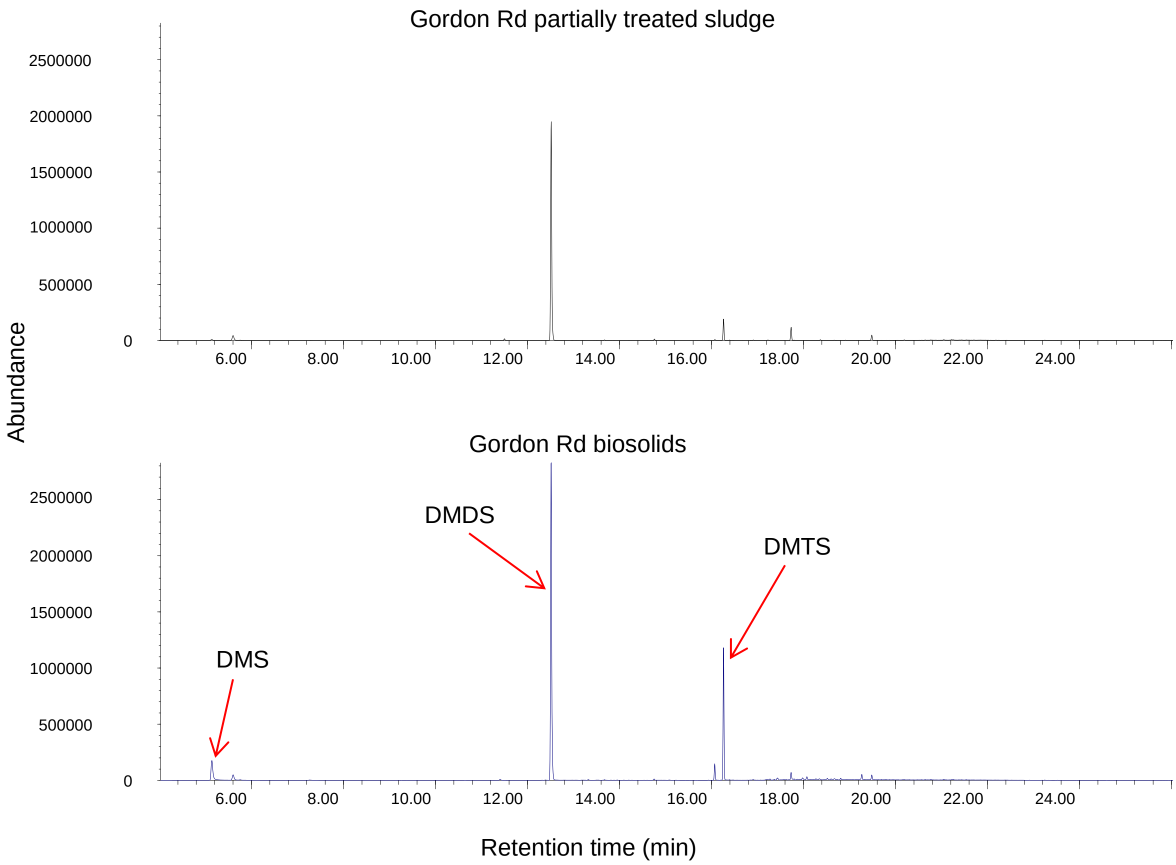

Figure 17. GC chromatograms of partially treated sludge and biosolids samples from Gordon Rd WWTP. Samples were collected and analysed on 3 October 2012. Samples were analysed using the HS SPME-GC-MS method for the analysis of sulphur compounds in selected ion monitoring mode (SIM) using a ZB5-MS capillary column.

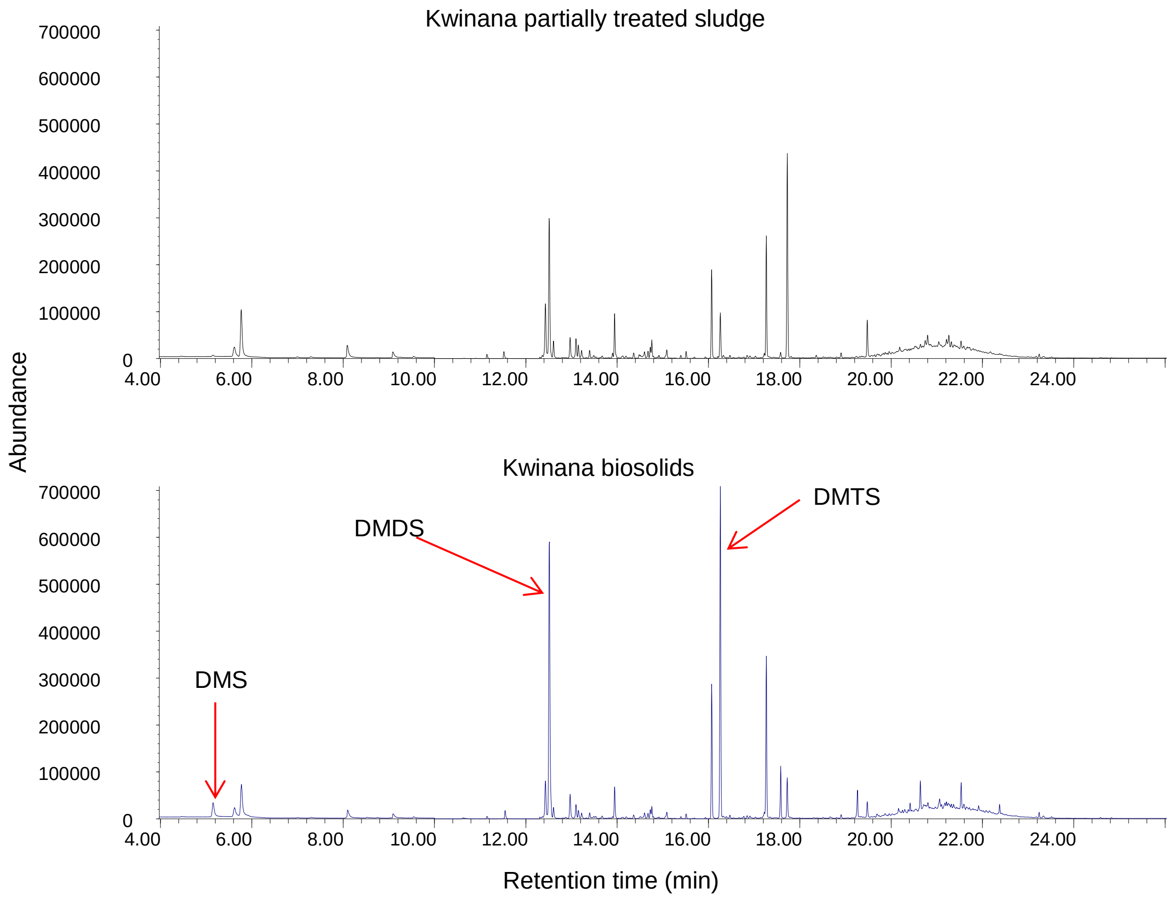

Figure 18. GC chromatograms of partially treated sludge and biosolids samples from Kwinana WWTP. Samples were collected on 17 October 2012 and analysed on 18 October 2012. Samples were analysed using the HS SPME-GC-MS method for the analysis of sulphur compounds in selected ion monitoring mode (SIM) using a ZB5-MS capillary column.

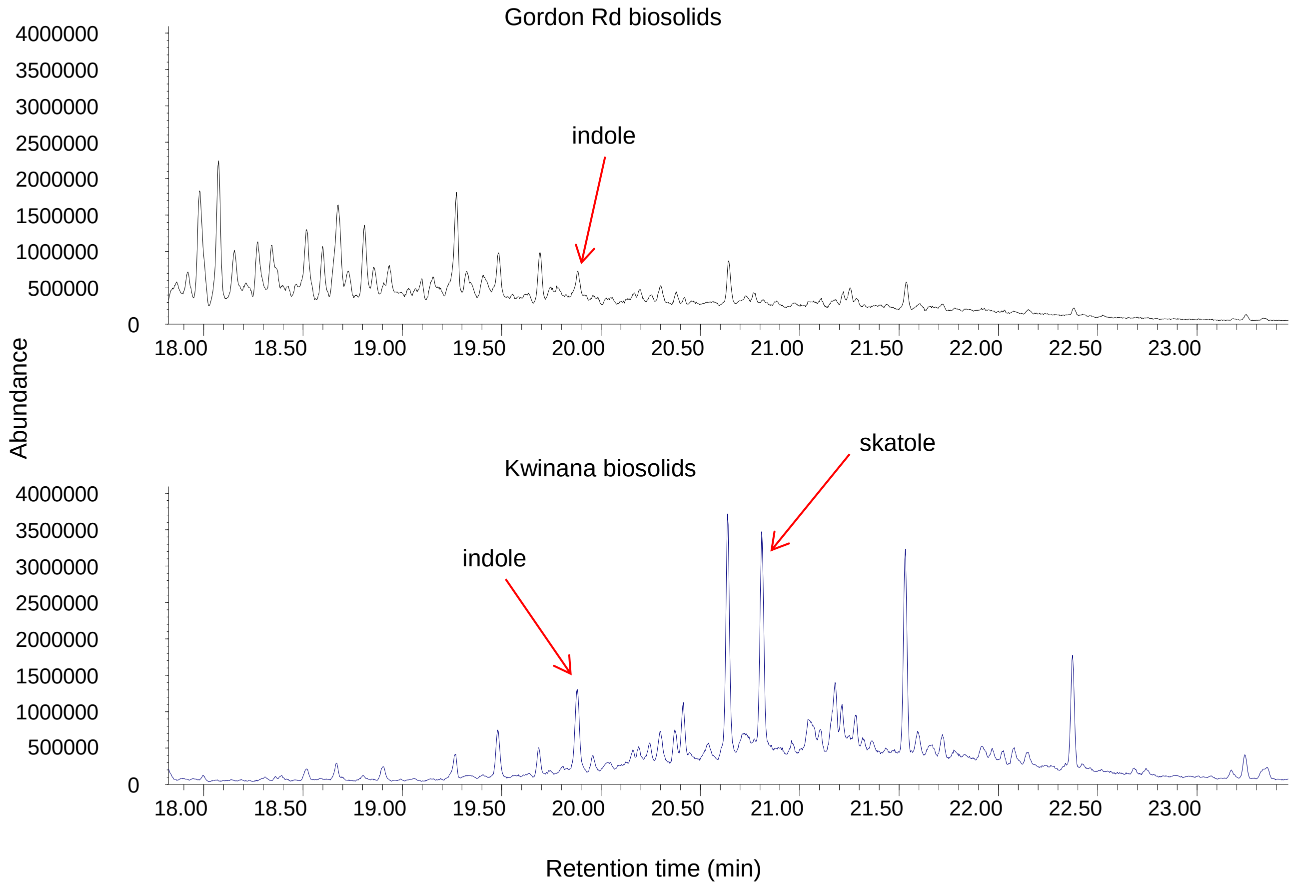

Figure 19. Expanded GC chromatograms of biosolids samples from Gordon Rd (sampled and analysed on 3 October 2012, i.e. day 1) and Kwinana (sampled on 17 October 2012 and analysed on 18 October 2012, i.e. day 2), showing the presence of indole and skatole. Samples were analysed using the HS SPME-GC-MS method for the analysis of sulphur compounds in full scan mode using a ZB5-MS capillary column.

NOTE: the method for the analysis of sulphur compounds is not optimised for the analysis of the OVACs and thus the amounts of indole and skatole may be higher than shown in the above chromatograms.