Odour reduction strategies for biosolids produced from a Western Australian wastewater treatment plant: results from Phase I laboratory trials

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ABSTRACT

This study investigated sources of odours from biosolids produced from a Western Australian wastewater treatment plant and examined possible strategies for odour reduction, specifically chemical additions and reduction of centrifuge speed on a laboratory scale. To identify the odorous compounds and assess the effectiveness of the odour reduction measures trialled in this study, headspace solid-phase microextraction gas chromatography-mass spectrometry (HS SPME-GC-MS) methods were developed. The target odour compounds included volatile sulphur compounds (e.g. dimethyl sulphide, dimethyl disulphide and dimethyl trisulphide) and other volatile organic compounds (e.g. toluene, ethylbenzene, styrene, p-cresol, indole and skatole). In our laboratory trials, aluminium sulphate added to anaerobically digested sludge prior to dewatering offered the best odour reduction strategy amongst the options that were investigated, resulting in approximately 40% reduction in the maximum concentration of the total volatile organic sulphur compounds, relative to control.

Key words | biosolids, HS SPME-GC-MS, odour, odour reduction, volatile sulphur compounds

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INTRODUCTION

The production of odours during biosolids storage and land application is a significant problem for wastewater utilities and can have a negative impact on beneficial re-use programmes. As such, considerable research has been carried out to understand the causes of odour formation in biosolids and to develop management strategies to minimise these odours (Adams et al. 2008). Odour production in biosolids is a complex process influenced by several factors including, but not limited to, process variables within anaerobic digestion and dewatering processes, as well as the relationships between odours and the concentrations of odorants (Adams et al. 2008). Due to differences in biosolids characteristics, treatment processes and operating conditions associated with different wastewater treatment plants (WWTPs), the chosen odour reduction strategies need to be based on the site-specific conditions at each WWTP. In most cases, laboratory and/or pilot-scale trials would be required to find the most suitable odour reduction strategy (Adams et al. 2008).

Compounds associated with odours from biosolids facilities include volatile organic sulphur compounds (VOSCs) such as methanethiol (MT), dimethyl sulphide (DMS), dimethyl disulphide (DMDS), dimethyl trisulphide (DMTS), as well as inorganic sulphur compounds such as hydrogen sulphide (H_2S) (Adams et al. 2008; Higgins et al. 2008). Nitrogenous compounds such as trimethylamine, ammonia, and volatile fatty acids can also be potential sources of odour (Higgins et al. 2008). Odorous volatile aromatic compounds (OVACs) such as toluene, ethylbenzene, styrene, p-cresol, indole and skatole have been identified in headspace samples from stored biosolids (Chen et al. 2006).

To assess the effectiveness of biosolids odour reduction strategies, appropriate sampling and analytical techniques are required to measure accurately the odorous compounds present in the biosolids cake. Glindemann et al. (2006) used a static headspace method for the analysis of odorous gases from dewatered sludge cakes in the

laboratory. This method utilised gas-tight bottles for incubating biosolids and involved manual sampling and injection of the headspace gases into the gas chromatography (GC) inlet using a gas-tight syringe (Glindemann *et al.* 2006). Although this method has been reported to be representative of the biosolids storage pile interior, easy to use and highly reproducible (Glindemann *et al.* 2006), manual injections of the headspace gases into the GC inlet are time consuming and laborious, and limited to analysis of only a few samples (Gruchlik *et al.* 2012). Solid-phase microextraction (SPME) coupled with GC-mass spectrometry (GC-MS) has also been used for the analysis of odorous compounds in several biosolids projects (e.g. Turkmen *et al.* 2004; Kim *et al.* 2005). However, many of these methods used complicated apparatus for the calibration and sampling of the gaseous odorants and involved manual injections of the SPME fibre into the GC injector.

In this study we used SPME-GC-MS for the analysis of odorous compounds in the headspace of wet biosolids. In this method biosolids samples were analysed as 'aqueous' samples, which do not require any complex sampling equipment, are reproducible, and the analysis is fully automated, allowing for a higher throughput of samples (Gruchlik *et al.* 2012).

The main objectives of this study were to: (a) develop analytical methods to identify the chemical compounds responsible for the odour in biosolids from our test site, (b) assess the effectiveness of trialled odour reduction measures, and (c) determine the most suitable odour reduction strategy for biosolids produced at our test site.

METHODOLOGY

Anaerobically digested sludge (10 L, dry solids (DS) 3.7%, sludge retention time (SRT) 19 days) and plant dewatered biosolids cake (1 kg, DS 16.9%) were obtained from a Perth metropolitan WWTP, which uses activated sludge and sequencing batch reactors, egg-shaped mesophilic anaerobic digestion and centrifuge dewatering. The samples were packed in coolers and transported to the laboratory for use in bench-scale trials (chemical additions and reduction of centrifuge speed). The same batch of anaerobically digested sludge and plant dewatered biosolids cake was used for all chemical addition and centrifuge-speed trials described in this study. The sludge and biosolids samples were stored in a cool room at 4 °C. Polymer used for dewatering was a powder polymer

FO4800SSH from SNF Pty Ltd (supplied by Water Corporation) with a molecular weight of approximately 8 million and a charge density of 80%. The polymer was dosed as a 0.3% (w/v) aqueous solution and the polymer dose was based on the average amount used at our test site and adjusted accordingly to the lab-scale experiments. Aluminium sulphate (alum; $Al_2(SO_4)_3 \cdot 14H_2O$) was used as a 56% w/v aqueous solution. Polyaluminium chloride (PAC23 from Orica) was used as a 23% w/v solution as aluminium oxide. Ferric chloride was used as a 36% w/v aqueous solution, prepared in-house from analytical grade ferric chloride (Sigma-Aldrich).

Laboratory-scale dewatering

We used a laboratory-scale dewatering procedure, previously developed, using a laboratory centrifuge (Heraeus Multifuge 3S with a maximum rotational radius of 18.2 cm) (Gruchlik *et al.* 2012). In order to simulate the dewatering conditions used at our test site the centrifuge was operated at 3850 rpm for 20 minutes. The resulting cake was then processed through a manual food mincer (Avanti food mincer #8) which pushed the cake through a 'scroll-conveyor', followed by extrusion through several openings. The laboratory-dewatered biosolids cake had a similar texture and odour to the plant dewatered sample. The solids content of the laboratory-dewatered cake was comparable to that of the plant dewatered cake.

Chemical addition to digested sludge prior to dewatering

Individual samples of anaerobically digested sludge (approximately 800 g each) in 1 L glass beakers were treated with alum, polyaluminium chloride and ferric chloride at doses of 2 and 4% of metal on a dry weight basis, followed by addition of polymer. The samples were mixed using a jar tester. The mixing regime used was based on that reported by Higgins (2010) and is shown in Figure 1. A control sample, with no chemical addition was also prepared. The samples were dewatered using the dewatering procedure developed in-house. The resulting biosolids cake samples (approximately 200 g) were incubated at room temperature in 1 L Schott bottles. The samples were wrapped in aluminium foil to protect them from light and were monitored for evolution of volatile sulphur compounds (DMS, EMS (ethyl methyl sulphide), DMDS, DEDS (diethyl disulphide) and DMTS) by headspace solid-phase microextraction gas chromatography-mass spectrometry
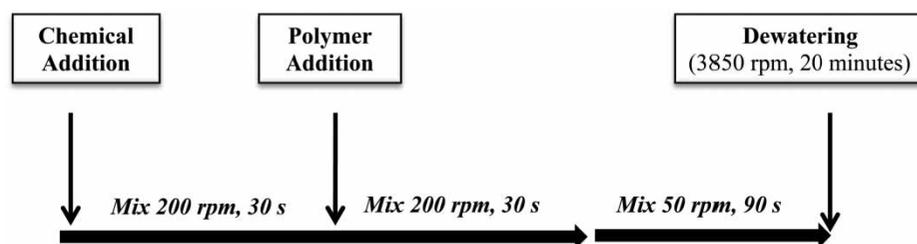
Figure 1 | The mixing regime used in laboratory-scale trials of chemical addition to digested sludge (adapted from Higgins (2010); Gruchlik et al. (2012)).

(HS SPME-GC-MS) every few days over a period of 20 days. Samples were also analysed for the production of OVACs (toluene, ethylbenzene, styrene, p-cresol, indole, and skatole) by HS SPME-GC-MS weekly over a period of 37 days.

Chemical addition to plant dewatered cake

Samples of the plant dewatered biosolids (approximately 85 g) in 400 mL glass beakers were treated with aluminium sulphate hydrate at doses of 2 and 4% of metal on dry weight basis and mixed manually with a stainless steel spatula for approximately 2 min. A control sample (no chemical addition) was prepared in the same way. Cake samples were incubated at room temperature in 250 mL Schott bottles. Samples were wrapped in aluminium foil to protect them from light and monitored for evolution of volatile sulphur compounds by HS SPME-GC-MS every few days over a period of 14 days. Samples were also analysed for the production of OVACs by HS SPME-GC-MS weekly over a period of 16 days.

Centrifuge-speed trials

Samples of untreated (i.e. no chemical addition except for polymer) anaerobically digested sludge (approximately 600 g each) were dewatered at 3850 rpm (control speed), 3460 rpm (10% reduction in speed, relative to control) and 3080 rpm (20% reduction in speed, relative to control) using the dewatering procedure developed in-house. The resulting biosolids cake samples (approximately 190 g) were incubated at room temperature in 500 mL Schott bottles. Samples were wrapped in aluminium foil and were monitored for evolution of volatile sulphur compounds by HS SPME-GC-MS every few days for a period of 20 days. Samples were also analysed for the production of OVACs by HS SPME-GC-MS weekly for a period of 23 days.

HS SPME-GC-MS procedure for the analysis of sulphur compounds

Sulphur compounds (DMS, EMS, DMDS, DEDS and DMTS) were analysed by headspace SPME using a 50/30 μm DVB-CAR-PDMS (divinylbenzene-carboxen-poly-dimethylsiloxane) fibre, followed by GC-MS analysis. SPME was performed using a Gerstel MPS2 Autosampler interfaced with a Hewlett Packard 6890N GC and a Hewlett Packard 5973 Network Mass Selective Detector. A sample of biosolids cake (50–80 mg) was placed into a Teflon-lined screw cap vial (20 mL) and 10 mL of a 500 ng/L DMDS-d_6 internal standard solution in MilliQ water was added, followed by anhydrous sodium sulphate (3 g). The SPME fibre was introduced into the headspace of the vials and extraction was carried out for 10 min at 40 °C. The fibre was then desorbed at 230 °C for 4 min in the injector port of the GC, while analytes were simultaneously cryofocused on the GC column at 0 °C. The oven temperature programme was set up as follows: 0 °C for 2.00 min, increased at 5 °/min to 35 °C then increased at 15 °/min to 315 °C and held at 315 °C for 10 min (run time = 37.67 min). GC separation of sulphur compounds was carried out using helium as the carrier gas at 1.0 mL/min, and a 30 m × 0.25 mm × 1 μm ZB-5MS (Phenomenex®) capillary column. The mass spectrometer (MS) was operated in selected ion monitoring mode and for each sulphur compound, the most abundant ion was used for quantitation and 1–2 characteristic m/z ions were selected for MS confirmation. Samples were analysed against standards of the pure compounds with deuterated DMDS (DMDS-d_6) as an internal standard.

HS SPME-GC-MS procedure for the analysis of OVACs

The OVACs (toluene, ethylbenzene, styrene, p-cresol, indole and skatole) were analysed using a similar procedure to that described above for the sulphur compounds except that a 65 μm PDMS-DVB (polydimethylsiloxane-divinylbenzene)

fibre was used, and extraction was carried out for 30 min at 60 °C. The fibre was desorbed at 250 °C for 5 min in the injector port of the GC and the analytes were not cryofocused. The oven temperature programme was set up as follows: 40 °C for 5.00 min, increased at 20 °C/min to 200 °C, then increased at 10 °C/min to 280 °C and then increased at 20 °C/min to 300 °C and held at 300 °C for 10 min (run time = 32.00 min). Samples were analysed against standards of pure compounds using deuterated ethylbenzene (ethylbenzene-d_{10}) as an internal standard.

Analysis of biosolids samples from the odour reduction trials

Odours from the biosolids samples obtained during odour reduction trials were assessed in terms of the concentration of the total volatile organic sulphur compounds (TVOSC), measured as the sum of the DMS, DMDS and DMTS concentrations present in the biosolids samples and compared with a control sample. Odour reduction (or increase) was considered to be the reduction (or increase) in the maximum TVOSC concentration relative to the control sample.

RESULTS AND DISCUSSION

Validation and optimisation of the HS SPME-GC-MS methods for the analysis of sulphur compounds and OVACs

GC-MS conditions (oven temperature, injector temperature, flow rate and MS parameters) and HS SPME parameters (fibre type, extraction temperature and time, and desorption conditions) for the analysis of sulphur compounds and OVACs were optimised, in order to achieve good baseline separation of analytes and maximise sensitivity and response, while minimising analyte degradation and carry-over (Gruchlik *et al.* 2012).

Degradation of analytes

Certain sulphur compounds such as mercaptans and dimethylpolysulphides (e.g. DMDS) can undergo degradation under certain conditions (Haberhauer-Troyer *et al.* 1999; Kristiana *et al.* 2010). 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. Analysis of a solution containing a mixture of MT and ET showed major peaks for DEDS and the 'scrambled' compound methyl ethyl disulphide (MEDS) formed as a result of a reaction between MT and ET, as well as smaller peaks for DMDS and DMTS. No degradation products were observed for any of the other analytes (Gruchlik *et al.* 2012). Since MT and ET were too reactive under our method conditions, they were excluded from the mixed standard solution. 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.

Odorous compounds identified in biosolids samples from our test site

The main odour compounds identified in fresh biosolids samples from our test site included: DMS, DMDS and DMTS. Indole and skatole were identified in older biosolids samples that were stored at room temperature for a few months and exhibited a strong faecal/nauseating odour. These compounds were not detected in the fresh biosolids samples. This finding is consistent with previous reports that one of the major sources of odours during the first 1–2 weeks of biosolids storage is due to the production of VOSCs by microbial degradation of sulphur-containing amino acids, while the OVACs start to accumulate only after VOSCs have been depleted (Chen *et al.* 2006). 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.

Chemical addition as an odour reduction strategy

Previous research has shown that many of the odour compounds associated with biosolids (e.g. MT, DMS, DMDS) can be formed during microbial degradation of bioavailable protein (Higgins *et al.* 2006). Thus, reducing the amount of bioavailable protein in dewatered biosolids may reduce the formation of odour causing compounds (Higgins 2010). Trivalent metals such as aluminium (Al) and iron (Fe) can bind labile proteins within the floc structure of sludge/biosolids (Subramanian *et al.* 2005). Therefore, addition of aluminium or iron (III) salts to biosolids could reduce the amount of bioavailable protein thereby reducing the production of odorous compounds (Higgins 2010). In a series of laboratory-scale experiments, researchers found that addition of alum to digested sludge prior to dewatering

significantly reduced the production of odour compounds (Adams *et al*. 2008; Higgins 2010). In some cases up to 80% reductions of the peak TVOSC concentrations were observed for aluminium doses as low as 0.5% (dry mass – mass basis) (Adams *et al*. 2008).

The chemicals could be added during conditioning and dewatering or directly to the dewatered cake: it was found that adding metal salts directly to the cake gave better TVOSC reductions compared to adding the salts during the conditioning and dewatering step. However, chemical addition to the cake also resulted in a greater reduction in the pH of the cake to levels below those desirable for land application (Higgins 2010).

In this study, laboratory-scale experiments of addition of aluminium sulphate, polyaluminium chloride and ferric chloride to digested sludge prior to dewatering at doses of 0% (control), 2 and 4% of metal on dry weight basis were conducted. In all of our experiments the primary coagulants were added prior to adding the polymer (Figure 1). The addition of aluminium sulphate to plant dewatered biosolids cakes was also investigated.

Chemical addition to digested sludge prior to dewatering

A 37% reduction of the maximum TVOSC concentration was observed for an aluminium dose of 2%, while a 4% Al dose resulted in a 40% reduction of the maximum TVOSC concentration, relative to the control sample (Figure 2(a)).

The odour reductions observed in our laboratory trials were lower than those reported by the Water Environment Research Foundation (WERF) research team (Adams *et al*. 2008). In one of their laboratory trials, using anaerobically digested sludge from a WWTP located in the east coast of the USA, the WERF researchers observed that the lowest alum dosage examined (0.5% as aluminium) reduced the maximum TVOSC concentration by approximately 83%, while a 2% Al dose resulted in approximately 90% reduction in the maximum TVOSC concentration (Adams *et al*. 2008). The observed differences in the odour reductions obtained in our laboratory trials and those reported by the WERF research team may be due to a number of factors such as differences in the sludge and biosolids characteristics, due
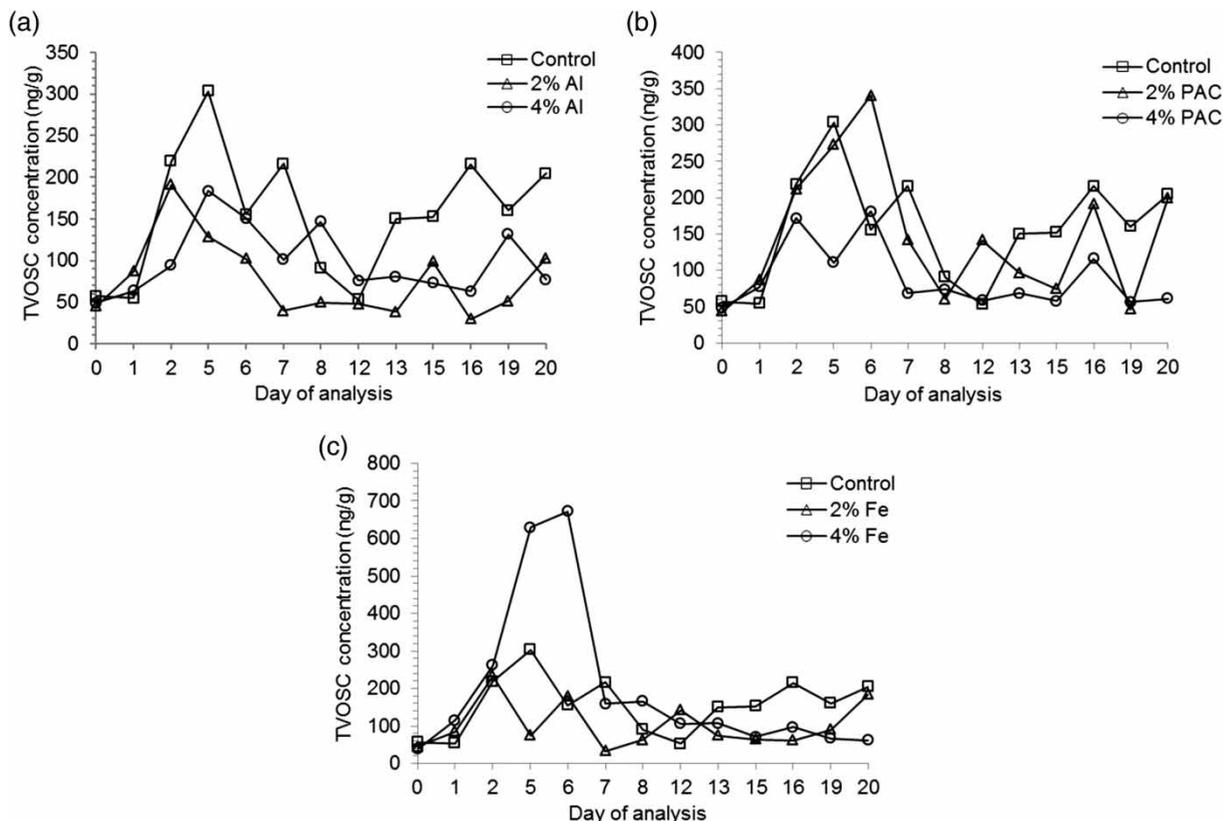

Figure 2 | TVOSC (total volatile organic sulphur compounds) production in biosolids cakes obtained from laboratory scale trials of: (a) aluminium sulphate (Al) addition; (b) polyaluminium chloride (PAC) addition, and (c) ferric chloride (Fe) addition to digested sludge prior to dewatering. (*Note:* TVOSC = the sum of DMS, DMDS and DMTS concentrations.)

to treatment processes and operating conditions associated with different WWTPs (e.g. sludge treatment upstream of anaerobic digestion, digestion conditions and SRT), type of polymer used, chemical contact time, mixing, shear and interactions between the metal and polymer (Adams *et al.* 2008; Gruchlik *et al.* 2012).

Addition of iron at the 2% dose resulted in only a slight decrease (23%) in the maximum TVOSC concentration, while addition of iron at the 4% dose resulted in a 50% increase in the maximum TVOSC concentration, relative to control (Figure 2(c)). It is unclear at this stage why the 4% iron dose resulted in such a large increase in the TVOSC concentration in our laboratory trials. Previously reported results with respect to iron addition as means of odour reduction have been somewhat inconsistent and contradictory, and dependent on the sludge properties and location of iron addition (Adams *et al.* 2008; Higgins 2010; Novak & Park 2010).

Comparison of TVOSC profiles in Figures 2(a)–(c) showed that in all three cases the TVOSC concentrations reached a maximum within the first week of incubation and then decreased, which was consistent with previously reported research (Adams *et al.* 2008).

Chemical addition to plant dewatered cake

In our laboratory trials, a 2% dose of aluminium sulphate (based on aluminium) resulted in a 24% increase in peak TVOSC concentration, while addition of 4% aluminium sulphate resulted in approximately 70% decrease in peak TVOSC concentration, relative to the control sample. However, the pH of the cake treated with 4% aluminium sulphate was also significantly reduced (pH 4.2) to levels that may not be suitable for land application. Therefore to overcome the problem of low pH, lime would need to be incorporated into the biosolids prior to land application to raise the pH to a suitable level (pH 6–8) for land application. These results are consistent with previously reported data (Higgins 2010).

Centrifuge-speed trials

It has been reported that odour emissions from cakes dewatered by high-solids centrifuges were higher than cakes dewatered by other means (Adams *et al.* 2008). This was attributed to the high shear imparted on biosolids during centrifuge dewatering, as a result of higher centrifuge speeds and/or torque conditions. The shear created by centrifugation may increase the amount of bioavailable protein

for odour production. Therefore reducing the centrifuge speed and/or torque can reduce the amount of shear imparted on biosolids, thereby reducing the odour of the dewatered cake (Adams *et al.* 2008). In a full-scale test, a 10% reduction in centrifuge bowl speed on one high-solids centrifuge resulted in 20% reduction of TVOSC emissions from dewatered cake with no observed reduction in cake solids concentration (Adams *et al.* 2008).

In this study we investigated reduction of centrifuge speed as means of odour reduction on a laboratory scale. Three centrifuge speeds were investigated: 3850 rpm (control speed), 3460 rpm (10% reduction in speed, relative to control) and 3080 rpm (20% reduction in speed, relative to control). In order to assess the impact of centrifuge speed on cake odour, the digested sludge used in these trials was left untreated (i.e. no chemical addition except for polymer). In our laboratory trials, a 20% reduction in centrifuge speed (3080 rpm) resulted in an approximate 30% decrease in the maximum TVOSC concentration, relative to the control. However, the solids content of the resulting cake was also significantly reduced from 15% at the control speed of 3850 rpm to 10% at 3080 rpm, which would not be desirable from the point of view of WWTP operations.

Analysis of OVACs in biosolids samples from the odour reduction trials

No significant concentrations of OVACs were observed in any of the cake samples analysed during the monitoring period. In most cases the compounds were either at or below limits of quantification for the method. This is most likely because the monitoring period was not long enough for significant amounts of OVACs to form. Research has shown that indole and skatole begin to form at approximately 40 days, peak at approximately 100 days, and begin to disappear after about 125–135 days of incubation (Novak 2012). The longest monitoring period over the course of the trials was 37 days.

CONCLUSIONS

In this study we identified some of the main odour compounds in biosolids samples obtained from a Western Australian WWTP and investigated chemical addition and reduction of centrifuge speed as potential odour reduction strategies. The main odour compounds identified in fresh biosolids samples from our test site included DMS, DMDS

and DMTS, while biosolids samples that were stored for several months contained indole and skatole.

Aluminium sulphate addition to digested sludge prior to dewatering offered the best odour reduction strategy amongst the options that were investigated, resulting in approximately 40% reduction in the maximum TVOSC concentration, relative to a control sample. From these preliminary trials, it appears that reduction of centrifuge speed would not be a viable odour reduction option for the test WWTP as it resulted in too much reduction in the solids content of the biosolids cake.

In most cases, results obtained from the HS SPME-GC-MS analyses were in general agreement with qualitative observations by a single trained odour assessor. In future studies, it would be beneficial to include dilution olfactometry measurements to obtain a more rigorous assessment of the overall odour generated from biosolids cake and to correlate/compare the results with measurements obtained using HS SPME-GC-MS.

While the investigations conducted in this Phase I study utilised sludge and biosolids samples from just one WWTP, Phase II studies will expand the scope to include biosolids and sludge sourced from different WWTPs. These Phase II studies will provide information on the odorous compounds present in biosolids produced at other WWTPs and whether the trialled odour reduction strategies are applicable to more than one type of wastewater treatment system.

ACKNOWLEDGEMENTS

REFERENCES

Adams, G. A., Witherspoon, J., Erdal, Z., Forbes, B., McEwen, D., Hargreaves, R., Higgins, M. J. & Novak, J. 2008 Identifying and Controlling Odour in the Municipal Wastewater Environment Phase 3: Biosolids Processing Modifications for Cake Odour Reduction. Water Environment Research Foundation Report No 03-CTS-9T, Alexandria, VA, USA.

Chen, Y., Higgins, M., Murthy, S., Maas, N., Covert, K. & Toffey, W. 2006 Production of odorous indole, skatole, *p*-cresol, toluene, styrene and ethylbenzene in biosolids. *Journal of Residuals Science and Technology* **3** (4), 193–202.

Glindemann, D., Murthy, S. N., Higgins, M. J., Chen, Y. C. & Novak, J. T. 2006 Biosolids incubation method for odorous gas measurement from dewatered sludge cakes. *Journal of Residuals Science and Technology* **3** (3), 153–160.

Gruchlik, Y., Heitz, A., Joll, C., Fouché, L., Penney, N. & Charrois, J. 2012 Laboratory scale investigations of potential odour reduction strategies in biosolids. *Water* **39** (7), 58–64.

Haberhauer-Troyer, C., Rosenberg, E. & Grasserbauer, M. 1999 Evaluation of solid phase microextraction for sampling of volatile organic sulphur compounds in air for subsequent gas chromatographic analysis with atomic emission detection. *Journal of Chromatography A* **848**, 305–315.

Higgins, M. J. 2010 Evaluation of Aluminium and Iron Addition During Conditioning and Dewatering for Odour Control. Phase IV. Water Environment Research Foundation Report No 03-CTS-9A, Alexandria, VA, USA.

Higgins, M. J., Chen, Y., Novak, J. T., Glindemann, D., Forbes, R., Erdal, Z., Witherspoon, J., McEwen, D., Murthy, S., Hargreaves, J. R. & 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 (accessed December 2012).

Higgins, M. J., Chen, Y., Yarosz, D. P., Murthy, S. N., Maas, N. A., Glindemann, D. & Novak, J. T. 2006 Cycling of volatile organic sulphur compounds in anaerobically digested biosolids and its implications for odours. *Water Environmental Research* **78**, 243–252.

Kim, H., McConnell, L. L., Ramirez, M., Abu-Orf, M., Choi, H. L. & Peot, C. 2005 Characterization of odours from limed biosolids treated with nitrate and anthraquinone. *Journal of Environmental Science and Health, Part A* **40** (1), 139–149.

Kristiana, I., Heitz, A., Joll, C. & Sathasivan, A. 2010 Analysis of polysulfides in drinking water distribution systems using headspace solid-phase microextraction and gas chromatography-mass spectrometry. *Journal of Chromatography A* **1217**, 5995–6001.

Novak, J. T. 2012 WERF ROSI Project: Regrowth, Odours and Sudden Increase (ROSI): Odours. WERF 2012 Webinar Series, 5 September 2012. www.werf.org/c/WebSeminars/9-5-12-ROSI.aspx (accessed 16 December 2012).

Novak, J. T. & Park, C. 2010 Effect of Aluminium and Iron on Odours, Digestion Efficiency and Dewatering Properties. Phase IV. Water Environment Research Foundation Report No 03-CTS-9b, Alexandria, VA, USA.

Subramanian, R., Novak, J. T., Murthy, S., Glindemann, D. & North, J. 2005 Investigating the role of process conditions in wastewater sludge odour generation. In: *Proceedings of the Water Environment Federation*, WEFTEC 2005, Washington, DC, USA, pp. 6582–6604.

Turkmen, M., Dentel, S. K., Chiu, P. C. & Hepner, S. 2004 Analysis of sulphur and nitrogen odorants using solid phase microextraction and GC-MS. *Water Science and Technology* **50** (4), 115–120.