Odorous volatile organic compound (VOC) emissions from ageing anaerobically stabilised biosolids

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ABSTRACT

Opportunities for the beneficial re-use of biosolids are limited by nuisance odour emissions. Volatile organic compounds (VOCs) from anaerobically stabilised biosolids were measured to identify compounds that could contribute to the overall odour character of nuisance emissions. Flux hood sampling and chemical analysis were used to identify VOCs emitted from biosolids as they were stored in ambient conditions. Compounds emitted varied as the biosolid cakes were stored for a period of 50 days. VOCs detected in the biosolids are likely to occur from catchment sources as well as abiotic and biotic generation in the wastewater processing and the biosolids as they are stored. Odour activity values (OAVs) were used to compare odorants. Trimethylamine was the only VOC detected that exceeded the sulfur compounds in terms of OAVs. Other compounds such as limonene, ethyl methyl benzene and acetic acid were detected at concentrations exceeding their olfactory detection limits, however at lower OAVs than sulfur compounds.

Key words | biosolids, odour, stabilisation, volatile organic compounds (VOCs)

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INTRODUCTION

Potential markets for biosolids re-use are limited by the risk of nuisance emissions impacting local communities (Hayes *et al.* 2014). Volatile sulfur compounds (VSCs) are widely attributed as the dominant odorants emitted from anaerobically stabilised biosolids, due to their offensive characters and low olfactory detection thresholds (Novak *et al.* 2006). Volatile organic compounds (VOCs) may also contribute to the perceivable odour character of emission streams, however these are not routinely measured as they are judged less sensorially important compared to VSCs and therefore have received less attention (Lehtinen & Veijanen 2011). The presence of VOCs in the biosolids may be due to effluent discharges through the catchment (industrial or household chemicals), biotic and abiotic generation during solids processing and/or as they are stored. Terpenes such as limonene or pinene may be emitted from household or industrial cleaners, halogenated compounds such as di or trichloroethylene from industrial processes, and hydrocarbons such as decane or dodecane from petrol residues (Escalas *et al.* 2003). These compounds have been detected in wastewater treatment plants (WWTPs) and may remain undegraded due to their association with the solid organic

fraction, due to their hydrophobicity or low solubility in water. The degradation of organic material biotically or abiotically is another source of odorous VOCs as well as VSCs. Volatile nitrogenous compounds such as ammonia and trimethylamine (TMA) are produced from the degradation of proteins, and have been reported to be emitted from land-applied dewatered sludge (Rosenfeld *et al.* 2001). Volatile aromatic compounds such as toluene, ethyl benzene, skatole, indole and p-cresol have been detected in the headspace of biosolids after a month of storage and were noted to dominate the odour profile (Chen *et al.* 2004). Determining if, and when, odorous VOCs are likely to be emitted from biosolids as they are stored can supplement the existing literature on the temporal emissions of VSCs to ensure compounds likely to contribute to nuisance emissions are identified.

Headspace sampling methods (Glindemann *et al.* 2006) aim to simulate the anaerobic conditions inside heaped biosolids piles. Ageing biosolids using this method has shown VSC emission patterns are based on microbial activity with the re-establishment of the methanogenic community causing a reduction in VSC emissions (Higgins *et al.* 2006).

Recommendations for minimum onsite storage of 6–10 days for anaerobically stabilised biosolids are based on the reductions after peak emissions (Novak *et al.* 2012). Volatile aromatic compounds noted in the aged sludge (Chen *et al.* 2004) were also found using the anaerobic headspace method. Dynamic flux hoods (such as the US EPA hood) can be used to identify potential emissions, as a known flow rate is applied to strip emissions from the surface of the source. Flux hood sampling can be used to measure emissions from porous media (such as biosolids) as they are stored in ambient conditions in storage sheds at WWTPs or when applied to land (Rosenfeld *et al.* 2001). The compounds measured using flux hoods may differ to those identified in headspace studies due to the aerobic conditions on the surface, differing microbial communities, abiotic (oxidative) transformations as well as different rates of volatilisation and diffusion of compounds from the surface of the biosolids. Additionally, the determination of area source emissions using flux hoods (over headspace sampling) allows the assessment of the potential impact of the emissions on the surrounding community using dispersion modelling (Hayes *et al.* 2014).

The objectives for this study are to identify VOCs associated with anaerobically stabilised biosolids, map emissions as the biosolids are stored and to identify sensorially relevant VOCs that have the potential to contribute to nuisance emissions from the biosolids at WWTPs.

MATERIAL AND METHODS

Sampling and analysis

A WWTP (Sydney, Australia) with a 90 day capacity for the storage of dewatered biosolids prior to being outloaded onto trucks for transport for land application was investigated. The average storage time for a full bay is 30 days, with the timing dependent on the scheduling of the contracted transport company.

Two 10 L biosolids samples were collected from a WWTP (catering for 200,000 equivalent population (EP)). The biosolids were collected directly after centrifugation of the anaerobically digested sludge. Both primary sludge and waste activated sludge were anaerobically digested, with a solids retention time (SRT) of about 20 days, in two parallel mesophilic digesters. Cake 1 was sampled after heavy rains, Cake 2 was sampled a week later, both were stored under ambient conditions.

US EPA-designed flux hoods were used to produce emissions from the stored biosolids after purging for 30 mins with a nitrogen flow of 5 L/min. Emissions were collected onto TenaxTA sorbent tubes (Markes International, UK) using a sampling flowrate of 100 mL/min (AirChek2000, SKM Inc.). All samples were collected in quadruplicate with measurements taken every few days for a total of 50 days. Sorbent tubes were also collected in series to ensure breakthrough of compounds did not occur. Cake 1 was sampled on days 1, 3, 6, 8, 10, 13, 15, 17, 20, 22, 24, 28, 30, 33, 40, 46 and 56, whilst Cake 2 was sampled on days 1, 3, 6, 8, 10, 13, 15, 17, 21, 23, 26, 33, 39 and 49 after they were collected from the WWTP. The temperature of the ambient air and the cake surface were recorded for each measurement. VOC analysis of the sorbent tubes was performed by thermal desorption gas chromatography mass spectrometry (TD-GC-MS) according the method described by Wang *et al.* (2012). On day 24 for Cake 1 and day 17 for Cake 2, the surfaces were disturbed and mixed to simulate the disturbance encountered during the out loading of biosolids. Emission measurements were continued after the surfaces were disturbed.

Data analysis

MS peaks were integrated and matched to the NIST11 mass spectral library, with matches greater than at least 70% being reported. The concentration(s) of the identified compounds were semi-quantified using toluene as a reference. Control studies for background VOC levels were measured by sorbent tubes sampling using an empty flux hood to determine the impact of the sampling equipment. VOCs identified frequently in the background sorbent tubes were excluded from the biosolids emission analysis as it could not be concluded they resulted only from the biosolids. Isomers for certain compounds were grouped and the total sum of compounds for each sorbent tube was compiled, including compounds noted in the breakthrough tubes. Compounds detected in more than 66% of replicates were reported.

RESULTS

VOCs and classes

VOCs emitted from the two cakes are shown in Table 1, while the measured concentrations emitted throughout storage for different classes of VOCs are shown in Figure 1.

Commonalities between the cakes were the high concentrations of cyclic alkanes emitted as the cakes were aged, with a slight decline being noted after periods of no

Table 1 │ VOCs emitted and identified throughout the storage experiment

Acid	Acetic acid
	Benzoic acid
Alcohol	Ethanol
Aldehyde	Benzaldehyde
Alkane	Decane
	Dodecane
	Heptane, 2,2,4,6,6-pentamethyl-
	Nonane
	Octane
	Tetradecane
	Total dimethyl heptane
	Total methyl decane
	Total methyl heptane
	Total methyl nonane
	Total methyl octane
	Tridecane
	Undecane
Alkyne	Pentadecyne
Aromatic	Acetaldehyde
	Benzene, 1,2,3-trimethyl-
	Benzene, 1,2,4-trimethyl-
	Benzene, 1,3,5-trimethyl-
	Benzonitrile
	Ethylbenzene
	p-Xylene
	o-Xylene
	m-Xylene
	Toluene
	Total cymene (methyl(methylethyl)-benzene)
	Total ethyl dimethyl benzene
	Total ethyl methyl benzene
Bicyclic alkane	Bicyclo[2.2.2]octane, 2-methyl-
	Bicyclo[3.1.1]heptane, 2,6,6-trimethyl-
	Bicyclo[4.1.0]heptane, methyl pentyl
	Total decalin, methyl
	Naphthalene, decahydro-
	Spiro[4.5]decane
	Total dimethyl decahydro naphthalene
	Total dimethylspiro[4.5]decane
	Total dimethylspiro[5.5]undecane
	Total ethyl decalin
	Total methyl decahydro-naphthalene
Cyclic alkane	1-Methyl-2-methylenecyclohexane
	Cyclohexane, pentyl-
	Cyclohexane, propyl-
	Cyclohexanone, ethenyl (methyl ethyl)
	Ketone, 2,2-dimethylcyclohexyl methyl
	Total cyclohexane, methyl-(methylethylidene)
	Total dimethyl cyclohexene
	Total ethyl methylcyclohexane
	Cyclohexane, ethyl-
	Cyclododecanemethanol
Cyclic alkene	Cyclopentene, octyl
	Methyl cyclodecene
Halogenated	Tetrachloroethylene
	Total dichloro benzene
	Trichloroethylene
Ketone	2-Butanone
	Acetone
	Methyl isobutyl ketone
	p-Benzoquinone
	Total pentanone
Nitrogenous	TMA
Sulfur compound	Carbon disulfide
	Dimethyl sulfide
	DMTS
	Disulfide, dimethyl
	Ethane, (methylthio)-
	Propane, 1-(methylthio)-
Terpene	Caryophyllene
	Total α-pinene
	Total β-citronellene
	Total β-pinene
	Total carene
	Total γ-terpinene
	Total limonene
	Total menthene

disturbance. Compounds such as bicyclic undecane, decahydro-naphthalene, methyl decahydro-naphthalene, dimethyl decahydro-naphthalene, methyl decalin and ethyl decalin were consistently emitted in all samples. Such bicyclic compounds have similar structures, and were seen at similar retention times in a densely matched region of the chromatogram.

High concentrations of aromatic compounds (1,3,5-trimethyl benzene, *p*-xylene, toluene, ethyl methyl benzene, cymene and ethylbenzene) were initially emitted and decreased as the biosolids were stored. Terpene emissions also showed this pattern, with the limonene, γ-terpinene, α-pinene, carene and caryophyllene concentrations decreasing over time. Menthene was consistently detected throughout storage. Cake 1 generally produced a higher concentration of terpene emissions than Cake 2, however mixing of Cake 2 increased the concentrations emitted for a few days.

Aliphatic groups, in particular alkanes, were detected in the initial days of storage, as well as after mixing for Cake 2. Emissions of straight chain and methylated alkanes from Cake 1 were four to seven times greater and lasted for longer compared to Cake 2. TMA was only detected in the first few days of storage for Cake 1. Other species were seen to be emitted after the alkane emissions reduced, namely acetic and benzoic acids, ethanol and butanone.
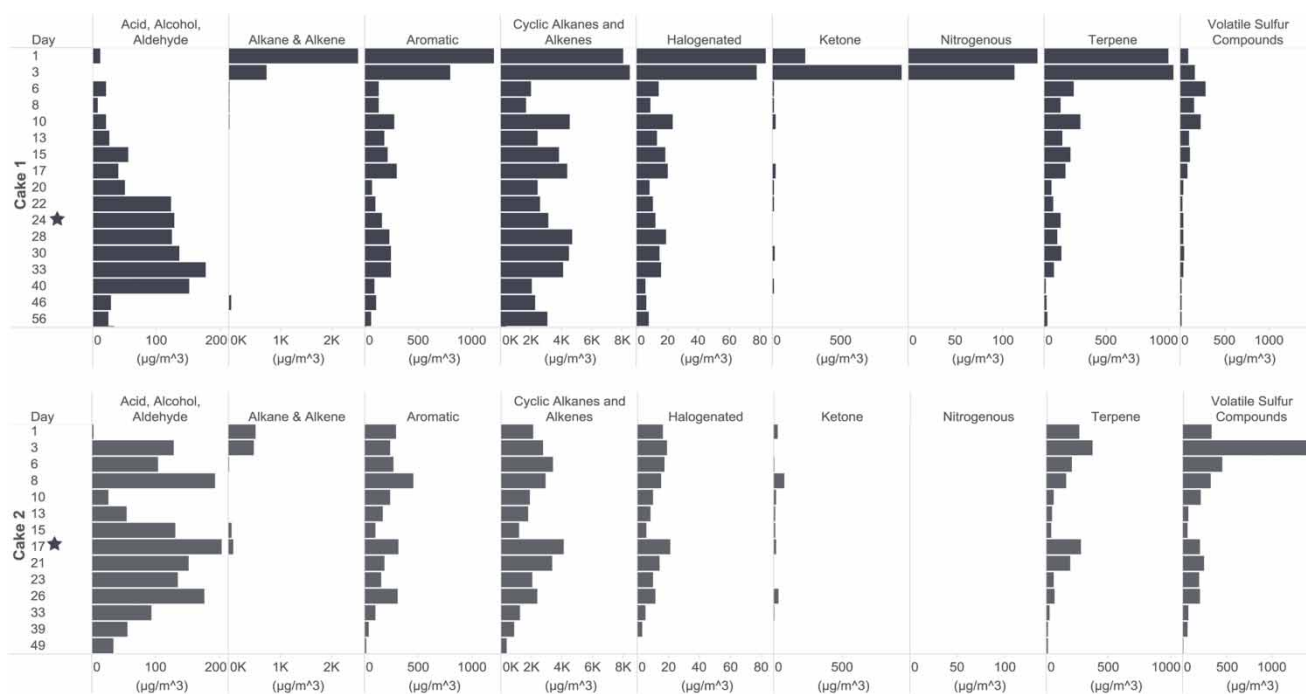
Figure 1 │ Emission patterns of VOC classes from the cakes as they were stored. The sum of individual VOC average concentrations are shown for the different classes emitted as the biosolids were stored. *Note:* different concentration ranges are used for each class. Stars indicate which day the cakes were mixed.

The range of sulfur compounds detected was dominated by dimethyl disulfide, with emissions peaking on days 3–6 and decreasing as the biosolids aged. Significantly higher levels of sulfur emissions were noted for Cake 2 compared to Cake 1. It should be noted that the full range of sulfur compounds cannot be accurately quantified using sorbent tubes (Murphy *et al.* 2012) as adsorption and desorption actions has been shown to convert mercaptans to sulfides (Sivret *et al.* 2010). No mercaptans were identified, however three sulfide species were identified, dimethyl sulfide, dimethyl disulfide and dimethyl trisulfide (DMTS).

Odour activity values

Odour activity values (OAVs) are a way of comparing odorants in terms of their contribution to nuisance emissions (Laor *et al.* 2014).

$$OAV = \frac{Concentration\ [\mu g/m^3]}{Odour\ Detection\ Threshold\ [\mu g/m^3]}$$

Compounds for which odour detection threshold data were available are shown in Table 2. Olfactory detection thresholds from Nagata (2003) were used as they were measured using the same methodology and panels.

However, DMTS was added as it is known to be sensorially important and was not reported by Nagata (2003). The OAVs for the emissions reported in Table 2 were calculated using the average concentration for each day of storage. Compounds for which the concentration exceeded the olfactory detection thresholds (OAV > 1) are shown in Figure 2.

DISCUSSION

Biosolids emission characteristics

General trends were detected in emission behaviour of the cakes as they are stored. Similarities in emission trends appeared to be dependent on whether the compounds are present initially, microbially generated or degraded, or a combination of both.

Commonalities between the two cakes were seen in the gradual reduction in emission rates of cyclic alkanes, aromatics, halogenated compounds and terpenes. These are typically larger sized compounds, and due to their hydrophobicity are likely to be associated with the organic fraction of the biosolids. Concentrations in the top layer decrease as the compounds are stripped with ageing, thereby

Table 2 | Select odorant detection thresholds used in OAV analysis (Nagata 2003)

		Detection threshold (µg/m³)
Nitrogenous	TMA	0.08
Acid	Acetic acid	15
Sulfur	Disulfide, dimethyl	8.5
	DMTS[a]	0.01
	Carbon disulfide	650
	Dimethyl sulfide	7.6
Ketone	Acetone	100,000
	2-Butanone	1,300
Cyclic alkane	Total methyl cyclohexane	600
Aromatic	*m*-Xylene	180
	p-Xylene	250
	o-Xylene	1.6
	Ethylbenzene	0.74
	Total ethyl methyl benzene	0.09
	Benzene, 1,2,4-trimethyl-	0.59
	Benzene, 1,3,5-trimethyl-	0.84
	Benzene, 1,2,3-trimethyl-	0.84
	Toluene	1,200
Alkane	Total methyl heptane	0.51
	Octane	7.9
	Pentane	4,100
	Decane	3.6
	Undecane	5.6
	Nonane	12
	Dodecane	0.77
Alkene	1,3-Butadiene, 2-methyl-	640
Terpene	Total alpha-pinene	0.1
	Total beta-pinene	0.18
	Total limonene	0.21
Halogenated	Trichloroethylene	21
	Tetrachloroethylene	5.2

[a]Ruth (1986).

limiting emission of compounds from the surface. Simulated mixing of the biosolid exposes the compounds in the underlying biosolids, increasing the emission of compounds from the biosolid.

Compared with aromatic, cyclic and halogenated compounds, a rapid reduction in the emissions of alkanes, alkenes, ketones and nitrogenous compounds was observed in the initial days of storage. These compounds have a lower affinity to organic matter, greater volatility and a greater tendency for degradation, which potentially accounts for the short emission period.

The emission of the acid, alcohol and aldehyde groups was quite significant until day 40 for Cake 1 and day 33 for Cake 2. This dominance suggests the availability of substrates in the biosolids and on-going microbial activity,

which results in the formation of these chemicals under anaerobic conditions.

VOC origins

The emission patterns of different groups of compounds as the biosolids are stored may suggest their origin. The degradation of readily biodegradable matter yields products such as ketones, acids, alcohols and aromatics, which may be used in further degradation pathways. Toluene is known to be produced in anaerobic digestion and has been detected previously in biosolids (Dincer & Muezzinoglu 2008). The reduction in toluene concentrations emitted from the biosolids in the first week may be due to microbial activity or dissipation. TMA and ethyl benzene, which can also be used in microbial pathways, showed the same rapid reduction. TMA emissions have previously been linked to the increase in pH due to liming, however its presence in these anaerobically stabilised biosolids may be due to the degradation of polymers used in dewatering (Kim *et al.* 2003), or choline and betaine during anaerobic digestion (Wang *et al.* 2010). TMA can be degraded by methanogens as a source of energy, or oxidised aerobically by a variety of methylotrophic bacteria (Wang *et al.* 2010).

Other compounds that are likely to be degradation products are acetic acid, benzoic acid and ethanol, which appear after day 3, suggesting they are formed in the biosolids. As the biosolids age, more recalcitrant compounds associated with microbial activity, such as certain terpenes used in signalling, may be produced (Kotowska *et al.* 2012). Therefore, the ongoing emissions of carene, menthene and limonene may be due to microbial activity (Korpi *et al.* 2009).

Some compounds noted in the emissions have also been detected in other biosolids studies, where they are attributed to anthropogenic sources. Alkanes such as decane and nonane from road runoff of fuels (Kotowska *et al.* 2012), terpenes such as limonene and pinene from household cleaners, and chlorinated compounds and certain aromatics (xylene and naphthalene) from industrial emissions (Wilson *et al.* 1994; Dincer & Muezzinoglu 2008). The large amount of bicyclic compounds may be due to the presence of other industrial discharges in the catchment, however their presence has not been reported in other studies.

Temporal emissions

The different VOC emission profiles of the cakes is likely due to upstream instabilities affecting the produced
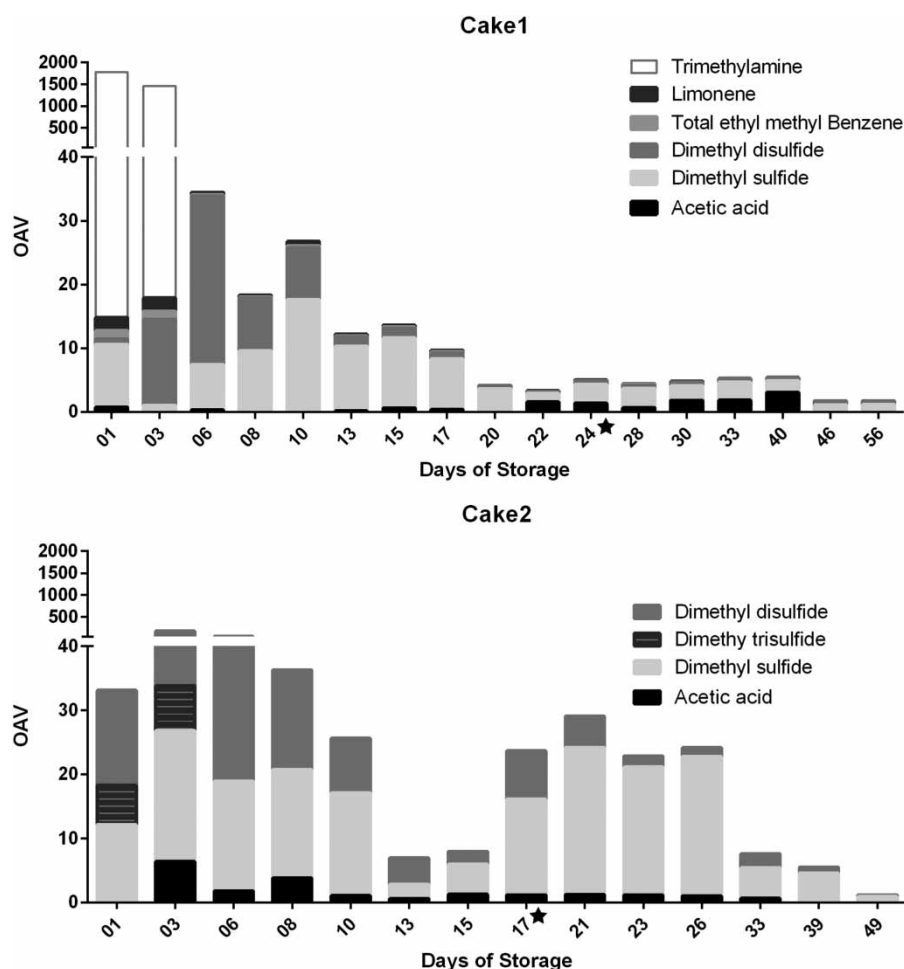
Figure 2 | OAVs calculated using selected odorants over biosolids ageing. Stars indicate the day the cakes were mixed.

biosolids, despite the buffering effect of the 20 day SRT of the anaerobic digesters. The greater concentrations of alkanes, terpenes and halogenated compounds detected in the initial days of storage for Cake 1 may be associated with heavy rains carrying compounds from roads and urban surfaces into the sewer catchment.

The mixing of each cake also had varied effects. The mixing of Cake 1 produced minimal changes in emissions. However, mixing of Cake 2 increased the emissions of terpenes, alkanes, aromatics and sulfur compounds. The mixing action exposes anaerobic regions, releasing sulfur compounds generated by microbes in the biosolids (Higgins et al. 2006), exposing VOCs like alkanes not previously stripped from the biosolids. Therefore, differences between the cakes' responses to mixing may be due to different microbial activity and/or composition. These differences may be attributable to the difference in storage age, variability in cake composition due to upstream instabilities, or

normal variability in biosolids quality. This apparent temporal variability in the odour quality of the stored biosolid may affect the management approaches, and is an area for further investigation.

Sensorial consequences

OAV analysis is used to provide an understanding of the potential odour levels emitted from the aging biosolids and highlight potentially significant odour-causing VOCs for odour mitigation/control purposes. The determination of OAVs showed that when compared to the VOCs (using detection thresholds reported by Nagata (2003)), the VSCs are the dominant class contributing to odour emissions as the biosolids are stored, particularly at longer storage times. This supports the focus of previous research on VSCs. However, TMA was detected in the initial days of the storage in Cake 1 at a concentration four orders of

magnitude greater than its odour detection threshold. The individual OAV of TMA exceeded the total OAVs of all other compounds for the days it was detected. Due to TMA's extremely low detection threshold and offensive character of rotten fish, its presence should be monitored. As TMA was only detected in Cake 1, it may be associated with plant upsets and the identification and control of process conditions that result in its formation should be further investigated.

Other VOCs were also detected at levels above their odour detection thresholds, however their OAVs were less than the VSCs and TMA. Acetic acid, which has a vinegar aroma, was found at intermediate sludge ages. Pinene, terpinene and menthene, as well as the aromatic trimethyl benzene, were noted in the initial days. These compounds have earthy, floral type aromas and may not contribute negatively to the overall odour character. Indole, skatole or p-cresol, which have been detected from biosolids in other studies (Kacker *et al.* 2011), were not detected suggesting catchment or operational conditions affect the types of compounds emitted. The reduction in OAV as the biosolids are stored suggests a reduction in likely odour load over time, however this is most likely due to reductions in the concentration of sulfur compounds.

CONCLUSION

The range of VOCs identified as the anaerobically stabilised cakes are aged suggests they are present due to a range of factors, such as biotic and/or abiotic production as well as household or industrial inputs to the sewer catchment. Variations between the types and concentration of compounds emitted from different biosolids emphasises the impact of upstream processes on biosolids emissions. The different response to mixing between the cakes suggests temporal variation in biosolids properties is an area of further research. Sensorially important VOCs such as TMA, acetic acid, limonene and ethyl methyl benzene were identified at levels above their detection thresholds. However, TMA was the only compound to exceed the OAVs of the sulfur compounds being detected, with a concentration four orders of magnitude greater than its odour detection threshold.

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