Biosolids Incubation Method for Odorous Gas Measurement from Dewatered Sludge Cakes

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ABSTRACT: In order to compare the odor potential of wastewater biosolids from individual dewatering technologies, a laboratory batch-test was developed that simulates the anaerobic incubation conditions in a sludge tank or cake pile, scaled down to a bottle sized test vessel. The method controls the quantity, incubation time and temperature of biosolids in the vessels, and analyzes the static headspace gases for the odor causing gas concentration. The odor concentration of an incubated specific biosolids sample is reproducible and robust to changes in vessel size and biosolids quantity, as long as the biosolids volume occupies 20% or more of the bottle volume. Incubation of digested cakes at 22°C in closed bottles to simulate non-aerated piles showed that cakes first produced volatile organic sulfide (VOS) odorants to a peak odor level that is highly specific for each dewatering technology. After peaking, VOC gases were consumed, usually within three additional weeks of incubation.

INTRODUCTION

A key aspect to successful biosolids land application programs is the reduction or elimination of nuisance odors. Odor complaints are often the biggest concern during biosolids management, however, understanding and quantifying odor production by biosolids is a difficult task. To understand the production of odors from biosolids, especially during storage, a rational, reproducible, and efficient method is needed to measure the odor production potential of the biosolids. In particular, a bench-scale method to mimic biosolids storage would be helpful in determining the mechanisms of odorant production as well as the factors which affect their production. In addition, this method would be useful for studying potential odor remediation methods at the lab scale prior to pilot or full-scale testing, as well as for determining the odor production potential of a particular biosolids.

Static headspace (Kolb and Ettre, 1997, Seto, 1994), flux chamber (Kim et al., 2002, Rosenfeld, 2001) and purge and trap methods have previously been used for bench-scale and full scale testing of biosolids odors. Fluxing as well as purging is a slow process that requires hours per sample and results in dilution of the odorous compounds. It also removes the gases from contact with the biosolids, thereby reducing the opportunity to investigate how the microbes in biosolids transform the odorous compounds over time. The use of a closed, static headspace system is a potential method to provide reliable and accurate bench-scale sludge storage simulation. This method eliminates the problems associated with flux chambers. In Figure 1, the closed bottle headspace method is compared to the flux chamber method and to the expected odor generation from a dewatered sludge cake storage pile. Although both methods work, the headspace method is thought to be representative of the storage pile interior, easier to perform, and highly reproducible. The static headspace method has the advantage of continuous contact between headspace and biosolids, ease of sampling, and less equipment needs. In this paper, the headspace method is explained and evaluated as a potential method to study odorant emissions from biosolids.

The goal of this study was to develop a reproducible bench-scale method that simulates full-scale storage piles. The methods utilize gas tight bottles for the com-

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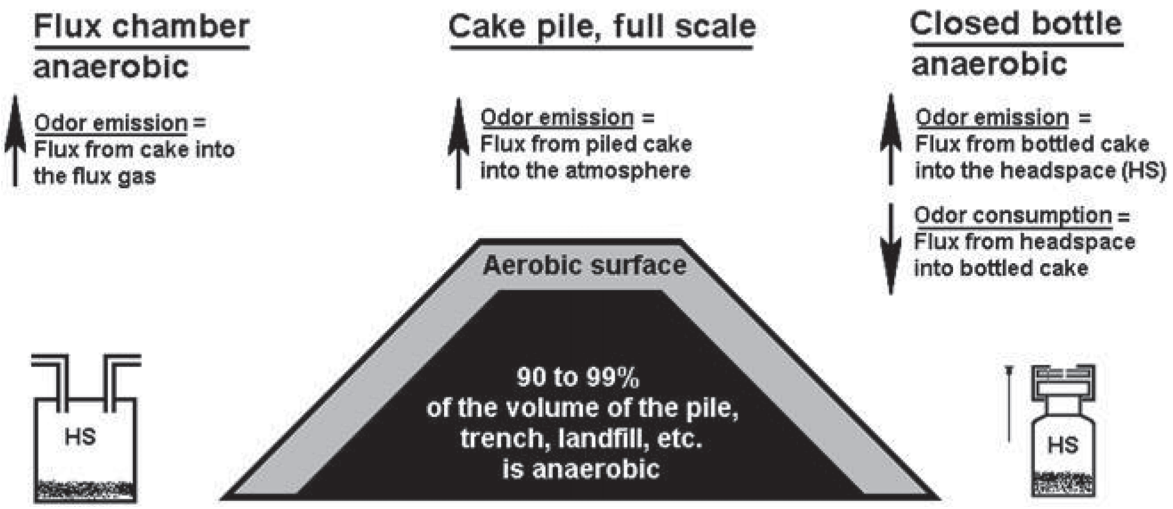

Figure 1. *Headspace (HS) bottle gas analysis versus a flux chamber analysis for odor evaluation from a sludge cake pile.*

bined purposes of sampling of biosolids at the wastewater plant, transport to the laboratory, and static headspace odor analysis during storage. This method allows comparison of the odor of samples from different wastewater plants and process locations. This method also mimics the aging of large biosolids storage piles, both the odor generation and consumption cycles. The method proved to be useful as a tool for several biosolids odor projects (Murthy et al., 2002; Higgins et al., 2002 and 2004; Novak et al., 2002; Adams et al., 2004; Abu-Orf et al., 2004) and is presented in detail.

MATERIALS AND METHODS

Some specific methodical details are described in the result section.

Sample Container Materials and Size

The types of headspace containers that were investigated were plastic polyethylene terephthalate (PET) beverage bottles with plastic screw caps, glass bottles with gas plastic screw caps, and glass laboratory serum bottles/vials (Fisher Scientific, Pittsburg, PA) with Teflon-lined butyl-rubber septum. In addition, one liter Tedlar bags (SKC, Inc., Eighty Four, PA) with polypropylene valve and septum tested. The size of the incubation vessels (vials, bottles and Tedlar bags) ranged from 20 to 1000 mL.

Cake Sample Collection and Incubation

Fresh dewatered sludge cake from mesophilic, anaerobic digestion and centrifuge dewatering was placed in headspace sample vessels, and shipped overnight in an insulated container to the respective laboratory. Alternatively, cake was shipped in plastic bags and distributed into appropriate headspace vessels. The quantity of cake per bottle was set to a predetermined weight (density ~ 1.1 g/mL), because mass can be more easily controlled than volume. Samples were preserved between 0°C (ice water) and 4°C (refrigerator), usually for one day or less, before the incubation was started. Once the headspace sample was placed in the container, the headspace vessel was incubated at constant temperature in the dark.

Headspace Sample Collection and Analysis by GC/MS

Headspace gas samples were collected with a gastight 100 µL syringe by puncturing the rubber septum or the polymer cap of the bottles and were immediately analyzed for odorants by GC. The static headspace gas samples were analyzed for odorous gases by cryo-trapping, gas-chromatography and mass spectrometry (GC/MS, GC 5890, MSD 5971, AGILENT). A 100 L gas sample was injected with a gastight syringe into a column inlet connected to a 30 m 0.32 mm ID 1 m column with 95% silicon 5% phenyl (Agilent) at a carrier gas flow of 2 mL/min helium. A linear oven temperature program of 20 K/min from 40°C up to 260°C was used. Cryo-trap gas sample injection: An unconventional method was used to inject the gas samples. The first meter of the column was unwound and partially immersed in liquid nitrogen prior

to gas injection in a Dewar jar containing liquid nitrogen. After the gas sample was injected into a normal injector (split/splitless or on-column), it was allowed to accumulate for three minutes in the cooled section of the GC column to trap and focus the analytical compounds and than the analysis was started. This method is able to cryo-trap odorous compounds with boiling temperatures as low as −88°C (H_2S) and to generate narrow chromatographic peaks (Figure 2).

Variability in the Method (Incubation and GC/MS)

The reproducibility of the complete procedure (cake incubation and GC/MS analysis of headspace gas within a concentration range from 0.5 to 1000 ppm (v/v) volatile sulfur) was evaluated by comparing duplicate incubation bottles is shown in Figure 3 and in Figures 7, 10, 11. The good precision of the method (repeated analysis of the same bottle) can also be seen in Figures 7, 10, 11. Precise headspace results can be achieved by controlling the shipping temperature, incubation temperature, incubation time, and ratio of biosolids to the total bottle volume. The accuracy (recovery of odorous gas standards of H_2S, methanethiol, dimethylsulfide and trimethylamine in PET bottles without biosolids) was found to be in the order of 70 to 90% after ten days of storage. Similar recovery can be obtained for biosolids in glass bottles. The detection limit of our GC/MS method is about 0.1 ppm(v/v), and the quantification limit is about 0.5 ppm(v/v) in the scan-mode and about 10 ppb (v/v), respectively 50 ppb (v/v) in the SIM mode. It was found that aliquots of cake samples that are incubated under similar conditions in two or more laboratories would yield sufficiently reproducible, precise and accurate headspace sulfide odor results, to track the observed large concentration differences between samples and incubation periods.

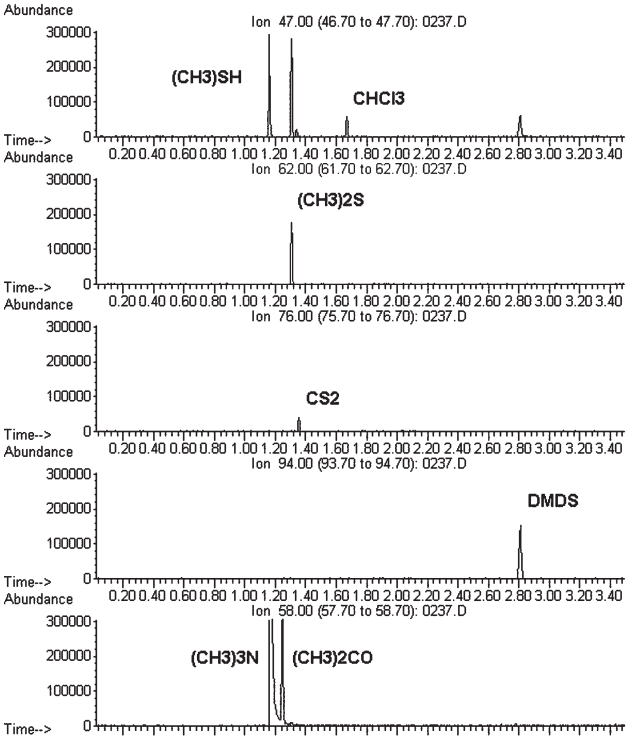

Figure 2. *Stacked GC/MS ion traces of odorous sulfides and trimethylamine (TMA) in headspace gas of digested, chlorinated and limed sludge cake. Abundance of 300000 translates into about 50 ppmv per compound in headspace gas. CH_3-SH methanethiol, $(CH_3)_2$-S dimethylsulfide, DMDS dimethyl disulfide, CS_2 carbon disulfide, $(CH_3)_3N$ trimethylamine TMA, $(CH_3)_2CO$ acetone, $CHCl_3$ chloroform. Cake from a municipal WWTP with 22% TS, 140 g, in a 710 mL PET-bottle. Analysis of 0.1 mL headspace gas by GC-MS after cryo-trapping on the un-winded inlet loop section of the GC column immersed in liquid nitrogen for 3 minutes.*

RESULTS AND DISCUSSION

The results from the different tests to develop an efficient, robust headspace procedure are discussed in the following sections.

Effect of Sample/Bottle Volume Ratio on the Headspace Odorant Concentration

A series of experiments were conducted in which the

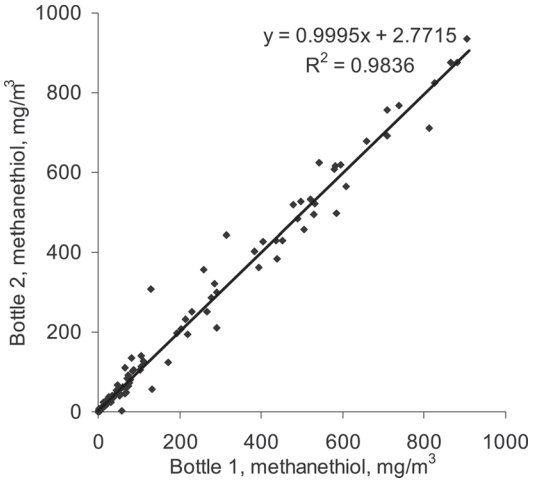

Figure 3. *Reproducibility of the headspace incubation procedure (cake incubation and headspace gas GC/MS analysis, N=140) by comparing aliquots of cake in duplicate incubation bottles. 710 mL PET bottles, 140 g digested cake, anaerobic pressurized incubation at 22°C, measurements on days 1, 3, 5, 7, 14 of incubation. The figure is a compilation of earlier published tabular data that represent cakes from 11 WWTP (Adams et al., 2003).*

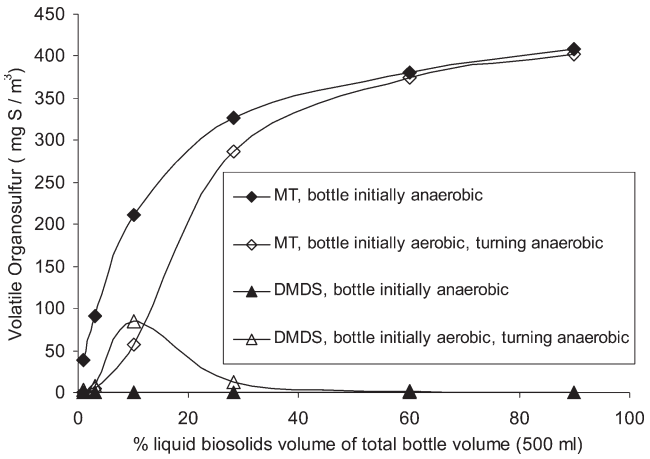

Figure 4. *Headspace concentration of Methanethiol (MT) and Dimethyldisulfide (DMDS) versus percent of bottle filled with sludge. Anaerobic centrifuge cake with 22% TS, 2 days of incubation at 22°C in 500 mL PET bottles.*

volume of sludge placed in the bottles was varied and the gas content analyzed after two days of incubation. One set of bottles started with the entrapped air, another set of bottles started after deaeration with nitrogen. The data in Figure 4 show that at higher than 20% sample/bottle volume ratio, the headspace gas content becomes more insensitive to the sample/bottle volume ratio, and less DMDS is formed (an oxidation product of MT with air oxygen trapped during cake sampling in the bottle). It was also found that all oxygen inside the oxic bottles that were 20% and more filled was consumed within one day (data not shown).

Thermodynamic Headspace Model

The relative constancy of VOS concentrations once more than 20% of the bottle is filled (data of anaerobic experiment in Figure 4) can be described by a simple model:

The molar balance of odorant in the initial sample with M_o = initial mole of odorant in sample before partitioning; M_s = mole in biosolids after partitioning; M_g = mole in headspace after partitioning, is:

$$M_o = M_s + M_g \tag{1}$$

Henry's Law: The concentration C_s of odorant in biosolids sample (mole/L); and the concentration C_g of odorant in gas phase or headspace (mole/L) are linked by K_H (akin to a unitless Henry's constant for a pure solvent, inverse of K_{sg} = cake/headspace partition coefficient):

$$K_H = 1/K_{sg} = C_g/C_S \tag{2}$$

The molar balance, Equation (1), of odorant in each phase can be written as the product of concentration and volume as follows: [V_s = sample volume (L); V_g = gas volume (L); C_o = initial concentration of odorant in sample before partitioning (mole/L)]:

$$V_s C_o = V_s C_s + V_g C_g \tag{3}$$

Finally, from Equations (2) and (3) and considering the total volume of the bottle $V_T = V_s + V_g$ as the sum of the sample and gas or headspace volume, we can derive the following equation for C_g, max = maximum odorant gas concentration in headspace of a near full bottle (mole/L):

$$\frac{C_g}{C_{g,max}} = \frac{K_H}{(V_T / V_s - 1) + K_H} \tag{4}$$

In Figure 5 the headspace gas concentration as a percentage of the maximum gas concentration is calculated from Equation (4) and plotted for three different partition coefficients. Based on the data in Figures 4 and 5, it was concluded that this incubation method is robust because liquid sludge and sludge cake can be sampled in the field and shipped for odor analysis without purging the oxygen as long as the sample size was in the range of 20% or higher of the container volume. The errors that arise from sludge-weighing error and different initial oxygen in the bottles are unimportant compared to the large concentration shifts of odorants in the

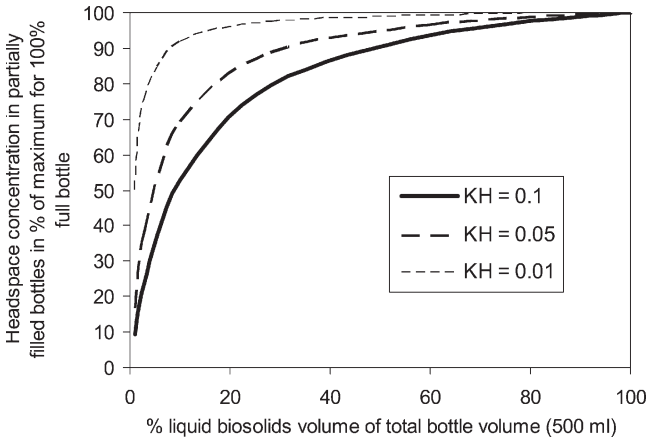

Figure 5. *Relative headspace concentration (concentration of gas in the headspace compared to the maximum concentration that occurs when the bottle is almost completely filled with sludge) versus percent of bottle volume occupied by sludge, for three different Henry coefficients* K_H *(model calculation, see Equation (4), results expressed as %). Range of Henry coefficients is representative for* H_2S *and typical VOS.*

course of incubation and to the different odor of different dewatering technologies. Sample/bottle volume ratios under 20% can be used with nitrogen purged samples and when they are used consistently for the comparison of different samples.

A link between headspace odorant concentration in a bottle and odorant emission of a static pile at the same temperature can theoretically be approximated by Equation 5, where E = Emission of odorant (mg/day), F = Flow of biogas (m^3/day) from the biosolids, $C_{g,max}$ = concentration of odorant in the biogas [as in Equation (4)] (mg/m^3).

$$E \sim C_{g,max} \, \mathrm{F} \qquad (5)$$

That assumes an ongoing flow of biogas by "solid state digestion" is the main driver of odorant transport from the pile core to its surface and of emission into the atmosphere.

The methanethiol (MT) that passes from the core of a pile through the gray aerobic surface layer (Figure 1) would momentarily be oxidized to dimethyldisulfide (DMDS). DMS i.e. methylated MT, was found more oxidation stable. Therefore, DMDS (and not MT) and DMS are major odorants emitted from static biosolids piles with oxic surface layers. More MT is emitted when a black portion of the cake with MT is exposed to the atmosphere once piles are turned inside out or moved into open trucks.

Comparison between Incubation in Pressure Tight (Closed) and Depressurized Bottles

Pressurized closed PET beverage bottles were used by the WERF 2 project (Adams et al., 2003; Novak et al., 2004) for cake incubation. On the day of measurement, the bottles were de-pressurized by venting the pressure through a needle in the cap. The headspace gases were then allowed to equilibrate for 30 minutes and were then used to fill a syringe for GC analysis. For each subsequent day, a new pressurized bottle was sampled and than sacrificed in that way, resulting in data with VOS odorants peaking and then disappearing within 14 days of incubation (Figure 6, pressure tight bottles).

The reason to keep the bottles closed was to show that digested cake can not only produce, but can also consume the VOS odorants over time. It was also chosen to sacrifice bottles after the first use because of the potential for changes resulting from decompression of the bottle and possible leakage. It was also important for accuracy to allow the depressurized bottle to equilibrate before filling the syringe because a biogas pressure of up to about 3 bar builds up upon incubation and decompression can change the headspace concentration by a factor of 1/3. Equilibration for at least 30 minutes (an empirically found value) regenerates the original equilibrium headspace VOS concentration.

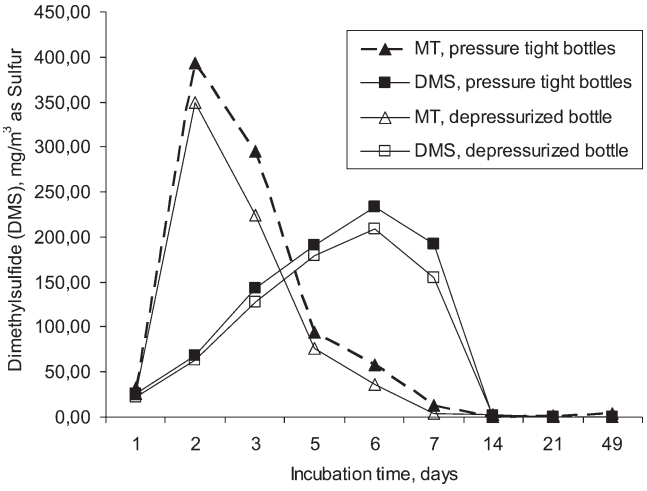

Figure 6. *Comparison of single sampling of multiple pressure tight bottles (a new closed bottle for each measurement day after depressurization) with multiple sampling of a single depressurized bottle on subsequent days. Anaerobic mesophilic digested centrifuge cake, 22% TS, 140 gram in a PET bottle of 710 mL, Incubation at 22°C.*

As a modification of the method, the gas sampling hole on the plastic cap of the PET bottle was sealed with clear-tape, between sampling days, to allow a controlled leak that is still oxygen tight, but allows pressurized biogas to escape and depressurize the bottle. The gas from the same bottle was repeatedly analyzed on subsequent days. As can be seen in Figure 6, the data for repeated sampling of a single depressurized bottle compared to single sampling of multiple pressurized bottles are very similar. Since the headspace gas content is used as an index of odor potential, it seemed unnecessary to continue to conduct incubation in multiple pressurized (closed) bottles that consume much labor for their set-up.

Comparison of Sampling Pressurized Glass Bottles and Depressurized Tedlar Gas Sampling Bags

Borosilicate glass serum bottles with Teflon lined septa and crimp caps were pressurized tested and compared with depressurized Tedlar bags with integrated valve and septa. The resulting pattern and extent of the

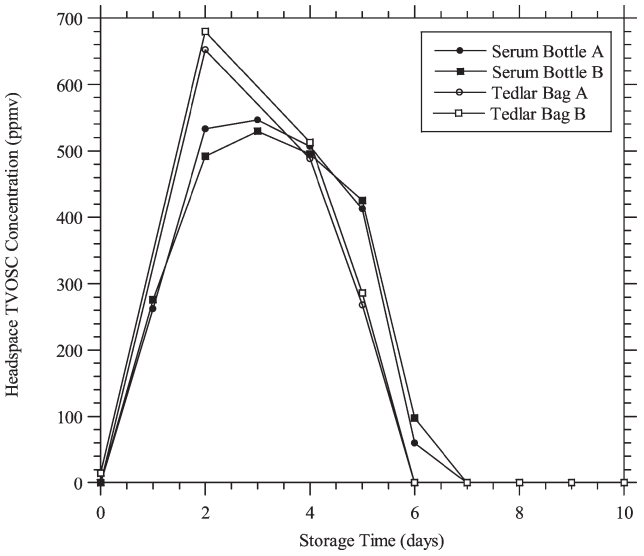

Figure 7. *Comparison of total volatile organic sulphur (TVOS) odorant concentration in headspace of glass serum bottles (pressurized) and Tedlar gas sampling bags (atmospheric pressure). Duplicate sample vessels and repeated analysis on subsequent days. Anaerobic mesophilic digested dewatered cake, 28% TS, incubation at 22°C. "Wheaton" serum bottles, glass, 125 mL, with rubber septum seal, pressurized, plus 25 g cake. Tedlar bags, 1 L, plus 200 g cake.*

total VOS production was very similar for the glass serum bottles and the Tedlar bags (Figure 7).

The question of why the differences between pressurized and depressurized incubation vessels within Figure 6 and within Figure 7 headspace data are so remarkably low can be answered by the thermodynamic model. Assuming a "pseudo" partition coefficient between cake and gas K_{sg} of 20, then there is a 20 times much larger pool of VOS odorants in cake compared to an equal volume of headspace. It is obvious that the flow of biogas that drives the odorants out of the cake is not large enough to deplete the cake significantly (more than 20%) from VOS, thus the headspace concentration of incubation vessels will not be significantly different. The use of Tedlar bags was uneconomical and was therefore discontinued after this experiment has shown the equivalence of this depressurized and closed system.

It can be concluded from these data, that full scale piles that are always depressurized, are well represented by depressurized sampling vessels (depressurized bottles, Figure 6, or Tedlar bags, Figure 7), and do emit only a small fraction of VOS odorants, while the major VOS fraction is consumed within the pile within 14 days. However, to prove this conclusion (VOS production and consumption by cake), it was necessary to perform experiments with closed (pressurized) bottles.

Inertness of PET Beverage Bottles for Odorant Stability

PET bottles were initially used in this work because they are economical, shockproof, and were reported to be inert for beverage flavor chemicals (Feron et al., 1994; Palzer and Franz, 1998). A PET inertness study with a headspace/water system shows (Figure 8) that PET bottles with anaerobic conditions and water saturated with the odorants provide a reasonable recovery (70 to 90%). It is obvious that water provides a large pool of gaseous odorants that replaces losses by oxidation or by PET wall effects. The replacement of biosolids by water for these tests was done to make sure that no biological processes interfered with the influence of the PET material.

A second PET inertness study with a headspace/biosolids system showed that the PET bottles were inert when biosolids were limed stored for 6 weeks in PET bottles as shown in Figure 9. This "analytical liming" made sure that biological activity was eliminated and no biological odorant production and consumption factors interfered with the investigated stability factor of the PET material.

It was found that PET bottles do not significantly affect the stability of odorants of limed and unlimed samples. Thus, the observed large concentration changes of unlimed samples (see Figures 6, 7, 10, 11) can be attributed to microbial production and consumption of gases. Any vessel (glass bottles, Tedlar bags, "silcosteel" inactivated metal etc.) that is reasonably odorant-tight, oxygen-tight and inert, will keep losses of the odorant headspace concentration low, in particular in the presence of a biosolids cake phase that provides a large pool

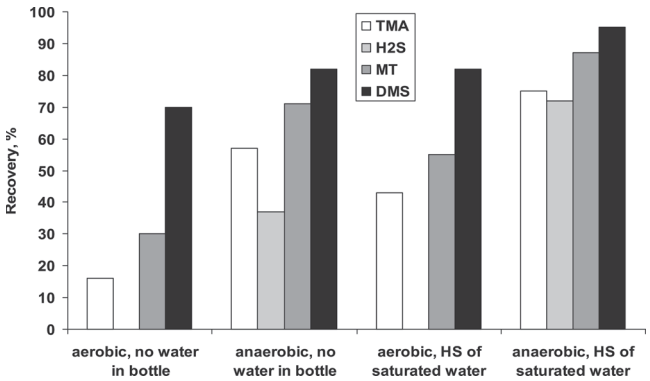

Figure 8. *Recovery of odorous compounds in 710 mL PET beverage bottles, no biosolids in the bottles. The odorants were initially added as 10 ppmv gas mixture in nitrogen or in air. Water (140 mL) was saturated by bubbling 20 L of the gas mixture. Bottles were analyzed by GC/MS after 10 days of storage at 20°C.*

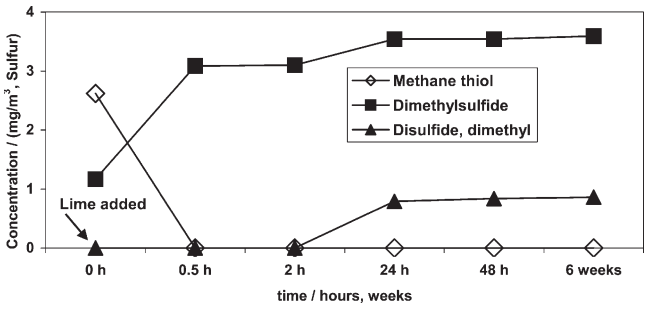

Figure 9. *Stability of the odorous compounds in PET bottles containing biosolids (dewatered WAS, 19% TS), before and after ôkillingö by liming. Incubation at 22°C. Repeated analysis of the same bottle on subsequent hours and weeks. Data for time 0 h represent state immediately before liming. The reason why lime addition caused an initial ca. 2 hour period of concentration change is sorption of the acid MT onto the lime, and subsequent oxidation of MT to form DMDS. DMDS and DMS are pH-neutral compounds that remained constant in headspace gas over 6 weeks of incubation.*

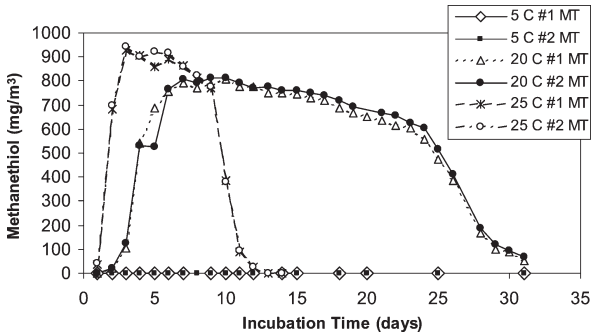

Figure 10. *Temperature dependence of methanethiol (MT) production and consumption (curing) during cake incubation. "Wheaton" serum bottles, glass, 125 mL, plus 25 g cake. Gas samples taken at the incubation temperature. Repeated analysis of the same bottles on subsequent days. Anaerobic mesophilic digested dewatered cake, 28% TS. Results mimic static curing of odorous field scale pile under different seasonal field conditions, where the necessary curing time is declining on rising pile temperature.*

of dissolved odorants to replace these losses. In addition, in real samples, biosolids will effectively and quickly eliminate small amounts of oxygen leakage.

Incubation Temperature, VOS Production and Consumption, and Curing of Sulfide (VOS) Odor

Control of the incubation temperature is not only of critical importance because it ensures comparability of odor results from cake samples, but it also affects spatial and seasonal variations in field sludge piles that can be mimicked in the test vessels to predict the necessary storage time to cure VOS odor by storage. "Curing" is defined as anaerobic (non-aerated) storage of cake over time to the point were the concentration of VOS odorants is peaks and then declines to very low levels due to anaerobic microbial production and consumption processes of VOS. The curing of the headspace concentration is equivalent to the curing of the cake, because the headspace concentration of MT is proportional to its concentration in the biosolids.

The curing of methanethiol (MT) by incubation in closed bottles shown in Figure 10 is typical of odorous digested cake. A decrease in the incubation temperature from 25 to 20°C causes the necessary odor curing time" to rise from 12 to 31 days. These data show also the importance of temperature control (suggested error range 0.5°C) for comparable incubation results at the same nominal temperature.

Low temperature incubation at 5°C inhibis the biochemical formation of MT in the cake. Shifting the temperature of the 5°C sample after 32 days to 20°C and subsequent incubation would result in a pattern similar to the 20°C experiment in Figure 10. Therefore, refrigeration (usually at 4°C) is a good method to preserve the odor potential of new biosolids samples until the start of incubation.

The bottle incubation at room temperature is yields the VOS odor potential of a specific cake in a reasonable time (Figure 10), and allows for identification of the most odorous cakes and was used for most of the incubation in the WERF II study (Adams, et al., 2003). Alternatively, incubation near the expected field storage temperature is advised to mimic the expected necessary "curing time" of cakes under field conditions.

Similar VOS production-consumption patterns as shown in Figures 6, 7, 10 and 11 were found for all digested and centrifuged cakes of 9 WWTP in the USA (Adams et al, 2003; Novak et al., 2004). Thus, it can be concluded that such cakes can be cured as non-aerated piles at appropriately high storage temperature and after sufficient storage time. The explanation for these

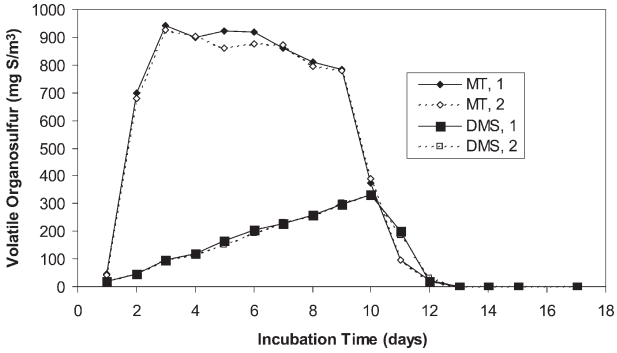

Figure 11. *VOSC concentrations in duplicate storage bottles (Wheaton serum bottles, glass, 125 mL, plus 10 g of digested cake, 28% TS. Incubation at 22°C. Repeated analysis of the same bottles on subsequent days.*

time patterns of VOS microbial production and consumption, and for the very different magnitude of the concentration peaks, is given by Higgins et al. (2006).

A very different pattern is found for limed biosolids (Novak et al., 2002), due to the high pH on both chemical and microbial activity. Limed sludges produce odorous compounds that are dominated by pH-neutral sulfur compounds (DMS and DMDS) and alkaline trimethylamine, and these gases will survive storage for weeks like in Figure 9 due to the absence of biological degradation.

CONCLUSIONS

This incubation and headspace method was found to be reproducible to compare the odor potential of anaerobically digested and dewatered sludge cakes. Specifically, it is recommended an incubation time be used to achieve production of VOS compounds to their peak concentration (the odor potential that is highly specific of the cake) and subsequently their consumption (the "odor curing time" that is highly specific of temperature).

For the purpose of comparing the odor potential of different biosolids, the experimental conditions must be controlled:

- Ship biosolids sample in airtight vessel overnight on ice. If the start of incubation is delayed, samples can be conserved in refrigerator between 1 and 4°C, for up to one week. Do not use frozen and thawed samples.
- Keep a volume ratio sample/bottle of at least 20%.
- Incubate samples in airtight bottles (glass, PET-plastic). Make sure that no excessive biogas pressure builds up. Test vessels must be air-tight, but not necessarily pressure tight.
- Incubate samples at a constant "room temperature" with a precision of 0.5°C in the absence of light. Sulfide (VOS) odor will peak and than disappear.
- Analyze headspace gas with any appropriate method for key odorants (GC, gas test tubes, chemical sensors) or overall odor (olfactometry) and use the headspace odor concentration as a measure of the odor potential of cake.

For the purpose of mimicking specific field conditions (influence of winter temperature, temperature fluctuation, freezing/thawing, static pile curing for many months, scale, cake porosity, etc.) the incubation conditions must be accordingly specified.

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