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Research Paper

VOC emissions from beef feedlot pen surfaces as affected by within-pen location, moisture and temperature☆

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A laboratory study was conducted to evaluate the effects of pen location, moisture, and temperature on emissions of volatile organic compounds (VOC) from surface materials obtained from feedlot pens where beef cattle were fed a diet containing 30% wet distillers grain plus solubles. Surface materials were collected from the feed trough (bunk), drainage, and raised areas (mounds) within three feedlot pens. The surface materials were mixed with water to represent dry, wet, or saturated conditions and then incubated at temperatures of 5, 15, 25 and 35 °C. A wind tunnel and gas chromatograph-mass spectrometer were used to collect and quantify emissions of eight volatile fatty acids (VFAs), five aromatics and two sulfur-containing compounds. Pen location significantly ($P < 0.05$) affected measurements of 10 of the VOC with the largest values occurring for materials collected near the mound area. The largest VFA and aromatic emissions resulted for the dry moisture condition while wet and saturated conditions produced the largest sulfide emissions. Temperature affected emission of each VOC except indole, with values generally increasing as temperature increased. Odour activity value (OAV), which was the ratio of measured concentration of a single compound normalised to the odour threshold for that compound, was calculated for each compound. Four VFAs contributed 7.5% of the total OAV but only one aromatic, 4-methylphenol, was a major contributor to total OAV at 2.5%. In comparison, sulfide compounds contributed 87.3% of the total OAV. This research shows VOC emissions are affected by pen location, moisture condition, and temperature.

Abbreviations: AFOs, animal feeding operations; DMDS, dimethyl disulfide; DMTS, dimethyl trisulfide; OAV, odour activity value; VFAs, volatile fatty acids; VOCs, volatile organic compounds; WDGS, wet distillers grains with solubles.

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1. Introduction

Airborne pollutants from animal feeding operations (AFO) may be a health concern to downwind populations (Donham et al., 2007; Heederik et al., 2007; Katja et al., 2007; Thorne, 2007; Wright et al., 2005). Chronic exposure to these pollutants has been associated with increased incidence of respiratory diseases, particularly for those responsible for the care of animals (Mitloehner & Calvo, 2008; Omland, 2002). Typical airborne pollutants from AFOs are comprised of particulate matter, biological materials, and chemicals including malodorous compounds. Malodorous compounds consisting of alcohols, amides, aromatics, sulfides, and volatile fatty acids (VFAs) are emitted during the microbial degradation of manure (Mackie, Stroot, & Varel, 1998; Miller & Berry, 2005; Miller & Varel, 2001; Rappert & Muller, 2005; Trabue et al., 2011).

Researchers have examined the effects of feeding ethanol by-products, including wet distillers grains plus solubles (WDGS), on odour characteristics of excreted manure. Introduction of ethanol by-products as a feed ingredient for beef cattle has modified the emission characteristics of manure (Gralapp, Powers, Faust, & Bundy, 2002; Spiehs & Varel, 2009; Varel, Wells, Berry, & Miller, 2010; Varel et al., 2008). Spiehs and Varel (2009) found that increasing the amount of WDGS in beef cattle rations increased phosphorus (P), nitrogen (N), and sulfur (S) intake and excretion. As a result, there was an increased production of odorous compounds (primarily long- and branched-chain VFAs and phenol) as well as increased ammonia (NH_3) and hydrogen sulfide (H_2S) emissions from a feedlot. Conversely, Hales, Parker, and Cole (2012) measured emissions of volatile organic compounds (VOC) from faeces and urine of cattle fed steam-flaked corn diets containing 0, 15, 30, or 45% WDGS, and reported no difference in VOC flux among the varying diets. Many of the previous studies investigated emissions from freshly excreted manure, which comprises only a small portion of the manure in feedlot pens. The focus of the present investigation was emissions from aged manure thoroughly mixed with soil from the pen surface, which should provide more realistic information regarding the characteristics of odorous emissions from open-lot feedlots.

Substantial research has been performed using different approaches to measure odour emission characteristics and rates from AFO (Auvermann, Paila, Hiranuma, & Bush, 2007; Kyoung, Hunt, Johnson, Szogi, & Vanotti, 2007; Todd, Cole, Harper, & Flesch, 2008; Trabue, Scoggin, Li, Burns, & Xin, 2008). Flux chambers and wind tunnels have been used to determine emissions at specific points on a pen surface (Hudson et al., 2009; Meisinger, Lefcourt, & Thompson, 2001). However, point measurements obtained using flux chambers have been shown to alter surface conditions and the measurements may not accurately estimate emissions (Cole, Todd, Parker, & Rhoades, 2007). Additionally, point measures may not adequately estimate large area emissions, particularly when there is considerable spatial variability (Cole et al., 2007; Parker, Rhoades, Schuster, Kiziel, & Perschbacher–Buser, 2005; Parker et al., 2008, 2009).

Micrometeorological theories and associated measurement technologies have been effectively used to measure emissions from larger areas (Flesch, Wilson, Harper, & Crenna, 2005; Flesch, Wilson, Harper, Todd, & Cole, 2007; Harper, Denmead, Freney, & Byers, 1999; McGinn, Janzen, & Coates, 2003; Todd et al., 2008). These methods generally have minimal impact on the pen surface and, therefore, are better suited for estimating total emissions. However, these techniques lack the resolution necessary to develop precision management practices for mitigating emissions from pen surfaces.

Physical, chemical and microbiological composition of feedlot surface materials has been shown to vary greatly both spatially and temporally (Cole, Mason, Todd, Rhoades, & Parker, 2009; Miller et al., 2006; Miller, Curtis, Larney, McAllister, & Olson, 2008; Rice, Mason, Cole, & Clark, 2007). Established geophysical methods have demonstrated that topography, pen design and layout can influence the pattern of manure accumulations (Eigenberg, Lesch, Woodbury, & Nienaber, 2008, 2010; Woodbury, Lesch, Eigenberg, Miller, & Spiehs, 2009; Woodbury, Eigenberg, Varel, Lesch, & Spiehs, 2011). Geophysical methods are useful in providing estimates of the percentage of the pen surface that is most responsible for malodorous emissions; however, predicting types and amounts of gas emissions requires more information than is provided using these techniques.

Pen location, precipitation and temperature are three important parameters that may influence emission types and amounts for a given diet. The influence of temperature on odour emissions is intuitive and has been documented. However, the combined effects of moisture, temperature and location within a pen on the types of odour that are emitted are not well known. Commercial-sized feedlot pens have designs and slopes that can influence the areas where manure accumulates on the pen surface. Typically manure, including manure that has been recently excreted, accumulates in greater quantities behind the bunk apron and near the water trough. The base of the mounds can have a relatively large manure content and greater mixing with soil. Down-gradient zones may have substantial manure accumulations which can be detached and transported by overland flow. All of these physical characteristics can have profound impacts on odour characteristics.

The objective of this study was to determine the effects of moisture, temperature and location in a pen on the types and amounts of odorous emissions from feedlot surface materials generated from cattle fed a diet containing 30% WDGS. This information will provide an improved understanding of emission characteristics from commercial-size pens so targeted mitigation practices can be developed. Additionally, the information from this study can be used to design larger *in-situ* studies evaluating odour mitigation practices.

2. Materials and methods

2.1. Collection of feedlot surface materials

Unconsolidated feedlot surface materials (FSM) were collected from three adjacent feedlot pens located at the U.S. Meat Animal Research Center near Clay Center, Nebraska (Fig. 1). The 30 × 90 m pens contained a central mound constructed

from manure and soil and a 3 m concrete apron located behind each feed bunk. Each pen had a slope gradient that ranged from 2 to 4%, and was stocked with approximately 80 bullocks (steers) that were fed a corn-based (dry-rolled) diet containing 30% WDGS. The bullocks entered the pens at 375 kg and were removed approximately 120 days later at 580 kg. Typical pen maintenance involved reshaping the mound, scraping and removing excess manure accumulation following animal removal. Three pen locations (bunk, base of the mound and drainage area) were selected to determine if feedlot location influenced odour characteristics (Fig. 1).

The manure was first removed from selected locations within the pens and placed in individual piles in an outdoor manure storage facility. The manure was allowed to air-dry for approximately 3 weeks in the facility until it could be ground using a portable wood chipper. The piles were not exposed to any appreciable rain during that period. The ground manure was stored indoors in separate 125 L plastic containers. The moisture content (gravimetric) of the manure during storage was 15.9%, 12.5%, and 11.8% for the bunk, base of the mound, and drainage area, respectively.

2.2. *Experimental procedures*

The experimental treatments included pen location (bunk, base of the mound, and drainage area), moisture condition (dry, wet, and saturated), and temperature (5, 15, 25 and 35 °C). Each of the experimental treatments was replicated three times and the feedlot surface material was placed in a temperature and humidity-controlled environmental chamber. Gas samples for measurements of VOC were collected within an environmental chamber at 0, 2 and 4 days. The temperature within the environmental chamber was controlled and relative humidity was maintained between 50 and 55% by a relative humidity sensor that was connected to a controller that either turned on/off a humidifier or de-humidifier as needed.

Approximately 6.0 kg of FSM (dry weight) from each pen location was mixed with distilled water. Water addition was adjusted slightly to account for moisture content of the surface material so that a total of 0, 1.5 or 3.0 L of water was present to represent dry, wet, and saturated treatment conditions, respectively. The FSM were placed and initially packed by hand within stainless steel pans (50 cm long × 30 cm wide × 6.5 cm deep) and then pressed into a uniform depth of 5 cm using a hydraulic press to obtain a consistent density among the treatments. Total pan weights were recorded after compaction and daily additions of water were added using a hand sprayer to compensate for evaporated water. Pans were arranged in a randomised block design on three identical racks located in the environmental chamber. Gas samples were collected 0, 2 and 4 days following treatment preparation. The VOC measurements were obtained for a specified temperature and then the material was discarded. The pans were re-packed with the same treatment and the temperature of the chamber was adjusted for additional collection of VOC.

2.3. *Wind tunnel flux measurements*

The use of wind tunnels is known to alter surface conditions and may not accurately measure actual emissions. However, wind tunnels are able to measure relative differences among treatments. Details on the operation of the small wind tunnel have been reported by Parker et al., 2010. To summarise, the wind tunnel had a 51 mm height, 305 mm length, and 152 mm width, with a footprint of 0.046 m^2 and internal volume of 2.36 l. The sweep air entered the wind tunnel through 17 holes (6-mm diameter) in three rows at heights of 17 mm (6 holes), 30 mm (5 holes), and 43 mm (6 holes) above the base. Air exited the wind tunnel through three 10-mm diameter holes equally spaced at a height of 27 mm above the surface at the opposite end of the tunnel. Sweep air (1 l min^{-1}) was supplied via Teflon® tubing from a compressed air system that was passed through a carbon filter and a humidity system that maintained relative humidity equivalent to the chamber air.

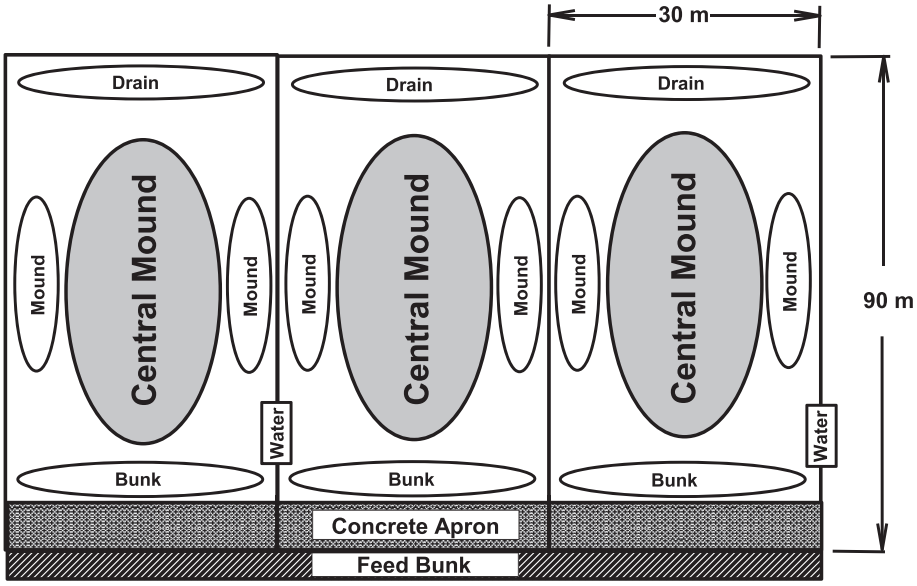

Fig. 1 – Schematic showing pen layout and the location of unconsolidated surface material collected for laboratory testing.

VOC samples were collected from the air exiting the wind tunnel following an equilibration period that allowed three volumes of sweep air to pass through the wind tunnel. Air samples were obtained in stainless steel sorbent tubes (89 mm × 6.4 mm outside diameter, Markes International Inc., Wilmington, DE, USA) filled with Tenax TA® sorbent. Prior to use, the sorbent tubes were conditioned for 30 min at 230 °C. A sample was pulled through the sorbent tubes at a flow rate of 75 ml min^{-1} for 30 min using a vacuum pump (Pocket pump 210 series, SKC Inc., Eighty Four, PA, USA).

Flux density, *J*, was calculated on a mass per unit area per unit time basis (μg m^{-2} min^{-1}) using Eq. (1):

$$J = \frac{Q\,C_{air}}{A} \quad (1)$$

where Q is sweep air flow rate (m^3 min^{-1}), C_{air} is VOC concentration of the exiting air (μg m^{-3}), and *A* is footprint area of the wind tunnel (m^2).

2.4. Gas chromatography/mass spectrometry analyses

Sorbent tube samples were collected in duplicate and measurements were averaged. A thermal desorption-gas chromatography-mass spectrometry (TD-GC-MS) system was used to analyse the sorbent tube samples. The TD-GC-MS system consisted of a Markes Unity 2 thermal desorber with Ultra 2 autosampler (Markes International Inc., Wilmington, DE, USA). Samples were quantified with an Agilent 7890A/5975C GC/MS (Agilent Technologies, Inc., Santa Clara, CA, USA). The system used an Agilent Innowax, 30 m × 0.25 mm internal diameter (ID) capillary column (polyethylene glycol, 0.25 μm film thickness) that was operated under a constant helium carrier gas flow rate of 1.4 ml min^{-1}.

Samples were first purged with helium for 1 min at 40 ml min^{-1} to remove water and air. The tube was then desorbed for 10 min at 280 °C with a helium carrier gas flow of 50 ml min^{-1} and emitted compounds were trapped on a cold trap maintained at −10 °C. The cold trap was heated to 320 °C for 1 min with a helium carrier gas flow of 20 ml min^{-1}, and transferred to the column using a split ratio of 13.3:1. The column was first held in the GC oven at 40 °C for 3 min, the temperature was then increased to 230 °C at a rate of 8 °C min^{-1}, and finally held at 230 °C for 5 min.

Samples were analysed for three basic categories of compounds, VFAs, aromatics and volatile sulfur compounds (VSC). The compounds were selected based on those compounds most prevalent in our previous sorbent tube studies on emissions from beef cattle manure (Woodbury et al., 2013). The eight VFAs were ethanoic, buoic, heptanoic, hexanoic, 2-methylpropanioc, 3-methylbutanoic, propionic, and pentanoic acids. The five aromatic compounds were 4-ethylphenol, 4-methyphenol, indole, phenol, and skatole. The two sulfur-containing VOC were dimethyl disulfide (DMDS) and dimethyl trisulfide (DMTS). Calibration standard solutions were prepared by diluting known masses of pure chemicals with methanol. Standards were prepared and analysed within 48 h to establish standard curves. The prepared standards were periodically checked with stored standards to identify any changes in sensitivity of analysis. All chemicals and solvents were FCC grade (Sigma Aldrich, St. Louis, MO, USA). Standards were prepared using serial dilutions, and then injected onto clean tubes while purified air was pulled through the tubes with a vacuum pump operated at 75 ml min^{-1}. Standard solutions were stored for periodic instrument calibrations.

Our experience has shown little evidence that VFAs are ionised and become non-volatile. We have seen nearly identical GC responses from freshly made standards when compared to standards that have been stored, which indicates minimal ionisation or response issues. Within the linear range, standard curves were fitted using linear regression with zero y-intercept. Coefficients of determination (r^2) for the standard curves ranged from 97.8 to 99.7% for the eight VFA, 97.8–99.3% for the five aromatics, and 99.8% for both DMDS and DMTS.

Recent work by Andersen, Hansen, and Feilberg (2012) has shown that thermal desorption procedures similar to those used in this research can dimerise methanethiol (MT) to other VSC like DMDS and DMTS. Because MT is sometimes reported in manure in anaerobic environments, it is difficult to definitively quantify whether VSC emissions are MT, DMDS, DMTS, or a combination thereof. For this reason, we have estimated odour impacts as discussed in the following section using two methods, first using measured DMDS/DMTS concentrations, and second assuming equal mass of methanethiol was converted to DMDS and/or DMTS.

2.5. Odour activity value and analyses

Concentrations of individual compounds were converted to their respective odour activity values (OAV). An assessment of the relative impact of each individual odour compound is provided by OAV analyses. Details of the conversion can be found in Parker et al. (2013); however, a brief description is provided below.

The OAV is a ratio of the measured concentration of a single compound normalised to the odour threshold (SCOT) for that compound (Friedrich & Acree, 1998; Trabue, Anhalt, & Zahn, 2006; Parker et al., 2010, 2013; Patton & Josephson, 1957). Therefore, the higher the OAV for an individual compound, the more likely that compound will contribute to the overall odour of a complex odour mixture.

The single compound odour threshold (SCOT) values for each compound were obtained using values from published odour thresholds (Table 1). The relative contribution of each compound was calculated by subtracting background emission for each compound and then dividing by the sum of the OAV for all measured compounds. It should be noted that this approach does not account for possible synergistic or other complex interactions of the compounds (Powers, 2001; Zahn et al., 2001).

2.6. Statistical analyses

The effects of pen location, moisture, and temperature on VOC measurements were determined using analysis of variance (ANOVA) (SAS Institute, 2011). By using ANOVA it was possible to test for significant differences among experimental variables. If a significant difference was identified, the least significant difference test (LSD) was used to identify

Table 1 – A summary of published single compound odour thresholds (SCOT) for the fifteen VOC's measured in this study. Different statistical measures of central tendency are provided. All units are in μg m^{-3}. All raw data on thresholds from van Gemert (2003) unless otherwise noted.

Compound	N[a]	Min	Max	Arithmetic mean	Std. dev.	Geometric mean	Harmonic mean	Median
Ethanoic acid	8	25	7500	2480	2754	578	85	2050
Propionic acid	7	3	890	303	344	106	18	80
2-Methylpropanoic acid	2	0.8	285	145	198	38	10	145
Buioc acid	11	0.4	105	25	34	6.9	1.4	13
3-Methylbutanioc acid	5	0.22	14	5.0	5.5	2.3	0.81	4.1
Pentanoic acid	6	0.8	75	24	30	8.8	3.0	9.0
Hexanoic acid	5	12	510	182	226	69	31	40
Heptanoic acid	3	22	300	118	157	60	38	33
Phenol	9	39	4000	734	1290	206	88	200
4-Methylphenol	4	0.05	24	9.2	11.5	1.3	0.16	6.3
4-Ethylphenol[b]	1	6.3	6.3	6.3	–	6.3	6.3	6.3
Indole	2	0.6	7.1	3.8	4.6	2.1	1.1	3.8
Skatole	4	0.35	0.78	0.51	0.19	0.48	0.46	0.45
Dimethyl disulfide	5	1.6	64	25	28	12	5.3	8.5
Dimethyl trisulfide	3	0.08	14	7.2	7.0	2.0	0.24	7.5
Dimethyl sulfide	15	0.3	750	113	242	14.0	2.8	9.4
Methanethiol	14	1E-9	1100	159	379	0.39	1.4E-8	0.50
Hydrogen sulfide	15	0.1	270	32.5	72.4	3.2	0.74	2.0

[a] n = number of independent odor threshold observations used in the calculations.
[b] 4-Ethylphenol threshold from Trabue et al. (2008).

differences among experimental treatments. A probability level <0.05 was considered significant.

3. Results and discussion

3.1. *Treatment effects on VOC*

Pen location significantly affected measurements of ethanoic acid, propanoic acid, 2-methylpropanoic acid, buoic acid, 3-methylbutanoic acid, 4-methyphenol, indole, skatols, and the two sulfide compounds (Tables 2 and 3). The largest measured values for each of these compounds occurred for FSM collected from the base of the mound. With the exceptions of isobutric acid and 4-methylphenol, no significant difference in emission measurements were found among FSM collected from the bunk and drainage areas.

Each of the VOCs except for two VFAs (hexanoic and heptanoic) and one aromatic (indole) were significantly affected by moisture content. The largest VFA and aromatic emissions occurred for the dry soil moisture condition, while the wet and saturated soil water conditions produced the largest sulfide emissions. Recent work by Andersen et al., (2012) has shown solid sorbent material used in this study can dimerise methanethiol (MT) to other VSC like DMDS and DMTS. As a result, we can't quantify specifically which VSC is driving the OAV. Although our analysis method has limitations quantifying specific sulfur-containing compounds, wet FSM conditions resulted in greater emissions of VSC.

The literature is incomplete regarding our understanding of the formation of volatile organic sulfur compounds on feedlot surfaces. A study by Beard and Guenzi (1983) indicated that the formation of DMDS from cattle manure slurry was redox-dependent. They showed that DMDS was produced appreciably from cattle manure slurries from redox +300 mV to −100 mV potential, with the greatest formation at or below 0 mV. They also showed the greatest formation of methanethiol occurred at −100 mV or less. Additional work by Higgins et al., (2006) on municipal waste water activated sludge systems indicated the formation of DMDS could be an abiotic process that converts methanethiol to DMDS in the presence of oxygen and a metal catalyst. Fritz and Bachofen (2000) have also suggested this abiotic pathway for the formation of DMDS and DMTS in natural, freshwater systems. Our work suggests the controlling factor for emission to the atmosphere is water solubility regardless of the pathway by which volatile organic sulfur compounds are formed. Generally, VFAs and aromatic compounds are more soluble than DMDS and DMTS. Therefore, these compounds remain in solution and are not emitted at higher soil moisture contents. Additional work needs to be performed to evaluate the effects of wetting and drying cycles on emissions.

Temperature affected all three categories of emissions and each of the individual VOCs except indole which had minimal emissions. The VOC emissions generally increased as temperature increased. For each of the compounds except indole and skatol, VOC measurements obtained at the 35 C temperature were significantly larger than values obtained at the other temperatures.

3.2. *Contributions of individual constituents to OAV*

The percentage of OAV for each constituent averaged over all pen locations, moisture conditions, and temperature is illustrated in Table 4. Also listed in Table 4 are the percentages for each category of odour. The following discussion will focus only on those compounds with OAV values greater than 1%. It was assumed that compounds with OAV values less than 1% were minor contributors to the overall odour activity.

Table 2 – Odorous VOC emissions in μg m^{-2} min^{-1} as affected by pen location, moisture condition, and temperature. For a given experimental treatment (location, moisture condition, or temperature), mean values within a column followed by different letters are significantly different at the 0.05 probability level based on the LSD test. Note description of individual odour compound abbreviation is listed in footnote below.

	Ethan	Propi	2-Meth	Buoic	3-Meth	Penta	Hexan	Hepta	Pheno	Methy	Ethyl	Indole	Skat	DMDS	DMTS
Location															
Bunk	0.961b	0.098b	0.025a	0.051b	0.009b	0.041	0.259	0.034	0.141	0.010a	0.003	0.002b	0.0004b	1.29b	0.125b
Mound	1.31a	0.119a	0.027a	0.066a	0.012a	0.053	0.405	0.047	0.142	0.011a	0.004	0.006a	0.0009a	3.89a	0.292a
Drain	1.04b	0.097b	0.024b	0.054b	0.009b	0.044	0.339	0.045	0.132	0.009b	0.003	0.002b	0.0004b	1.69b	0.094b
Moisture															
Dry	1.49a	0.132a	0.030a	0.065a	0.011a	0.057a	0.416	0.050	0.180a	0.011a	0.004a	0.006	0.0009a	0.162c	0.025b
Wet	0.885b	0.089b	0.024b	0.053b	0.009b	0.039b	0.290	0.036	0.114b	0.009b	0.003b	0.002	0.0005b	2.90b	0.218a
Saturated	0.932b	0.092b	0.023b	0.054b	0.009b	0.041b	0.298	0.040	0.122b	0.009b	0.003b	0.003	0.0003b	3.81a	0.269a
Temp. (°C)															
5	0.644c	0.057c	0.013d	0.028d	0.005d	0.010c	0.018c	0.008c	0.067c	0.004c	0.001c	0.002	0.0002b	0.892b	0.040b
15	0.978b	0.091b	0.018c	0.047c	0.008c	0.021c	0.074c	0.015c	0.080b	0.007b	0.002b	0.006	0.0007a	1.72b	0.084b
25	0.883bc	0.103b	0.026b	0.061b	0.011b	0.058b	0.440b	0.048b	0.082b	0.008b	0.002b	0.002	0.0003b	0.931b	0.059b
35	1.90a	0.168a	0.046a	0.092a	0.016a	0.095a	0.805a	0.096a	0.324a	0.021a	0.006a	0.005	0.0010a	5.61a	0.500a

Ethan = Ethanoic acid; Propi = propionic acid; 2-Meth 2-Methylpropanoic acid; 3-Meth = 3-Methylbutanoic acid; Penta = Pentanoic acid; Hexan = hexanoic acid; Hepta = heptanoic acid; Pheno = phenol; Methy = 4-methyphenol; Ethyl = 4-ethylphenol; Indole = indole; Skat = skatole; DMDS = dimethyl disulfide; DMTS = dimethyl trisulfide.

Table 3 – ANOVA showing the effects of pen location, moisture condition, and temperature on VOC emissions. Note description of individual odour compound abbreviation is listed below in a footnote.

	Ethan	Propi	2-Meth	Buoic	3-Meth	Pentan	Hexan	Hepta	Pheno	Methy	Ethyl	Indole	Skat	DMDS	DMTS
ANOVA	Pr > F														
Location	**0.01**	**0.01**	**0.02**	**0.01**	**0.01**	0.07	0.13	0.16	0.09	**0.01**	0.08	**0.03**	**0.01**	**0.01**	**0.01**
Moisture	**0.01**	**0.01**	**0.01**	**0.03**	**0.03**	**0.01**	0.16	0.15	**0.01**	**0.01**	**0.02**	0.07	**0.01**	**0.01**	**0.01**
Location × Moisture	**0.01**	0.03	**0.01**	0.30	**0.01**	0.34	0.33	0.19	**0.05**	**0.01**	**0.03**	**0.01**	**0.01**	**0.01**	0.23
Temperature	**0.01**	**0.01**	**0.01**	**0.01**	**0.01**	**0.01**	**0.01**	**0.01**	**0.01**	**0.01**	**0.01**	0.11	**0.01**	**0.01**	**0.01**
Location × Temperature	0.76	0.90	0.22	0.86	0.17	0.78	0.43	0.16	**0.01**	0.29	0.28	0.16	0.07	**0.01**	**0.01**
Moisture × Temperature	**0.01**	**0.01**	**0.01**	0.06	0.42	**0.01**	0.12	0.06	**0.01**	**0.01**	0.15	0.08	0.11	**0.01**	**0.01**
Location × Moisture × Temperature	0.15	0.28	**0.01**	0.12	0.06	**0.10**	0.07	**0.01**	**0.01**	0.06	0.08	0.10	**0.05**	**0.01**	0.24

Ethan = Ethanoic acid; Propi = propionic acid; 2-Meth 2-Methypropanoic acid; 3-Meth = 3-Methylbutanoic acid; Pentan = Pentanoic acid; Hexan = hexanoic acid; Hepta = heptanoic acid; Pheno = phenol; Methy = 4-methyphenol; Ethyl = 4-ethylphenol; Indole = indole; Skat = skatole; DMDS = dimethyl disulfide; DMTS = dimethyl trisulfide.
Bolded values indicate statistical significance at the P = 0.05 level.

Compounds with OAV above 1% accounted for over 97% of the total odour activity.

Four VFA compounds (buoic (2.8%), 3-methylbutanoic (1.4%), pentanoic (1.7%) and hexanoic (1.6%) acid) contributed 7.5% of the total OAV. The relatively small contribution of VFA to the total OAV is similar to what others have found when manure is derived from diets containing WDGS. Spiehs and Varel (2009) found in an incubation study of recently deposited manure and urine that total VFA concentration in faeces decreased as the percentage of WDGS in the diet increased. They also noted that feedlot cattle fed increasing amounts of WDGS had greater P, N, and S intake and excretion, which may have contributed to the production of odorous compounds (primarily long- and branched-chain VFA). Varel et al. (2010) reported reduced total VFA production with the inclusion of 40% WDGS in cattle diets during a study using beef manure collected from pen surfaces. Miller and Varel (2001, 2002) found that once starch is depleted, protein fermentation in aged manure is likely and the fermentation may result in the production of objectionable odours like branch-chained VFA, including phenolic compounds which have a very low odour threshold.

There was only one aromatic, 4-methylphenol, that at approximately 2.5% was a major contributor to the total OAV (Table 4). In a recent study, Hales et al. (2012) evaluated diets containing 0, 15, 30 and 45% WDGS and measured odour emissions from faeces using wind tunnel technology. The faeces and urine were collected separately and never mixed together or with soil during the study. They found that varying amounts of WDGS in the diet did not significantly affect the emission of selected aromatic compounds (4-methylphenol, indole, and skatole) from faeces.

Spiehs and Varel (2009) also found no significant difference in the concentration of selected aromatic compounds (4-ethyphenol, indole, and skatole) in freshly excreted (not mixed with soil) faeces from cattle fed four selected dietary treatments. However, Varel et al. (2010) found an increase in 4-methylphenol concentration during 28 day slurry incubations of manure collected from soil pen surfaces where cattle were fed diets containing 0 and 40% WDGS. The increase was greater for the 40% WDGS diet than for the corn-based diet and concentrations increased with incubation time. Miller and Varel (2001) measured manure odour emissions and indicated the potential for the formation of aromatics resulting from the on-going fermentation of aged manure once most of the starch had been metabolised by the microbial population in the manure.

The two sulfide compounds, DMDS (60.4%) and DMTS (26.8%), were the largest contributors to total OAV (Table 4). As previously stated, we can't quantify specifically which VSC was driving the OAV. However, assuming complete (equal mass) dimerization of MT, the assumed OAV attributed to VSC would be over 99% (Table 4). Unfortunately, our analysis method lacks the resolution to adequately specify which sulfide compound is the primary contributor to the overall OAV, but it was apparent that VSCs are a very important consideration when developing odour mitigation practices.

Additionally, other reduced sulfur compounds, such as hydrogen sulfide, may contribute to odour emissions. Hydrogen sulfide is a primary odorant measured in animal housing facilities, particularly swine (Hansen, Adamsen, Pedersen, & Feilberg, 2012; Kim et al., 2008; Ni et al., 2002). However, hydrogen sulfide from open lot beef feedlots is not as well established. Miller, Varel, Woodbury, and Spiehs (2010) showed hydrogen sulfide emission were well below the 0.1 ppm (30 min average) Nebraska regulatory limits when cattle are fed diets containing up to 40% WDGS. However, Miller did find increasing the percentage of WDGS in the diet increased hydrogen sulfide emissions. These increases were during the warmer summer months from areas of the pens that were chronically wet, such as behind the feed bunk or near the water trough. Additional work looking at downwind measures of hydrogen sulfide found levels to be well below regulatory levels. Koelsch, Woodbury, Stenberg, Miller, and Schulte (2004) performed an ambient air quality investigation on feedlots where a corn-based (non-WDGS) diet was fed, and showed that total reduced sulfur (TRS), as measured using a Jerome meter, in the vicinity of beef cattle feedlots did not exceed current regulatory thresholds used in the Midwestern United States. The concentration of TRS was found to vary with air temperature and time of day. However, wet feedlot surface conditions and wind speed had minimal impact upon measured concentrations of TRS.

Sulfide emissions from manure generated from diets containing WDGS were found to be influenced by the higher S content typically found in the WDGS feed stock when

Table 4 – Relative percent contribution of odorant to the total activity value (OAV) averaged for pen location, moisture condition, and temperature. Each compound is normalised for its specific odour threshold. The OAV for VSC was calculated using two methods, first using measured DMDS and DMTS concentrations, and second assuming equal mass of methanethiol was converted to DMDS and/or DMTS due to analysis method.

	OAV as DMDS/DMTS	OAV as methanethiol[a]
VFA		
Ethanoic acid	0.6	
Propanoic acid	0.3	
2-Methylpropanoic acid	0.2	
Buoic acid	2.8	
3-Methylbutanoic acid	1.4	
Pentanoic acid	1.7	
Hexanoic acid	1.6	
Heptanoic acid	0.3	
Subtotal	**8.9%**	**0.4%**
Aromatics		
Phenol	0.2	
4-Methylphenol	2.5	
4-Ethylphenol	0.2	
Indole	0.6	
Skatole	0.4	
Subtotal	**3.9%**	**0.3%**
Sulfides		
Dimethyl disulfide	60.4	92.5%
Dimethyl trisulfide	26.8	6.8%
Subtotal	**87.2%**	**99.3%**
Total	**100.0%**	**100.0%**

[a] Assuming equal mass of methanethiol was converted to DMDS and/or DMTS.

compared to corn diets (Spiehs & Varel, 2009; Varel et al., 2010). Higher concentrations of S-containing compounds in the distiller's by-product results from the removal of the corn starch component and the addition of S compounds during ethanol production. The production of volatile S compounds from manure slurry under conditions where the redox potential was controlled was investigated by Beard and Guenzi (1983). They found DMDS and methanethiol (MT) were produced at all the evaluated redox levels. However, the greatest production of DMS and DMDS occurred at 0 mV and, as the redox potential decreased to −100 mV, the reaction favoured the production of H_2S and MT. Diverse levels of redox potentials can be found on any active pen surface. Wet conditions typically found behind the bunk apron and within the drainage end of the pen may produce redox potentials that are more favourable for the production of VSC. The wetting and drying cycles typically found at the base of the mound may also have redox potentials that would favour VSC production during selected periods.

3.3. *Treatment effects on OAV*

The total OAV based on pen location, moisture, and temperature are presented in Table 5. The percentage of total OAV by location was 22.8, 52.2, and 25.0% for the bunk, base of the mound and drainage area respectively (Table 5). Woodbury, Miller, Nienaber, and Eigenberg (2001) evaluated microbial denitrification enzyme activity (DEA) across feedlot pens and found DEA to be the largest near the base of the mound. This increased DEA concentration remained throughout the winter months even after the surface was frozen. One of the locations where the unconsolidated surface material was collected for this study was the base of the mound, which is affected by a variety of environmental conditions. This area is typified by high faeces and urine accumulations because any material deposited on the mound eventually accumulates at the base. Also, this area experiences very dry and aerated periods as well as periods of saturation during and shortly after a precipitation event. The areas behind the bunk apron and the drainage area generally are not as aerated and typically experience much longer wet periods. Environmental conditions near the base of the mound may result in a more active and diverse microbial population resulting from greater variations in redox potentials due to the wetting and drying cycles. Additional work is required to determine the reasons the base of the mound has a greater OAV than the other pen locations.

The percentage of total OAV for varying moisture conditions was 8.1, 37.4 and 54.5% for dry, wet, and saturated conditions, respectively (Table 5). In general, the addition of water decreased emissions of VFA and aromatics (Table 6). However, the addition of water increased the emission of VSC. As mentioned previously, the VFA and aromatics are generally more water soluble than VSC. The addition of water may cause the VFA and aromatics to remain within the FSM and promote the emission of sulfide compounds. Increased amounts of water would decrease the relative emission rates for the VFA and aromatics while increasing the relative emission rates of VSC. Also, the odour thresholds for most VSC are low and any relative increase in their emissions would have a substantial impact on the total OAV. Miller and Berry (2005) found that substantial odour production occurred when both high manure concentration and high moisture content were present. However, when high manure content was present in the absence of high moisture conditions, odour production has been found to be minimal (Liang, Das, & McClendon, 2003).

The percentage of total OAV for varying temperature was 9.2, 12.1, 17.5 and 61.2% at 5, 15, 25 and 35 C, respectively (Table 5). The percentage OAV consistently increased as the temperature varied from 5 C to 35 C. The largest increase in OAV resulted when temperature was raised from 25 C to 35 C. Odour emission is the result of microbial degradation of organic materials deposited on feedlot surfaces. Temperature has a large impact on microbial activity and temperatures

Table 5 – Contribution to total odour activity value (OAV) for each feedlot pen location, moisture condition, and temperature. All within treatment odour activity values sum to 100%.

Treatment	OAV (%)
Location within pen	
Bunk	22.8
Mound	52.2
Drainage	25.0
	100
Moisture status	
Dry	8.1
Wet	37.4
Saturated	54.5
	100
Temperature (°C)	
5	9.2
15	12.1
25	17.5
35	61.2
	100

Table 6 – Contribution of volatile fatty acids (VFA), aromatic, and volatile sulfur compounds (VSC) to total odour activity value (OAV) (using measured DMDS/DMTS concentrations) for each feedlot location, moisture condition, and temperature. All within treatment odour activity values sum to 100%.

Treatment	Odor category		
	VFA (%)	Aromatic (%)	VSC (%)
Location within pen			
Bunk	11.5	5.2	83.3
Mound	6.5	3.0	90.5
Drainage	11.4	4.3	84.3
Moisture status			
Dry	44.4	20.2	35.4
Wet	6.9	2.9	90.2
Saturated	5.0	2.0	93.0
Temperature (°C)			
5	18.9	4.5	76.6
15	20.7	5.7	73.6
25	7.8	4.7	87.5
35	6.8	3.1	90.1

below 25 C appear to limit the microbial activity that results in odour emissions.

The total OAV based on location, moisture, and temperature as contributed by VFA, aromatic and sulfide compounds are listed in Table 6. The contribution to total OAV at each pen location, moisture content, and temperature is dominated by the sulfide compounds (approx. or greater than 75%) with the exception of the dry soil moisture condition. The total percent OAV for the dry soil moisture condition for VFA, aromatic, and VSC were 44.4, 20.2 and 35.4%, respectively. The dry moisture condition had less soil water to retain the VFA and aromatics in solution; therefore, the contribution of these compounds to total OAV was not significantly inhibited.

3.4. *Limitations of the laboratory study*

This study was conducted under idealised laboratory conditions using unconsolidated materials collected from feedlot surfaces. The experimental treatments which included pen location, moisture, and temperature could not have been easily reproduced within individual feedlot pens in which cattle were present. In feedlot pens containing cattle, other variables not examined in this study including varying climatic conditions, surface disturbance by cattle hooves, and the input of recently deposited manure would be expected to influence VOC emissions.

4. Conclusions

A laboratory study was conducted investigating the effects of within pen location, soil moisture content and temperature on VOC emissions from pen surface materials collected from commercial-sized feedlot pens. This information can be used to help develop field-scale studies examining management practices for mitigating odorous emissions from pen surfaces. Evaluation of these parameters was performed on individually measured compounds and a normalising approach was used to calculate odour activity. When the odour compounds were normalised with respect to their activity value, many of the measured compounds contributed minimally to the overall odour activity. Approximately 10% of the OAV was contributed by VFA (buoic, 3-methylbutanoic, pentanoic, and hexanoic acid) and one aromatic (4-methylphenol). Sulfides contributed the most with 87.2% of the total OAV.

The design of the pens used for this study, the raised mound area comprised approximately 50% of the area and the bunk and drainage regions were each approximately 25% of the area. When evaluating the effects of pen location on OAV, more than half of the OAV occurred at the base of the mound with the bunk and drainage contributing approximately equally to the remainder. The more frequent wetting and drying cycles occurring near the base of the mound may have resulted in a more diverse microbial population when compared with the chronically wet to saturated conditions existing behind the feed bunk. Additional work is being performed to develop geophysical measures as a tool for predicting the types and amounts of emissions occurring from feedlot pen surfaces (Woodbury, Eigenberg, Varel, Lesch, & Spiehs, 2011).

The addition of water significantly increased the OAV when compared with dry soil moisture conditions. Approximately 92% of the OAV was accounted for by wet and saturated conditions. In general, the addition of water decreased emissions of VFA and aromatics, and increased the emission of sulfides. This is likely to be due to two reasons. First, the greater solubility of the VFA and aromatics allowed them to be retained in the solution fraction of the FSM and, therefore, they were not emitted. Second, the addition of water resulted in an anaerobic environment and reducing conditions, which are conducive to production of sulfide compounds. Additional work will need to be performed to evaluate the effects of wetting and drying cycles on emissions.

Temperature significantly affected OAV with over 60% of the total OAV occurring at the 35 C treatment. The 35 C temperature treatment also had a significant effect on each of the measured odour compounds, with the exception of indole. The increase in emissions at the 35 C temperature was greatest for the sulfide compounds.

Management of feedlot pen surfaces for odour emission can present difficult challenges for operators. This laboratory study illustrates that odour emissions from pen surface materials are affected by pen location, moisture condition, and temperature. This study also confirms results reported by others that the base of the mound is more biologically active than other pen locations. Understanding the spatial variability of odour emission is important in the development of effective management practices. Based on the results from this investigation, larger *in situ* studies can be conducted to develop targeted management practices for mitigating malodorous emissions.

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