Effects of Sodium Bisulfate on Alcohol, Amine, and Ammonia Emissions from Dairy Slurry

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Sodium bisulfate (SBS) is extensively used in the poultry industry to reduce ammonia and bacterial levels in litter. It is also used in the dairy industry to reduce bacterial counts in bedding and ammonia emissions, preventing environmental mastitis and calf respiratory stress. The present study measured the effect of SBS on the air emission of ammonia, amine, and alcohol from a dairy slurry mix. Amine flux was undetectable (<5 ng L^{-1}) across treatments. Application of SBS decreased ammonia, methanol, and ethanol emissions from fresh dairy slurry. Ammonia emissions decreased with increasing levels of SBS treatment. The 3-d average ammonia flux from the control (no SBS applied) and the three different SBS surface application levels of 0.125, 0.250, and 0.375 kg m^{-2} were 513.4, 407.2, 294.8, and 204.5 mg h^{-1} m^{-2}, respectively. The ammonia emission reduction potentials were 0, 21, 43, and 60%, respectively. Methanol and ethanol emissions decreased with an increase in the amount of SBS applied. The 3-d average methanol emissions were 223.7, 178.0, 131.6, and 87.0 mg h^{-1} m^{-2} for SBS surface application level of 0, 0.125, 0.250, and 0.375 kg m^{-2}, with corresponding reduction potentials of 0, 20, 41, and 61, respectively. Similar emission reduction potentials of 0, 18, 35, and 58% were obtained for ethanol. Sodium bisulfate was shown to be effective in the mitigation of ammonia and alcohol emissions from fresh dairy slurry.

California is the leading dairy state in the USA, producing 21% of the nation's milk supply. The state is also home to two of the three worst air-sheds with respect to ozone (smog) pollution (Schwehr, 2004). Ozone is formed through the interaction of volatile organic compounds (VOCs) and oxides of nitrogen in the presence of sunlight. Volatile organic compound emissions from San Joaquin Valley (SJV) stationary sources have been reduced by 85% since 1975 (California Air Resources Board, 2006). However, further reductions in SJV air district emissions of oxides of nitrogen are required (by the state) to reach the ozone attainment standard.

Air emissions from agricultural processes in California have been regulated since 2003. Current regulatory estimates suggest that SJV dairies emit VOCs (aka reactive organic gas) at even higher rates than passenger vehicles (San Joaquin Valley Air Pollution Control District, 2004). Current SJV air district rules are intended to reduce VOC emissions from dairies, cattle feedlots, poultry ranches, and other animal husbandry operations by 15.8 tons or 26% d^{-1} (San Joaquin Valley Air Pollution Control District, 2005).

A total of 1.74 million lactating dairy cows are housed on 2107 dairies in California, and within the last 10 yr the number of cows per dairy has more than doubled to an average of 825. The majority of SJV dairies house lactating dairy cows in concrete-floored, open-sided, freestall barns. Each cow produces approximately 60 kg of manure daily, which is flushed with water from the milking parlor and freestalls into large manure ponds. Nonlactating dry cows and heifers are generally housed in dirt-floored drylot corrals, which are not flushed with water but scraped several times per year. Emission studies conducted in our lab have shown that fresh slurry and fermented feed (silage) are the main sources of VOCs from dairies, with the main compound groups being alcohols (ethanol and methanol). Furthermore, dairy slurry is known to emit substantial amounts of ammonia shortly after excretion of urine, which mixes with feces (Lefcourt and Meisinger, 2001). These gaseous releases are mainly produced by microbial and enzymatic activity on excreted nitrogenous and carbonaceous compounds in the feces and/or urine (Carey et al., 2005; Mutlu et al., 2005).

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Abbreviations: SBS, sodium bisulfate; VOC, volatile organic compound; SJV, San Joaquin Valley.

After the passage of California State Senate Bill 700 and subsequent Environmental Protection Agency (EPA) Title V permitting requirements of dairies in California, farmers began identifying practical and economical control technologies for VOC and ammonia. Although research efforts in the past have focused on control technologies that deal with liquid dairy waste storage and treatment, ongoing research identified fresh slurry in the animal housing areas to be a major source of VOC and ammonia. Therefore, management practices have to be implemented to effectively address emissions from fresh slurry (Dragosits et al., 2002).

Ehanol and methanol are produced during anaerobic fermentation in the cow's rumen by microbial strains like *Streptococcus bovis* and *Ruminococcus albus* (J. Russell, personal communication, 2006). Fresh slurry contains both of these alcohols in the liquid phase. Environmental drivers like pH, temperature, and oxygenation of the slurry influence the microbial and physical processes that determine which alcohols are produced, metabolized by bacteria, and transferred from liquid to gas phase. The production and emissions of gaseous ammonia from animal manure is dependent on urea content in urine, the pH and temperature of the manure, and urease activity (Monteny et al., 1998; Gay and Knowlton, 2005). Therefore, mitigation must address at least one of the main environmental drivers (e.g., pH) to effectively disrupt microbial and enzymatic activity and reduce atmospheric releases (Jongebreur and Monteny, 2001).

Streptococcus bovis does not grow in the presence of elevated sodium concentrations (5–6%), and there is a cessation of *R. albus* growth at a pH of 6.0 or below, so the application of acidifying sodium bisulfate (SBS) could conceivably reduce the growth and survival of these alcohol-producing organisms (Montefiore Medical Center and Albert Einstein College of Medicine, 2001; Schlegel et al., 2003; Thurston et al., 1993). Alcohol, amine, and ammonia losses from freshly excreted manures to the atmosphere occur very rapidly, and effective mitigation needs to be implemented shortly after excretion (Meisinger et al., 2001). Acidification of barn floors and gutters has been suggested as one possible intervention strategy (Ferguson et al., 2001). The selection of acids would need to be done carefully because few are compatible with the presence of animals. Acidification of manure slurry has also been suggested in the literature (Meisinger et al., 2001; Clemens et al., 2002; Lefcourt and Meisinger, 2001).

Sodium Bisulfate

Sodium bisulfate is a dry, granular acid salt that has been used for many years as a pH reducer in a variety of agricultural, industrial, and food applications (Sweeney et al., 1996; Sweeney et al., 2000). It is categorized as a mineral acid by the EPA (Registration Eligibility Decision Mineral Acids, EPA 738-R-029). In general, mineral acids dissociate and release hydrogen ions, thus decreasing the pH. The extent and duration of the pH decrease depends on the amount of neutralizing ions present, the buffering capacity of the medium to which it is applied, and the amount of dilution. The antibacterial properties of SBS have been exploited in its application as a sanitizer (EPA Reg #1913-24-AA) and as a preservative (EPA method #5035) to prevent microbial activity leading to VOC release. These pH-reducing and antimicrobial properties have led to its use for ammonia binding and bacterial reduction in poultry, dairy, and equine manure and bedding materials (Ullman et al., 2004; Sweeney et al., 1996; Harper, 2002). Currently, 30 to 40% of all broiler chickens produced in the USA are raised on SBS-treated litter (PLT litter acidifier; Jones-Hamilton Co., Walbridge, OH). Sodium bisulfate has been established as an effective surface amendment to reduce ammonia and bacterial counts in poultry systems (Pope and Cherry, 2000; Terzich, 1997).

Sodium bisulfate is hygroscopic, and as ambient moisture is adsorbed into the SBS bead, the compound dissolves into its sodium (Na^+), hydrogen (H^+), and sulfate (SO_4^-) constituents. The hydrogen ion reduces the pH of the litter or bedding and protonates the ammonia molecule, converting it to ammonium ($NH_3 + H^+ \rightarrow NH_4^+$). The ammonium is then bound by the sulfate component (Ullman et al., 2004). The newly formed ammonium sulfate does not aerosolize but is retained in the manure in its solid form (similar to ammonium sulfate inorganic fertilizer). Theoretically, 100 kg SBS can bind 14 kg ammonia based on the reaction $2\ NaHSO_4 + 2\ NH_4OH \rightarrow (NH_4)_2SO_4 + Na_2SO_4 + 2\ H_2O$. Sodium and hydrogen ions exert synergistic negative pressure on the bacterial populations within the manure, decreasing total aerobic population counts by 2 to 3 logs (Pope and Cherry, 2000). This may also serve to further decrease urease concentrations in the manure slurry, leading to additional ammonia reductions (Ullman et al., 2004).

Material and Methods

General Design

The study was conducted using experimental flux chambers, a range of potential SBS treatment levels, and freshly excreted dairy cow slurry. The slurry and treatments were placed in open containers and covered by surface isolation flux chambers. The four flux chambers (Odotech Inc., Montreal, Quebec, Canada) were built from acrylic resin with a volume of 64.5 L. Each chamber consisted of a cylindrical enclosure with a spherical top. Teflon tubing (50 cm, 6.35 mm OD) was installed around the inside circumference of the chamber. This tubing was perforated to allow air to circulate throughout the chamber when connected to a compressed air distribution system. An opening on each chamber (fitted with a stainless steel Swagelok connector) was used to sample air for ammonia, amine, and alcohol. Of the remaining two openings on the flux chamber top, the smaller one was used for the thermocouple, and the larger opening to allowed extra air to escape and equalized inside pressure while sweeping air and sampling. A square piece of Teflon sheet was used underneath the flux chambers to minimize absorption of the emitted gases by the counter surface.

Urine and fecal samples were collected directly from mid-lactating Holstein cows that were on a California typical total mixed ration diet. A combination (1:1) of feces and was mixed in a homogenizer to ensure a homogeneous slurry mixture. The slurry mixture was divided (each containing 1 kg) into four circular trays (25.4 × 3 cm) for each of the four SBS treatments. The depth of the slurry in the tray was approximately 2 cm, which simulates the depth of the slurry in freestall barns in the dairy industry. The slurry pH was measured and recorded immediately

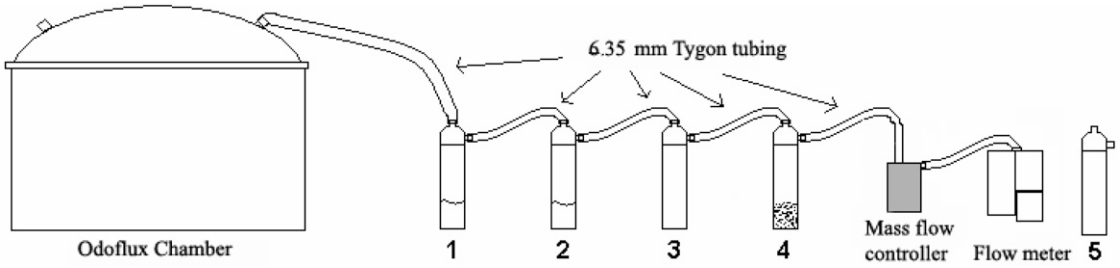

Fig. 1. Amine sampling system.

after mixing using the Accumet Research AR 15 pH meter (Fisher Scientific, Pittsburgh, PA). Appropriate amounts of SBS (0, 6.4, 12.8, and 19.2 g) were added to each tray (i.e., to the slurry) to bring the treatments to an equivalent of 0, 0.125, 0.250, and 0.375 kg m^{-2}. Sodium bisulfate was applied to the surface of the slurry. The pH was measured 15 min after SBS treatments were applied. After the pH was recorded and slurry samples were taken, each tray was placed under its respective flux chamber.

Experimental Design and Statistical Analysis

The experimental design was a 4 × 4 Latin Square with four treatment levels and four manure series; thus, each treatment experienced each flux chamber (n = 4).

The SAS PROC MIXED procedure (SAS Inst. Inc, Cary, NC) was used for statistical analysis. The model included the fixed effects of SBS treatment, time, and SBS treatment by time interaction with the flux chamber as the random factor. For all measures, the predicted difference test in PROC MIXED procedure in SAS was used to separate means when the overall F-value was significant (P < 0.05). All data are presented as least squares means, and variability is expressed as pooled SEM.

Air Sampling

The air inside the flux chamber was continuously replaced with clean air at a rate of 10 L min^{-1}. Sampled amine species were methylamine, dimethylamine, ethylamine, trimethylamine, isoprophylamine, propylamine, and butylamine. Amines were collected with an impinger sampling train (Fig. 1) connected to the sampling port of the flux chamber. The sampling train consisted of two midget bubblers and two midget impingers (Kimble/Kontes Vineland, NJ). The first two impingers (#1 and #2) each contained 15 mL of 0.1N H_2SO_4 solution. The first bubbler captured most of the amines emitted from the sample source. However, if the acid solution were to become saturated due to high amine concentrations, the second bubbler would retain the surplus amines. The third impinger (#3) was empty to trap any overflow of sulfuric acid from the second bubbler. The fourth impinger (#4) was filled with 15 mL tarred silica gel (6–12 mesh). A field blank was used (fifth impinger) and filled with 15 mL of 0.1N sulfuric acid to absorb any background ammonia and amines in the room. Sampling ports between impingers were connected with Tygon tubing (6.35 mm ID; Saint-Gobain Performance Plastics Corp., Akron, OH). The sampling train was assembled in a test tube rack for stability and placed into a plastic container containing sufficient crushed ice to cover the 15-mL line of the impingers. Air was pulled through the sampling train at a rate of 1 L min^{-1}. Each sampling period was 2 h. Each treatment chamber was sampled independently, and all four chambers were measured simultaneously. Amine concentrations were sampled every 24 h for 72 h. After 72 h, each bubbler was rinsed three times with 0.1N H_2SO_4 and brought to volume in a 50-mL Falcon tube. The samples were diluted using USEPA Method 3500B, and the amine concentration was measured by ion-chromatography (ICS-2000; Dionex Corporation, Sunnyvale, CA) using a 4-mm IonPac CS17 cation exchange column and a Cation Self-Regenerating Suppressor (CSRS ULTRA). The minimum detectable level of methylamine was 10 μg N L^{-1}, which is comparable to results reported by Hutchinson et al. (1983) and Schade and Crutzen (1995). The minimum detectable methylamine concentration in air corresponding to 0.12 m^3 sampling volume was approximately 5 ng L^{-1}.

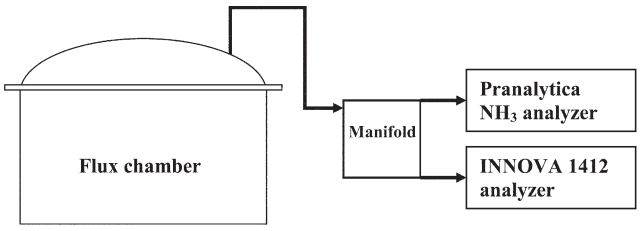

Fig. 2. Ammonia and alcohol sampling system.

Ammonia emissions from four flux chambers and one inlet were sequentially and continuously measured for 20 min each using a NITROLUX ammonia analyzer (Pranalytica, Santa Monica, CA) (Fig. 2). The NITROLUX analyzer is based on near-infrared diode lasers and fiber-amplifier–enhanced photoacoustic spectroscopy that was designed to address the problem of monitoring ambient agricultural ammonia (Webber et al., 2005).

Alcohols measured were ethanol and methanol. The four flux chambers and one inlet were sequentially sampled for these two compounds and analyzed for 20 min each, using a photoacoustic INNOVA model 1412 field gas monitor (Fig. 2). The instrument has been approved as a standard reference method by California Air Resource Board (CARB, MSO 2000-08) and the Environmental Protection Agency (EPA-VS-SCM-28) for measurement of ethanol and chlorinated VOC.

The emission flux rate was calculated using the following equation:

$$E = \frac{\sum_{n} Q \times (C_{out} - C_{in})}{n} \quad [1]$$

where E = gas emission rate from the chamber (mg h^{-1}), C_{out} = mass concentration in the exhaust air (mg m^{-3}), C_{in} = mass concentration in the compressed air (mg m^{-3}), Q = ventilation rate at 20°C and 1 atm (m^3 h^{-1}), and n = total effective measurement numbers.

In this study, reduction potential was used to evaluate the effect of SBS on ammonia and alcohols emissions from slurry. Reduction potential was calculated using the following equation:

$$RP=\frac{EF_C - EF_T}{EF_C}\times 100\% \quad [2]$$

Where EF_C = ammonia or alcohol emission flux from the control (mg h^{-1} m^{-2}), and EF_T = ammonia or alcohol emission flux from the SBS treatment (mg h^{-1} m^{-2}).

Binding efficiency (BE) was used to illustrate the fraction of applied SBS that ties up ammonia. The BE was calculated by:

$$BE=\frac{(EF_C - EF_T)\times T}{W_{SBS}\times BC}\times 100\% \quad [3]$$

where T = time (h); W_{SBS} = weight of SBS used per m^2 (mg SBS m^{-2}); and BC = theoretical binding capacity of SBS to ammonia, which equals 14 mg NH_3 per 100 mg SBS.

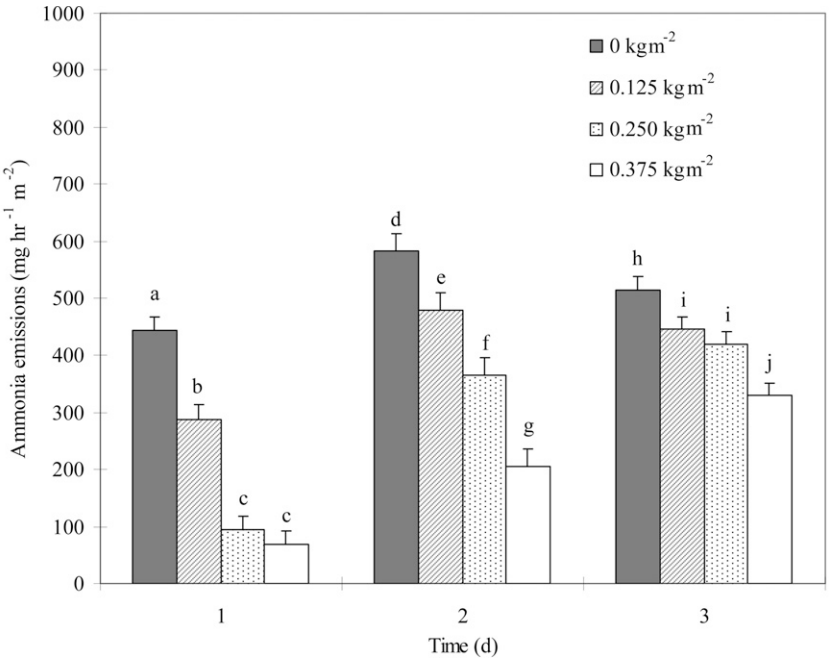

Fig. 3. Least squares means and pooled SEM of ammonia emission flux from three levels of sodium bisulfate–treated vs. untreated control slurry over a 3-d period. Least squares means with different superscripts differ ($P < 0.01$).

Results and Discussion

Surface application of SBS significantly decreased ($P < 0.01$) ammonia emission flux from fresh dairy slurry (Fig. 3) in a dose-response manner. Ammonia emission reduction potentials were 0, 21, 43, and 60% from application levels of 0, 0.125, 0.250, and 0.375 kg SBS m^{-2}, respectively. The binding efficiencies of SBS across treatments were almost identical (average, 44%), which indicated a linear relationship between the amounts of SBS applied per unit area of slurry and the ammonia flux over the entire experimental period. Ammonia emissions from the control (0 SBS) treatment reached a peak between the first and second day, after which the emission flux decreased. This pattern was also found in the lower SBS treatment (0.125 kg m^{-2}) but not in the two higher (0.250 and 0.375 kg m^{-2}) treatments. After 72 h, all four treatments had an ammonia flux of approximately 420 mg h^{-1} m^{-2} ($P > 0.01$). The most effective reduction of ammonia emission occurred during the first day (Fig. 3). The emission reduction potentials were 35, 79, and 84%, respectively, from application levels of 0.125, 0.250, and 0.375 kg m^{-2}. After 24 h, the reduction rate decreased ($P < 0.01$). By day 3, the reduction rates between 0.125 and 0.250 kg m^{-2} treatments were no longer different ($P > 0.05$).

Amine emissions were undetectable in the present study. Previous studies have shown very low amine fluxes from feedyards (Schade and Crutzen, 1995; Hutchinson et al., 1983). Schade and Crutzen (1995) and Hutchinson et al. (1983) conducted work in open feedyards where additional non-waste–related sources (e.g., feed) could have contributed to amine emissions.

Surface application of SBS also decreased ($P < 0.01$) methanol and ethanol emissions from fresh dairy slurry (Fig. 4 and 5). The 3-d average methanol emissions were 223.7, 178.0, 131.6, and 87.0 mg h^{-1} m^{-2} for SBS surface application levels of 0, 0.125, 0.250, and 0.375 kg m^{-2}, respectively, and the corresponding methanol emission reduction potentials were 0, 20, 41, and 61%, respectively (Fig. 4). The 3-d average ethanol emissions were 356.4, 291.2, 232.4, and 150.8 mg h^{-1} m^{-2} for SBS surface application levels of 0, 0.125, 0.250, and 0.375 kg m^{-2}, respectively. The corresponding ethanol reduction potentials were 0, 18, 35, and 58% (Fig. 5).

Like ammonia, methanol and ethanol emission flux was a function of the SBS treatment level and time. Methanol and ethanol emissions were most effectively reduced during the first treatment day (Fig. 4 and 5). For methanol, the initial emission reduction potentials were 40, 70, and 86% from application levels 0.125, 0.250, and 0.375 kg SBS m^{-2}, respectively. For ethanol, they were 39, 63, and 87%. By the third day, however, methanol and ethanol emissions were not well controlled by SBS treatment. Emission flux from the lowest treatment level (12.5 kg 100 m^{-2}) was the same as that from the control ($P > 0.05$).

The change of pH in the slurry after applying SBS may explain the reduction of ammonia, methanol, and ethanol emissions from cow slurry. Within 15 min, SBS decreased the slurry pH from 7.8 to 1.4–3.5 ($P < 0.01$) (Table 1). Based on the ammonia dissociation constant in water at a pH <5, ammonia in the slurry was most likely converted to ammonium ($NH_3 + H^+ \Leftrightarrow NH_4^+$) and combined with SO_4^{2-}. Furthermore, the low pH may have quickly deactivated the enzyme urease and microorgan-

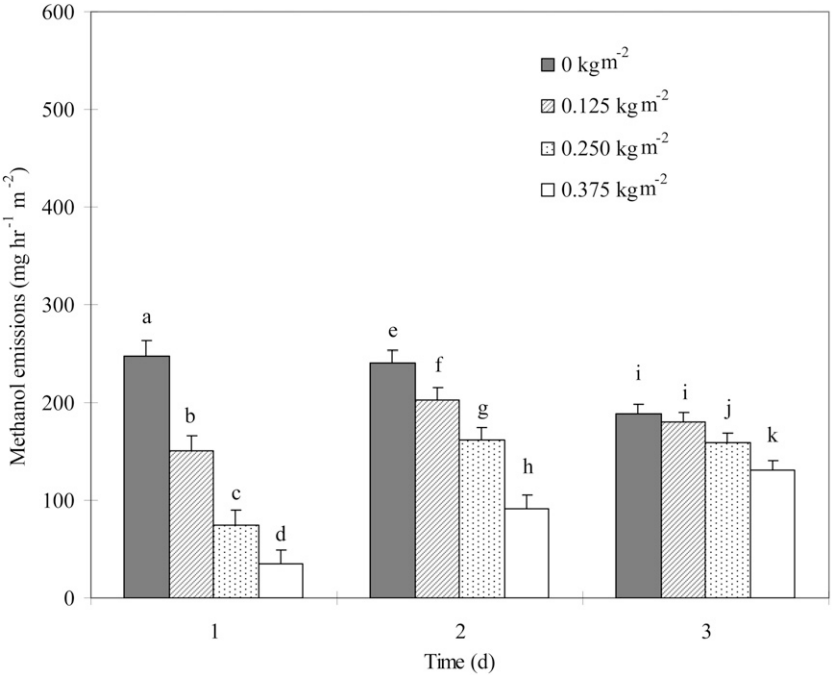

Fig. 4. Least squares means and pooled SEM of methanol emissions from three levels of sodium bisulfate–treated vs. untreated control slurry over a 3-d period. Least squares means with different superscripts differ ($P < 0.01$).

isms in the slurry (Mulrooney et al., 2001; Pearson et al., 2000; Laidler, 1955). Ammonium and alcohols would stay in the liquid phase and not be released into the gas phase. Molloy and Tunney (1983) found that ammonia volatilization of cattle slurry effectively stopped at pH 4.0. Stevens et al. (1989) demonstrated that the treatment of manure with sulfuric acid to a pH range of 4.0 to 5.5 reduced ammonia emissions. In work by Shi et al. (2001), ammonia emissions in a simulated beef cattle feedyard dropped to almost zero at a pH of 4.2. Ammonia reductions of up to 60% were achieved on a dairy by Monteny and Erisman (1998) using a combination of the acidification of slurry in a shallow pit with regular flushing of the slats with the acidified slurry. Acidification of dairy slurry to a pH <5 reduced ammonia volatization by 60% in a study conducted by Meisinger et al. (2001). In swine confinement buildings, Jensen (2002) sprayed a dilute sulfuric acid mixture on the floor and added sulfuric acid to the pooled manure. Ambient ammonia concentrations in the building were reduced from 5.6 to 7.0 mg L^{-1} to 0.7 to 1.4 mg L^{-1} for the duration of the study. Acidification of manure to a pH below 7.0 before land application has also been shown to be effective in reducing ammonia emissions (Sommer and Hutchings, 1995; Meisinger and Jokela, 2000). Miller and Varel (2001) reported the inhibition of VFA production from fresh manure when the pH decreased below 4.5. Additionally, acidification of slurry seems to reduce greenhouse gases. Keeping the pH of manure at approximately 4.5 almost completely eliminated CH_4, CO_2, and N_2O losses (Clemens et al., 2002). Berg et al. (1998) and Clemens and Huschka (2001) reported similar reductions in greenhouse gases when the pH of manure was kept below a pH of 5.0 to 6.0.

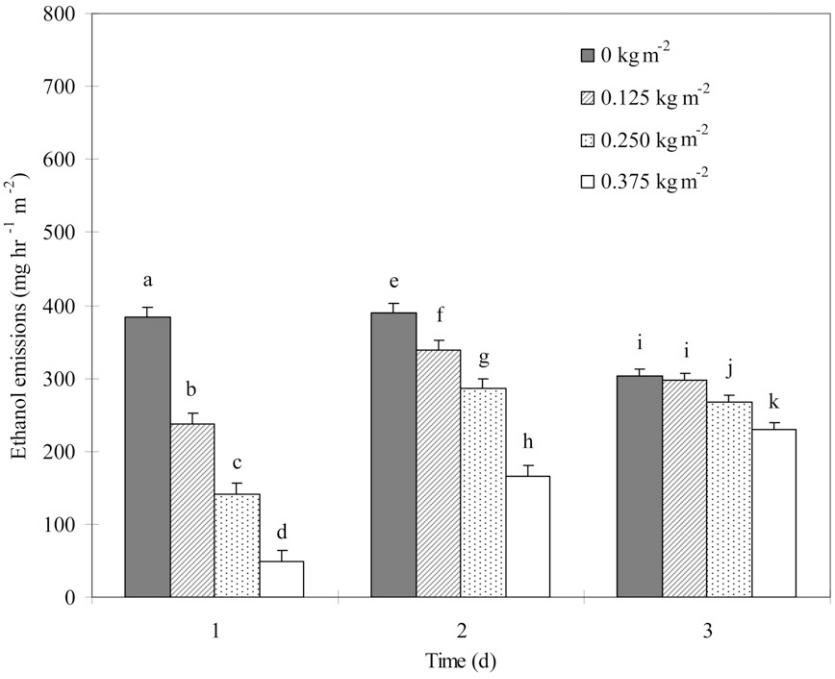

Fig. 5. Least squares means and pooled SEM of ethanol emissions from three levels of sodium bisulfate–treated vs. untreated control slurry over a 3-d period. Least squares means with different superscripts differ ($P < 0.01$).

In the present study, SBS reduced the pH of the slurry mix, which may have led to inactivation of enzymes and microbes associated with emissions of ammonia and VOCs (Mulrooney et al., 2001; Pearson et al., 2000; Laidler, 1955). After approximately 72 h, the pH of the slurry increased to pretreatment levels. The portion of ammonia in the slurry that had been converted to ammonium sulfate prevented a sharp increase in ammonia flux after the pH of the treated samples had returned to neutral conditions. Alcohol emissions were remarkably reduced during treatment with the acidifying agent. In summary, the acidification of the slurry has been shown to be effective to reduce ammonia and alcohol emissions from fresh slurry.

Conclusions

Federal, state, and regional air regulatory agencies view commercial dairies as a major source of regulated air pollutants. Recent dairy emission research conducted in our lab has identified alcohols (ethanol and methanol) as

Table 1. Least squares means and standard errors of initial pH, 15-min pH, and final pH after 72 h from sodium bisulfate–treated and untreated slurry.

Parameter	SBS treatment†			
	0	0.12.5	0.250	0.375
	kg m^{-2}			
Initial pH	7.7 ± 0.1	7.8 ± 0.1	7.8 ± 0.1	7.8 ± 0.1
pH after 15 min	7.7 ± 0.2	3.5 ± 0.2	1.9 ± 0.2	1.4 ± 0.2
pH after 72 h	9.0 ± 0.1	8.7 ± 0.1	8.9 ± 0.1	8.7 ± 0.1

† SBS, sodium bisulfate.

the major volatile organic compound group originating from fresh slurry and fermented feedstuffs. Effective control of alcohols and ammonia emissions will help meet regulatory standards, satisfy public concerns, and improve local and regional air quality. Sodium bisulfate may provide an effective, low-cost management practice for the reduction of alcohol and ammonia emissions from dairy housing conditions. Sodium bisulfate has been shown here to be effective in the mitigation of ammonia and alcohol emissions from fresh slurry.

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