



2950 Niles Road, St. Joseph, MI 49085-9659, USA
269.429.0300 fax 269.429.3852 hq@asabe.org www.asabe.org

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Three-Phase Foam Analysis and the Development of a Lab-Scale Foaming Capacity and Stability Test for Swine Manures

Mark B. Van Weelden, Iowa State University, 3252 NSRIC; Ames, IA 50011

Daniel S. Andersen, Iowa State University, 3165 NSRIC; Ames, IA 50011

Kurt A. Rosentrater, Iowa State University, 3167 NSRIC; Ames, IA 50011

Steven L. Trabue, USDA-ARS, 2101 NSRIC; Ames, IA 50011

Brian J. Kerr, USDA-ARS, 2165 NSRIC; Ames, IA 50011

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Abstract. Foam accumulation on the manure slurry at deep pit swine facilities has been linked to flash fire incidents, making it a serious safety concern for pork producers. In order to investigate this phenomenon, samples of swine manure were collected from over 50 swine production facilities in Iowa with varying levels of foam accumulation over a seven month period. These samples were tested for a number of physical and chemical parameters including temperature, pH, total solids, volatile solids, volatile fatty acid concentration, biochemical methane potential, and methane production rate. After establishing these parameters, a “foaming capacity and stability” test was performed where samples were placed in clear PVC tubes with air diffusers at the bottom to simulate biogas production. The amount of foam produced at a set aeration rate was recorded as a measure of foaming capacity, and foam stability was assessed by measuring the height of foam remaining at certain time intervals after aeration had ceased. The results of this test indicated that samples collected from foaming barns showed a greater capacity to produce and stabilize foam. In addition, statistical analysis indicated that manures with foam produced methane at significantly greater rates than non-foaming manures (0.154 ± 0.010 and 0.052 ± 0.003 L CH₄/L slurry*day respectively, average \pm standard error), and consequently had significantly greater fluxes of methane moving through the manure volume. On the other hand, the biochemical methane production assay suggested that manure from foaming pits had less potential to generate methane (112 ± 9 mL CH₄/g VS) than non-foaming pits (129 ± 9 mL CH₄/g VS), and the VFA analysis showed significantly lower concentrations in foaming pits (4472, 3486, and 1439 μ g/g for the surface level and descending depths of the pit, respectively) as compared to non-foaming pits (9385, 8931, and 6938 μ g/g for the same sample depths). When taken together, these assays suggest enhanced anaerobic digestion efficiency from foaming barns, as well as the possible accumulation of a surfactant at the manure-air interface of foaming deep pits. Overall, this work supports a three-phase system conceptualization of foam production in swine manure deep pits, and that the control of one or more of these phases will be required for mitigation.

Keywords. Swine manure, foaming, deep pit manure storage, anaerobic digestion, methane production

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Introduction

The appearance of foam on the surface of deep pit manure storages is a serious concern for pork producers in the Midwest United States. Among several other environmental concerns associated with deep pit storage systems, biological foam accumulation poses a number of problems that must be addressed from a managerial and safety standpoint. For example, foam accumulation can significantly reduce the amount of storage available in deep pits. As a result, the manure pumping and application cycle is stressed, forcing the producer to apply manure during untimely seasonal windows or seek other means of storage.

The accumulation of biological foam on deep pit storages also has serious implications for the gaseous emissions and overall safety at swine facilities. During the foaming phenomenon, flammable gases (most significantly methane) produced by the anaerobic decomposition of the manure are captured in the foam matrix. Depending on the amount of gas that is trapped in a given barn, dangerous gas concentrations are possible during a sudden breakage of foam. In some cases, foam agitation or breakage along with a spark source has caused flash fires or explosions in swine facilities (Moody et al., 2009).

Examples of foam accumulation in the field as well as foam collected for lab analysis are shown in Figure 1. In general, foam observed in the field is dark-brown, viscous fluid with mid-sized bubbles entrained throughout (Robert et al., 2011). Lab experimentation indicated that there was in fact significant methane concentrations entrained in this biological foam. In addition, monthly observation of facilities monitored in this study indicated that the foam accumulation varied by month, especially as a result of temperature trends and management practices. For example, surface accumulation in the winter months was much more condensed than the accumulation observed in the late fall, and the pumping of deep pits played a large role in the amount of surface accumulation and other characteristics of the pits.

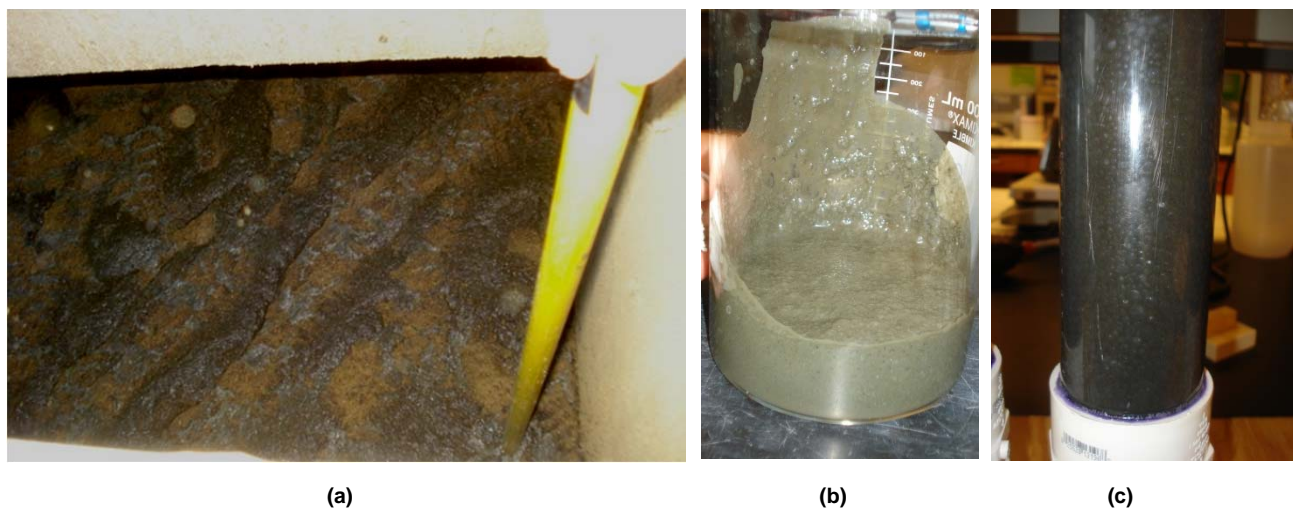


Figure 1. (a) Biological foam accumulation on the surface of a deep pit storage system in Central Iowa, (b) a sample of foam taken from a swine finishing barn in Central Iowa, and (c) foam distribution of a sample after aeration.

Foam Accumulation: A Three-Phase System Approach

The foaming of anaerobic systems is not exclusive to livestock production facilities. The foaming of anaerobic digesters in municipal treatment facilities is a serious concern that has been researched extensively for some time. In these systems, biological foam accumulation has serious implications for process efficiency, and is considered a nuisance for a number of reasons surrounding the safety and aesthetics of digester operation (Ganidi et al., 2009). The research established in this subject area is helpful in developing a framework of understanding for foaming deep pits.

Davenport and Curtis (2002) helped to establish a useful means of characterizing the production of foam as a three-phase system for wastewater. The initiation of foam production occurs as a result of both the gas and liquid phases working together to capture bubbles produced by the system at the interface of the slurry layer and the atmosphere. In anaerobic systems, the gas phase is a result of biogas production due to methanogenic activity. When a significant concentration of surface active agents is present in the slurry, the liquid phase facilitates foam production by lowering the surface tension of the solution (Glaser et al., 2007; Davenport et al., 2008). Finally, solids in the form of hydrophobic substances are thought to stabilize the foam that is produced by preventing liquid drainage back into the slurry layer (Bindal et al., 2002; Horozov, 2008; Heard et al., 2009).

The sustained presence of foam occurs only after all aspects of this three-phase system are optimized for the production and stabilization of foam.

This study will use this conceptual framework to seek to better understand the foaming of deep pit manure storages. To this end, samples collected from swine finisher facilities were analyzed for a number of baseline parameters to evaluate the gas, liquid, and physical phases. The gas phase was investigated by determining the methane production rate (MPR) and biochemical methane potential (BMP) of the samples. The liquid phase was characterized with pH, viscosity, surface tension, and density measurements, along with an analysis of volatile fatty acid concentration. Finally, the total and volatile solids contents were determined to better understand the solid phase. With these baseline parameters established, samples were aerated with a lab-scale apparatus that was developed to determine the capacity of the sample to foam, as well as the ability for the foam to stabilize.

It was hypothesized that samples collected from barns with existing foam layers would exhibit significantly different values for key parameters such as the rate of biogas production, the concentration of volatile fatty acids, and the solids content of samples. In addition, this group predicted that the lab-scale foaming capacity and stability test would successfully model the foaming activity of the deep pit manure storages studied, and reinforce the trends shown by the other parameters measured in this study.

Materials and Methods

Samples of swine manure were obtained from over 50 swine finishing facilities in Central and Southeastern Iowa. At each site, samples were taken from the same pump out location once a month for seven months. Samples were extracted from multiple depths of the deep pit depending on the total depth of manure in the pit at the time of sampling. These depths were designated with letters A through D. The letter “A” corresponded to the foam layer itself or a “crust” on the manure surface, “B” represented the thin liquid layer at the interface of surface accumulation and the depth of manure, and “C” and “D” designated descending depths of the deep pit, with the “C” designation representing manure 61 cm (24 inches) below the B layer and the “D” designation representing manure 122 cm (48 inches) below the B layer. Throughout this paper, these various depths are referred to as “strata”.

The total depth of the pit and the height of surface accumulation were measured on site. In addition, the temperature of the manure at each facility was measured with a digital temperature probe from a sample collected six inches from the bottom of the pit. Finally, pH values were collected for each sample with a pH electrode according to EPA SW-846, Method 9040 within a day of sample collection.

Total Solids and Volatile Solids

The total solids and volatile solids contents of manure samples were tested according to the Standard Methods for the Examination of Water and Wastewater 2540B and 2540E (APHA, 2000). Approximately 30 mL of a manure sample was poured into a pre-weighed porcelain dish after thorough mixing. After obtaining the weight of the full crucible, the sample was dried in a 104°C oven for approximately 24 hours. After drying the sample was weighed again. The percent of total solids was determined by equation 1 below.

$$\% \text{ Total Solids} = \frac{\text{Weight of Dried Sample and Dish} - \text{Weight of Crucible}}{\text{Weight of Wet Sample and Dish} - \text{Weight of Crucible}} \times 100 \quad (1)$$

After obtaining the dried weight of the sample, the crucible with the dried contents was placed in a muffle furnace at 550°C for approximately 8 hours. Once cooled, the final weight of the ash and crucible was obtained, and the volatile solids content was determined by equation 2.

$$\% \text{ Volatile Solids} = \frac{\text{Weight of Dried Sample and Dish} - \text{Weight of Ash and Dish}}{\text{Weight of Wet Sample and Dish} - \text{Weight of Crucible}} \times 100 \quad (2)$$

Volatile Fatty Acid Analysis

The concentration of short chain fatty acids was determined as follows. Approximately 5 g of the sample was inserted into a 15 mL centrifuge vial and centrifuged at 21,000 x g for 23 minutes at 4°C to remove sample debris. Then, approximately 100 µL of concentrated phosphoric acid was added to the supernatant to obtain a pH of 2.0 to 2.5. 1 mL of the acidified sample was added to a 20 mL headspace vial (Agilent Technologies, Wilmington, DE) along with 0.3 g of NaCl and sealed.

Each sample was then placed in a gas chromatography-flame ionization detector (GC-FID) system with multipurpose autosampler (MPS2A, Gerstel Inc., Linthicum, MD) containing a solid phase microextraction (SPME) attachment. This automated SPME method used a Carbowax/Divinylbenzene fiber (Supelco, Inc.,

Bellefonte, PA). The samples were heated for 15 minutes at 70°C and extracted 5 minutes prior to injection in the GC-FID. Samples were then analyzed on an Agilent 7890A GC-FID system that was equipped with a HP-FFAP column (30 m × 0.25 mm × 0.25 µm; Agilent Technologies).

The GC parameters were set as follows: splitless mode; inlet temperature, 230°C; inlet pressure, 24.56 psi; septum purge flow, 30 mL/min; constant column flow 1 mL/min (helium); and detector temperature, 300°C. The GC oven temperature program was initial temperature, 100°C, 2 min hold; ramp of 10°C/min to the final temperature of 240°C, hold for 2 min. All calibration standards were based on external calibration.

Biochemical Methane Potential Assay

The biochemical methane potential (BMP) of a sample defines the anaerobic biodegradability of a given material (Owen et al., 1979). Specifically, the BMP test yields the total volume of methane able to be produced over a long-term digestion period and the potential efficiency of anaerobic digestion a particular sample could achieve. Typically the results are normalized to methane produced per gram of volatile solid added.

The procedure in assessing the BMP of the swine manure samples collected for this study was to add 10 to 15 grams of sample to a 250 mL serum bottle (Wheaton Science Products No.:223950), with the exact mass being recorded. This mass of sample was selected based on an estimated 300 mL of CH₄ produced per gram of volatile solids added as suggested by Vedreene et al. (2008), Hashimoto (1984), and Burton and Turner (2003) who suggested a range of 244 to 480 L CH₄ per kg volatile solids. Next, 50 mL of inoculum was added from an active anaerobic digester maintained in the Agricultural Waste Management Laboratory (AWML) at Iowa State University. This volume of inoculum was added to approximately achieve a 2:1 mass ratio of volatile solids from the manure to inoculum, with the actually ratio varying due to the exact volatile solids content of the manure. Finally, the solution was diluted to approximately 150 mL and sealed with a sleeve stopper septa (Sigma-Aldrich Z564729).

Once the sample was set up, it was incubated at 35°C while being constantly agitated. The samples were regularly checked for biogas production with a gas-tight syringe (Micro-Mate interchangeable hypodermic Syringe 50cc Lock Tip, Popper & Sons, Inc. New Hyde Park, New York). When the syringe was filled with sampled biogas, it was injected into an infrared methane analyzer (NDIR-CH₄ Gasanalyzer University Kiel, Germany) to obtain the percent of methane present in the sample.

When the sample ceased making biogas (typically within 45-60 days), one final sample of biogas was extracted from the bottle to determine the final methane content of the headspace. The cumulative volume of methane produced from each sample was standardized per mass of volatile solids added to the bottle to obtain the BMP.

Methane Production Rate Assay

The goal of the methane production rate assay is to provide a short term biogas production measurement with a relatively simple procedure. While the methane production rate (MPR) test is similar to the BMP assay, it is unique in a number of ways. First, the test is conducted over a much shorter incubation time (approximately 3 to 7 days compared to over 40 days for the BMP assay) to ensure that the sample does not approach substrate limiting conditions. Also, the manure sample used for the MPR assay is not inoculated or diluted; rather, the ability of the endogenous bacteria to produce biogas is evaluated by adding a single volume of the sample. Finally, the sample was incubated at room temperature rather than at 35°C, and the sample was kept stationary rather than agitated. Keeping the sample stationary allowed the observer to record the amount of surface accumulation, foam or otherwise, that developed on the sample.

The procedure for the MPR test involved adding approximately 100 mL of well-mixed sample to a 250 mL serum bottle similar to that used for the BMP assay. Upon the sealing of the sample with a sleeve stopper septa, the exact time was recorded along with the mass of sample added to the bottle. Next, the sample was incubated at room temperature (approximately 23°C). An incubation period of approximately three days was selected based on preliminary trials by Baptista et al. (2013). Once the three day incubation period was over, the sample was checked for biogas production with the gas-tight syringe and analyzed for methane content using the NDIR-CH₄ Gasanalyzer. During the analysis of the biogas produced, the accumulation of foam and/or solids on the surface of the sample was observed and recorded. Figure 2 shows a set of samples after three days of incubation.



Figure 2. A set of samples after three days of incubation for the methane production rate assay.

The rate of biogas production and the rate of methane production were calculated using equations 3 and 4. In order to correct for the additional volume of methane remaining in the headspace, the volume of the headspace of the 250 mL bottles was necessary. The total volume of each bottle was determined to be approximately 283.3 mL using a water displacement method. Then, the volume of manure added was subtracted to obtain the volume of headspace. This headspace correction is seen in equation 4, as the final percent of methane is multiplied by both the volume of biogas produced as well as the headspace volume.

$$\text{BPR} \left(\frac{\text{L}}{\text{L} \cdot \text{day}} \right) = \frac{\text{Biogas Produced (mL)} \times \rho_{\text{manure}} \left(\frac{\text{g}}{\text{mL}} \right)}{\text{Mass of sample (g)} \times \text{incubation period (minutes)}} \times \frac{1440 \text{ minutes}}{\text{day}} \quad (3)$$

$$\text{MPR} \left(\frac{\text{L}}{\text{L} \cdot \text{day}} \right) = \frac{\left(\text{Methane \%} \times \frac{1}{100} \right) (\text{Biogas Produced (mL)} + V_{\text{headspace}}) \times \rho_{\text{manure}} \left(\frac{\text{g}}{\text{mL}} \right)}{\text{Mass of sample (g)} \times \text{incubation period (minutes)}} \times \frac{1440 \text{ minutes}}{\text{day}} \quad (4)$$

Temperature Correction of the MPR and Gas Flux Estimates

The methane production rate assay is performed at room temperature; however, as this group was most interested in the methane flux from the manure pit, a method to adjust the measured MPR to that expected at the in situ temperature of the manure pit was required. Batista et al. (2013) researched the effects of temperature and agitation on the methane production rate of manure samples collected from the same group of facilities used in this study. This group used the Arrhenius equation to model the impact of temperature on methane production rate. This equation was used to adjust the methane production rate values measured in the AWML at 23°C to the in situ pit temperature recorded during field sample collection. The temperature adjusted values were averaged across layers of manure collected during sampling (with the exception of the foam or crust layer) to attain an average MPR for each facility each month. In order to calculate the methane flux, the average, temperature-corrected MPR was multiplied by the recorded depth of manure for the pit. The flux was converted appropriately to give units of liters of methane per area in m² per day.

Foaming Capacity and Stability Testing

As mentioned previously, the methods highlighted above sought to establish baseline parameters in all three phases involved in a foaming anaerobic system. The purpose of the lab-scale foaming capacity and stability test, then, was to establish empirical parameters related to the inherent foaming characteristics of manure samples. These parameters were able to be compared to the field data regarding the foaming status of facilities during collection as well as the other laboratory tests.

The foaming capacity and stability apparatus used in this study, as well as the parameters used to evaluate the foaming characteristics of swine manure, were adapted from a number of other studies, including Ross et al. (1992), Bindal et al. (2002), Bamforth (2004), and Hutzler (2011). The apparatus developed for the lab-scale test is shown in figure 3. Air was passed through an in-line gas regulator (Restek Model 21666) directly into a 2-inch diameter clear PVC column. The flow rate of air through the column was measured and controlled with a variable area flow meter (Dwyer RMA-SSV). For the purposes of this experiment, it was determined that a flow rate of 200 cubic centimeters per minute (0.0033 L/s) was appropriate based on preliminary trials. In order to conduct the foaming capacity experiment, a sample volume of approximately 300 mL was poured into the column and the initial level was recorded based on measuring tape placed on the columns. The sample was then aerated through a cylindrical air stone at 0.0033 L/s until a steady state height was reached or the foam

layer reached the maximum height of the column. The time of aeration was recorded along with the height of foam produced and the level of the foam-liquid interface. A foaming capacity index was calculated as the height of foam produced divided by the initial manure level and multiplied by a factor of 100.

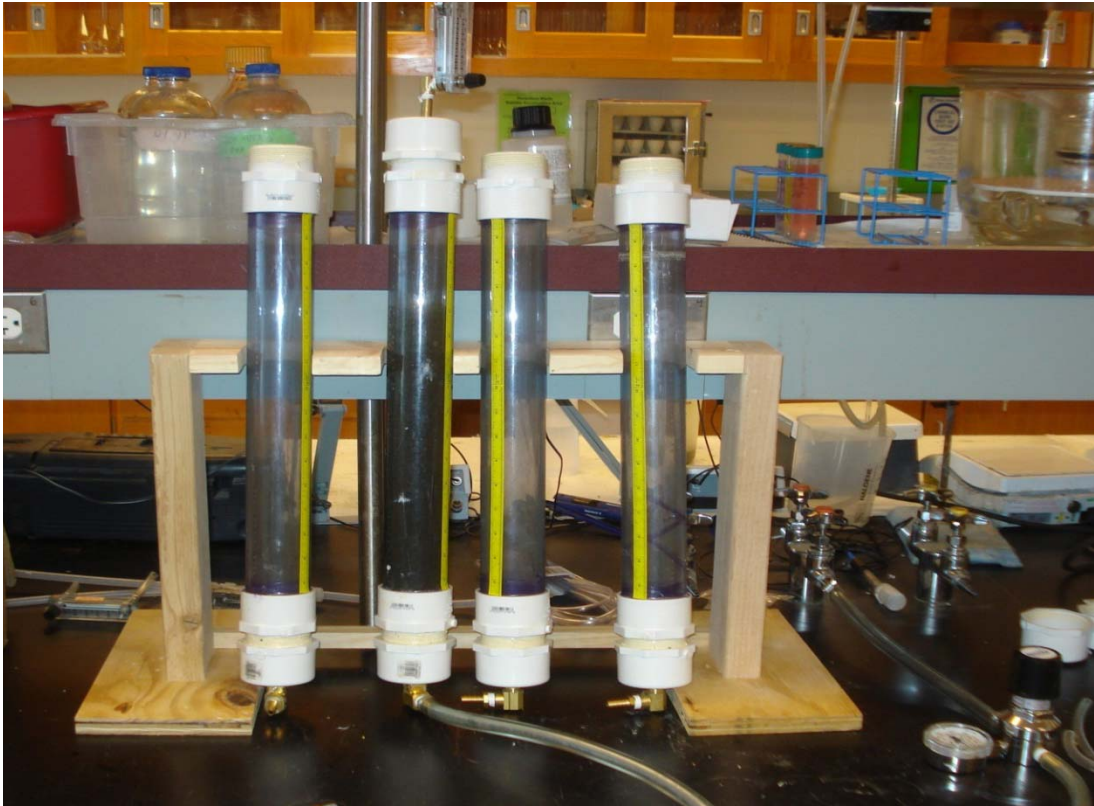


Figure 3. Apparatus for foaming capacity and stability testing at a lab scale.

The foam stability measurement occurred immediately after the foaming capacity was determined. Once aeration ceased, the final height of foam became the initial level recorded at time zero. Once this level was established, the descending height of the foam was recorded at expanding time intervals. Simultaneously, the ascending level of the foam-liquid interface was recorded at the same time intervals. The descending height of foam was normalized to percent of initial foam height and plotted as a function of time (figure 4). A first-order exponential decay model fit the data well in most cases. The half-life of the foam was determined with equation 5 in terms of the coefficient obtained from the first order graph as a measure of the foam stability.

$$t_{1/2}(\text{minutes}) = \frac{\ln(2)}{\text{decay coefficient } k} \quad (5)$$

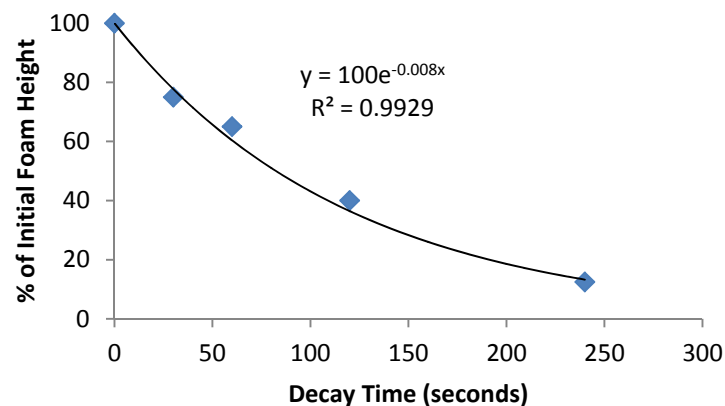


Figure 4. Example of foam decay for a swine manure sample taken from a swine finishing barn in Central Iowa.

Statistical Analysis

Statistical analysis was performed using JMP Pro 10 (JMP Pro, Version 10. SAS Institute Inc., Cary, NC, 1989-2012). Fixed factors were established according to data collected on site, including the surface condition (foaming, non-foaming, or foaming crust), stratum the manure was collected from (B, C, or D), and the month during which samples were collected. Note that a designation of “foaming crust” was added in the month of December to describe surface accumulation composed of crust-like material with some bubbles entrained throughout or trapped beneath.

Results and Discussion

Pit Temperature pH Trends

Pit temperatures of each facility were monitored throughout the seven months of sampling conducted thus far. The temperature was measured from a sample collected six inches from the bottom of the pit. Temperature trends are shown in figure 5 based on the month and type of surface accumulation on the pit. A statistical analysis indicated that both month ($p < 0.0001$), surface conditions ($p < 0.0001$), as well as the month*surface condition interaction ($p = 0.0078$) were all significant. In this case the significant interaction indicates that pits with different surface conditions tended to warm and cool at slightly different rates. For example, non-foaming pits cooled more quickly than foaming pits from October to November, while pits with foaming crusts cooled more quickly than foaming pits from December to January. These differences could have been caused by differences in the timing of when pits were pumped and in the classification of pits as foaming, non-foaming, or foaming crust. More importantly, there was a strong seasonal pattern, with pits warmest in October (20.1 and 19.2°C for foaming and non-foaming pits, respectively) and coolest in February (12.9, 12.1, and 10.9°C for foaming, foaming crust, and non-foaming barns, respectively). This analysis indicates that barns with surface accumulation of foam or foamy crust have significantly higher temperatures than those without accumulation.

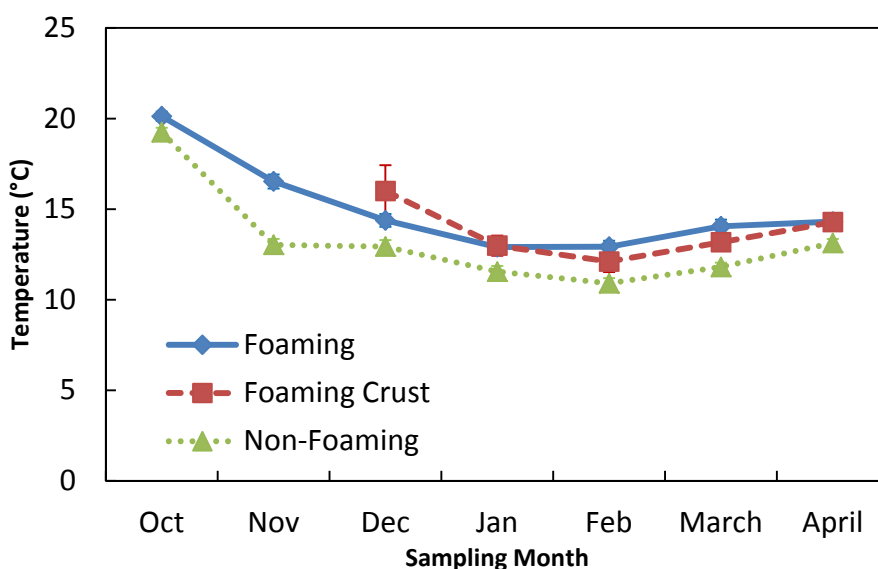


Figure 5. Manure pit temperatures taken from the bottom of the pit for foaming, foaming crust, and non-foaming barns by month. Error bars represent the standard error of the mean for the month*surface interaction.

An analysis of variance for pH was conducted by considering barn surface status (foaming, non-foaming, foaming crust), month, and stratum and the interaction of status*stratum as fixed factors. The ANOVA indicated that status and month were significant ($p < 0.0001$ for both) while stratum was not ($p = 0.7276$). The interaction between status and stratum was significant ($p = 0.0306$). In general, the results (figure 6) indicated that the pH of foaming barns was more basic (pH = 7.62 on average) than for barns with other statuses (foaming crust pH = 7.41 and non-foaming pH = 7.42). The significance of the status*strata interaction was entirely due to pH differences between strata in foaming barns, specifically the A strata, while no differences by strata existed for non-foaming barns. In foaming barns, manure near the surface tended to be more basic (7.74 on average) when compared to manure at lower depths (7.63, 7.59, and 7.54 for layers B, C, and D respectively). No statistical differences by strata were found for other barn statuses. Monthly differences were primarily due to more basic pH conditions in the fall months (October and November) as compared to winter and spring months.

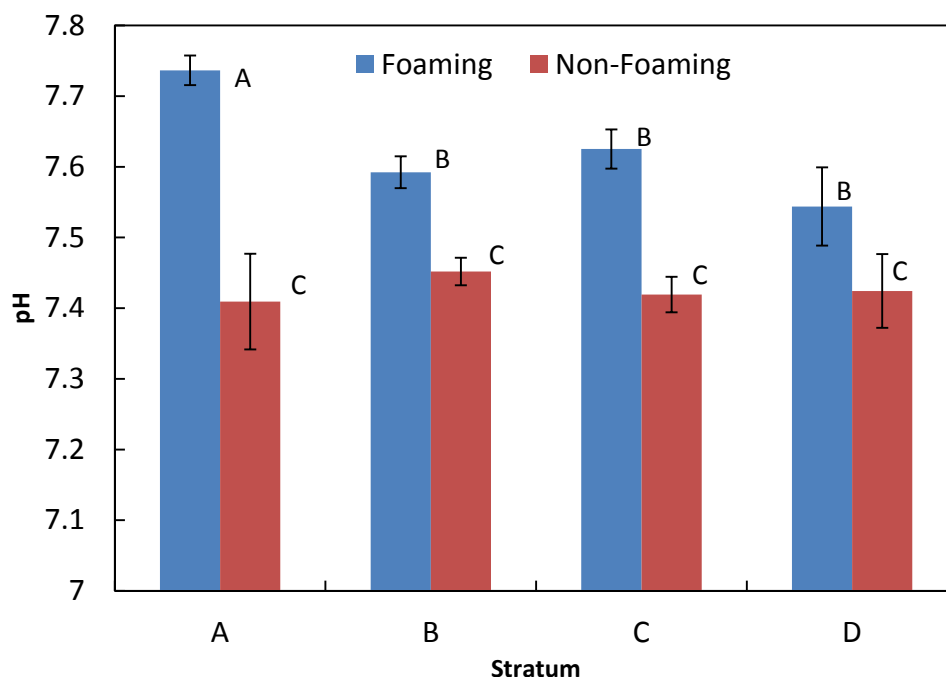


Figure 6. Manure pH by average over all month sorted by stratum and surface condition. Error barns represent one standard error of the mean. Letters are used to denote significant differences with respect to barn surface status and strata.

Total Solids and Volatile Solids

Total solids and volatile solids contents were strongly correlated to each other as shown in figure 7. A regression of total solids and volatile solids indicated that total solids concentration explained 98% of the variation in volatile solids content. The regression line indicated that there was a baseline total solids content of 0.98%, and that for every additional unit increase in total solids concentration, volatile solids concentrations would increase by 0.88 units. This relationship is relatively robust as it is generated from solids contents ranging from 1.3 to 22.5%. A relatively consistent fraction of solids were volatile ($74.3\% \pm 6.7\%$, average \pm standard deviation). However, a statistical model with sample month, surface condition, and strata as factors did not fit well with either total solids or volatile solids. In this way, the solids content of a sample was not significantly different based on the surface condition of the facility from which it was collected. Samples from the B stratum had significantly less solids content than those from the C and D strata, however, no difference in solids content by strata were seen between foaming and non-foaming barns

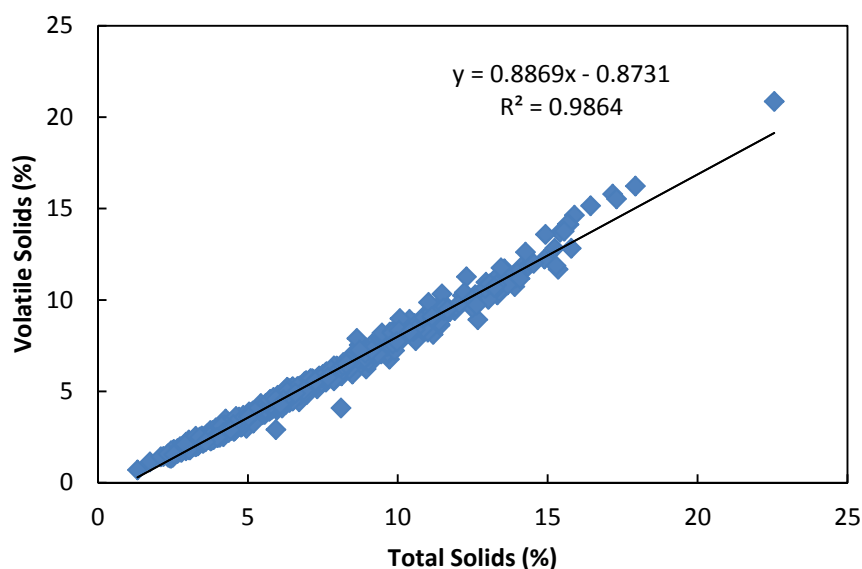


Figure 7. Volatile Solids vs. Total Solids content for each sample over 7 month trial period.

Volatile Fatty Acid Analysis

The average volatile fatty acid (VFA) concentration of selected samples from the months of October, November, and December are shown in figure 8. The first figure shows the monthly averages from barns with reported foam layers and those without (which includes samples with crust-like accumulation). Figure 7(b) again groups samples by surface condition, showing the averages by the strata that the samples were collected from. Both figures show the standard error of the mean.

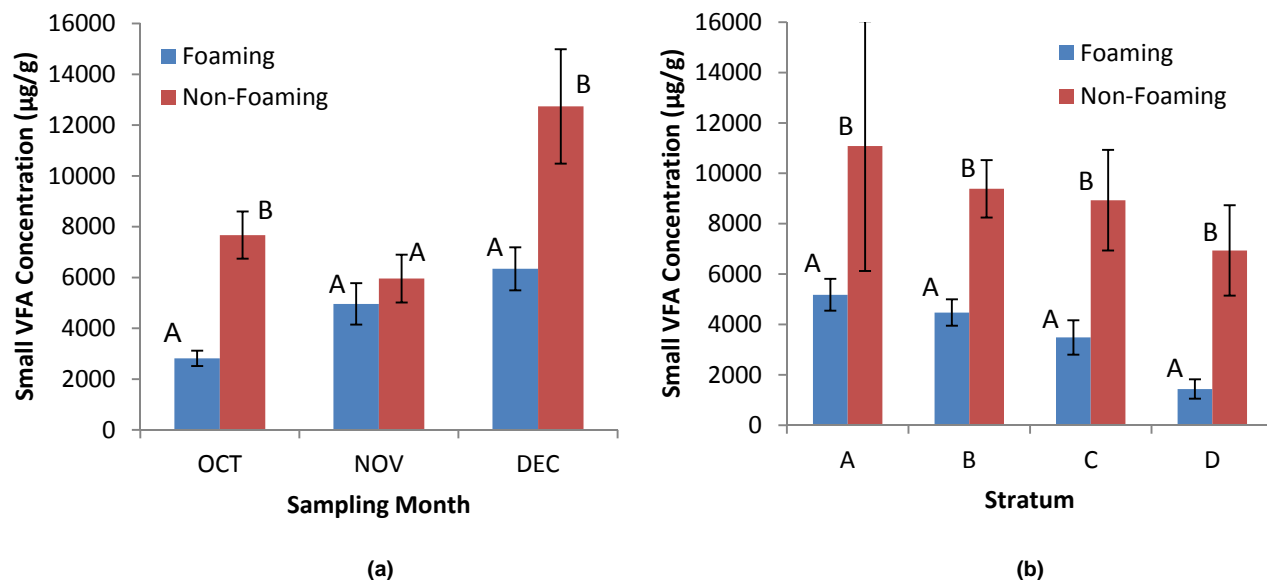


Figure 8. (a) Average total VFA concentrations of foaming and non-foaming samples by month and (b) by layer from which the sample was collected with standard error of mean shown. Letters show statistical differences between foaming and non-foaming samples.

In both graphs, the total VFA concentration in non-foaming barns was greater than that in foaming barns. This difference was most clear in the months of October and December. In addition, the foaming barns showed an increasing trend in VFA concentration, while the non-foaming barns fluctuated unpredictably. In terms of concentration per layer of sample collection, the average VFA concentrations of both foaming and non-foaming barns was the greatest at the surface layer of manure, and gradually decreased as samples came from greater depths of the pit.

The statistical model indicated that the impact of month ($p < 0.0001$) and surface ($p < 0.0001$) was significant, but that strata ($p = 0.5569$) was not. The overall results indicated that foaming barns had significantly lower VFA concentrations (4356 µg/g) than non-foaming barns (9039 µg/g).

Biochemical Methane Potential Assay

The BMP assay provided an estimate of the potential methane production a material could generate under ideal digestion conditions. Previous research by Moody et al. (2011) suggests that swine manure slurry taken from a deep pit should have an approximate methane production potential of 132 mL CH₄/g VS. On average this group found a methane production potential of 121 ± 86 mL CH₄/g VS across all samples collected. A statistical analysis was performed to evaluate the impact of surface status, sample month, and strata. Results from the analysis indicated that only month was significant ($p = 0.0029$); however, the other factors status ($p = 0.0591$) and strata ($p = 0.0678$) were nearly significant. Graphs comparing foaming and nonfoaming barns and the impact of strata are shown in figures 9a and 9b, respectively. The near significant difference in remaining biochemical methane production potential between foaming and non-foaming barns could indicate that foaming barns are operating as more effective anaerobic digesters than non-foaming barns as more of the potential for methane production has already been consumed. Similar tests conducted by this group with manure sampled directly from feeding trials indicated significantly higher values for BMP, supporting this hypothesis.

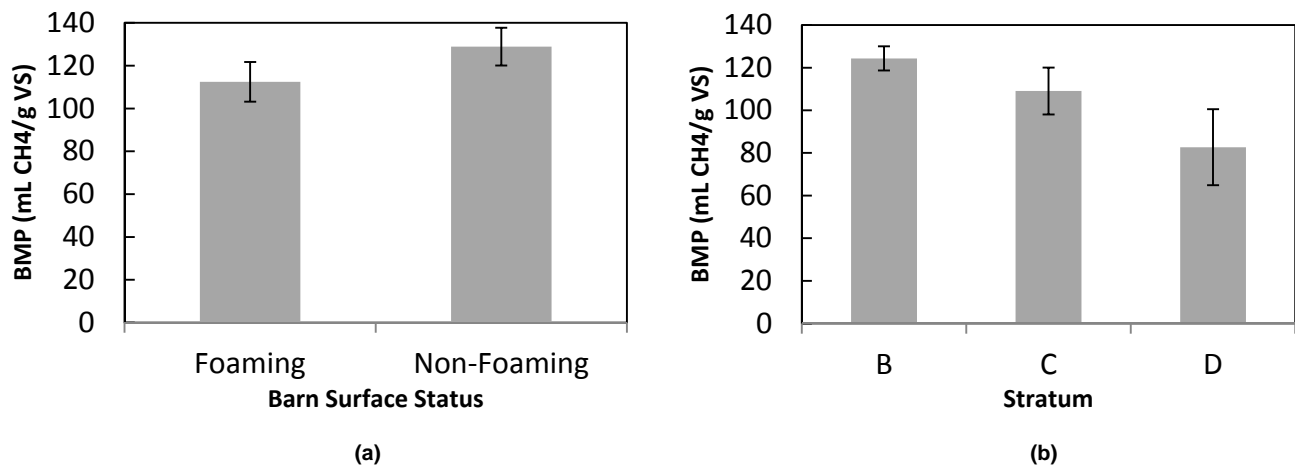


Figure 9. (a) Average biochemical methane potential of foaming and non-foaming samples and (b) by layer from which the sample was collected. Error bars represent the standard error of the mean.

Methane Production Rate

Results for the methane production rate test again showed contrasts between barns that exhibited foaming characteristics during sampling and those with no foam accumulation. Trends for foaming and non-foaming barns by month and stratum are shown in figure 10; error bars represent one standard deviation.

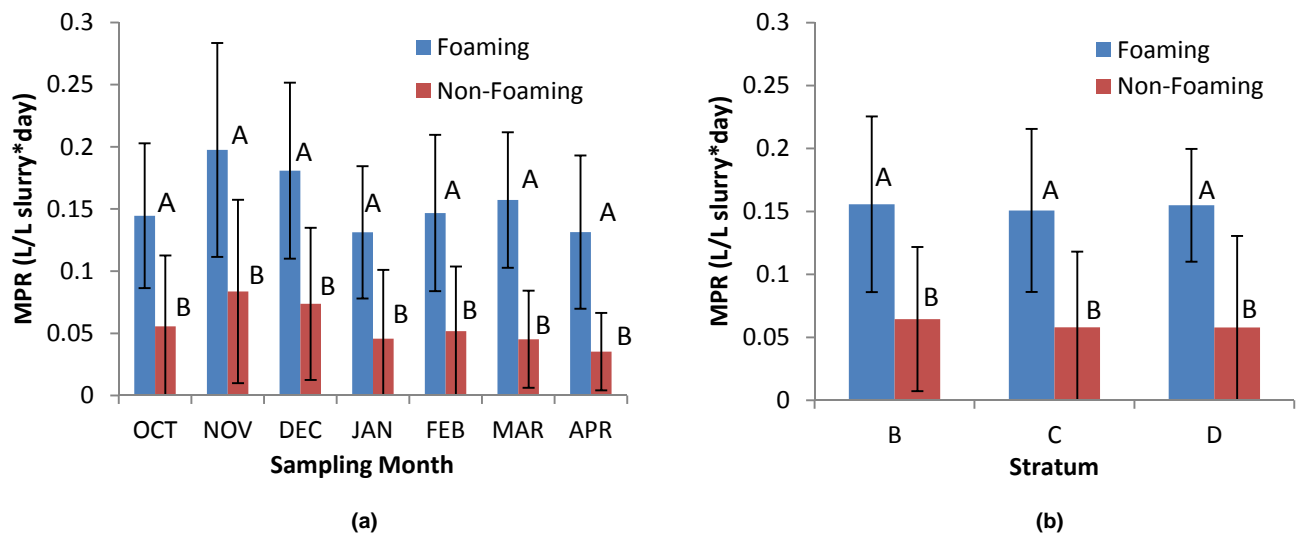


Figure 10. (a) Average methane production rate of foaming and non-foaming samples by month and (b) by layer from which the sample was collected with one standard deviation shown. Letters show statistical differences between foaming and non-foaming samples.

Figure 10 reflects a significant difference between foaming and non-foaming barns with regard to methane production rates during each month. The statistical model also showed that the samples collected from facilities with foamy crust showed a significantly different methane production rate compared to other surface types (Least Square Mean values equal to 0.147, 0.120, and 0.048 L/L*day for foaming, foaming crust, and non-foaming facilities, respectively). However, differences between month (with the exception of November and December) and stratum from which the samples were collected were not significant prior to adjusting the bench-top MPR test for temperature.

Methane Flux

The conversion from the MPR values reported during bench top experiments to values adjusted for temperature made a significant impact on the monthly trend for MPR. The average methane flux of foaming and non-foaming barns is shown in figure 11. An additional line representing the new designation of "foamy crust" is also represented starting in the sample month of January. The methane flux values from each facility

were derived from a temperature-corrected term averaged across sampled strata, which yielded a more intuitive line reflecting the temperature effects of the sample month compared to that in figure 10. At the same time, the curves in figure 11 continue to reflect the enhanced rate at which samples from foaming barns produced methane. Again results indicated a significantly greater methane flux through foaming barns than non-foaming barns. Overall, there was not a significant difference between foaming barns and barns with a foamy crust.

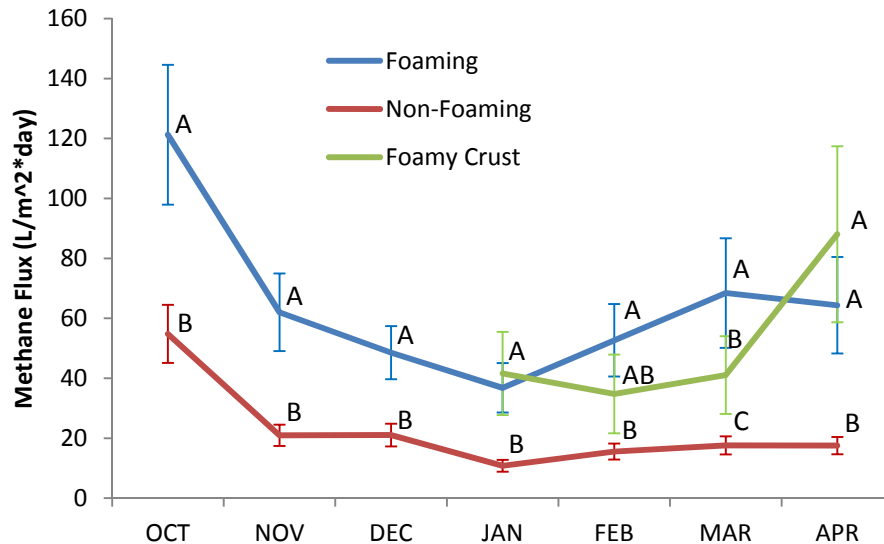


Figure 11. The average methane flux for foaming and non-foaming facilities, as well as the flux from barns with a “foamy crust” from the months of January to April. Error bars show the standard error of the mean. Letters show statistical differences between surface statuses within each month.

Foaming Capacity and Stability

For the most part, the capacity for samples to foam follows trends in previous examples, with samples from foaming barns showing a significantly greater foaming capacity than those from non-foaming barns. The average foaming indices for each sample from foaming and non-foaming facilities are shown in figure 12 by month and by sample stratum. Interestingly, the foaming barns showed an increasing capacity to foam by month, which was the opposite trend of the level of foam accumulation on barns in the field. However, this group hypothesizes that this may have been due to lower gas production levels in the field, as lower gas production rates wouldn’t provide the required gas flux to keep the foam bubbles enriched. Moreover, the lower gas flux may have allowed any biologically generated surfactant to stay more distributed in the manure. The non-foaming barns, at the same time, showed a slightly downward trend. With respect to strata, samples collected closer to surface of the slurry layer showed a significantly higher capacity to foam than that of the manure collected from the bottom of foaming barns. Meanwhile, non-foaming barns showed similar trends in foaming capacity per stratum. This could indicate that the greater flux of gas through foaming pits is helping to lift some surfactant to the surface of the manure.

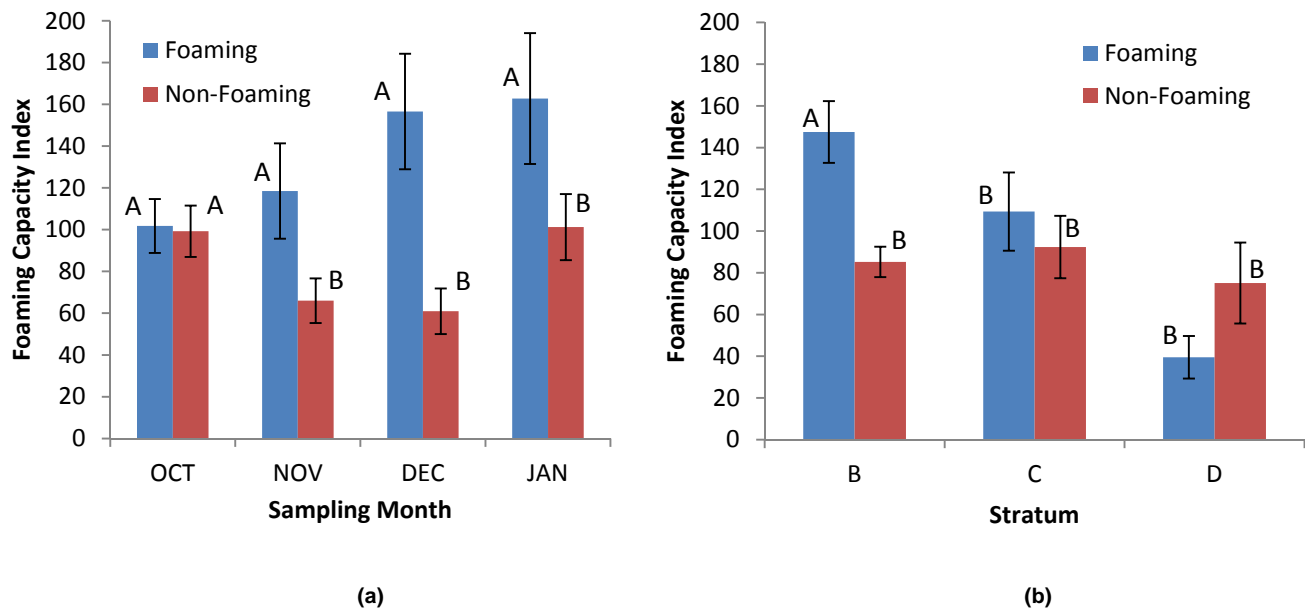


Figure 12. (a) Average foaming capacity index of foaming and non-foaming samples by month and (b) by layer from which the sample was collected with standard error of the mean shown. Letters show statistical differences between foaming and non-foaming samples.

The corresponding graphs representing the half-life of samples after being aerated are shown in figure 13.

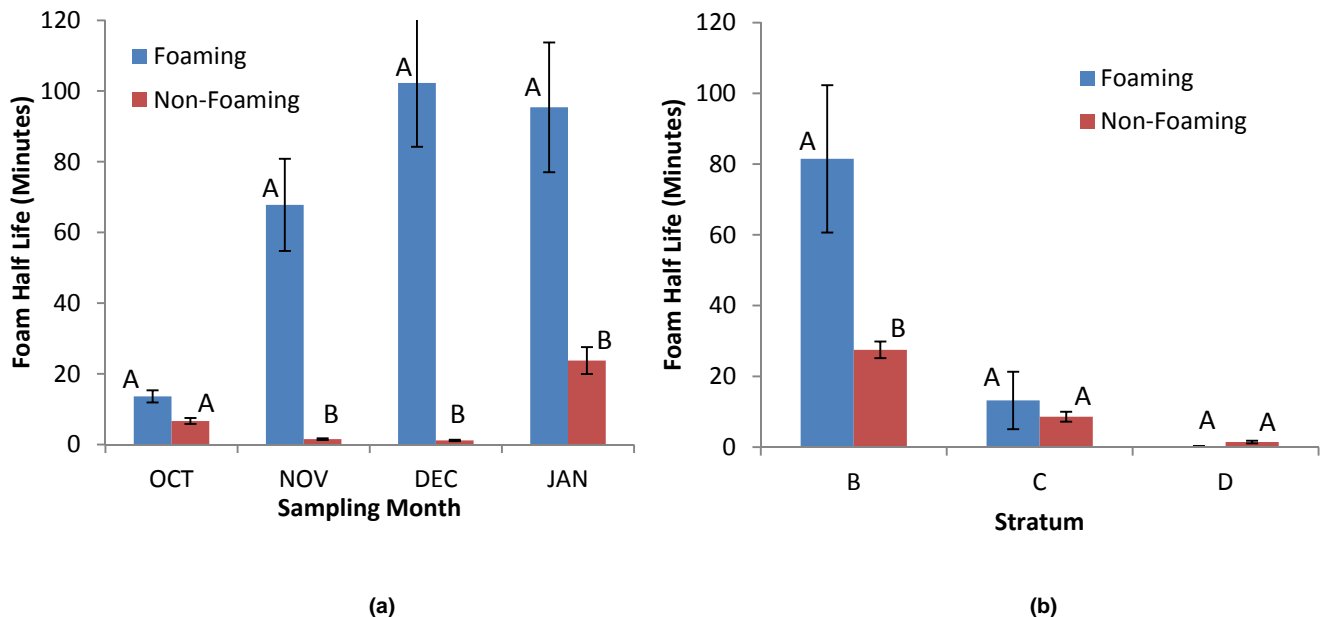


Figure 13. (a) Average half-life of foaming and non-foaming samples by month and (b) by layer from which the sample was collected with standard error of the mean shown. Letters show statistical differences between foaming and non-foaming samples.

The trends in figure 12 are similar to those in figure 13, with increasing foam stability for samples from foaming facilities according to month, and decreasing foam stability as the depth of sample collection is increased. Also, the half-life of samples from foaming barns was significantly longer than those from the other surface groupings when the data was analyzed as a whole.

Conclusion

There are several key observations that can be made with this study's data up to this point. First, the testing of the gas phase of foaming systems has proven a significantly enhanced rate of methane production of barns with foam accumulation in comparison to barns with no foam or crust. At the same time, the biochemical methane potential assay has indicated that samples from foaming barns have less potential to generate additional methane than those from non-foaming barns on a per gram of VS basis. Also, it was shown to be helpful to correct MPR data for the in situ pit temperature to formulate an average methane flux for each facility per month. The resulting monthly flux curve showed an intuitive monthly trend with respect to microbial activity in pits over the winter months as well as a significantly greater flux curve for foaming barns. As sampling continues through the spring and into the summer, MPR results will continue to be monitored closely to investigate trends as the temperature of deep pits increases.

It was initially hypothesized that foaming barns would show a relative accumulation of volatile fatty acids with respect to non-foaming barns. However, the results indicated the opposite, which could have implications for the microbial utilization of VFAs in the methanogenesis process.

The lab scale test developed for this study showed interesting results in comparison to the three-phase data accumulated for the samples. First, the test reflected the difference in behavior of samples collected from facilities with different types of surface accumulation, or no accumulation at all. However, samples collected from foaming facilities during months where foam in the field was more frequently depressed or non-existent (i.e. the cold winter months such as January and December) showed a greater capacity to foam up and stabilize. This trend may indicate the presence of a surfactant and/or stabilizing agent that is suspended in the foam layer in the field. Once the foam is broken down, this agent may exist in a great concentration at the foam-slurry interface, explaining the trends shown in the discussion. Other data showing a greater foaming capacity and stability for foaming facility samples collected closer to the surface may support this theory of an accumulation of a surface active agent near the surface of foaming facilities.

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