A procedure and its application in evaluating pit additives for odor control

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Zhu, J., Bundy, D.S., Li, X. and Rashid, N. 1997. **A procedure and its application in evaluating pit additives for odor control**. Can. Agric. Eng. **39**:207-214. A procedure for testing swine manure pit additive products in terms of the effectiveness of reducing malodor and volatile substances from a manure storage area is presented. This procedure combines many features of the past methods. It includes considerations in many respects to ensure that the test results correctly reflect the real performance of the pit additive products tested. This is necessary for providing reliable information to pork producers in selecting appropriate pit additive products for odor control. Five commercial pit additive products were tested using this procedure and the data for both odor threshold and liquid chemical analysis were obtained. Based on this study, all five products significantly reduced odor threshold levels as compared with the control samples. Four of the five products reduced odor threshold by 83% to 87% and the other reduced by 58%. Liquid chemical analysis showed that three of the five products accomplished significant reductions in volatile fatty acids. The reduction of aerial ammonia and hydrogen sulfide emissions was not determined.

Une méthode fut vérifiée pour évaluer l'effet d'additifs sur le niveau d'odeur et les substances volatiles du lisiers de porcs à l'entreposage. Cette méthode utilise plusieurs techniques développées plus tôt mais considère plusieurs aspects directement reliés à la performance réelle des additifs. Ces aspects doivent être inclus dans la méthode dans le but d'informer les éleveurs de porcs afin de leur permettre de sélectionner un additif pour le contrôle des odeurs des lisiers de leur troupeau. Cinq additifs commerciaux à lisiers furent testés avec la méthode proposée en comparant leur effet sur le contrôle des odeurs et la composition chimique de lisier de porc. Cette étude a démontré que les 5 additifs peuvent réduire de façon significative le taux d'odeur dégagé par les lisiers de porcs à l'entreposage. Quatre des additifs ont réduit le point de détection de 83% à 87% alors que le cinquième réduisait celui-ci de 58%. L'analyse des lisiers a démontré que 3 des 5 additifs pouvaient réduire significativement le taux d'acides gras volatiles des lisiers. Aucune réduction de la volatilisation du NH_3 et du H_2S n'a pu être détecté.

INTRODUCTION

Of the available odor control techniques, adding amendments into swine manure is still considered a feasible, inexpensive way to reduce odor release. Many pork producers are considering the use of commercial manure additives in their efforts to abate or control odor (Williams and Schiffman 1996). Numerous companies are manufacturing chemical and/or biological products for the purpose of odor control. Pork producers lack criteria to evaluate the effectiveness of these products. Due to the complexity of odor generating components in swine manure, it is likely that those products may or may not be effective for reducing odor intensity under diverse environment and management conditions. Therefore, corresponding procedures aimed at evaluating pit additive products have been developed to provide the swine industry with performance information so the claims made by the manufacturers can be verified (Williams and Schiffman 1996; Riskowski et al. 1991; Warburton et al. 1980; Sobel et al. 1972).

Inevitably, different procedures used to evaluate commercial products may yield different results in terms of the effectiveness of odor reduction. To date there is no consensus of how pit additive products should be tested. It is thus imperative to develop and standardize a test procedure which would be accurate, easy to use, adaptable, and commonly recognized.

LITERATURE REVIEW

An odor evaluation procedure is composed of two parts, i.e., odor sensory techniques and odorous gas sampling techniques. Although an electronic nose is in the development stage, the human nose is still the best odor sensing equipment available and on it all measurements currently are based. There are five basic techniques that have been used for sensory odor analysis: ranking, rating, magnitude estimation, dilution, and forced choice (Riskowski et al. 1991). A panel composed of 4 to 50 people were used in the different procedures for the olfactory test. Among these methods, only the forced choice technique is considered a standard U.S. practice recommended by ASTM (ASTM 1989). The second part of the odor evaluation procedure is the odorous gas sampling. Past studies used different sampling techniques to collect air samples for their odor evaluation tests, resulting in large discrepancies in the test results.

Ritter and Eastburn (1980) reported a study in which the organoleptic rating test proposed by Sobel (1972) was used to evaluate five different commercial chemical products for odor control. A total of at least six panelists selected at random were employed in each rating test. The manure for the test was collected and stored in a 114 L drum for 30-40 days before the manure additives were added. The odor evaluation was conducted 24-48 hours after the addition of additives. Obviously, this method is not appropriate for biological products as microbes can not reach a dominant level within 48 hours.

Warburton et al. (1980) evaluated 22 pit additive products provided by 22 companies. Manure was collected from experimental hog units once a day at the same time each day during the test and was agitated before samples were added to the 208 L drums with a diameter of 568 mm. The manure for the odor evaluation was collected from the top 50 mm at the center of the liquid surface in each drum. Odors were evaluated weekly with the rating method by a panel of human sniffers by judging the odor strength and acceptability emitted from the manure. The manure was contained in black bottles covered with gauze to eliminate any visual comparisons. A total of 40 to 50 people were involved in the weekly evaluation, with most of them participating every week. A problem with this testing set-up is that the manure source was disturbed when collecting manure samples for odor evaluation. Riskowski et al. (1991), by using an experimental design similar to that of Warburton et al. (1980), compared the magnitude estimation rating method with the ranking method with a panel of 12 to 18 people to evaluate a commercial manure additive product.

Williams and Schiffman (1996) reported a laboratory rating test with a 0 to 8 scale for evaluating five manure additive products in terms of odor irritation, odor intensity, and odor pleasantness. A cotton fabric swatch, placed in a nitrogen rich environment, was exposed for 24 hours to the gas from manure slurry contained in a vessel and then transferred to a sealed amber glass jar for odor evaluation. This swatch was used as the odor source for panelists to sniff. The odor panel was comprised of 10 individuals and, in general, the same panelists were utilized for all analyses. This method is simple and easy to use; however, whether the cotton swatch can represent an odor source with complex emission characteristics is questionable.

The differences in procedures used by these researchers in providing odor samples to the panelists for evaluation may affect the evaluation results. The possible discrepancies may result from both the air sampling and the odor evaluation. For air sampling, the conditions of the manure sources where sampling is conducted will affect the quality of the odor gas collected and, thus, will affect the subsequent odor evaluation tests. The emission of odorous compounds from liquid manure is closely related to many factors such as temperature, humidity, ventilation rates, total manure amount, total solids content, partial pressure of each volatile substance in the gas phase, and the concentrations of the volatile substance in both the air and liquid, thus any direct or indirect disturbance of the odor sources may affect the odor emission rate from manure under normal operating conditions and eventually interfere with the evaluation results. Past experience has proven that when manure is disturbed, a strong odor will be released into the air. Thus, it may be reasonable to assume that the closer the test settings are to the real environment, the more accurate the collected odor samples will be and more reliable evaluation results will be obtained.

The evaluation methods also are critical in determining the odor intensities. The above mentioned studies evaluated the odor offensiveness based on a rating scale of either 0 to 8 or 1 to 10 with references. This methodology, or others similar to this, is subject to the sensitivity of human noses. It is virtually impossible for the human nose to detect the slight difference in intensity between the adjacent levels even when sensing fatigue is ignored. Comparatively, "presence or absence" logic is much easier for the human nose to detect, which is the advantage that dynamic forced choice olfactometers provide.

To evaluate a procedure, several aspects should be taken into account. First of all, it is important that the experimental design reflects the real environment to the greatest extent. This includes considerations of temperature, humidity, ventilation rates, manure storage time, floor cleaning frequency, and bacterial properties. Secondly, the odor air collected for the evaluation should represent as closely as possible the odor air released from a real pit. Any intermediate steps which could cause errors in the final results should be avoided. Thirdly, the procedure should use a commonly recognized technique, such as ASTM recommended sensory technique - dynamic forced-choice olfactory, to evaluate odor samples. The olfactometer for such uses needs to be designed following the principles presented in the ASTM standards (ASTM 1989). The sensing ability of all the panelists should be calibrated on a regular basis (currently, *n*-butanol is usually used in calibration). Fourthly, the procedure should employ statistical methods. This encompasses both the experiment and the sample analysis so that errors from the test as well as from the odor sample analysis can be reduced to a minimum. Fifthly, in addition to the odor analysis, a comprehensive liquid manure analysis including total solids, total volatile solids, volatile fatty acids, pH, ammonia nitrogen, and total nitrogen also should be a part of the procedure because these factors are closely related to the malodor production. This information is also useful in evaluating additive products in terms of nutrient balance in the treated manure and environmental protection. Finally, it would be better, although it is not critical, to make the procedure simple and easy to use, provided that all the requirements mentioned above are met. The purpose of this paper is to introduce a procedure proposed by Bundy et al. (1996) for testing pit additive products. An application of this procedure based on the testing of five commercial pit additive products is presented as well.

PROCEDURE AND TESTING

Experimental design

This procedure uses polyvinyl chloride columns to simulate the pit where manure is stored. Each column is 1225 mm high and 380 mm in diameter (Fig. 1). The maximum manure level in the column is 920 mm with a head space of 305 mm for air circulation. The height of the column is basically the same as the depth of a shallow pit that is about 1200 mm (commonly-used pits in US have a depth between 1200 and 2400 mm). The purpose of using this design is to obtain air samples that may closely reflect the real characteristics of the odor air in an operating pit. Air continuously passes through the column head space at a rate of 14 L/min to represent the typical air exchange rate in a pit underneath a slatted floor. This rate was calculated based on the minimum pit ventilation rate provided in "Swine Housing and Equipment Handbook" (MWPS 1983) and then scaled down according to the manure surface area in the testing columns. The column is sealed during the test except for air circulation, sampling, and manure loading ports. The columns are housed in an

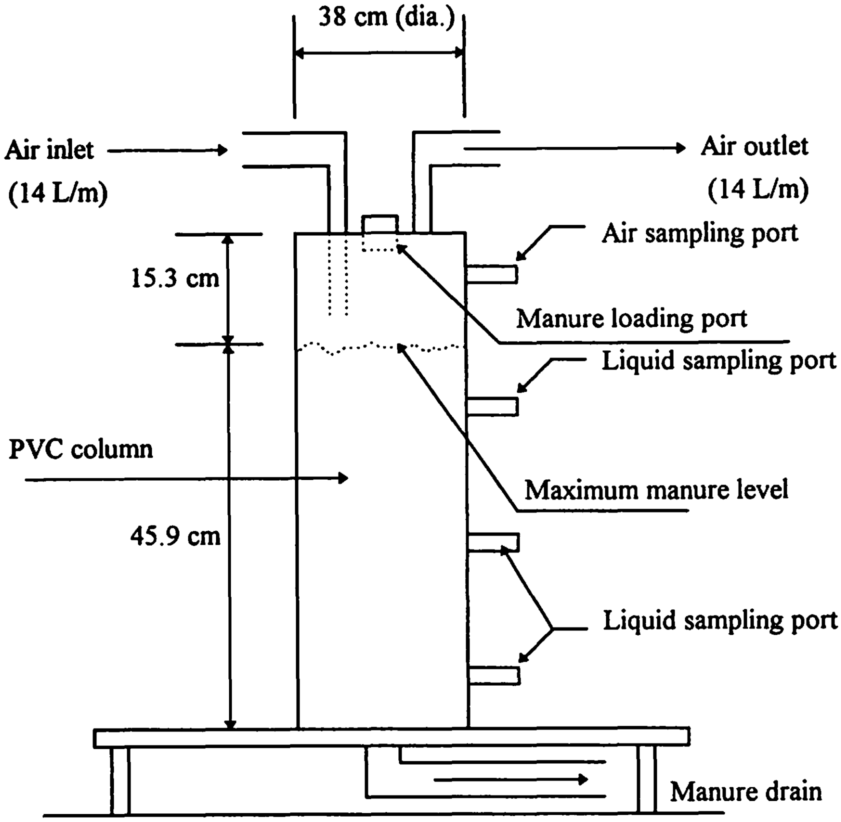


Fig. 1. Testing column apparatus.

environment-controlled building with temperature ranging from 15 to 21°C during the test. A completely randomized design is used with two treatments (1 control and 1 treatment) with three replications for each treatment so the results can be analyzed statistically.

Manure source and loading

Fresh manure is added into the columns weekly to simulate manure accumulation in the pit. A total of five loadings of manure is made with the first loading being 305 mm high (assuming there always is some manure left after emptying the pit) and the rest 153 mm each. After the last loading, a total of 106 L of manure is stored in the columns. The manure will be well mixed and adjusted to approximately 4.0% of total solids content (common level in a real pit) before being added to the testing column. There is no agitation or aeration induced to the manure in the testing columns during the test. The full test lasts for five weeks so biological products can also be examined.

Air sampling and analysis

The odorous air for odor threshold analysis is collected from the head space of each column without any disturbance made to the manure in the column. This offers a possibility to study, under operating conditions, the odor air released from the surface of a manure pit under the slatted floor. In air sampling, polyethylene bags of about 25 L each in volume are employed. There has been an argument regarding the use of polyethylene material since this material is suspected to absorb odorous compounds, hence influencing the odor evaluation results. However, little quantitative information is available in relation to how fast the absorption process proceeds and how much the influence is. In this test, since the air samples are analyzed immediately after collection, it could be assumed that the material impact on odor evaluation results should not be significant. To keep the air samples from being contaminated, the bag is kept in a sealed container and the odorous air is drawn into the sampling bag by the negative pressure created by an external vacuum pump (known as "lung principle"). Small vacuum pumps are used to produce a low incoming airflow rate to the bag (it takes about 15 min for each 25 L bag sampling). This makes it possible to collect odorous air from a less disturbed source and the quality of the odor can be obtained. Besides odorous air analysis, the aerial ammonia and hydrogen sulfide levels are also included in this procedure and are taken directly from the head space of each column by using MSA detector tubes (ammonia: MSA5085-845, NH_3-2, range: 2 - 500 ppm; hydrogen sulfide: MSA5085-826, H_2S-1, range: 1 - 200 ppm).

The odorous air is collected weekly and the samples are analyzed immediately after collection to avoid any potential changes in properties which might occur due to the interactions between the odorous air and the environment. A panel consisting of four people randomly selected from a panelist pool of about 20 are employed to run the odor evaluation test using a triangle dynamic forced choice olfactometer developed at Iowa State University according to ASTM Standards (ASTM 1989). The sensing ability of all the panelists is calibrated on a regular basis using *n* -butanol.

Liquid manure sampling and analysis

The procedure includes a comprehensive liquid manure analysis to study the relationship between odor threshold and the levels of various chemical compounds in the manure. Since the levels of volatile fatty acids, total solids, total volatile solids, ammonia nitrogen, pH, and total nitrogen are, in various degrees, related to the odor generation, these parameters are targeted. At the end of the test, the manure in each column is drained out into a 132 L container and then repumped into the column to flush out all the solids which might have settled at the bottom of the column. This cycle is repeated until the column is clear of sediment. The liquid sample is collected from the center of the manure volume in the container after the manure is well mixed. American Public Health Association (APHA 1992) Standard methods are used to measure these variables (Table I).

Testing

Five commercial pit additive products were tested using the

Table I: Measuring methods for different variables (APHA 1992)

Variables	Measuring methods
pH	Combination electrodes with Ag/AgCl references (Fisher Scientific)
Total solids (TS)	Drying at 105°C
Volatile solids (VS)	Combustion at 550 °C
Ammonia nitrogen	Ammonia Ion Selective Electrode (Fisher Scientific)
Total nitrogen	Kjeldahl
Volatile fatty acids (VFA)	distillation

above procedure. These additives included chemical, enzymatic, and bacterial products, each of which is described in Table II. The fresh manure for testing was collected from a swine finishing building with a scraper system for providing uniform manure samples. The pit additives were added into the testing columns based on the instructions provided by the manufacturers. A complete randomized design with six treatments were employed (1 control and 5 treatments). There were three replications for each treatment. Student *t* pairwise test was used throughout the analysis of the testing data.

Table II: Information for the tested pit additives

Trade name	Classification	Functions
MPC	Chemical emulsifier	Helps eliminate the odor causing bacteria
Bio-Safe	Enzymes and microorganisms	Stimulates bacteria to break down odor generating compounds
Shac	Natural coal product (chemical)	Enhances biological and chemical processes to reduce odor
X-Stink (LF1)	Aerobic bacteria	Breaks down volatile organic compounds
CPPD	Chemical oxidizing agent	Increases oxygen level in liquid to support bacterial activities

RESULTS AND DISCUSSION

Odor threshold analysis

All the products significantly reduced odor threshold levels as compared with the control samples at a significance level of $P < 0.05$ (Fig. 2). Further comparison between the treatments showed that the first four treatments had achieved significant reductions from the fifth treatment, CPPD. The percentages of odor threshold reduction for products MPC, Bio-Safe, Shac, and X-Stink ranged from 83% to 87%, while the percentage for CPPD was 58%.

The idea of using manure additives to control odors was proposed about twenty years ago and a considerable amount of research effort has been spent in this field. Past researchers rarely found any of the pit additive products to be effective in reducing odor levels of swine manure (Warburton et al. 1980; Ritter and Eastburn 1980; Al-Kanani et al. 1992). However, a study conducted by Barrington (1994) showed that some of the commercial products did reduce swine manure odor. Another study done by Bundy and Greene (1995) also showed promise when alkaline by-products were used as manure additives to control odor. The results obtained from these two studies provided evidence that some of the pit additive products might be able to debilitate odor intensities.

Aerial ammonia and hydrogen sulfide

The levels of ammonia and hydrogen sulfide in the gas phase were low during the entire test period. The concentrations of ammonia detected by MSA detector tubes ranged from 2 to 5 ppm for all the testing columns (including controls) and the release of hydrogen sulfide was not detectable (below 1 ppm). These results are consistent with the data obtained from another pit additives evaluation study (Riskowski 1991). Thus, it could be inferred that these pit additive products will not affect the emissions of ammonia and hydrogen sulfide into the air.

Chemical analysis of liquid manure

pH values The measurements of pH for the liquid manure collected at the end of the test (Fig. 3) show some differences in pH values for the treatments as compared with the control. MPC and Bio-Safe are not significantly different from the control ($P < 0.05$), while Shac, X-Stink, and CPPD are. Shac has a higher pH and X-Stink and CPPD have lower pH values than the control. Although these differences are significant from the statistical point of view due to small standard deviations, the overall range of pH for all the treatments and the control does not vary widely (6.0 -6.5) and is slightly lower than the normal pH range (6.5 -7.0) in liquid swine manure (Donham et al. 1985). At low pH values, nitrogen is present mainly as the ammonium ion and the formation of ammonia is hindered. That may in part account for the aerial ammonia levels being low for all the columns in this study. Since the differences in pH values between treatments and control is small, whether those pit additives affect the emission rate of ammonia can hardly be determined by the pH data in this test.

Volatile fatty acids The first three treatments (MPC, Bio-Safe, and Shac) had significantly lower volatile fatty acids ($P < 0.05$), while the other two (X-Stink and CPPD) did not (Fig. 4). Comparing Fig. 4 with Fig. 2, the reduction of odor threshold for the first three treatments and the control was consistent with the reductions of volatile fatty acids. It is reasonable that the odor threshold is reduced due to the decrease in the amount of volatile fatty acids which was identified as offensive odor indicators by past research (Spoelstra 1980). Nonetheless, if further comparisons are made between the two figures regarding X-Stink and CPPD, the results look inconsistent, i.e., the levels of volatile fatty acids for X-Stink and CPPD are not statistically different

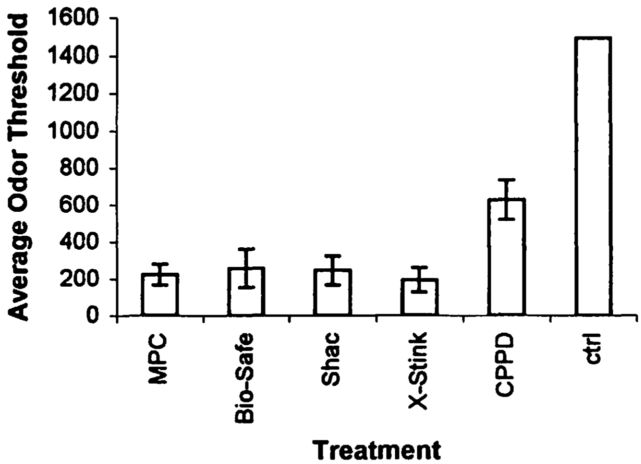


Fig. 2. Odor threshold means and standard deviations for the treatments and the control.

from that of the control, while the levels of odor threshold shown in Fig. 2 are significantly lower than that of the control. It is somewhat confusing that the odor levels can be reduced without reducing the level of volatile fatty acids. A possible explanation is that the X-Stink and CPPD may reduce the levels of sulfur-containing compounds, hence, reducing odor. This hypothesis could not be verified by this study because of the lack of liquid manure analysis for sulfur related compounds. The aerial hydrogen sulfide was low for both these two treatments and the control, thus there wasn't any evidence to support this hypothesis.

A study conducted by Hartung (1988) showed the types of volatile fatty acids in the gas phase as well as their quantities detected in the air of pig barns (Table III). Since most of the gases have escaped from swine manure, the volatilization equilibrium of each gas component in the air should follow Henry's law for dilute systems and should thus be proportionally related to the quantity of this component in the swine manure. According to Mackie (1994), the greater the chain

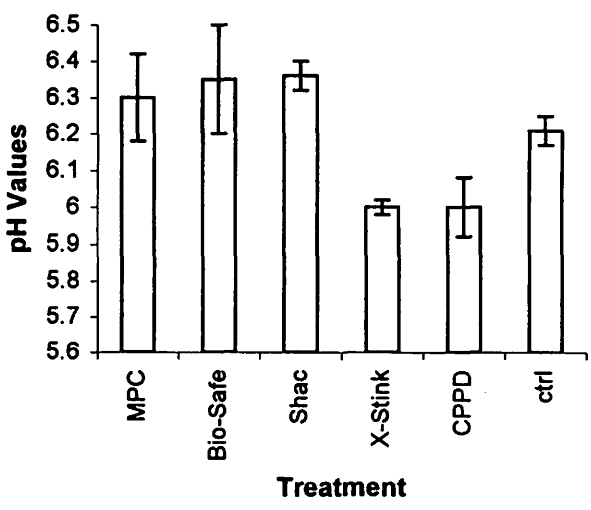


Fig. 3. pH values for the treated and control samples.

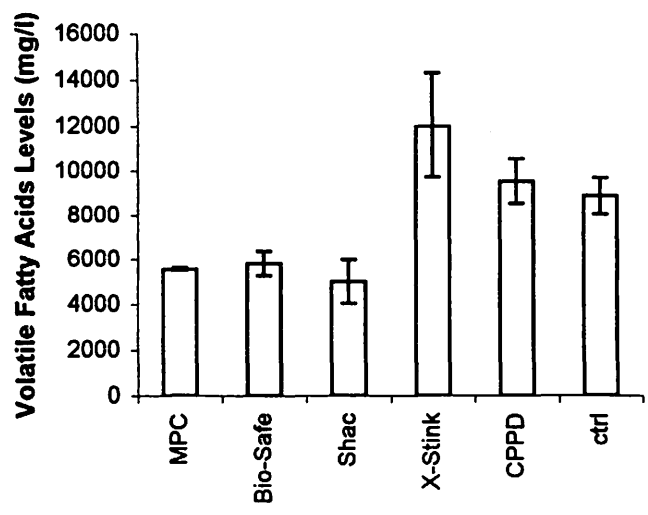


Fig. 4. Volatile fatty acids variations between samples.

length of the volatile fatty acids (C_5 -C_{10} as compared with C_1 - C_4), as well as the branching, the more offensive is the nature of odor associated with these acids. Quantitatively, the short chain acids (acetate, propionate, and butyrate) contribute less to odor potential even if they are present in much higher concentrations. Another study conducted by O'Neill and Phillips (1992) also showed that the volatile fatty acids with higher carbon numbers (Table III) have lower odor detection threshold, thus are more offensive in nature. Although the odors of the lower aliphatic acids are more volatile and smell pungent, the odorous nature progresses from pungent odors of formic and acetic acids to the distinctly unpleasant and offensive odors of valeric and caproic acids (Morrison 1987). Thus, it could be inferred that the offensive odor potential is not directly associated with the total amount of the volatile fatty acids in the manure, but rather it is dependent upon the types and characteristics of certain acids regardless of the quantities. In other words, those volatile fatty acids which have higher concentrations may not be the major sources of odor emission from the swine manure.

The above discussion could explain the results presented in Fig. 2 and Fig. 4. A simple calculation indicates that the first four acids in Table III account for about 88% of the total amount of the VFAs which may be considered less offensive in producing malodor. The reduction of odor thresholds by X-Stink and CPPD could be due to the effective breakdown of the rest, which are 12% of the total amount and are likely responsible for malodor generation. Therefore, to reduce odors caused by VFAs in swine manure, it seems more important and effective to reduce the levels of those which constitute the major portion of the malodor than to reduce the overall concentrations of VFAs. Odor components may be more offensive when mixed and smell differently than pure compounds; however, little is known concerning the extent of single volatile fatty acid contributions to the overall odor intensity. More research effort seems needed in ascertaining the targeted volatile fatty acids so that the corresponding pit additive products can be targeted to the purpose.

Table III: Volatile fatty acids gases in the air of pig houses (Hartung 1988)

Volatile fatty acids	Concentrations (μg/m^3)
Acetic acid	326
Propionic acid	290
n-butyric acid	617
i-butyric acid	78
n-valeric acid	63
i-valeric acid	92
n-hexanoic acid	10
i-hexanoic acid	4
Heptanoic acid	3
Octanoic acid	5
Pelargonic acid	4
Total	1492

Total solids and total volatile solids Volatile solids also may affect odor intensities (Fig. 5). For MPC, Bio-Safe, and CPPD, there appear significant changes in total volatile solids as compared with the control ($P < 0.05$). Products MPC and Bio-Safe reduced total volatile solids levels and, on the contrary, CPPD raised them. It is difficult to determine if the deviations for Shac and X-Stink from the control are significant due to large variations in the test data. For the control samples the amount of total volatile solids accounts for large percentages of the total solids. This means most of the solids in the liquid are volatile so the control of total volatile solids will be of great help in reducing the emission of volatile substances. According to this test, only products MPC and Bio-Safe can effectively abate the total volatile solids levels. In addition, comparisons between the ratios of total volatile solids to total solids of different treatments show a large variation (33, 43, 71, 78, 77, and 66% for MPC, Bio-Safe, Shac, X-Stink, CPPD, and the control, respectively). This variation is possibly due to the uneven distribution of total solids when filling the columns.

One point that needs to be addressed here is that these changes in the volatile solids level could also be caused by the uneven total solids distribution in the initial manure. Difficulties were often experienced in obtaining uniform liquid manure when loading test columns. A further examination on the original total solids levels for all the treatments is needed to determine if the changes in volatile solids levels for different treatments are due to the products tested. If each treatment received the same amount of total solids, the ash levels (total solids minus volatile solids) should be the same for all the treatments. According to Fig. 5, however, the calculated ash levels for MPC, Bio-Safe, Shac, X-Stink, CPPD, and control are 11000, 12000, 6000, 6000, 5000, and 10000 mg/L, respectively. Thus, the columns for MPC, Bio-Safe, and control received manure with roughly the same levels of total solids, while the columns for Shac, X-Stink, and CPPD received manure with a lower total solids level. This situation creates difficulties in making comparisons among Shac, X-Stink, CPPD, and the control. According to this analysis, it can be concluded that products MPC and Bio-Safe significantly reduced total volatile solids levels as compared with the control. The changes in volatile solids for products Shac, X-Stink, and CPPD could not be determined.

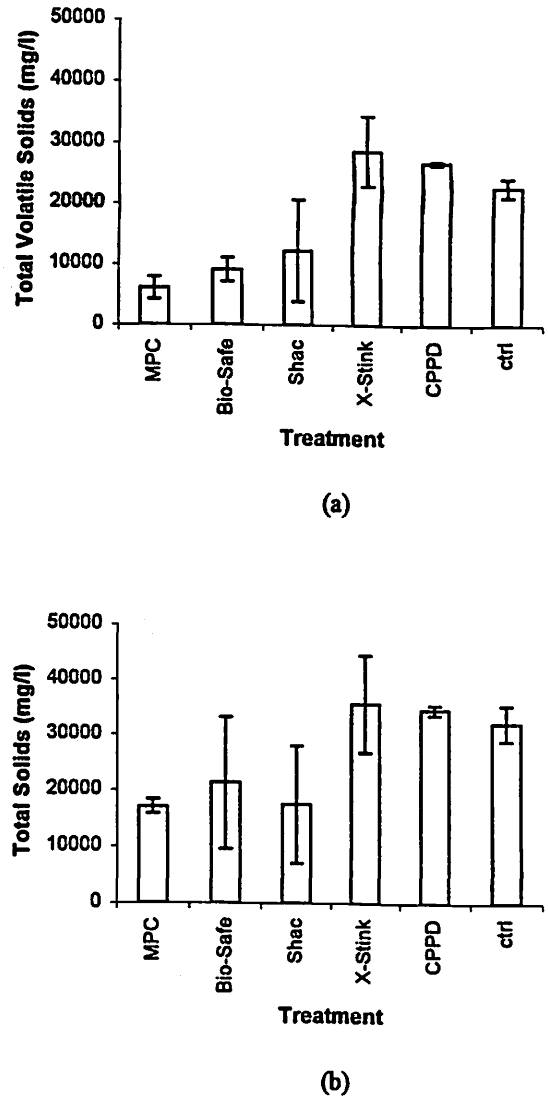
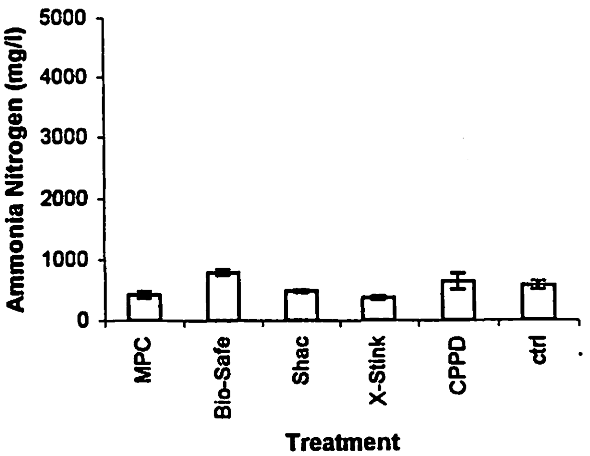
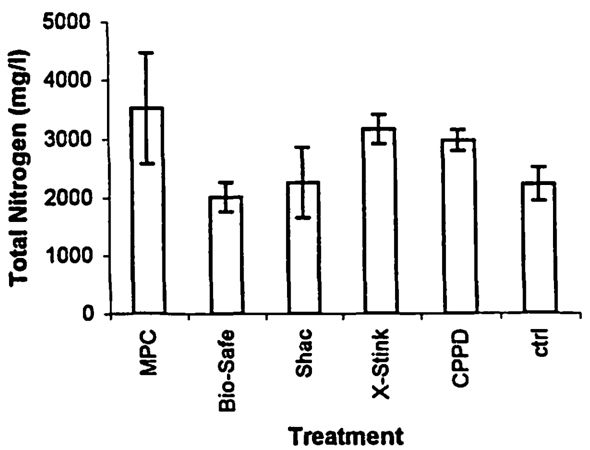


Fig. 5. Total volatile solids and total solids between samples; a) total volatile solids; b) total solids.

Total nitrogen and total ammonia nitrogen The changes in levels of ammonia nitrogen and total nitrogen are presented in Fig. 6. Only a small part of the total nitrogen is ammonia nitrogen and this implies that the potential for ammonia emission is low because most of the nitrogen exists in the form of nitrites, nitrates, or other organic compounds and is not available for volatilization (Groot Koerkamp 1994). According to Fig. 6(a), there is no significant difference in ammonia nitrogen for most of the products (with X-Stink being the exception) between the treated and the control in this test so the effect of these products on controlling ammonia emission from the manure could not be verified. For X-Stink, the ammonia nitrogen level is significantly lower than the control ($P < 0.05$). This product could reduce the ammonia emission. However, since the aerial ammonia measurement in this test did not provide conclusive evidence of significant reduction as compared with the con-



(a)



(b)

Fig. 6. Total ammonia nitrogen and total nitrogen; a) total ammonia nitrogen; b) total nitrogen.

trol, the reduction of ammonia emission from swine manure by X-Stink under the testing environment could not be quantitatively determined.

SUMMARY

As more and more swine producers are considering the use of pit additives for controlling unpleasant odors emitted from their swine production facilities, there is a great need to develop a proper protocol which would combine the best features of past evaluation methods and could be used to provide swine producers with valuable information concerning the performance of pit additives in odor control. The procedure addressed in detail in this paper is an effort to pave the way towards the standardization of the pit additive testing protocol. This procedure examines the performance of pit additives under an environment relatively similar to the actual environment for pit additives so that, to great extent, it can reflect the effectiveness of odor reduction due to the pit additive products tested.

The five commercial pit additive products evaluated using this procedure show evidence that the emission of odor from swine liquid manure can be abated by using pit additives. The reduction in odor intensities appears not directly related to the total amount of volatile fatty acids. All treatments reduced odor levels significantly, but MPC, Bio-Safe, and Shac performed better than the other two in terms of reducing the total amount of volatile fatty acids. The reduction of ammonia emission from swine manure was not apparent for any treatments tested in this study.

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