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Assessment of a Two-Stage Wood Chip-Based Biofilter Using Solid-Phase Microextraction and Gas Chromatography-Mass Spectrometry-Olfactometry

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***Abstract*.** A mobile pilot-scale biofilter testing laboratory, which consisted of one- and two-stage biofilter reactor barrels, was developed where two types of wood chips (western cedar and hard wood) were examined to treat odor emissions from a deep-pit swine finishing facility in central Iowa. The biofilters were operated continuously from July 20 to October 17, 2007 at different air flow rates resulting in variable empty bed residence times. During this test period, solid-phase microextraction 85 µm Carboxen/PDMS fibers were used to extract volatile organic compounds (VOCs) from both the control plenum and biofilter treatment. Analyses of VOCs were carried out using a multidimensional gas chromatography-mass spectrometry-olfactometry system. Reductions of nine odorous compounds were reported. An overall average reduction efficiency of 98.9% and 96.4% was achieved for two-stage western cedar and hardwood biofilters, respectively. The results showed that maintaining proper moisture content is critical to the success of wood chip-based biofilter.

Keywords. Air quality, Animal facility, Biofilter, GC-MS, SPME, Wood chips.

Introduction

With increasing human population and the intensification of animal production in many countries throughout the world, the odor produced and emitted from such intensive animal production can cause nuisance to individuals living in the vicinity of livestock farms. The reduction of odors emitted from livestock and poultry production systems continues to present challenges for researchers. Most odors and gas emissions from building and manure storage sources are by-products of anaerobic decomposition and transformation of organic matter in manure by microorganisms (Nicolai et al., 2006). These by-products result in a complex mixture of over 168 volatile compounds of which 30 have a detection threshold of 0.001 mg/m^3 or less, and hence are most likely to be associated with downwind odor nuisance (O'Neil and Philips, 1992). These compounds cover a broad spectrum and generally exist in low concentrations. Any technology used to reduce emissions must be able to treat a broad spectrum of airborne compounds. Various air pollution control technologies have been invented and applied, such as activated carbon adsorption, wet scrubbing, and masking agents. These methods, however, often transfer odor-causing materials from the gas phase to scrubbing liquids or solid adsorbents, and their derivatives have resulted in wastewater and solid waste concerns (Day, 1996; Lin et al., 2001; Chung et al., 2007). Biofiltration, which can be cost effective and has the ability to treat a broad spectrum of gaseous compounds (Devinny et al., 1999; Janni et al., 2001; O'Neil et al., 1992), has been regarded as a promising odor and gas treatment technology that is gaining acceptance in animal agriculture. Several research studies using compost-based biofilters have been conducted with significant reductions in odor and specific gases reported. Nicolai and Janni (1997) reported a compost/bean straw biofilter that achieved average odor and H_2S removal rates of 75% to 90%, respectively. Sun et al. (2000) observed an average H_2S removal efficiency between 92.8% and 94.2%, and an average NH_3 removal efficiency between 90.3% and 75.8% with 50% media moisture content and 20 sec gas residence time. Martinec et al. (2001) also found odor reduction efficiency up to 95%. The mixture of wood chips and compost (70:30 to 50:50 percent by weight) has been recommended as biofilter media (Nicolai and Janni, 2001a). However, special care is needed to screen fines from compost/wood chip mixtures to reduce operating static pressure. A wood chip media eliminates the need for mixing multiple media but little is known about the performance of wood chip-based biofilters.

To date, studies have mainly focused on odor, NH_3 and H_2S reductions when evaluating biofilters tested at animal sites. More studies are needed to better understand the biofilter's effects on volatile organic compounds (VOCs). Therefore, the objective of this research was to investigate the fate of selected chemicals when subjected to two distinct wood-chip based

biofilters operating at various moisture contents and different empty bed residence times (EBRT), defined as the volume of the biofilter media divided by the air flow rate passing through the media. This paper describes some initial testing conducted using one- and two-stage biofilters with the intention of future studies investigating selectivity of gas scrubbing by biofilter stage and the potential for reducing compaction of media through multi-stage biofilter designs.

MATERIALS AND METHODS

Experiment Site

This research project was conducted at a 1,000-head curtain-sided deep-pit swine finishing facility located in central Iowa, from July 20 to October 17, 2007. The building monitored was approximately 14 x 55 m with 25 cm and 61 cm diameter fans pulling pit-gases from the barn pump-out locations.

Mobile Pilot-Scale Biofilter System

A mobile pilot-scale biofilter system, which consisted of a biofilter testing laboratory (BTL) and a biofilter monitoring laboratory (BML), was constructed for this research project. The system set-up is shown in figure 1a. The BML was used to house all instrumentation hardware, calibration gases, and data acquisition hardware required to measure and store temperature, biofilter moisture content, wind speed, wind direction, NH_3 and H_2S concentrations. The BTL was covered at the top and sides to eliminate wind and rain effects on the biofilters being tested. The layout of the BTL is shown in figure 1b.

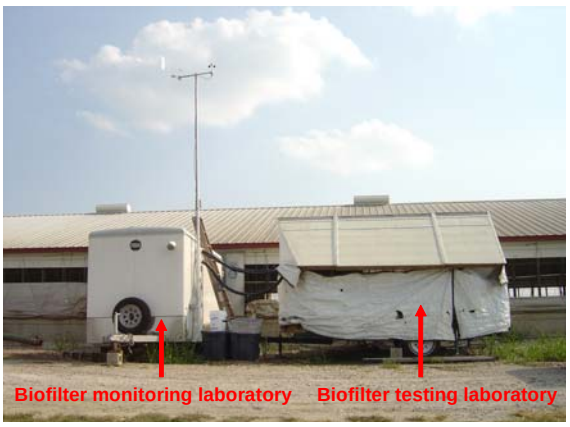

Figure 1a. The system set up.

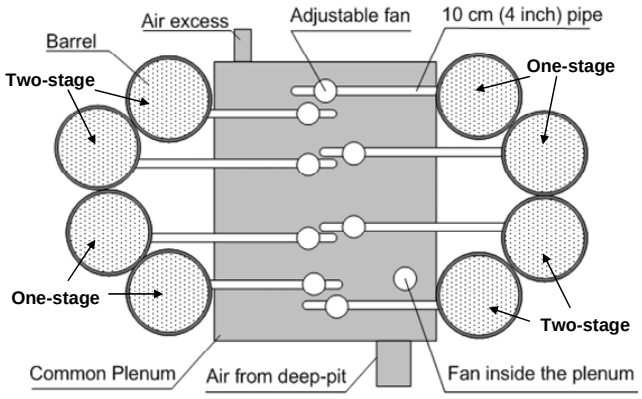

Figure 1b. The layout of the biofilter testing laboratory.

The BTL (figure 2a) consisted of eight parallel plastic reactor barrels, four (two of each two-stage and one-stage) of which were randomly selected to be filled with western cedar (WC)

chips, and the remaining four (two of each two-stage and one-stage) were filled with hardwood (HW) chips (figure 2b). Water holding capacity (wet basis) was measured as 74.8% ± 2.9% and 67.3% ± 1.5% for WC and HW, respectively. The WC and HW media porosity was 56.5%±3.3% and 53.7%±1.6% respectively, using the bucket test method (Nicolai and Janni, 2001a). There was a common plenum below the barrels directly connected to a fan from one of the barn pump-out locations. Eight adjustable fans (AXC 100b; Continental Fan Manufacturing, Buffalo, New York) and 10 cm (4 in) PVC pipes were used to connect the common plenum with the eight barrels. In order to homogenize the exhaust air in the plenum, a small fan (4C442; Dayton Fans) was installed inside the plenum for mixing purposes.

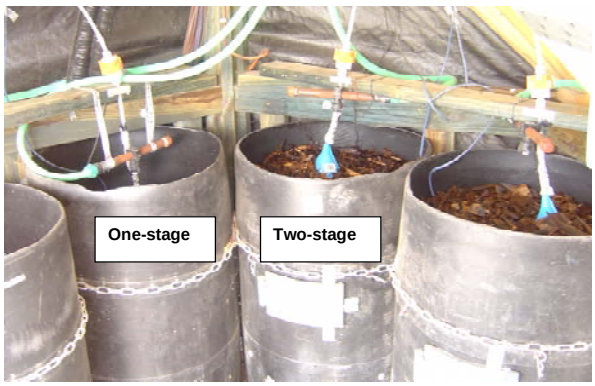

Figure 2a. Inside the BTL showing four (two of each one-stage and two-stage) of eight reactor barrels.

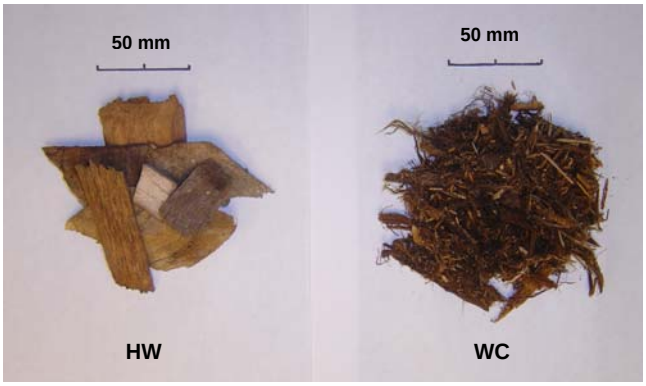

Figure 2b. Hardwood and western cedar chips.

The one-stage reactor barrels (56 cm diameter, 86 cm in depth) were designed with a 25 cm air space at the bottom of the barrels, with a height of 38 cm biofilter media located above this airspace separated by a metal mesh support (figure 3). The two-stage reactor barrels (56 cm diameter, 86 cm in depth) were designed with a 25 cm air space at the bottom of the barrels, with a height of 20 cm first-stage biofilter media located above this airspace separated by a metal mesh support (figure 4). There was another 25 cm air space above the first-stage biofilter, with a height of 18 cm second-stage biofilter media above this airspace separated by another metal mesh support. Water was added automatically *via* a spray nozzle and solenoid at the top of each barrel with a 9 sec water supply time at cycling times that varied from 30 to 45 min. Biofilter media moisture was measured with commercially available soil moisture sensors (Model ECH2O EC-20; Decagon Devices, Inc. Pullman, WA) combined with an oven method. The soil moisture sensors were first calibrated in the laboratory. The oven method involved placing the chip samples into an oven for 24 hours at 110℃. The variable speed fans were used to adjust the EBRT to 3.1, 3.7, 4.1, and 5.5 sec.

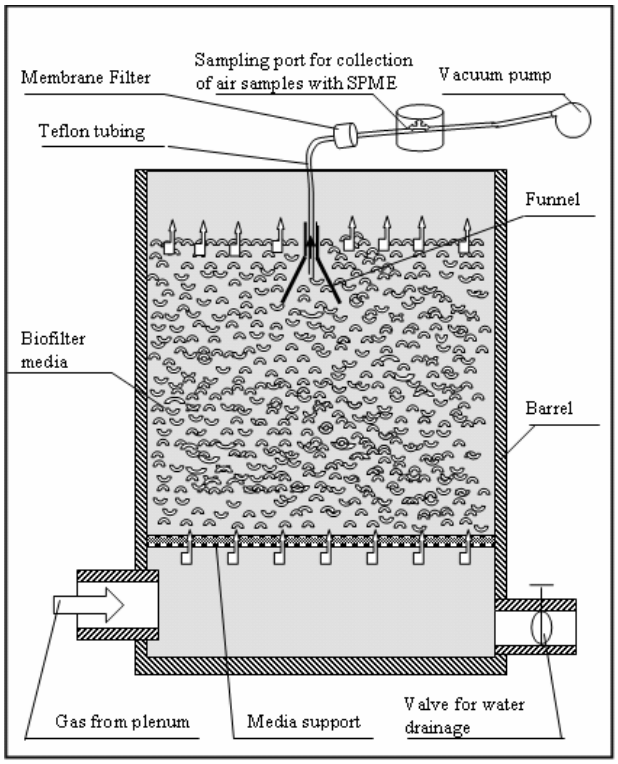

Figure 3 Schematic of one-stage biofilter reactor and gas/ solid-phase microextraction (SPME) sampling systems.

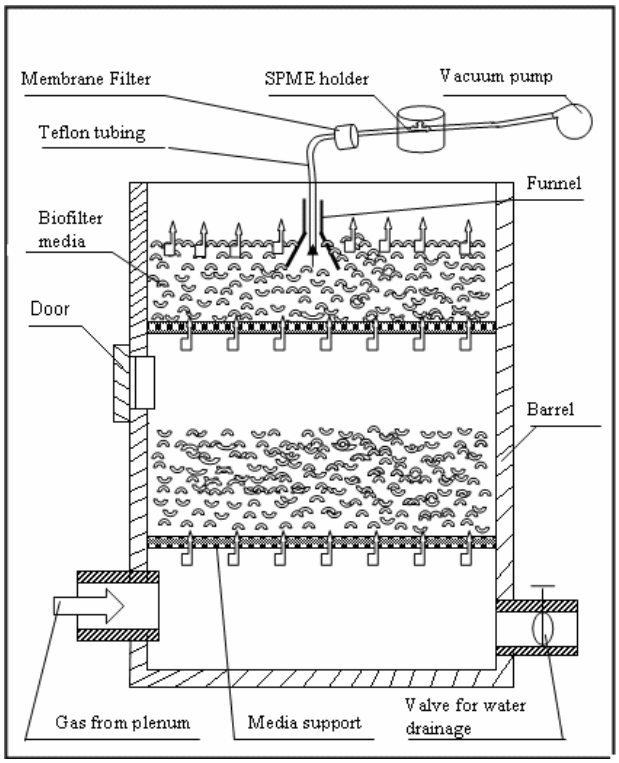

Figure 4. Schematic of the two-stage biofilter reactor and gas/ solid-phase microextraction (SPME) sampling systems.

Biofilter Operation

The biofilter media in each reactor was allowed to stabilize by passing pit-gas air through each reactor with a maintained moisture content in the 50-60% range (wet based) and an air flow rate of 1354 L/min. The stabilization period was one month during which static odor samples were taken weekly and solid-phase microextraction (SPME) fiber selection and time series tests were conducted. After the one month stabilization period, four levels of air flow rate (1804 L/min, 1512L/min, 1354 L/min and 1014 L/min) were randomly set to run in specified reactors for about one week during which SPME and static odor samples were collected and analyzed. SPME and static odor samples were also collected and analyzed at three different media moisture levels (71.5%, 56.3%, and 17.3%) with a fixed air flow rate of 1354 L/min (4.1 sec EBRT).

SPME Sampling

The SPME sampling system consisted of a funnel, PFA 6 mm (¼ inch) inside diameter Teflon® tubing, a 47 mm diameter membrane filter with a 0.45µm pore size, a custom-built PTFE (Teflon®) sampling port for the collection of air samples with SPME and a vacuum pump (figures 3, 4). All sample tubing was heated to prevent condensation within the tubes. The SPME sampling ports were cleaned and dried at 110℃ overnight before installing. When the SPME samples were collected, the SPME fibers were placed into the customized SPME sampling ports which allowed fiber contact with the sample air. Three commercially available fibers including 85 µm Carboxen/PDMS, 65 µm PDMS/DVB, and 50/30 µm DVB/Carboxen/PDMS (Supelco, Bellefonte, PA) were first tested to select the most suitable SPME coating for extracting VOCs associated with the pit-gas exhaust air. Before use, each fiber was conditioned in a heated GC splitless injection port under helium flow according to the manufacturer's instructions. SPME sampling time was varied from five minutes to two hours to determine the optimal SPME sampling time. The system was first allowed to run for two minutes to equilibrate and then a SPME fiber was placed into the sampling port where the SPME fiber was exposed to sample air for the preset sampling time. The fibers were then removed from the sampling port, wrapped in clean aluminum foil and stored in a cooler for shipping to an on-campus laboratory for analysis. All SPME samples were analyzed within 48 hours of collection. The 85 µm Carboxen/PDMS fiber and 60 min extraction time were finally selected for this study based on the results (not shown here) of fiber selection and time tests.

Chemical Analysis

The compounds attracted by the SPME fiber were analyzed using a multidimentional gas chromatography-mass spectrometry-olfactometry (MDGC-MS-O) (Microanalytics, Round Rock, TX) which integrates GC-O with conventional GC-MS (Model 6890N GC/5973 MS; Agilent, Inc Wilgton, DE) as the base platform with the addition of an olfactory port and flame ionization detector (FID). The system was equipped with a non-polar pre-column and a polar column in series as well as system automation and data acquisition software (MultiTraxTM V. 6.00 and AromaTraxTM V. 6.61, from Microanalytics; ChemStationTM, from Agilent). The general run parameters used were the same as those described by Chen et al. (2008).

Compounds were identified with three sets of criteria: (1) matching of the retention time on the GC capillary column with the retention time of pure compounds run as standards, (2) matching mass spectrums of unknown compounds with Bench-Top/PBM (from Palisade Mass Spectrometry, Ithaca, NY) and (3) matching odor character.

There are several chemical compounds which are the main sources of offensive odors from swine buildings. Hammond et al. (1979) identified propanoic acid, and butanoic acid, as well as phenol, p-cresol, and 4-ethyl phenol, as important odor contributors. Wright et al. (2005) ranked p-cresol, indole, and skatole as the major odorants and assigned lower ranking to acetic acid and phenol. However, acetic acid and phenol are typically present at higher concentrations in these environments. Cai et al. (2006) also reported key malodorants associated with swine barn particulate matter including isovaleric acid, p-cresol, indole and skatole. In this study, qualitative assessment of VOC abundance was measured as area counts under peaks for separated VOCs. The mean peak area counts, which were calculated using the integrated area of a single ion, of the main odorous compounds as mentioned above (acetic acid, propanoic acid, butanoic acid, isovaleric acid, phenol, p-cresol, and 4-ethyl phenol, indole, and skatole) were used to compare the reduction efficiencies between treatments, as defined in equation (1) (Cai et al, 2007),

$$\%Reduction = \frac{C_i - T_i}{C_i} \times 100\% \qquad (1)$$

where:

C_i = peak area count of compound “i” for the control, and

T_i = peak area count of compound “i” for the treatment.

RESULTS AND DISCUSSION

Reduction Efficiency and Mean Peak Area Counts

The reduction efficiency of the two-stage biofilters as a function of EBRT is shown in figures 5a and 5b for WC and HW, respectively. For WC (figure 5a.), variations in EBRT from 3.1s to 5.5 sec did not result in discernible differences in reduction efficiency for all the main odorous compounds except for a drop from 98% to 80% for phenol at the 3.7 sec EBRT with a media moisture content of 75%±1% (wet basis). The overall average reduction efficiency was 98.9%, 97.6%, 98.2%, and 97.5% for EBRT levels of 3.1, 3.7, 4.1, and 5.5 sec, respectively. For HW (figure 5b.), the differences in reduction efficiency for all compounds tested were small at the 3.1 and 3.7 sec EBRT with a media moisture content of 65%±2% (wet basis). There was a slight increase in reduction efficiency when the EBRT increased from 3.1 to 3.7 sec for all compounds except phenol, where the reduction efficiency decreased from 96.6% to 93.5%. The overall average reduction efficiency for all compounds increased from 96.4% to 98.4%.

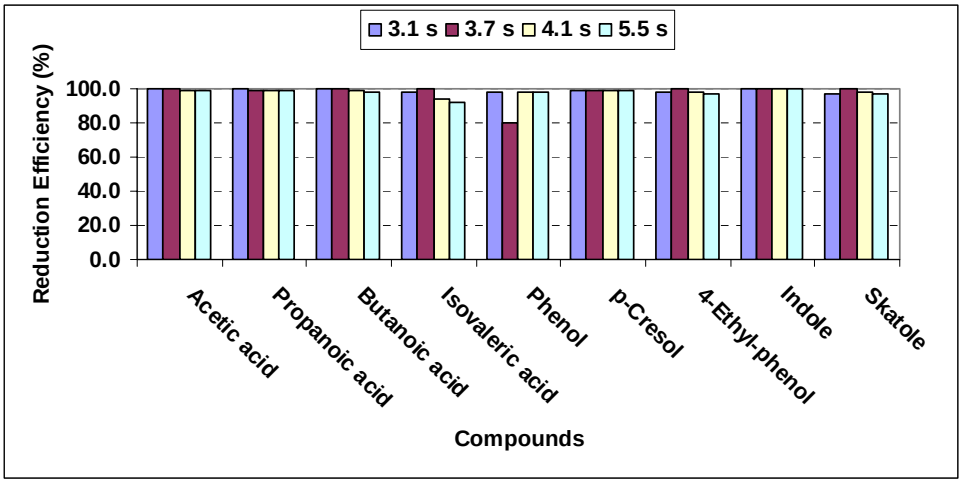

Figure 5a. Reduction efficiency for two-stage WC at four different EBRT levels.

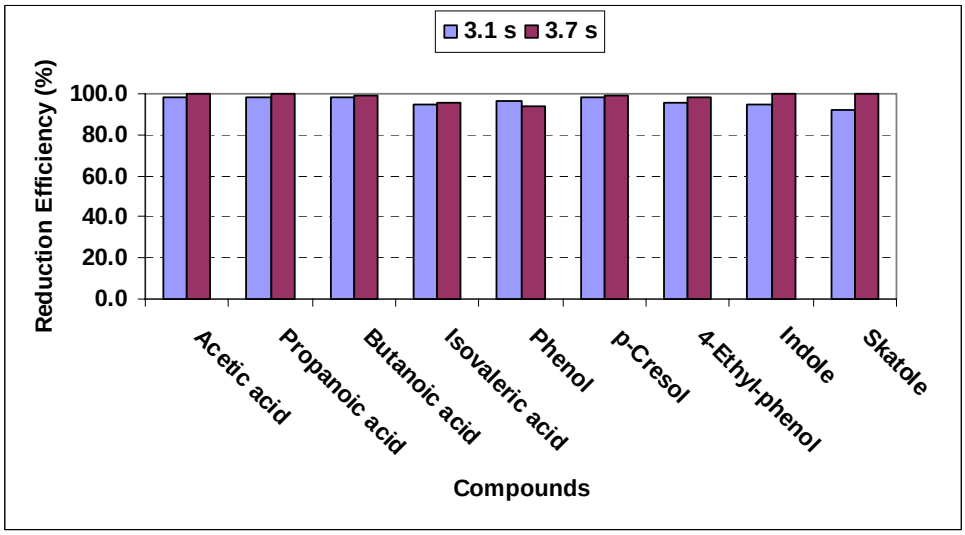

Figure 5b. Reduction efficiency for two-stage HW at two different EBRT levels.

The two-stage WC biofilter performed better than the one-stage for all compounds tested (figure 6a.). Two-stage HW biofilter reached a higher reduction efficiency compared to the one-stage biofilter except for indole (figure 6b.). The average reduction efficiency of WC was 98.9% and 72.4% for the two- and one-stage biofilters, respectively. Average reduction efficiency of HW was 96.4% and 93.8% for the two- and one-stage biofilters, respectively. Chung et al. (2007) reported that a two-stage biofilter achieved higher NH_3 removal efficiency compared with a conventional single-stage biofilter. They attributed this to a high H_2S concentration inhibiting nitrification when H_2S and NH_3 are simultaneously treated in a single biofilter.

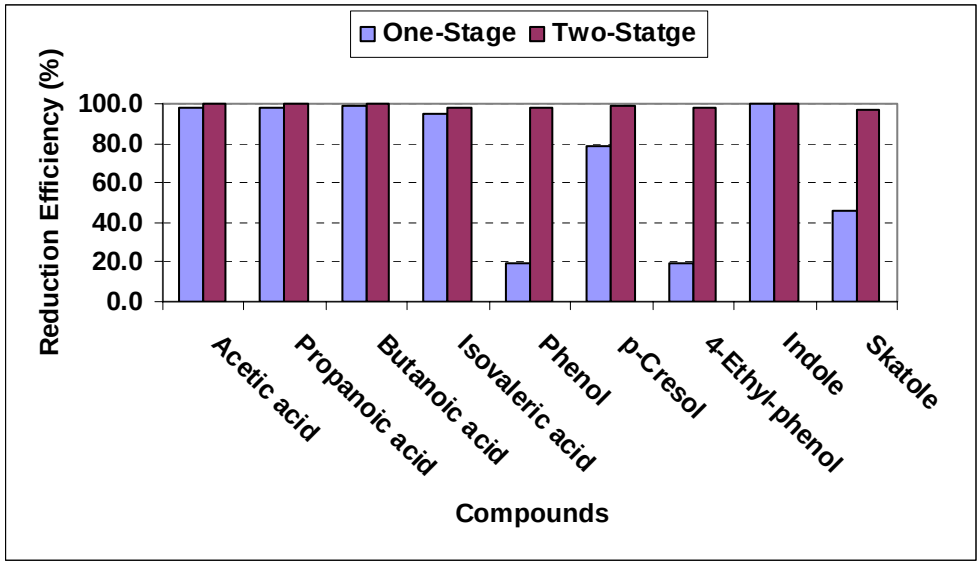

Figure 6a. Reduction efficiency comparison between one-stage and two-stage WC.

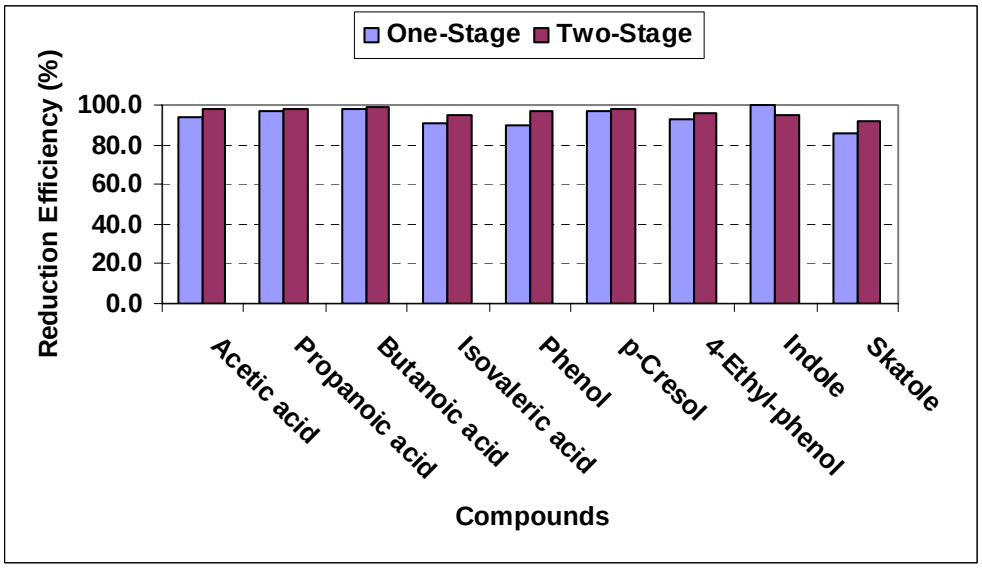

Figure 6b. Reduction efficiency comparison between one-stage and two-stage HW.

A summary of the two-stage biofilter reduction efficiency, estimated with equation (1), for the nine main odorous compounds is given in table 1. The removal efficiencies, based on overall average, for both types of biofilter media ranged from 96.4% to 98.9%. Particularly noteworthy is the removal of p-cresol which has been cited as the major odorant responsible for downwind swine odor (Koziel et al., 2006). The reduction of p-cresol, averaged over all EBRTs, was 98.7% and 99.1 % for HW and WC, respectively. The reduction efficiencies shown in table 1 have no discernable trend relative to EBRT.

Table 1. Summary of reduction efficiency as a function of EBRT.

\Chips (Moisture Content)	HW (63% ± 2%)		WC (75% ± 1%)				HW Average over EBRT (%)	WC Average over EBRT (%)
Compounds\EBRT (s)	3.1	3.7	3.1	3.7	4.1	5.5		
Acetic acid (%)	97.9	100.0	100.0	100.0	98.6	98.8	99.0	99.3
Propanoic acid (%)	98.2	100.0	99.8	99.3	99.3	98.6	99.1	99.2
Butanoic acid (%)	98.5	99.5	99.6	100.0	98.8	98.0	99.0	99.1
Isovaleric acid (%)	95.1	95.3	98.2	100.0	93.7	91.7	95.2	95.9
Phenol (%)	96.6	93.5	98.0	80.1	97.5	98.2	95.1	93.5
p-Cresol (%)	98.1	99.4	99.4	98.6	99.3	98.9	98.7	99.1
4-Ethyl-phenol (%)	95.8	98.2	97.9	100.0	98.2	96.8	97.0	98.2
Indole (%)	95.1	100.0	100.0	100.0	100.0	100.0	97.5	100.0
Skatole (%)	92.2	100.0	97.1	100.0	98.3	97.0	96.1	98.1
Overall Average (%)	96.4	98.4	98.9	97.6	98.2	97.5	97.4	98.1

Reduction Efficiency versus Media Moisture Content

Moisture is needed to maintain microbial activity during biofiltration processes. Several studies have reported that biofilter media moisture is one of the key factors when biofilters are used for treating odors (Hartung et al., 2001; Nicolai et al., 2006; Sun et al., 2000). Moisture levels between 40%-60% (wet basis) have been suggested for biofilter operation (Kastner, 2004; Nicolai and Janni, 2001b). In this study, SPME samples were collected and analyzed at three levels of media moisture content (71.5%, 56.3% and 17.3% wet basis) with a fixed 4.1 s EBRT.

Decreasing WC media moisture content resulted in lower reduction efficiencies for the nine main odorous compounds as shown in figure 7. Several studies conducted on odor, H_2S and NH_3 reductions obtained similar trends. Sun et al. (2000) reported that a higher media moisture content (compost-based biofilter) resulted in a higher removal efficiency for H_2S (47%-94%) and NH_3 (25%-90%) corresponding to moisture contents between 30-50%, respectively. Nicolai et al. (2006) observed that increasing the moisture content from 40% to 50% (wet basis) increased removal efficiency of NH_3 from an average of 76.7% to 82.3% and further increasing the moisture content to 60% did not significantly change the removal efficiency with a compost/wood chip biofilter.

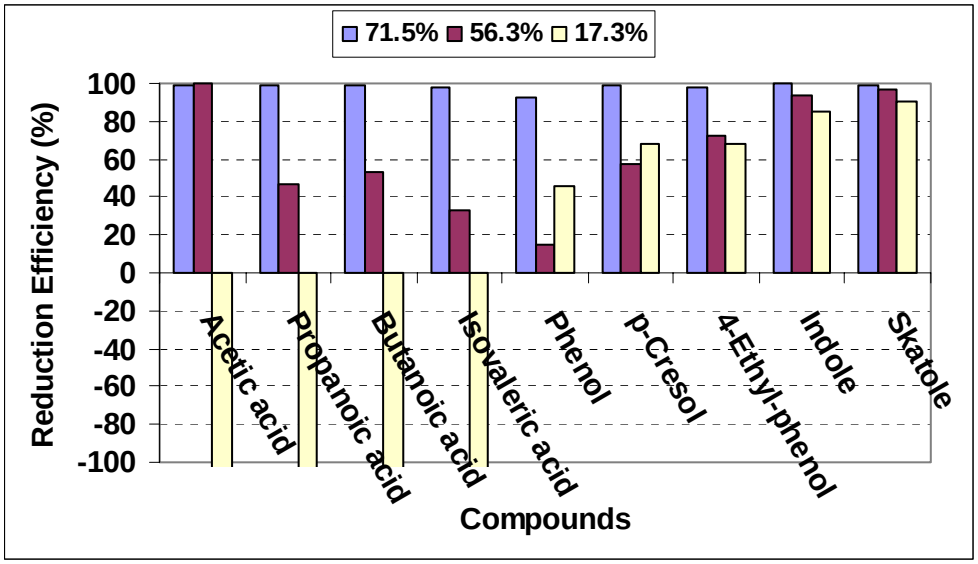

Figure 7. Reduction efficiency at different media moisture content.

The overall reduction efficiency was higher than 63.1% when media moisture content was kept above 56% (wet basis), even though the decrease of media moisture from 71.5% to 56.3% resulted in an overall reduction efficiency drop from 98.1% to 63.1%. Further decreasing the media moisture content from 56.3% to 17.3% resulted in negative efficiencies. For p-cresol and 4-ethyl-phenol, decreasing media moisture content from 71.5% to 56.3% led to a large difference in reduction efficiency, but further decreasing media moisture content from 56.3% to 17.3% did not result in a large change. For indole and skatole, there was a decreasing trend for the reduction efficiency with decreasing media moisture content. Overall, keeping the biofilter at higher media moisture content is critical for successful performance.

CONCLUSIONS

A mobile pilot-scale biofilter laboratory, which consisted of one- and two-stage biofilter reactors, was developed where WC and HW chips were examined to treat odor emissions from a deep-pit swine finishing facility. The fate of nine odorous compounds was investigated. The results of this study demonstrated that a two-stage biofilter for both the WC and HW chips was comparable to the one-stage biofilters tested. The reduction efficiency results at three media moisture levels indicated that the biofilter, whether WC or HW, was more sensitive to the media moisture content than media depth or EBRT. Therefore, maintaining proper moisture content is critical to the proper operation of wood chip-based biofilters. The high reduction efficiency obtained with the wood chip-based biofilter media studied in this research suggests that these

materials can be used effectively as biofilter media for reducing swine building odorants. However, more studies at full scale biofilters are needed.

ACKNOWLEDGEMENTS

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