Reduction of Malodorous Compounds from a Treated Swine Anaerobic Lagoon

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ABSTRACT

There is a need for treatment technologies that can eliminate environmental problems associated with anaerobic lagoons. These technologies must be able to capture nutrients, kill pathogens, and reduce emissions of ammonia and nuisance odors. To meet these needs, a full-scale wastewater treatment plant was installed as a demonstration project on one of three 4360-pig (*Sus scrofa*) production units in a finishing farm in Duplin County, North Carolina. Once the treatment plant was operational, flow of raw manure into the unit's corresponding lagoon was discontinued and the lagoon was used to store treated wastewater. Water quality was monitored in the converted lagoon and in the two conventional lagoons. A gas chromatographic method was developed to measure concentration of five selected malodorous compounds (phenol, *p*-cresol, 4-ethylphenol, indole, and skatole) in liquid lagoon samples. Dramatic improvements in the water quality parameters TKN, NH_3–N, solids, COD, and BOD in the converted waste lagoon paralleled reductions in malodorous compounds. Nine months after conversion, identified malodorous compounds in liquid extractions averaged 6.6 and 38.8 ng mL^{-1} in water from the converted lagoon and the conventional lagoons, respectively. The reduction was particularly marked for *p*-cresol, 4-ethylphenol, and skatole, all of which make important contributions to swine waste odors due to their characteristic odors and low detection thresholds.

Anaerobic lagoons are widely used to store and treat liquid effluents from confined swine production facilities in the southeastern USA. During lagoon treatment, anaerobic processes contribute to emission of volatile compounds, some of which produce offensive odors (Schiffman et al., 2001). Odorous emissions concerns about human health in rural communities are due mainly to the large concentration in small geographic areas of confined animal feeding operations. Because of this, there is major interest in developing swine manure treatment systems that can substantially reduce odor emissions (Williams, 2001).

Several studies described efforts to reduce odor emissions from swine lagoons that included: management of waste loading (Lim et al., 2003), use of additives (McCrory and Hobbs, 2001), mechanical aeration (Heber et al., 2002; Westerman and Zhang, 1997), or the use of lagoon covers (Clanton et al., 1999; Funk et al., 2004; Miner et al., 2003; Zahn et al., 2001b). However, widespread objection to the use of anaerobic lagoons for swine manure treatment in North Carolina prompted a state government–industry framework that gave preference to alternative technologies that would eliminate anaerobic lagoons. This framework established an agreement between government and swine industry to develop and demonstrate environmentally superior waste management technologies (EST) that could meet the following five environmental performance standards: (i) eliminate the discharge of animal waste to surface waters and ground water through direct discharge, seepage, or runoff; (ii) substantially eliminate atmospheric emissions of ammonia; (iii) substantially eliminate the emission of odor that is detectable beyond the boundaries of farm; (iv) substantially eliminate the release of disease-transmitting vectors and airborne pathogens; and (v) substantially eliminate nutrient and heavy metal contamination of soil and ground water (Williams, 2001).

In July 2004, 2 out of 16 technologies were determined to meet the environmental performance criteria necessary for EST. One of these technologies treated separated solids using a high solids anaerobic digestion system, while the other one was designed to treat the entire waste stream from a swine farm using a solids separation, nitrification–denitrification, and soluble P removal system (Williams, 2004). This second technology effectively replaced anaerobic lagoon treatment by discontinuing loading of liquid manure into the lagoon. In turn, recycled clean water promoted the conversion of the anaerobic lagoon into an aerobic pond in less than a year (Vanotti, 2004). The objective of this study was to evaluate the converted lagoon for odor reduction by comparing it with adjacent conventional lagoons with similar production management. The evaluation consisted of quantifying the levels of selected malodorous volatile compounds from both converted and conventional anaerobic swine lagoons under the hypothesis that water quality improvement in lagoons would substantially reduce the concentration of malodorous compounds.

MATERIALS AND METHODS

Site Description

The test site had three finishing hog production units with 4400 animals each. Each unit consisted of six houses with a 1.0-ha anaerobic lagoon. Hog houses had pit recharge systems to handle the liquid manure. The typical management was to drain the pit content by gravity to a lagoon weekly, and then recharge the pit with new lagoon-treated liquid. As part of the project to demonstrate a new technology to replace treatment lagoons, one of the three production units was retrofitted with a full-scale waste management system that combined solid–liquid separation with removal of N and P from the

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Abbreviations: BOD, biological oxygen demand; COD, chemical oxygen demand; EST, environmentally superior technology; GC-MS, gas chromatography–mass spectroscopy; NH_3–N, ammonia nitrogen; TKN, total Kjeldahl nitrogen; TS, total solids, TSS, total suspended solids, VSS, volatile suspended solids.

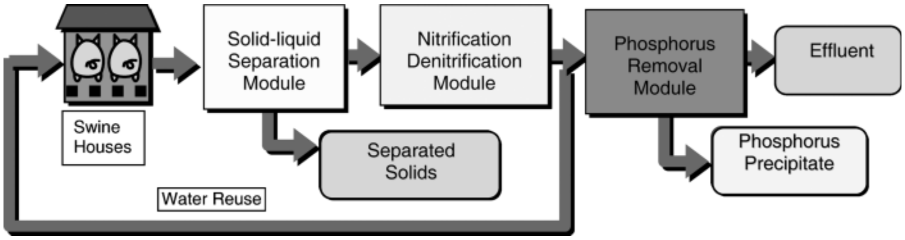

Fig. 1. Schematic diagram of wastewater treatment system.

liquid phase. The system made use of three modules (Fig. 1). The first module quickly separated solids and liquids. This module produced 596 Mg of separated solids per year that were converted to organic plant fertilizer, soil amendments, or energy (Vanotti, 2004). The second module treated the liquids after solid separation using a biological N removal system. The excess liquid then went to the final step, where P was recovered as calcium phosphate with the addition of small quantities of liquid lime (Vanotti et al., 2005). The system treated an average of 39 m^3 d^{-1} of raw manure flushed from the barns. The treated water (after N removal) was recycled to refill the barns (13 m^3 d^{-1}) or stored (26 m^3 d^{-1}) in the lagoon and later used for crop irrigation. As the treatment system recovered the manure solids and replaced the anaerobic lagoon liquid with clean water, it transformed the anaerobic lagoon into an aerobic pond during the treatment technology evaluation (March 2003–January 2004). For a detailed description of the full-scale treatment system see Vanotti (2004).

Water Analysis

Samples for water analysis were taken from lagoon supernatant within a 0.30-m depth. Two 1.0-L composite samples were obtained by mixing in two separate buckets four subsamples that were collected using a 500-mL polyethylene dipper with a 3.6-m long handle. All analyses were performed according to Standard Methods (APHA, 1998). Total solids (TS), total suspended solids (TSS), and volatile suspended solids (VSS) were determined according to Standard Methods 2540 B, D and E, respectively. The TSS were that portion of TS retained on a glass microfiber filter (Whatman grade 934-AH, Whatman, Clifton, NJ) after filtration and drying to constant weight at 105°C whereas VSS was that portion of TSS that was lost on ignition in a muffle furnace at 500°C for 15 min. Chemical analyses consisted of chemical oxygen demand (COD) and 5-d biochemical oxygen demand (BOD), ammonia-N (NH_3–N), total Kjeldahl N (TKN), and pH. For COD determination, we used the closed reflux, colorimetric method (Standard Method 5520 D), while BOD was determined using the 5-d BOD test (Standard Method 5210 B). Ammonia-N was determined using the automated phenate method (Standard Method 4500-NH_3 G). Total Kjeldahl N was determined by the same phenate method adapted to digested extracts (Technicon Instruments Corp., 1977) and pH electrometrically (Standard Method 4500-H^+ B).

Extraction of Volatile Compounds from Water

Five aliquots of supernatant liquid were collected at various sites on the converted and two conventional lagoons except the side adjacent to the swine housing and each separated by at least 20 m. Water was collected from the top 30 cm of the lagoon surface in a 500-mL polyethylene bottle attached to a 3.6-m pole and a subsample of the liquid stored in a 40-mL glass vial so that no headspace remained. Care was taken to avoid roiling of the lagoon surface during sample collection while at the same time avoiding collection of scum on the water surface. The vials were refrigerated until the samples were extracted. Extraction columns containing 100 mg of Tenax TA (Supelco, Bellefonte, PA) enclosed within glass wool plugs were prepared in Pasteur-type pipettes. Each column was prerinsed with high-purity methanol and deionized water before use. The lagoon water samples were centrifuged and the supernatant was gravity-fed through the Tenax TA column. The sediment was resuspended in 5 mL of water, vortexed, and centrifuged again. The resulting supernatant was also passed through the column. The columns were rinsed with 2-mL deionized water, and excess water on the columns was expressed using a stream of high-purity N_2. Retained compounds were eluted into 2-mL vials with 400 μL of a 50:50 mixture of CH_2Cl_2 and hexane. An internal standard of pentyl acetate (750 ng) was added to each vial. Chromatographic separation of the samples was performed on a Varian Model 3800 GC (Varian Associates, Walnut Creek, CA) by injection of 1.0 μL in splitless mode for 1 min and with a 75:1 split thereafter onto a 60 m by 0.32 mm DB-5 column (Supelco, Bellefonte, PA) with a film thickness of 1.0 μm. Injector temperature was 220°C, FID 240°C, column oven initial temperature was 40°C for 2 min, then programmed at 3°C min^{-1} to 150°C and held for 2 min. The column oven was then programmed at 5°C min^{-1} to 220°C and held for 20 min. Injector and detector temperatures and gas flow rates were as described previously. Compounds were quantified by injection of external standards of high purity compounds obtained from Aldrich (Milwaukee, WI), and the resulting values were adjusted on the basis of the peak size of the pentyl acetate internal standard. Results were expressed as nanograms of compound per milliliter of water.

Compound Identification

Gas chromatography–mass spectroscopy (GC-MS) was performed using a Hewlett-Packard model 6890 gas chromatograph equipped with a 30 m by 0.25 mm HP-5 column (Hewlett-Packard, Palo Alto, CA) interfaced to a Model 1800 mass selective detector. One-μL aliquots were injected onto the GC in splitless mode for 1 min, and the mass ion detector used a scanning range of 40 to 450 amu. Operating conditions for the GC-MS were: injector 220°C, column oven 40°C for 1 min, then programmed at 3°C min^{-1} to 180°C. Compound identifications were performed by computer database searches and retention time matches of authentic samples of compounds on the SPB-5 column.

All data for concentration of malodorous compounds were analyzed by means and standard errors of the mean (PROC MEAN), analysis of variance (PROC ANOVA) after log-transformation of individual values, and linear regression (PROC REG) using the SAS system for Windows version 6.12 (SAS Institute, 1996).

Table 1. Lagoon liquid analyses of three swine lagoons performed before and after conversion of Lagoon 1 to storage pond.

Lagoon	TKN	NH_3–N	TS	TSS	VSS	COD	BOD	pH
	mg L^{-1}							
	January–December 2002 (before conversion of Lagoon 1)							
1	505†	464	4217	273	192	1700	207	8.03
2	521	467	3526	315	225	1700	170	7.96
3	517	469	3578	328	225	1700	196	7.98
	September–October 2003 (after conversion of Lagoon 1)							
1	114	98	2319	103	80	435	14	8.14
2	372	332	2794	263	83	1150	47	7.85
3	364	315	2537	245	170	1110	62	7.77

† Data are average of monthly samples.

RESULTS AND DISCUSSION

Water Quality

Before conversion of conventional Lagoon 1 to a storage pond, all three anaerobic lagoons in this study received flushed manure. Table 1 shows that average annual (January–December 2002) water quality parameters of the lagoons before the conversion of Lagoon 1 were very similar because the three lagoons had the same size and management. Beginning in February 2003, manure flush to Lagoon 1 was halted and 100% of the manure generated was processed through the treatment plant. Water constituent levels (such as TKN, NH_3–H, TSS, VSS, COD, and BOD) in Lagoon 2 and 3 were lower than annual average during the September–October 2003 sampling period due to annual variation in liquid concentration, typical of conventional anaerobic lagoons (Bicudo et al., 1999). Except for pH, all measured water quality constituents declined in the converted lagoon 9 mo after conversion (Table 1). Due to lagoon conversion, the levels of TKN and NH_3–N declined to about one-third of those of the conventional waste lagoons. In addition, levels of TSS and VSS in the converted lagoon were reduced by 60 and 37% with respect to those of the conventional lagoons. For instance, VSS averaged 80 mg L^{-1} for the converted lagoon and 127 mg L^{-1} for the conventional swine-waste lagoons. In the converted lagoon, the decline in organic load caused a large decline of 62% for COD and 75% for BOD with respect to conventional lagoons levels. This decline in COD and BOD levels may have a major effect on the microbial production of volatile compounds by both reducing the amount of organic substrate and increased aeration levels in the converted lagoon liquid. A study by Williams (1984) on pig slurry odors related NH_3–N, BOD, sulfides, volatile fatty acids (C2–C5), as well as total indoles and phenols of raw and aerobically treated slurries to offensiveness as determined by an odor panel. Ammonium N and sulfide concentration had poor correlation with odor offensiveness, but log BOD was correlated with offensiveness ratings ($r = 0.86$), as were fatty acids and total indoles and phenols concentrations ($r = 0.84$ and 0.85, respectively). Therefore, results from Williams (1984) indicate that reductions in the offensiveness of swine wastes may parallel declines in BOD levels, as were noted in Lagoon 1 after conversion.

Selection of Odorous Compounds

Odorous emissions from manure consist of a complex array of compounds such as volatile fatty acids, indoles and phenols, ammonia, volatile amines, and volatile S-containing compounds (Zhu, 2000). Evaluation of odor-abating waste handling methods consist of sensory analysis by olfactometry with human subjects or analytical methods. The olfactometry approach is cumbersome for routine analysis because rather large odor panels are needed to obtain reliable and reproducible results (Gostelow et al., 2001). As an alternative, Spoelstra (1980) proposed the use *p*-cresol and volatile fatty acids concentrations as indicators of objectionable odors to evaluate odor-abating waste handling methods. Zahn et al. (2001a) made the case for using volatile fatty acids concentrations, specifically C2 through C9 chains, as indicators for decreased air quality. Their study also included phenol, *p*-cresol, 4-ethyl phenol, indole, and skatole. Air samples from lagoons showed a consistently lower concentration of volatile fatty acids, as well as phenol, *p*-cresol, 4-ethyl phenol, indole, and skatole levels in treatment lagoons with respect to manure storage basins. One potential problem for using volatile fatty acids as malodor indicators is that their volatility and concentration may be reduced under alkaline conditions (Spoelstra, 1980; Zhu, 2000). Because alkaline conditions (pH > 7.6) are typical of anaerobic lagoons in North Carolina (Bicudo et al., 1999), we assumed that other compounds less affected than volatile fatty acid by alkaline conditions (phenol, *p*-cresol, 4-ethyl phenol, indole, and skatole) might serve as more reliable malodor indicators in the traditional and converted lagoons.

Although there is not a single compound that serves as a reliable indicator of malodors from swine manure, the five key malodorous compounds (phenol, *p*-cresol, 4-ethylphenol, indole, and skatole) selected for our study are frequently cited as related to fecal matter (Spoelstra, 1977; Gralapp et al., 2001; Hobbs et al., 1995; Schiffman et al., 2001; Yasuhara et al., 1984; Zahn et al., 2001a). Table 2 shows some characteristics of these five selected malodor indicators including physical constants (octanol–water coefficient and water solubility) calculated using the EPI suite for Windows (USEPA, 2000). These five compounds are microbial degradation products produced in the digestive tract (Nagata and Takeuchi, 1990; Zhu, 2000). Phenol and *p*-cresol are degradation products of tyrosine and phenylalanine (Elsden et al., 1976; Ishaque et al., 1985), whereas 4-ethylphenol is a likely decarboxylation product of *p*-coumaric acid (van Beek and Priest, 2000). Indole and skatole are degradation products of tryptophan (Elsden et al., 1976). Indole, skatole, and *p*-cresol have very low odor

Table 2. Characteristic of malodorous compounds.

Compound	Odor characteristic	Odor threshold†	$\log_{10}(K_{ow})$‡	Solubility in water
		mg m^{-3}		ng mL^{-1}
Phenol	**aromatic, irritant**	**0.43**	**1.51**	**45.9×10^6**
***p*-Cresol**	**fecal**	**8.32×10^{-3}**	**2.06**	**$15{,}7 \times 10^6$**
4-Ethylphenol	**pungent**	**–**	**2.55**	**5.16×10^6**
Indole	**fecal/stench**	**1.55×10^{-5}**	**2.05**	**5.61×10^5**
Skatole	**fecal/nauseating**	**3.09×10^{-3}**	**2.6**	**1.88×10^5**

† Data from Schiffman et al. (2001).

‡ Octanol–water solubility coefficient and solubility in water estimated using EPI Suite for Windows (USEPA, 2000).

thresholds, so reduction in their levels by treatment may have an effect on odor quality. In addition, these five compounds follow the five following selection criteria for manure odor indicators (Spoelstra, 1980): (i) the components must be products of protein degradation; (ii) the components should be stable end products under normal conditions of waste storage; (iii) the formation of the components must reflect the kinetics of waste degradation; (iv) the components must respond in a representative way to environmental changes; and (v) the concentrations of the components must be easy to measure.

Extraction of Volatile Compounds from Water

We first tried a dynamic headspace sampling procedure for quantification of odor but results were not reliable. This procedure consisted of a collection chamber containing lagoon liquid and an air headspace that received purified air and passing the exhaust air through a column containing Super Q absorbent (Supelco, Bellefonte, PA). We were not able to quantify treatment differences in most compounds analyzed. Conditions such as relative humidity, temperature, air velocity, individual thresholds, and column trapping efficiency are all known factors that could have caused the poor results obtained with the headspace procedure. For this reason, we wished to develop a simplified approach to odor quantification that was independent of factors affecting odor dispersion by measuring concentration of selected odor compounds in water samples. In addition, water extraction allows direct comparison of the odors with water quality indicators commonly used in wastewater treatment monitoring.

We chose solid-phase extraction of the lagoon samples for the analysis of malodors. Although solid-phase extraction of organic compounds is a common technique, this method has not been used routinely for odor compound analysis of lagoon wastewater. We chose Tenax TA as the extraction polymer because of its low affinity for water and relatively high affinity for compounds such as phenol and *p*-cresol (Chen et al., 2001; Sato et al., 2001). Although we did not measure the efficiency of Tenax TA for the recovery of volatile compounds from water, preliminary extractions of lagoon samples showed that the selected compounds were retained on the Tenax column in that we were not able to detect any of the targeted analytes in re-extracted samples. Furthermore, we were able to note consistently clear differences in the concentration of the selected volatile compounds in the converted lagoon compared with the two conventional lagoons. Figure 2 shows chromatograms of solid-phase extracts of the converted waste lagoon and one of the conventional waste lagoons. The five odorous compounds selected for this study are all highly soluble in water, and we found them in concentrations well below the limit of their predicted solubility (Table 2). In addition to the five selected odor compounds, 12 additional compounds were identified (n-alkanes, unsaturated hydrocarbons, and brominated alkanes). Yields of these 12 compounds from Tenax-extracted water were below 10 ng mL^{-1} and did not significantly differ between the converted lagoon and conventional lagoons. However, the extraction method effectively showed relative differences in the concentrations of the selected malodorous compounds present in the converted lagoon and conventional lagoons. There was a marked (83%) reduction in identified malodorous compounds in water from the converted lagoon compared with liquid from the two conventional lagoons (Table 3). The reduction was especially marked in the case of *p*-cresol (83%), 4-ethylphenol (93%), and skatole (97%), all of which make important contributions to swine waste odors due to their distinctive odors and low detection thresholds. An independent analysis by a trained odor panel confirmed that odor dispersion from this production unit with the converted lagoon was greatly reduced when odor from the animal housing was subtracted from the total odor contribution of the production unit (Schiffman and Graham, 2004).

The remarkable reduction of selected malodor compound levels in the converted lagoon was due probably to the combined effect of discontinuing loading of liquid manure and storing excess clean water generated by the full-scale wastewater treatment system into the converted lagoon. This management reduced lagoon liquid strength by both reducing organic loads and improving aeration of lagoon liquid (Vanotti, 2004). Therefore, considerable reduction of anaerobic microbial breakdown of the amino acid precursors for malodor compounds were expected as indicated by declining TKN and NH_3–N values in the converted lagoon from May to October 2003 (Table 1). According to Williams (1984), the most reliable water quality applicable indicator for odor reduction in liquid swine manure is supernatant BOD, both during aerobic treatment and post-treatment storage. Similarly, we found that BOD was the best predictor for total odor concentration in lagoon liquid samples (Fig. 3). This relationship ($r^2 = 0.79$, $n = 8$, $P < 0.01$) shows that total odor concentration linearly decreased with the decrease in organic substrate (Fig. 3).

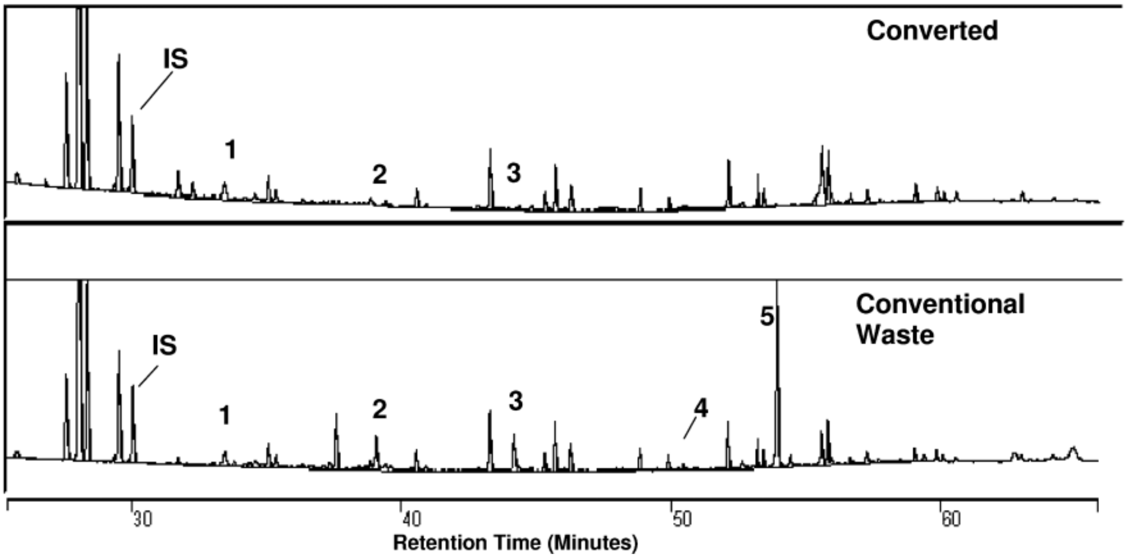

Fig. 2. Portions of chromatograms of liquid from converted lagoon and conventional anaerobic lagoon. Peak identities: IS, pentyl acetate internal standard. 1, Phenol. 2, *para*-Cresol. 3, 4-Ethylphenol. 4, Indole. 5, Skatole.

Table 3. Reduction of odor compounds in the liquid of converted lagoon as compared to conventional lagoons.

Compound	Converted Lagoon 1†	Conventional Lagoon 2	Conventional Lagoon 3	Odor compound reduction‡
	ng mL^{-1}			%
Phenol	**3.89 ± 1.42 a§**	**3.03 ± 1.50 a**	**9.02 ± 3.67 a**	**35.4**
***p*-Cresol**	**0.69 ± 0.60 b**	**4.38 ± 3.86 a**	**3.73 ± 2.20 ab**	**83.0**
4-Ethylphenol	**0.24 ± 0.15 b**	**2.76 ± 1.78 a**	**3.76 ± 2.22 a**	**92.6**
Indole	**1.06 ± 0.40 a**	**0.86 ± 0.37 a**	**1.69 ± 1.17 a**	**16.9**
Skatole	**0.67 ± 0.35 b**	**15.2 ± 4.18 a**	**32.9 ± 10.3 a**	**97.2**
Total identified	**6.64 b**	**26.44 a**	**51.24 a**	**82.9**

† Data are means ±SE for the period September–October 2003 ($n = 5$).
‡ Odor compound reduction compares concentration in Lagoon 1 with the average concentration of Lagoons 2 and 3.
§ Means followed by the same letter are not significantly different by a least significant difference test of log-transformed values ($\alpha = 0.05$).

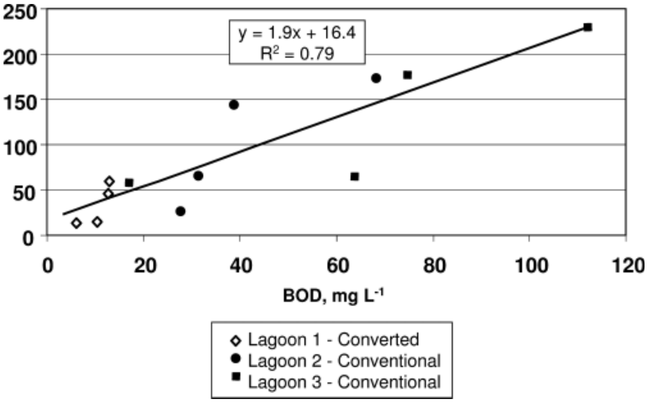

Fig. 3. Linear regressions of biological oxygen demand of the lagoons vs. concentrations of malodorous compounds in water samples.

Therefore, a decline in organic substrate for microbial growth probably paralleled a decline in the pool of precursors to malodor compounds to the point where differences in odor compound concentrations of the converted and conventional waste lagoon were highly significant.

CONCLUSIONS

Differences in water quality characteristics among lagoons were observed starting in 2003 after manure flush to Lagoon 1 was halted and 100% of the manure generated was processed through the treatment plant. Later, analysis of water showed a marked (83%) reduction in total concentration of malodorous compounds in water from the converted lagoon compared with liquid from the two conventional lagoons. The reduction was particularly marked in the case of *p*-cresol, 4-ethylphenol, and skatole, all of which make important contributions to swine waste odors due to their low detection thresholds and distinctive odors.

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