Odorous compounds from treated pig manure

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Yu, J.C., Isaac, C.E., Coleman, R.N., Feddes, J.J.R. and West, B.S. 1991. **Odorous compounds from treated pig manure.** Can. Agric. Eng. **33**:131-136. Three treatments of pig manure slurry were evaluated for their effectiveness in the reduction of odorous compounds associated with stored slurry. These treatments included oligolysis (an electrolytic process), Bio-gest™ and Nature-aid™ (proprietary odour reducing agents). In addition, a control was included as a reference to the three treatments. Manure was stored in 205 L bioreactors for an eight-week period. Volatile organic acids were found to be highest and the ammonia levels were found to be the lowest in the liquid phase of the Bio-gest and Nature-aid treatments when compared to the oligolysis and control treatments. Little difference between the measured parameters (with the exception of propionic acid) was observed between the control and oligolysis treatment. Volatile organic acids, and phenolics and H_2S were measured in the gas phase. The gas generated from the oligolysis treatment contained 94% less H_2S than gas from the other treatments and odour was reduced. There were no statistical differences among the treatment means for volatile organic acids and phenolics for the gas phase of treated pig manure.

On a évalué trois méthodes de traitement du lisier de porcs pour leur efficacité à réduire les odeurs associées aux boues entreposées : l'«oligolyse» (un procédé électrolytique), le Bio-gest[md] et le Nature-aid[md] (des agents de réduction brevetés). En outre, un traitement de contrôle servait de référence aux trois autres. Le lisier a été entreposé dans des bioréacteurs de 205 L pendant huit semaines. On a découvert que les acides organiques volatils étaient les plus élevés et que les niveaux d'ammoniac étaient les plus bas à la phase liquide des traitements Bio-gest et Nature-aid, comparativement aux traitements d'oligolyse et de contrôle. Les paramètres mesurés (à l'exception de l'acide propionique) présentaient peu de différences entre le traitement de contrôle et d'oligolyse. Les acides organiques volatils, les dérivés phénoliques et le H_2S ont été mesurés à la phase gazeuse. Le gaz produit par l'oligolyse contenait 94 % de H_2S de moins que le gaz des autres traitements, et l'odeur était réduite. On ne releva aucune différence statistique entre les moyens de traitement des acides organiques volatils et des composés phénoliques à la phase gazeuse du lisier de porc traité.

INTRODUCTION

Nuisance odour emanating from stored liquid pig manure continues to be a major concern for intensive confinement operations. Various techniques have been applied to reduce or eliminate odour production. Manure odour control strategies can be classified into four categories, i.e., physical treatment, chemical treatment, biological treatment and electro-chemical treatment. Physical treatment focuses on the control of the physical conditions of the manure to reduce the release of odours, such as lagoon covers or mechanical aeration that prohibits anaerobic conditions (Gunn and Kolstee 1974). Chemical treatments use chemicals to maintain unfavorable conditions for releasing odours, such as the addition of ozone to the manure, or unfavorable conditions for bacterial activity (Barth and Hill 1976). Biological treatments typically employ aeration for aerobic processes (Williams 1984a) and anaerobic fermentation processes (Welsh et al. 1976). Electrochemical treatment supplies ionized metal through electrical current within the liquid manure for the odour control (Chiumanti et al. 1987). However, the most common type of treatment used in pig operations is anaerobic in nature and results in the production of odorous compounds. Technology for odour control during anaerobic treatment is available in the form of additives and oligolysis.

Odour causing compounds have been quantified using various extraction and detection methods. Extraction and concentration have typically used dichloromethane and flash distillation (Yasuhara and Fuwa 1979) as well as vacuum distillation at -80°C followed by extraction of the distillate into diethyl ether and quantified by gas chromatography (GC) and mass spectrometry (MS) (Yasuhara and Fuwa 1983). Detection of odorous compounds using various chemical techniques and technologies does not necessarily indicate the overall odour or smell as subjectively perceived by any person. The relationship between the offensiveness of swine slurry odour and various indicators within the slurry have previously been investigated. Using linear regression analysis, BOD, Total Organic Acids, Total Indoles and Phenols, Volatile Organic Acids (VOA) and Sulfide were found to correlate highly with odour offensiveness (Williams 1984b). From a subjective analysis of defined mixtures of known odorous pig slurry compounds, Yasuhara (1980) found that carboxylic acids, VOA and sulfides were the main contributors to odour.

Current technology for odour control during anaerobic treatment is the use of commercial additives and oligolysis. Two commercially available manure additives, Bio-gest™ and Nature-Aid™ have been marketed for odour control for more than twenty years. An electro-chemical treatment, oligolysis, has also been used for this purpose. In this study, oligolysis, Bio-gest and Nature-aid were evaluated for odour causing compounds along with a control that served as a reference. Odour causing compounds such as volatile organic acids, phenols, ammonia and sulfides were measured in the liquid and gas phases.

EXPERIMENTAL FACILITIES AND PROCEDURES

Animals

Eighteen pigs (3 groups of 6) were housed in pens with rubber covered solid floor pens with meshed dunging areas at one end. Feces, urine and water spillage were collected in a sealed

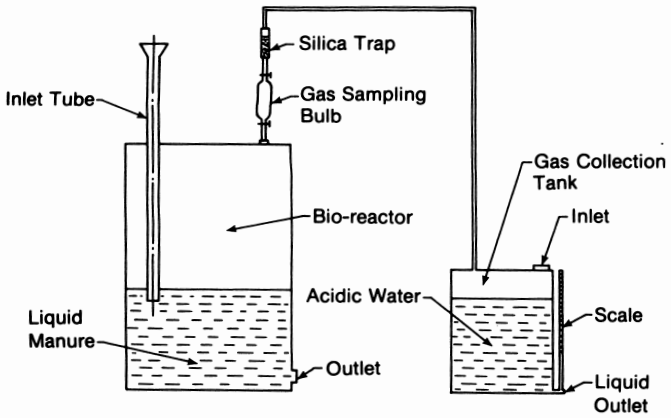

Fig. 1. Bioreactor (plastic barrel) and gas collection tank.

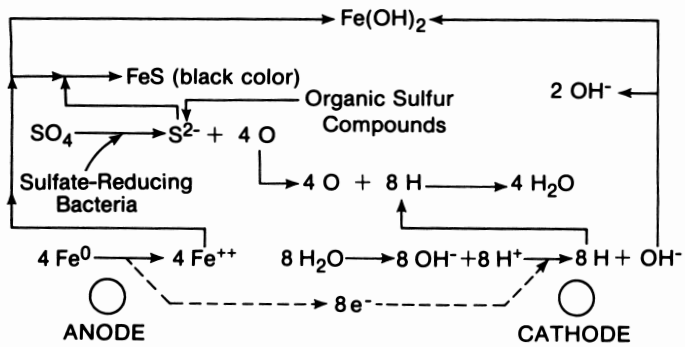

Fig. 2. A graphical presentation of the oligolysis treatment (adapted from Barber and McQuitty 1974).

container beneath the dunging area. Manure from the three containers was collected every second day, combined, mixed and separated into twelve equivalent fractions (4 treatments x 3 replicates) and introduced to the bioreactors. The initial mean weight of the pigs was 50 kg and the mean final weight was 95 kg.

Manure was stored in 12 bioreactors that were closed plastic barrels with a volume of 2.36 m^3. Their total cross sectional areas were similar to that of a slatted floor over a storage pit storing manure for 18 pigs (50 to 95 kg). The configuration of the bioreactor is shown in Fig. 1. Inlet tube height was adjustable so that the end of the tube could be located about 100 mm below the liquid level before adding manure to avoid gas loss to the inlet. The location of the tube insured that the gas exited the gas outlet. A thermistor was located approximately mid point within the liquid contents.

Experimental design

The experiment consisted of three treatments plus a control. Each treatment had three replicates. The treatments were as follows:

Control: a conventional anaerobic fermentation process of pig manure with no modification that simulated a typical storage pit.

Oligolysis: an electrolytic process in which two iron bar electrodes were placed in the liquid manure 30 cm apart in the bioreactor. A DC current (12 V, 0.5 Amp) was passed through the slurry using a automobile battery charger. The three bioreactors were electrically connected in series.

Bio-gest: Bio-gest was added to the fresh slurry at an equivalent rate of 1 kg of dry material per 30 m^3 of manure (approximately 33 ppm). Bio-gest is a light brown-colored dry powder. No information was available on the formulation of Bio-gest.

Nature-aid: Nature-aid was added to the combined fresh slurry at a rate of 1 kg/250 m^3. Nature-aid is a liquid with a dark brown color. No information was available on the formulation of Nature-aid.

The experiment was originally set up to analyze both the liquid and gas phase concurrently. The silica traps used to absorb the VOA, phenolics and H_2S did not collect a large enough sample to obtain an accurate measurement; consequently, the experiment was repeated under the same environmental conditions and sampling procedure to evaluate only the gas phase. Prior to the experiment the bioreactors were filled with manure and drained to within 10 cm of the bottom, similar to filling and draining pits beneath slatted floors. Thus the first part of the experiment deals with the liquid phase while the second part of the experiment deals with the gas phase. Liquid and gas samples were obtained on a weekly basis from each bioreactor. The samples were kept in plastic bottles or gas traps at 4°C prior to extraction or analysis.

Selection of odour causing compounds

More than 100 odour causing compounds have been identified by researchers in the past. However, to measure all of these compounds concurrently is not feasible due to both technical and economical constraints. Therefore, odour causing compounds were selected that represented odour from treated pig manure most closely.

Organic acids are prevalent contributors to pig manure odours. Vapor pressures decrease with increasing molecular weights. The smaller members of a specific series are more evident in the ambient air. Therefore, eight VOAs of lower molecular weights were selected for this study. These were acetic, propionic, isobutyric, butyric, isovaleric, valeric, caproic and heptanoic acids. Phenol, another contributor, was found to have a rather pleasant and sweet odour. Although it does not contribute any offensiveness to the odour of pig manure, it most certainly contributes to the total odour (Yasuhara and Fuwa 1977). Previous research conducted in the Netherlands showed p-cresol concentration to have the highest correlation with odour concentration (Schaefer 1980). Indole and skatole have fecal smells that contribute to the total odours of pig manure. Ammonia was another well-known odour causing compound. Therefore, phenol, p-cresol, indole, skatole and ammonia, along with the organic acids above, were selected for this study.

As a matter of interest, the thirteen compounds selected above were obtained commercially in their pure form and placed in a closed container with the lid sealed. The odour from this container when uncovered was perceived to be similar to that of anaerobically treated pig manure.

LABORATORY PROCEDURES

Liquid phase

Approximately 40 mL of sample were centrifuged (3000 g's) for 10 minutes. Exactly 25 mL of supernatant were removed and placed in a 250 mL round bottom flask containing 20 g NaCl. Following acidification of the mixture to pH 2 using 50% H_2S0_4, 30 mL of diethyl ether were added. The contents of the flask were heated and refluxed for 1 hour (condenser temperature was 4 to 8°C). After cooling, the contents were partitioned (using the neck) in a 50 mL volumetric flask, the ether layer transferred to another 50 ml volumetric flask along with 50 µL of 1,4 Dioxane (internal standard) followed by addition of diethyl ether up to the mark.

Extraction efficiency

Not all of the identified odour causing compounds extracted at the same efficiency thus a correction was required. The correction factors were determined as follows. Triplicate samples of slurry were refluxed and extracted three times followed by discarding the ether layer to remove all traces of the compounds of interest. The extracted slurry was spiked with all purified compounds of interest (at appropriate concentrations), reextracted and assayed for odour components by gas chromatography. Analysis of an equal amount of pure standard mixture was performed and compared to the spiked sample to determine extraction efficiency.

Gas chromatography

All gas chromatographic analyses were performed using a Hewlett Packard 5840A Gas Chromatograph complete with 18835B capillary inlet system.

Instrument conditions were:

a) Column: Nukol fused silica wide bore column, 0.53 mm ID, 30 m in length, 0.5 m film thickness (Supelco Ltd.)
b) Injection System: Split mode using He carrier gas
c) Detection: Flame Ionization detector (FID, single channel mode)
d) Quantification Method: Internal standard method using 1,4 dioxane as a reference peak.

Operating conditions were:

a) Capillary system set up:

Column flow:	7.2 mL/min
Column head pressure:	34.5 kPa
Split flow:	100 mL/min
Split Ratio:	107.2/7.2 = 14.9/1
FID Make-up:	85 mL/min (total He flow including column flow)
Hydrogen:	50 mL/min (regulator at 163.9 kPa)
Septum purge:	5 mL/min
Air Flow:	240 mL/min (regulator at 207.0 kPa)

b) Separation conditions:

Temperature gradient: 2 min at 125°C then 125°C to 220°C at 5°C/min and hold for 10 min.
Injection temperature: 125°C
FID temperature: 250°C
Chart speed: 10 mm/min.
Zero: 10%
Attenuation: 4
Slope Sensitivity: 0.1

Ammonia determination

Ammonia was determined using a specific ammonia ion electrode (Orion Research Inc., MA) and referenced with appropriate NH_4Cl standards. All dilutions of the liquid phase of the slurry were made with ASTM Type II water.

Gas phase

Silica traps were used to absorb the VOAs and phenolics but not NH_3 in the gas phase. The silica traps were constructed using glass tubes with a length of 150 mm and an internal diameter of 10 mm. The tubes were loosely packed with 5.0 g of activated silica gel. The activation process involved drying the silica for 1 to 2 hours at a temperature of 160°C. The silica traps were connected to the bioreactor for various time periods. During this time the gases generated from the bioreactor passed through the silica trap and the NH_3 passed through to the water displacement tanks which contained acidified water.

The gas samples for H_2S analysis were obtained by using glass gas sampling bulbs (Fig. 1). Before each sampling, the bulbs were cleaned using chromic acid, water and acetone and were flushed with nitrogen gas. After being connected to the bioreactors, the valves of the bulb were opened and bioreactor gas flowed through the bulb for 24 hours. This allowed at least 10 volumes of gas to pass through the bulb before the sample was taken by closing the inlet and outlet valves. After sampling, the bulbs were retained at room temperature so that there was no condensation formed which might alter the contents of the sample. Ammonia was analyzed by determining the NH_3 content of the acidified water from the gas collection tanks.

After disconnection from the bioreactor, silica gel was placed in an 18 mL bottle. Ten µL of pentanol (internal standard) and 10 mL of deionized distilled water were added and the bottle was sealed with a teflon lined cap. The mixture was shaken on a wrist action shaker for 1 hour, then centrifuged at 5000 g's for 10 min. The supernatant was transferred to a 5 mL Mininert vial to which was added 25 µL of concentrated formic acid. The contents were analyzed by GC under conditions previously described. The sample for H_2S analysis was directly taken from the injection port on the glass bulb. Another GC equipped with a flame photometric detector (FPD) was used for H_2S determination. Separation of sulfur-containing compounds was carried out on a 3m x 3mm ID Teflon column packed with 80/100 mesh porepack QS (Supelco Canada Inc., Oakville, ON).

Ammonia

Ammonia in the gas phase was removed by acidified water (pH 4.5 with phosphoric acid). As the exhausted gases from the bioreactors passed through the acidified water, ammonia was ionized to NH_4+ and trapped in solution. The pH of the liquid samples was increased to 10 by the addition of NaOH and the ionized ammonium was converted back to ammonia. The ammonia content of the solution was determined using an ammonia specific ion electrode and comparing the potentials to those of known standards.

Table I. Mean concentrations of compounds in the liquid pig manure, g/L

Compound	Treatment			
	Control	Oligolysis	Bio-gest	Nature-aid
Acetic acid	5.672	5.266	6.282	7.413
Propionic acid	3.186	2.957	3.450	3.918
Isobutyric acid	0.399	0.425	0.622	0.558
Butyric acid	2.921	2.804	3.538	3.951
Isovaleric acid	0.752	0.812	0.956	1.054
Valeric acid	0.577	0.524	0.654	0.703
Caproic acid	0.135	0.166	0.264	0.386
Heptanoic acid	0.020	0.026	0.034	0.066
VOA*	13.622	12.980	15.800	18.048
Phenol	0.029	0.031	0.036	0.037
p-Cresol	0.095	0.077	0.099	0.140
Phenols*	0.124	0.104	0.135	0.177
Indole	0.004	ND	ND	0.066
Skatole	0.039	0.012	ND	0.011
Ammonia	6.925	6.773	6.006	5.715

* These values are the summation of Volatile Organic Acids and Phenols in the liquid phase, respectively.
ND not detected

Table II. Analysis of variance

Compound	Treatment
Acetic acid	***
Propionic acid	***
Isobutyric acid	NS
Butyric acid	***
Isovaleric acid	***
Valeric acid	***
Caproic acid	***
Heptanoic acid	***
Phenol	*
p-Cresol	**
Indole	NS
Skatole	NS
Ammonia	***

*** significant at 0.01 level of probability
** significant at 0.05 level of probability
* significant at 0.10 level of probability
NS not significant

RESULTS AND DISCUSSION

Liquid phase

An assumption was made that all treatment processes were typical sequence batch anaerobic reactors to which all treatments could be reliably intercompared. Mean concentrations (W/V) of the thirteen compounds in the liquid phase of manure over an eight-week experiment for each treatment are tabulated in Table I. Analysis of Variance and Multicomparison of Means (Student Newman Keuls (SNK) Test) were carried out on the experimental data and summarized in Table II and Table III. Since indole and skatole were either not detected or present at very low levels (Table I) in the treated pig manure slurry, indole and skatole were not considered in the discussion.

The statistical results in Table II indicate that the treatments were significantly different at $P<0.001$ for the concentrations of nine compounds in the liquid manure. Phenol and p-cresol were significantly different at levels of $P<0.05$ and $P<0.01$, respectively.

The results in Table III indicate that the control and oligolysis treatments were not significantly different with an exception of propionic acid. However, there were statistical differences between the control and the other two additive treatments, specifically in the medium molecular weight VOA (Table III).

The control treatment produced the lowest mean concentrations of isobutyric, isovaleric, caproic, heptanoic acids and phenol. However the highest levels of ammonia were found in the control treatment. The second lowest mean concentrations of acetic, propionic, butyric, valeric acids and p-cresol were observed in the control treatment (Table I).

Oligolysis treatment yielded the lowest mean concentrations of acetic, propionic, butyric, valeric acids and p-cresol. The second lowest mean concentrations of isobutyric, isovaleric, caproic, heptanoic acids and phenol also were observed in the control. The second highest ammonia level was found in the liquid phase. The SNK test results in Table III indicated that there were no differences between the control and oligolysis treatments for the mean concentrations of all compounds determined in the liquid phase.

All mean concentrations in the Bio-gest treatment were higher than both the control and oligolysis treatments, with the exception of ammonia. The highest level of isobutyric acid was found in the Bio-gest treatment. Compared to the control

Table III. Student Newman Keuls (SNK) test for testing difference among means

Compound	Treatment			
	Control	Oligolysis	Bio-gest	Nature-aid
Acetic acid	ab	b	a	c
Propionic acid	a	b	c	d
Isobutyric acid	a	a	a	a
Butyric acid	a	a	b	b
Isovaleric acid	a	a	b	b
Valeric acid	a	a	b	b
Caproic acid	a	a	b	b
Heptanoic acid	a	a	a	b
VOA*	A	A	B	C
Phenol	a	a	a	a
p-Cresol	a	a	a	b
PHENOLS**	A	A	A	B
Ammonia	a	a	b	b

Note: Different letters along rows indicate difference ($P<0.05$)

* Volatile Organic Acids grouped

** Total of Phenol plus p-Cresol

and oligolysis treatments, the concentrations of organic acids were increased in the Bio-gest treatment; however, the mean concentration of ammonia in the liquid phase decreased.

The mean concentrations of nine compounds (acetic, propionic, butyric, isovaleric, valeric, caproic, heptanoic acids, phenol and p-cresol) observed from the liquid phase were highest in the Nature-aid treatment (Table 1). Whereas, ammonia in the liquid phase of this treatment was lowest among all four treatments.

The VOAs determined in the liquid phase were collectively considered as was phenol and p-cresol. The mean values and comparisons for VOAs and Phenols are included in Table I and Table III, respectively. The data in Table III indicate that the mean concentrations of the VOAs are all significantly different among treatments except between the control and oligolysis treatments. The mean concentration of Phenols in the Nature-aid treatment is significantly different from the other three treatments, whereas, the differences among the control, oligolysis and Bio-gest treatments are not significant.

In general terms, statistically significant higher concentrations of volatile organic acids and phenols were determined in both additive treatments compared to both the control and oligolysis treatments. However, the opposite trend was observed with levels of ammonia (Table I). These observations may relate to odour production since in the additive treatments the odour causing compounds may not be as readily released to the gas phase above the liquid manure and thus not contribute as much to perceived odour levels in the latter phase. Temperature and pH value of the liquid pig manure were also measured during the experiment. There were no statistical differences in these two parameters between the treatments. The observed temperatures and pH were typically 19.5 °C and 7.0, respectively.

Gas phase

In this study, the compounds were analyzed collectively, i.e., the eight organic acids were considered as VOA, phenol and p-cresol as phenolics. Also, hydrogen sulfide and NH_3 were analyzed in the gas phase. The mean concentrations of the odour causing compounds over the entire experiment are summarized in Table IV. Ammonia concentrations in the gas phase are not reported in the table, since the ammonia concentration values were at the lower detectable limits. Problems were encountered throughout the experiment in bubbling the gas through the acidified water in the gas collection tank.

Analysis of Variance and Multiple Comparison of Means were carried out on the experiment data. The results indicated that the treatments were only significantly different ($P<0.001$) in the H_2S production. The total VOA and phenolics in each treatment were not significantly different at any probability. The Student Newman Keuls (SNK) test showed that the mean concentrations of H_2S in the oligolysis treatment were significantly different from the other three treatments.

The removal of H_2S from the gas phase of the treated pig manure was 94.9% relative to that contained in the control treatment (Table IV). There were no statistical differences between the control, Bio-gest and Nature-aid in relation to the mean concentrations of H_2S. Hydrogen sulfide is one of the most odorous components produced in anaerobically treated pig manure. Table IV illustrates that H_2S was predominant among the odour causing compounds in the gas phase. The concentrations of the other odour causing compounds were merely several ppm, whereas, H_2S was thousands of ppm in the oligolysis, Biogest and Nature-aid treatments. Therefore, extra attention should be paid to H_2S in the pig manure odour. The reason for the removal of H_2S in the oligolysis treatment may be attributed to the following possibilities:

1. Small quantities of ferrous ions dissolved by electrolysis may inhibit microbial activity (Chiumenti et al. 1988).
2. The reaction of H_2S with ferrous ions resulting in insoluble FeS and thus the removal of H_2S (Fig. 2).

Table IV. Concentrations of various compound groups in the gas phase

Compound	Mean concentration in each treatment[1]			
	Control	Oligolysis	Bio-gest	Nature-aid
VOA[2]	7.9	3.3	3.2	2.2
Phenolics	1.1	1.4	3.7	1.2
H^2S	8020	410	8480	7740

[1] Mean of three replicates
[2] Volatile Organic Acids

The black color of the treated manure from oligolysis treatment suggested the formation of FeS.

CONCLUSIONS

The following conclusions may be drawn from this study:

1. Oligolysis treatment yielded no statistical differences from the results of the control treatment with the exception of propionic acid being lower in the oligolysis treatment in the liquid phase.

2. Bio-gest and Nature-aid increased the concentration of organic acids in the liquid phase of treated pig manure indicating an increase in microbial activity.

3. Bio-gest and Nature-aid appeared to reduce the level of ammonia in the liquid pig manure.

4. Oligolysis treatment removed hydrogen sulfide in the gas phase of treated pig manure by 94.9% and reduced the odour nuisance of the treated pig manure.

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