

# **On-farm pilot-scale testing of black ultraviolet light and photocatalytic coating for mitigation of odor, odorous VOCs, and greenhouse gases**

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## **Abstract**

Technologies for controlling gaseous emissions of livestock is of interest to producers, the public, and regulatory agencies. In our previous lab-scale study, the use of a photocatalytic coating on surfaces subjected to black ultraviolet light reduced emissions of key odorant compounds relevant to the livestock industry. Thus, an on-farm pilot-scale experiment was conducted at a commercial swine barn to evaluate a photocatalytic coating on surfaces subjected to ultraviolet light under field conditions. A flow-through reactor was constructed with a TiO<sub>2</sub>-based photocatalytic coating on the interior surfaces and black ultraviolet light fixtures. The reactor was deployed in a room downstream of the entire swine barn exhaust. Gas samples were collected from three sampling ports in the reactor, one at the inlet (control), the midpoint (half treatment) and the outlet (treatment). Compared to the control, significant reductions in emissions were observed for *p*-cresol (22%), odor (16%) and nitrous oxide (9%). A significant increase in carbon dioxide (3%) was also measured. Results show that the TiO<sub>2</sub>-based photocatalytic coating and black UV light are effective in mitigating odor, a key VOC responsible for downwind swine odor, and one important greenhouse effect gas when subjected to swine barn exhaust.

## ***Keywords***

Odor, ultraviolet light, photocatalysis, swine manure, emissions control.

## **1. Introduction**

Mitigation of gaseous emissions that are the unwanted side effect of pork production is of importance for improving sustainability of the industry. Emissions of volatile organic compounds (VOCs) pose odor nuisance to the local air quality. Emissions of greenhouse gases (GHG; i.e., CO<sub>2</sub>, CH<sub>4</sub>, N<sub>2</sub>O) are of concern due to their contribution to global climate change. Emissions of odorous ammonia (NH<sub>3</sub>) and hydrogen sulfide (H<sub>2</sub>S) are also of concern due mainly to quantity and toxicity, respectively.

Technologies explored for mitigating such emissions were summarized in Maurer et al. (2016) and the scientific literature database on the Iowa State University Extension and Outreach website (Air Management Practices Assessment Tool; AMPAT, 2016). The mitigation technologies fall in to twelve categories; barriers (Ikeguchi et al., 2003), biofilters (Dumont et al., 2014; Chen et al., 2012; Hansen et al., 2012; Hoff et al., 2009; Chen et al., 2009; Mann et al., 2002), chimneys (Kai et al., 2003; Mortensen et al., 1995), diet manipulation (Eriksen et al., 2010; Amon et al., 1995), electrostatic precipitation (Nicolai et al., 2008; Ritz et al., 2008), landscaping (Hernandez et al., 2012; Parker et al., 2012), oil sprinkling/spraying/additives (Kalus et al., 2017; Maurer et al., 2017a, 2017b, 2017c; Blanes-Vidal et al., 2008; Paszek et al., 2001; Zhang et al., 1996), ventilation (Jacobson et al., 2008), scrubbers (Melse et al., 2012; Aarnink et al., 2011), siting (Hoff et al., 2008; Jacobson et al., 2005), urine/feces segregation (Koger et al., 2014; von Bernuth et al., 2005), biogas collection/purification (Lin et al., 2013) and ultraviolet

(UV) light (Costa et al., 2012; Guarino et al., 2008; Zhu et al., 2017). Ultraviolet light and photocatalysis research has demonstrated significant reductions in odor and VOCs (Yang et al., 2008; Yang et al., 2007. Liu et al., 2015), NH<sub>3</sub> (Rockafellow et al., 2012), and GHGs emissions (Costa et al., 2012; Guarino et al., 2008) in lab- and farm-scale studies.

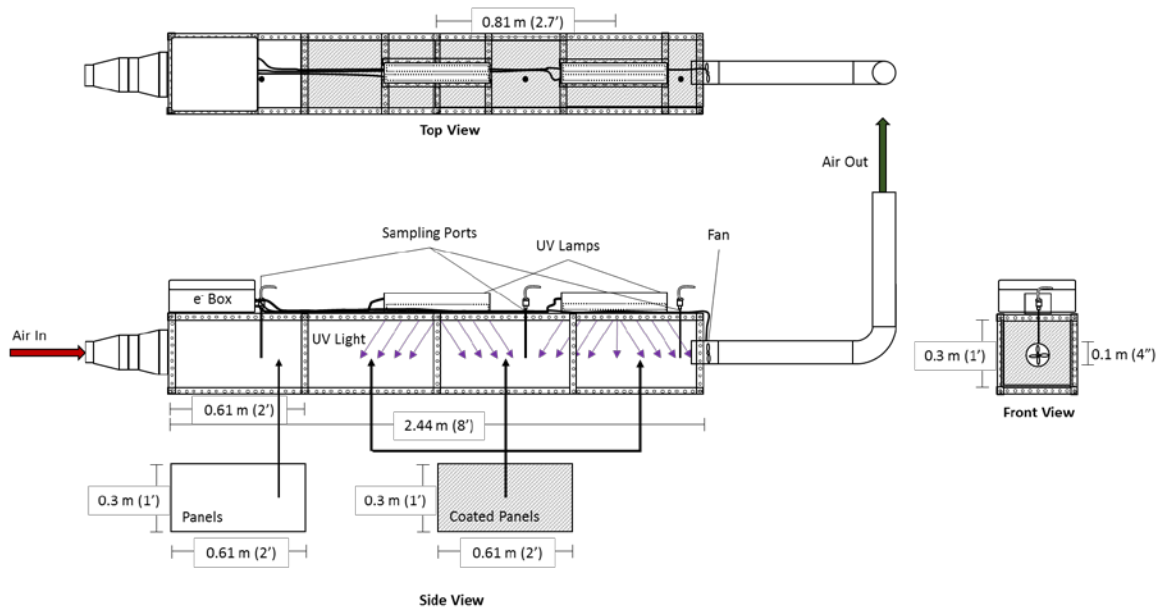
In our previous lab-scale study (Zhu et al., 2017), the use of a new TiO<sub>2</sub>-based special photocatalytic coating on surfaces subjected to black ultraviolet light reduced emissions of key odorant compounds relevant to the livestock industry. Dimethyl disulfide (DMDS), diethyl disulfide (DEDS), dimethyl trisulfide (DMTS), butyric acid, p-cresol, and guaiacol were selected as model odorants. The performance of the new catalyst at 10 µg·cm<sup>-2</sup> was comparable to that of conventional TiO<sub>2</sub> (P25, Evonik) at 250 µg·cm<sup>-2</sup>. The odorant reduction ranged from 100.0 ± 0.0% to 40.4 ± 24.8% at a treatment time of 200 s, simulating wintertime barn ventilation. At a treatment time of 40 s (simulating summertime barn ventilation), the reductions were lower (from 27.4 ± 8.3% to 62.2 ± 7.5%). The swine dust layer on the catalyst surface blocked 15.06 ± 5.30% of UV<sub>365</sub> and did not have a significant impact ( $p > 0.23$ ) on the catalyst performance.

Thus, the motivation for this experiment was to scale up the use of new TiO<sub>2</sub>-based special photocatalytic coating to pilot conditions. This study aimed to evaluate the on farm pilot-scale efficacy of UV photocatalytic reactor in reducing gaseous emissions using real swine barn exhaust air. Results are needed to inform the design of future farm-scale work, where the barn interior walls will be covered with the photocatalyst and foul air will be passively treated as it moves through the barn.

## **2. Materials and Methods**

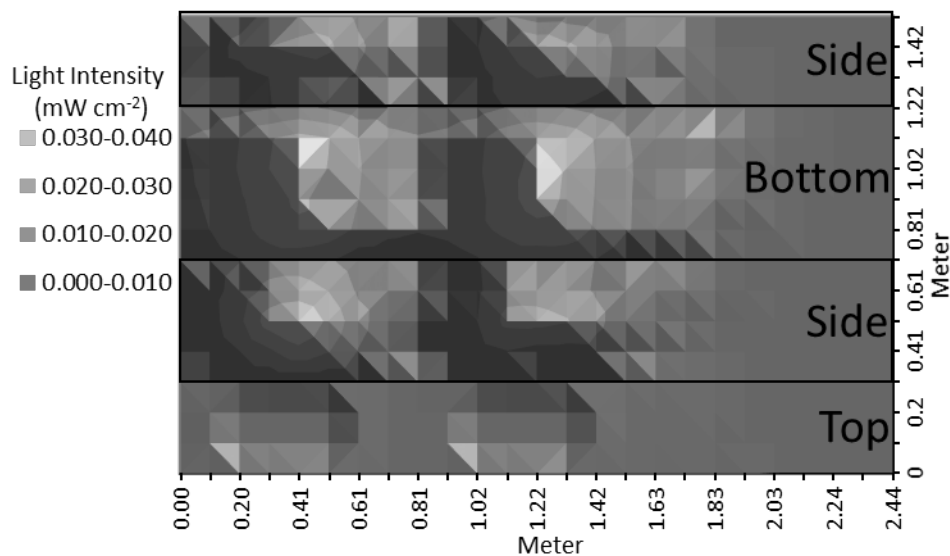
### *2.1 Experimental reactor setup*

The flow-through reactor (2.44 x 0.3 x 0.3 m) was designed and assembled for all experiments. The frame was constructed of plated steel slotted angle (Lowes, Mooresville, NC, USA) (Figure 1) to support interchangeable wall panels. The panels (0.3 x 0.61 x 0.002 m) were constructed of embossed white fiberglass reinforced plastic wall panel (Lowes, Mooresville, NC, USA), i.e., the common type of interior wall material used in livestock barns. The reactor air inlet and outlet was constructed from 0.20 m diameter to 0.15 m diameter duct reducer and a 0.15 m diameter to 0.10 m diameter duct reducer, respectively (Lowes, Mooresville, NC, USA). A small fan was installed in a 0.10 m diameter steel axial duct (Lowes, Mooresville, NC, USA) at the outlet to draw air inside reactor and to remove UV-treated air. The fan had a a dimmer switch to achieve a desired air flows and therefore a controlled treatment time consistent with typical air exchange rates inside mechanically-ventilated barns. The wind tunnel was illuminated with four 15W black light XX-15A UV-A lamps (Spectroline, Westbury, NY, USA) with a major output at 365 nm. PureTi Clean coating was applied to all the panels of the last section (1.83 m from the air inlet) of the reactor at 0.5%,  $\sim 10.8 \text{ mL}\cdot\text{m}^{-2}$ .



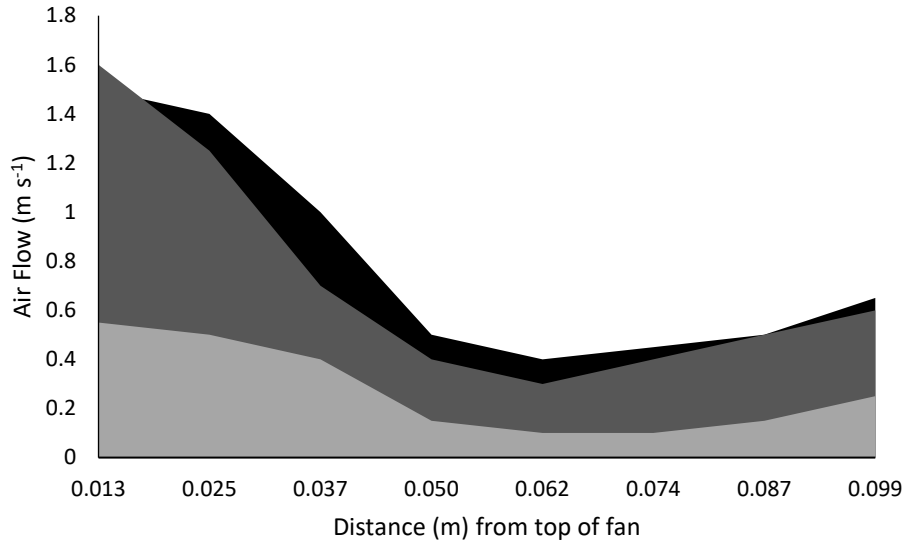
**Figure 1.** Schematic of UV photocatalytic reactor for on-farm experiments.

The effective dosage of UV<sub>365</sub> was mapped on the interior surfaces of the wind tunnel (Figure 2). The mapping was done using a ILT 1700 radiometer (International Light Technologies, Peabody, MA, USA) equipped with a X-ray diffraction (XRD) 340B UV-A detector (International Light Technologies, Peabody, MA, USA).



**Figure 2.** UV<sub>365</sub> effective dosage surface map.

The air velocity inside the reactor the fan was mapped (Figure 3) to control the treatment time of the barn air to the UV and photocatalytic coated panels. The air velocity was measured using a hot wire anemometer (Extech, Nashua, NH, USA). Settings 1, 2 and 3 resulted in a estimated treatment times of 26.2 sec, 29.2 sec, and 76.2 sec, respectively. With these calibration results, the treatment time at 2.5 setting used for all experiments was estimated at ~47.2 sec, i.e., consistent with typical warm season air exchange rates in a typical mechanically-ventilated barn in Midwest U.S.

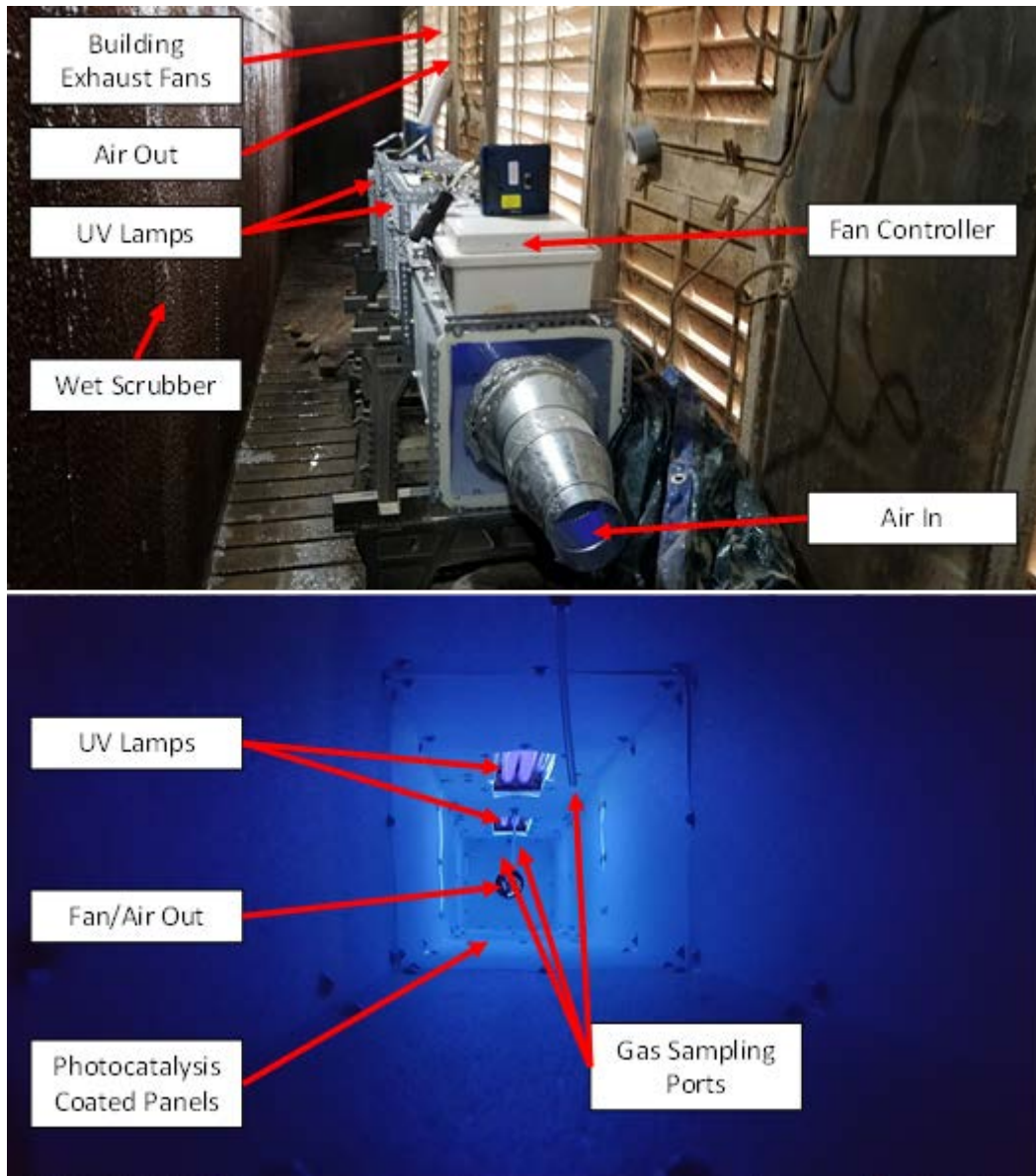


**Figure 3.** Mapping reactor airflow for estimation of mean treatment time. Black: setting 1, Gray: setting 2 and Light Gray: setting 3.

On farm pilot-scale testing was conducted at a commercial swine production facility. The research was conducted in a 1200 pig capacity, deep pit manure storage type, swine barn equipped with a wet scrubber on the barn exhaust. The wind tunnel was setup in the room after the wet scrubber in the swine barn exhaust stream (Figure 4).

Measurement of  $\text{NH}_3$  and  $\text{H}_2\text{S}$  concentrations, temperature and RH were conducted in real-time. Gas samples for GHGs and odorous VOCs were collected and subsequently analyzed in the lab. Emission rates were calculated as a product of measured gas concentrations and the total airflow rate through the wind tunnel, adjusted for standard conditions and dry air using collected environmental data. Percent RH was monitored via an 850071 Environmental Quality meter (Sper Scientific, Scottsdale, AZ, USA). Volatile organic compounds were measured three days with the uncoated panels (UV treatment only, i.e., photolysis) and five days after the panels had been coated with PureTi Clean photocatalytic coating (treatment can be attributed to both

photolysis and photocatalysis). Odor samples were collected three days after the panels had been coated. Ammonia and hydrogen sulfide were measured four days with uncoated panels and five days once the panels were coated. Methane, CO<sub>2</sub> and N<sub>2</sub>O were measured three days with uncoated panels and 4 days after the panels had been coated.



**Figure 4.** On-farm UV photocatalytic reactor setup. View from the air inlet (top). Experiments tested the effects of black light on odorous volatile organic compounds,

odor, GHGs, ammonia and hydrogen sulfide. Thin film of photocatalyst (bottom) covered plastic material typically used for indoor barn wall cover.

### *2.2 Volatile Organic Compounds (VOCs)*

Air samples for VOC measurement were collected using 1 L glass gas sampling bulbs (Supelco, Bellefonte, PA, USA). Air samples were taken using a portable vacuum sampling pump (Leland Legacy; SKC Inc., Eighty Four, PA, USA) with a set flow rate of 5 L min<sup>-1</sup> for 1 min, and analyzed within two days. Chemical analyses of swine odorants were completed using a solid phase microextraction (SPME) (50/30um DVB/CAR/PDMS; 2 cm-long fibers, Supelco, Bellefonte, PA, USA) using static extraction for 1 h at room temperature and gas chromatography - mass spectrometer (GC-MS) system for analyses (Agilent 6890 GC; Microanalytics, Round Rock, TX, USA).

### *2.3 Odor*

Odor samples were collected from the incoming and outgoing air sampling ports of the UV reactor in 10 L Tedlar sample bags using a Vac-U-Chamber (SKC Inc., Eighty Four, PA, USA) and sampling pump. Tedlar sampling bags were pre-cleaned by flushing with clean air three times before use. Odor samples were analyzed using a dynamic triangular forced-choice olfactometer (St. Croix Sensory Inc., Stillwater, MN, USA). Four trained panelists at two repetitions each were used in the analysis of each sample. Each sample was presented to the panelists from low concentration to higher concentrations, each dilution level doubled the concentration of the odor.

### *2.4 Ammonia and Hydrogen Sulfide*



Ammonia and H<sub>2</sub>S concentrations were measured as previously described in Maurer et al. (2017) using a Drager X-am 5600 portable gas analyzer (Luebeck, Germany) with NH<sub>3</sub> and low range H<sub>2</sub>S XS sensors. The analyzer was calibrated using Drager calibration software and standard gases (Praxair, Ames, IA, USA) (NH<sub>3</sub>: 298 ppm and H<sub>2</sub>S: 50.4 ppm).

### *2.5 Greenhouse Gases*

Gas samples were collected as previously described in Maurer et al. (2017a,b) via syringe and 5.9 mL Exetainer vials (Labco Limited, UK) and were analyzed for GHG concentrations on a GHG GC equipped with FID and ECD detectors (SRI Instruments, Torrance, CA, USA). Standard calibrations were constructed daily using 10.3 ppm and 20.5 ppm CH<sub>4</sub>; 1005 ppm, 4010 ppm and 8020 ppm CO<sub>2</sub>; and 0.101 ppm, 1.01 ppm and 10.0 ppm N<sub>2</sub>O (Air Liquide America, Plumsteadville, PA, USA). Standards used for calibrations were completed in duplicate for CH<sub>4</sub> and CO<sub>2</sub> while N<sub>2</sub>O standards were done in triplicate.

### *2.6 Concentration Reduction Estimations*

Overall mean % reduction for each measured gas was estimated using Eq. 1.

$$\% R = \frac{E_{Con} - E_{Treat}}{E_{Con}} * 100$$

Eq. 1

where: %*R* is the % of reduction, *E*<sub>Con</sub> is the mean measured concentrations of the of the control, and *E*<sub>Treat</sub> is the mean concentrations of the treated air. Two treatments were tested:

- Photolysis, UV lights only (no photocatalytic coating on plastic panels), and
- Photolysis and photocatalysis (with the photocatalytic coating on panels).

Results of the treatment were evaluated halfway through the reactor and at the end of reactor (

## 2.7 Statistical Analyses

The Standard Least Squares in a REML method, in JMP Pro (version 11, SAS Institute, Inc., Cary, NC, USA) was used to analyze the data by comparing mean values to determine the p values,  $p < 0.05$  was used as the significance level.

## 3. Results

### 3.1 Environmental Parameters

Over the course of the on farm pilot-scale UV only trial the measured temperature ranged from 18.9 to 22.0°C and over the course of the UV plus photocatalytic coating trial the measured temperature ranged from 21.8 to 26.0°C. The trials started mid-May 2017 and ended at the end of June 2017. The relative humidity (RH) of the UV only trial ranged from 46 to 84% and over the course of the UV plus photocatalytic coating trial the measured RH ranged from 36 to 80%. The average measured atmospheric pressure was recorded over the course of the trials was 758 mmHg.

### 3.2 Volatile Organic Compounds

Significant reduction of *p*-cresol (22.0%,  $p = 0.0280$ ) was observed for the full treatment with UV and the photocatalytic coating. Other treatments had no significant effect on *p*-cresol. The measured values of sulfur-containing compounds, DMDS and DMTS, and indolic compounds, indole and skatole, resulted in no significant effect neither due to the photolysis nor the photocatalysis (Table 1). Phenol was not significantly reduced or increased by the UV with or without photocatalytic coating. Measured volatile organic acids, *n*-butyric, isovaleric acid and valeric acid also were not significantly affected by these treatments. Acetic acid significantly

increased (-52.9 %,  $p=0.0408$ ) only after half treatment with the photocatalytic coating. Other treatments had no significant effect on acetic acid.

**Table 1.** Measured % reductions of treated barn emissions.

	Target Compounds	Half Treatment (~23.6 sec mean treatment time (in mid section of reactor))		Full Treatment (~47.2 sec mean treatment time at the end of the reactor)	
		UV Only (photolysis)	UV plus photocatalytic coating (photolysis and photocatalysis)	UV Only (photolysis)	UV plus photocatalytic coating (photolysis and photocatalysis)
% Reduction	Odor	NA	NA	NA	<b>16.3 (0.0390)</b>
	DMDS	-30.6 (0.4409)	27.6 (0.2264)	10.5 (0.8762)	23.6 (0.1372)
	DMTS	-95.4 (0.2546)	3.0 (0.9364)	2.2 (0.9750)	41.1 (0.1456)
	Acetic Acid	-79.5 (0.2800)	<b>-52.9 (0.0408)</b>	20.5 (0.6406)	-19.7 (0.4928)
	<i>n</i> -Butyric Acid	-25.1 (0.5814)	-48.4 (0.0868)	35.5 (0.3903)	6.8 (0.6910)
	Isovaleric Acid	-19.8 (0.6356)	-23.7 (0.1543)	38.6 (0.2787)	5.6 (0.6022)
	Valeric Acid	-131.2 (0.1753)	-92.9 (0.0813)	21.9 (0.7360)	-18.5 (0.4579)
	Phenol	32.7 (0.0779)	-15.5 (0.3395)	42.0 (0.0976)	10.9 (0.4095)
	<i>p</i> -Cresol	-5.9 (0.5707)	-21.4 (0.2136)	16.8 (0.1156)	<b>22.0 (0.0280)</b>
	Indole	-58.8 (0.3289)	11.8 (0.6928)	-8.8 (0.8060)	47.5 (0.1157)
	Skatole	100 (0.4226)	16.5 (0.5537)	-410.2 (0.5445)	-16.1 (0.7627)
	Methane	-3.7 (0.3103)	-1.4 (0.6678)	-4.7 (0.1491)	-2.2 (0.5244)
	Carbon Dioxide	2.6 (0.4307)	0.0 (0.9965)	1.9 (0.2920)	<b>-3.1 (0.0178)</b>
	Nitrous Oxide	<b>4.2 (0.0409)</b>	<b>7.3 (0.0252)</b>	<b>7.6 (0.0181)</b>	<b>8.7 (0.0200)</b>

Note: Values in parenthesis are p values, Bold values are significant.

### 3.3 Odor

Odor samples were collected and measured only for the full treatment with UV and the photocatalytic coating and significant reduction was 16.3% ( $p=0.0390$ ) (Table 1).

### 3.4 Ammonia and Hydrogen Sulfide

Ammonia and hydrogen sulfide concentrations were too low for measurement with instrumentation used, thus reductions could not be estimated.

### *3.5 Greenhouse Gases*

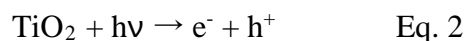
Nitrous oxide was significantly reduced; 4.2% ( $p=0.0409$ ), 7.3% ( $p=0.0252$ ), 7.6% ( $p=0.0181$ ) and 8.7% ( $p=0.0200$ ) for half treatment of UV only, half treatment of UV and photocatalytic coating, full treatment of UV only and full treatment of UV and photocatalytic coating, respectively. The reduction of  $N_2O$  was greater for the full treatments compared to the half treatments for both UV only and UV with photocatalytic coating treatments. The photocatalytic coating also resulted in greater reduction of  $N_2O$  compared to photolysis only trials. Treatments had not significant effect on  $CH_4$ .  $CO_2$  increased, -3.1% ( $p=0.0178$ ) for the full treatment with UV and the photocatalytic coating (Table 1), likely a breakdown product of more complex VOCs.

## **4. Discussion**

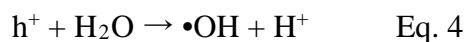
### *4.1 UV Modes of Action*

Sources of  $TiO_2$  are generally anatase, rutile, and ilmenite. Anatase and rutile are the commonly used types of  $TiO_2$  crystal for photocatalysis process, while anatase has a higher photocatalytic activity. Titanium dioxide is a semiconductor. A band gap exists between the top of the filled valence band and the bottom of the vacant conduction band. The band gap energy of anatase is 3.2 eV.

Band-gap photoexcitation firstly occurs during heterogeneous photocatalysis with semiconductor particles. Photons with energy higher than 3.2 eV can be absorbed by anatase. An electron is excited from the valence band to the conduction band when absorption occurs, leaving a hole with high oxidative potential in the valence band. The band gap impedes the recombination of excited electron and hole. This process can be expressed as following:



Charge transfer occurs secondarily during photocatalysis. The generated electrons and holes separate after excitation and transfer to catalyst surfaces to react with adsorbed electron acceptors and electron donors. In photocatalysis air purification reactions, electron acceptors are usually  $\text{O}_2$ , while electron donors are ambient moisture. The oxidative holes react with  $\text{OH}^-$  and  $\text{H}_2\text{O}$  on catalyst surfaces and produce hydroxyl radicals  $\bullet\text{OH}$ .



The electrons reduce  $\text{O}_2$  to  $\bullet\text{O}_2^-$ .



These produced radicals can, in turn, degrade organic pollutants.

Degradations in gas phase reactions of the pollutants in this study with significant reductions, *p*-cresol and nitrous oxide have been reported in the literature. Gas phase reactions of *p*-cresol and hydroxyl radicals were observed to be degraded to 4-methyl-2-nitrophenol by Coeur-Tourneur (2006). The reduction of *p*-cresol, a major odorant, may explain the reduction of odor observed by the panelists. Nitrous oxide in the atmosphere exposed to UV light produce free radicals such as nitric oxide (Clark and Brunick, 2015). The observed increase of carbon dioxide, 3%, is likely the result of breakdown reactions of VOCs with UV light and/or free radicals produced by the UV light.

#### *4.2 Comparison with pilot and laboratory-scale experiments.*

In a previous study (Zhu et al., 2017) completed on laboratory-scale, the same TiO<sub>2</sub> photocatalyst and UV light source was evaluated in regard to reduction efficiency of six odorous VOCs. The *p*-cresol reduction observed in this farm pilot-scale study (22%) was very similar to that observed on laboratory-scale (27%) at a similar treatment time of 47 sec and 40 sec respectively. The other VOCs that were also monitored in both studies, DMDS, DMTS and *n*-butyric acid resulted in reductions on the laboratory-scale (35-62%) but no significant reductions were observed on the farm pilot-scale study.

## **5. Conclusions**

In this on farm pilot-scale study, we observed significant reductions in emissions were observed for *p*-cresol (22%), odor (16%) and N<sub>2</sub>O (9%). Significant increase in CO<sub>2</sub> (3%) was also observed. The UV plus photocatalytic coating had no significant effect on DMDS, DMTS,

acetic acid, *n*-butyric acid, isovaleric acid, valeric acid, phenol, indole, skatole and CH<sub>4</sub> emissions.

## **Author contributions**

The manuscript was written through contributions of all authors. All authors have given approval to the final version of the manuscript. All authors contributed equally.

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## Figure Captions

**Figure 1.** Schematic of UV photocatalytic reactor for on-farm experiments.

**Figure 2.** UV<sub>365</sub> effective dosage surface map.

**Figure 3.** Mapping reactor airflow for estimation of mean treatment time. Black: setting 1, Gray: setting 2 and Light Gray: setting 3.

**Figure 4.** On-farm UV photocatalytic reactor setup. View from the air inlet (top). Experiments tested the effects of black light on odorous volatile organic compounds, odor, GHGs, ammonia and hydrogen sulfide. Thin film of photocatalyst (bottom) covered plastic material typically used for indoor barn wall cover.