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Swine slurry characteristics as affected by selected additives and disinfectants[☆]

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ABSTRACT

Current swine industry practice is to house animals in confinement facilities which capture and store feces and urine as slurry in pits below the production area. Additives and disinfectants may be introduced into the manure pits. This study was conducted to measure the effects of additives and disinfectants on temporal changes in swine slurry characteristics. Slurry from a commercial swine production facility in southeast Nebraska, USA was collected and transferred to 57 L reactors located within a greenhouse. Selected additives and disinfectants were added to the reactors and physical properties, chemical characteristics, and antibiotic concentrations were monitored for 40 days. Concentrations of dry matter (DM), total nitrogen (TN), phosphorus pentoxide (P₂O₅), calcium (Ca), magnesium (Mg), zinc (Zn), iron (Fe), manganese (Mn), and copper (Cu) were significantly greater than the Control in each of the reactors containing additives. The reactors in which the additives MOC-7, More Than Manure®, Sludge Away, and Sulfi-Doxx were introduced had significantly greater values of chemical oxygen demand (COD), total volatile solids (TVS), total suspended solids (TSS), total solids (TS), dry matter (DM), TN, P₂O₅, Ca, Mg, Zn, Fe, Mn, Cu and chlortetracycline than the other additive treatments. Concentrations of TVS and TSS were significantly lower in the reactors containing Clorox® and Virkon™ than the other disinfectant treatments. The total dissolved solids (TDS) concentration of 26,500 mg L⁻¹ and pH value of 7.27 obtained for the reactors containing Tek-Trol were significantly greater than measurements obtained for the other treatments. Concentrations of chlortetracycline and tiamulin of 8840 and 28.8 ng g⁻¹, respectively, were significantly lower for the treatments containing Tek-Trol. The sodium (Na) concentration of 1070 mg L⁻¹ measured in the reactors containing Clorox® was significantly greater than values for the other disinfectant treatments. The introduction of selected additives and disinfectants may influence certain physical properties, chemical characteristics, and antibiotic concentrations of swine slurry.

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1. Introduction

1.1. Background

Current swine industry practice is to house animals in

confinement facilities which capture and store animal feces and urine as slurry in pits below the production area. Additives may be placed in swine manure pits to aid in solids digestion, preserve nutrients, reduce odor, minimize foaming, and decrease crusting. The types of additives used usually include acidifiers, adsorbents, oxidizing agents, or urease inhibitors. Additives containing enzymes or selected microbial strains have been developed to enhance the biodegradation of manure, and some have even been used to replace processes such as aeration, anaerobic digestion, and composting. (McCroly and Hobbs, 2001). Disinfectants are used in swine production facilities to reduce disease transmission and for

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improved animal health and welfare.

Antibiotics and other pharmaceuticals are administered to swine for treatment of disease (Spielmeyer, 2018). The antibiotics may not be completely absorbed in the animal digestive system and antibiotic residues may be released with livestock wastes. Antibiotics and their metabolites may accumulate during swine manure storage and can enter the environment following land application of manure. There is concern about the occurrence of veterinary pharmaceuticals in manure and their potential environmental impacts. Little information is currently available on the effects of selected additives or disinfectants on the physical properties, chemical characteristics, and antibiotic concentrations in swine slurry.

1.2. Additive effects on physical and chemical characteristics of slurry

Zhu et al. (2006) examined the effects of the microbial additive Sporzyme combined with aeration on reduction of nutrients in swine manure over a 15-day period. After one day of aeration, all aerated treatments experienced a 42% decrease in total soluble phosphorus and an increase in total insoluble phosphorus. Total Kjeldahl nitrogen decreased by approximately 40% in all treatments except the control.

The effects of the More Than Manure® amendment, anaerobic digestion, and coarse solids on the solids and nitrogen content of dairy manure slurry were examined by Sun et al. (2014). Addition of More Than Manure® and coarse solids to raw manure slurry resulted in statistically significant increases in total nitrogen, total solids, and volatile solids. A statistically significant increase in total solids and a decrease in volatile solids was found when More Than Manure® was added to raw manure with no coarse solids present.

Holly and Larson (2017) investigated the effects of More Than Manure® on solids and nitrogen composition of dairy manure slurry. The additive was tested at the manufacturer recommended dosage as well as at ten times the suggested rate. The solids and nitrogen content of the slurry was not significantly reduced by the additive at either of the application rates.

1.3. Additive effects on antibiotic concentrations in slurry

The degradation of chlortetracycline and tylosin during anaerobic digestion of swine manure was investigated over a 216-day incubation by Stone et al. (2009). The digester temperature during the period was gradually increased from 10 to 20 °C between days 0 and 56 to simulate the transition from winter to summer. The concentration of chlortetracycline decreased from 27.0 to 11.6 mg L⁻¹ during the incubation period while the concentration of tylosin remained relatively stable until day 109 after which it decreased from 30 to 0 mg L⁻¹.

Alvarez et al. (2010) measured the degradation of chlortetracycline and oxytetracycline at concentrations of 10, 50, and 100 mg L⁻¹ during a 21-day anaerobic incubation at a temperature of 35 °C. At concentrations of 10, 50, and 100 mg L⁻¹, half-lives for chlortetracycline and oxytetracycline were 3.8, 3.2, and 4.1 days and 13.3, 15.4, and 11.9 days, respectively. The initial concentration of antibiotics was found to influence the degradation effectiveness of anaerobic digestion.

The effect of temperatures on concentrations of chlortetracycline in swine manure during anaerobic digestion was also investigated by Varel et al. (2012). After 21 days, the concentration of chlortetracycline in digesters maintained at 22, 38, and 55 °C were reduced by 7, 80, and 98%, respectively. It was concluded that anaerobic digestion at 38 or 55 °C was an effective treatment for reducing concentrations of chlortetracycline in swine manure.

Joy et al. (2014) simulated swine manure slurry storage under aerobic conditions during a 40-day incubation. The fate of bacitracin, chlortetracycline, and tylosin at initial concentrations of 50, 300, and 10 mg kg⁻¹ (dry weight basis) was examined. First order degradation models were fitted to the decay of each antibiotic, with half lives of 1.9, 1.0, and 9.7 days measured for bacitracin, chlortetracycline, and tylosin, respectively.

The persistence of selected antibiotics during swine manure storage was measured by Berendsen et al. (2018). Swine manure samples were fortified with antibiotics and then incubated. The length of time for 50% of the chlortetracycline, lincomycin, and tiamulin in swine manure to degrade was 19, 269, and 101 days, respectively, while 90% dissipation required 62, 892, and 335 days, respectively. The present investigation was conducted to identify the effects of selected additives and disinfectants on the physical properties, chemical characteristics, and antibiotic concentrations of swine slurry.

2. Materials and methods

2.1. Additives and disinfectants

Swine manure pit additives are marketed for several purposes including: crust prevention, foam reduction, nutrient preservation, odor control, and solids reduction. Although there is often one primary function for which an additive is advertised, many are sold to serve multiple purposes. The additives that were examined in this investigation included: Coban® 90, Manure Magic®, MOC-7, More Than Manure®, Sludge Away, and Sulfi-Doxx.

Coban® 90 is marketed as a feed additive for chickens, quail, and turkeys to prevent the intestinal ailment coccidiosis and its active ingredient, sodium monensin, is also sold to improve feed efficiency and control coccidiosis and bloat in feedlot cattle by altering microbial composition of the rumen. Sodium monensin has also been used for swine pit foam control, although this off-label use has not been approved by the U.S. Food and Drug Administration.

Manure Magic® is marketed to reduce excess solids, foaming, and odor in swine pits and lagoons while MOC-7 is advertised as an odor reduction product that also minimizes crust formation and solids accumulation. The active ingredients in Manure Magic® and MOC-7 are proprietary.

More Than Manure® is advertised to reduce nitrogen losses from stored manure due to volatilization and denitrification and is also marketed to minimize solids accumulation and phosphorus losses in stored manure. More Than Manure® consists of a maleic-itaconic copolymer as a partial salt with calcium and ammonium. Sludge Away is sold to reduce organic solids and biogases produced during anaerobic digestion of manure. Some of the products contained in Sludge Away include humic acid based lignins which absorb odorous compounds and strains of purple sulfur bacteria used to sequester volatile sulfur-containing compounds. Sulfi-Doxx is advertised as a control for the emission of hydrogen sulfide and other odor producing compounds from stored manure. A mixture of *Bacillus* spp (bacteria) and *Trichoderma* spp (fungi) in a humate liquid carrier are contained in Sulfi-Doxx.

The disinfectant products tested in this investigation included: Clorox®, Pi Quat, Tek-Trol, and Virkon™. Clorox® is a halogen-based disinfectant containing sodium hypochlorite that is used for a variety of disinfectant purposes inside and outside of the animal production industry. Pi Quat is often utilized in the animal industry during cleaning and sanitation of production facilities. Tek-Trol is a phenol-based disinfectant used for sanitation. Virkon™ is an oxidant that is frequently employed as a disinfectant.

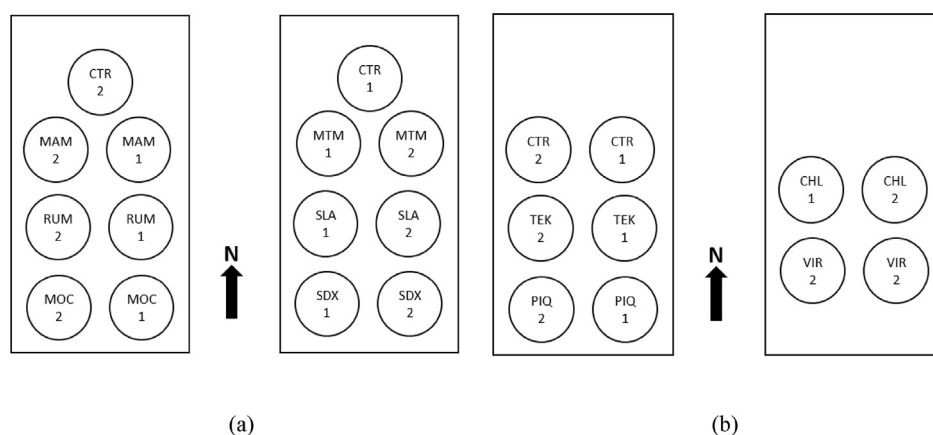


Fig. 1. A schematic of the reactor arrangement for the additives (a) and disinfectants (b) portions of the experiment. Additives: RUM = Coban® 90; CTR = Control; MAM = Manure Magic®; MTM = More Than Manure®; MOC = MOC-7; SLA = Sludge Away; SDX = Sulfi-Doxx. Disinfectants: CHL = Clorox®; CTR = Control; PIQ = Pi-Quat; TEK = Tek-Trol; VIR = Vikron.

2.2. Swine slurry collection

Slurry was collected from the deep pit of a commercial wean-to-finish swine production facility in southeast Nebraska. A sump pump was lowered into the deep pit to transfer slurry into 19 L buckets. Slurry samples for the additive and disinfectant experiments were obtained on January 25, 2018 and March 17, 2018, respectively. Approximately 687 L of slurry was required for the additives experiment and 490 L was needed for the disinfectants experiment. The slurry was transported to a temperature-controlled hoop house style greenhouse on the University of Nebraska – Lincoln campus and approximately 50 L was placed in stainless steel pots, which served as reactors (Fig. 1).

Before the start of the study, the temperature effects of reactor placement in the greenhouse was investigated to ensure the temperatures of the reactors were similar. An awning and tarps covered the reactors, protecting them from direct sunlight. The arrangement of the reactors for the additive and disinfectant portions of the study are shown in Fig. 1(a) and (b), respectively. Each of the treatments in the additive and disinfectant experiments were replicated twice. Neither an additive or disinfectant were added to the Control treatment.

2.3. Dosing procedures

Dosing procedures for the additives were determined from product instructions as well as from personal correspondence with

individual manufacturers (Table 1). Product literature for Coban® 90, Manure Magic®, Sludge Away, and Sulfi-Doxx provided recommended application rates as volume or weight of product per volume of slurry. The dosing amount for More Than Manure® was identified by first assuming the nitrogen requirement for a unit area of corn, determining the nitrogen content of the manure slurry, and then calculating the amount of product to be dosed. Technical representatives of MOC-7 recommended that 177 mL of their product be added to 50 L of swine slurry.

Dosing requirements for the disinfectants was identified by calculating the volume necessary to achieve saturation of the internal surface area of the reactors at the desired concentration. The amount of disinfectant used in a swine production facility depends upon the surface area being cleaned. Surface area can be estimated by multiplying the floor space by 2.5 to account for walls and other surfaces. The calculated area was then multiplied by the depth required for surface saturation, approximately 0.03 cm. A rinse was recommended for Clorox® and Tek-Trol but not for Pi-Quat and Vikron. An equal volume of distilled water was added to those disinfectants which required a rinse. The disinfectant product dose was reduced by 1/2 and the difference made up with the rinse water to maintain consistency in volume of the treatments.

2.4. Slurry sampling

Each reactor was stirred using a large cast iron paint mixer operated with a cordless power drill prior to slurry collection. The

Table 1
Dosing requirements for additives and disinfectants.

Product	Manufacturers Dosing Requirement	Prepared Dose	Concentration in Slurry
Additives			
Coban® 90	2.27 kg per 379,000 L manure slurry	0.295 g product	0.006 g/L slurry
Control	Not applicable	Not applicable	Not applicable
Manure Magic®	22.7 kg per 3,790,000 L manure slurry	0.295 g product	0.006 g/L slurry
MOC-7	177 mL per 56.8 L manure slurry	1.7 mL product	3.0 mL/L slurry
More Than Manure®	215 mL per ha of manure land application	3.1 mL product	0.05 mL/L slurry
Sludge Away	5.68 L per 28,400 L manure slurry	9.8 mL product	0.20 mL/L slurry
Sulfi-Doxx	3.79 mL per 56,800 L manure slurry	0.82 mL product	0.067 mL/L slurry
Disinfectants			
Control	Not applicable	360 mL De-Ionized water	Not applicable
Clorox®	Surface Saturation	180 mL product +180 mL De-Ionized water	3.4 mL/L slurry
Pi-Quat	Surface Saturation	360 mL product	3.4 mL/L slurry
Tek-Trol	Surface Saturation	180 mL product +180 mL De-Ionized water	3.4 mL/L slurry
Virkon™	300 mL/m ²	1.80 g of product in 360 mL De-Ionized water	3.4 mL/L slurry

solution was mixed for 30 s or until the foam crust on the top of the reactor was completely incorporated into solution. Separate mixers were used for each reactor.

After blending, approximately 1 L of slurry was transferred from every reactor during each sampling date into clear plastic bottles which were immediately taken to a laboratory. Slurry was retrieved from the reactors using a 500 mL container that was assigned to each reactor. Once in the laboratory, every 1 L sample was mixed for 30 s on high in a lab blender. After mixing, each sample was distributed into smaller subsamples to be used for determination of physical properties, chemical characteristics, and antimicrobial concentrations. Blenders were cleaned between processing of each sample by a thorough rinse, followed by washing with a 50:50 solution by volume of isopropyl alcohol and water which remained on the surface of the blender for 30 s, followed by a second thorough rinse.

2.5. Laboratory analyses

Slurry samples to be examined for chemical oxygen demand (COD) and physical properties were stored in clear plastic bottles in a freezer maintained at 4 °C and were processed within 48 h following collection. Samples were prepared by first pipetting 0.25 mL of slurry into a 25 mL beaker. The samples were then diluted by a factor of 20 by adding 4.75 mL of deionized water to the beaker. Next, 0.20 mL of diluted slurry was pipetted into a Hach High Range Plus COD Digestion Vial (Hach Company, Loveland, Colorado). Once all samples were prepared, the digestion vials were inverted several times to enhance mixing and then added to a heating block at 150 °C for 2 h. At the end of 2 h, the heating block was turned off and the vials were cooled to 120 °C before they were removed and allowed to cool to room temperature. Once at room temperature, the samples were analyzed using a Hach DR 2800 Spectrophotometer (Hach Company, Loveland, Colorado) using program 435 for HR COD. As recommended by the manufacturer, the COD provided values were multiplied by 10 to convert from HR to HR + analysis. This result was then multiplied by 20 to account for the earlier factor 20 dilution.

Samples used for analyses of total volatile solids (TVS), total suspended solids (TSS), total dissolved solids (TDS), and total solids (TS) were stored in the same manner as previously described for COD analysis. Testing for TS was performed by pipetting 8 mL of slurry into a tared 70 mm aluminum pan and the wet weight was then recorded. The pan was then transferred to a steam table for approximately 30 min until the slurry was dry. The pan was next placed in a 41 °C oven for at least 24 h and then cooled in a desiccator and weighed again. The second weight provided the TS content. After weighing, the pan was transferred to a 288 °C furnace for 30 min. After a second cooling sequence was performed in a desiccator, the pan was weighed a final time, yielding the TVS content.

To find the TSS and TDS content of the slurry, 0.25 mL of slurry was pipetted into a 25 mL beaker and diluted with 20 mL of distilled–deionized water. The diluted slurry solution was then filtered through a pre-weighed 47 mm glass fiber filter. If needed, an extra 5 mL of distilled–deionized water was used to rinse any remaining solids from the beaker onto the filter. The filter was then placed in a 41 °C oven for at least 24 h. Finally, 10 mL of filtrate were transferred to a tared aluminum solids handling pan and placed in a 41 °C oven for at least 24 h.

Samples to be analyzed for electrical conductivity (EC), pH, dry matter (DM), and chemical constituents were stored in clear 500 mL plastic bottles in a cooler maintained at 4 °C until the completion of either the additive or disinfectant portions of the experiment. The samples were then transported to a commercial

laboratory for analyses.

Values for potassium oxide (K_2O) and phosphorus pentoxide (P_2O_5) are reported in this paper since these constituents are often used when determining the fertilizer value of manure. The concentration of K_2O should be multiplied by 0.830 to find the K content of the slurry. The total P (TP) content of the slurry can be determined by multiplying the P_2O_5 concentration by 0.436.

2.6. Antimicrobial analyses

Antimicrobials were extracted from manure using solvent removal followed by solid phase extraction (SPE) cleanup. Approximately 0.2 g of sample was mixed with 5 g of clean sand and spiked with 16 ng oleandomycin as a surrogate to monitor analyte recovery, followed by the addition of 14 mL of 5 mM ammonium citrate (buffered to pH 6 using ammonium hydroxide) and 6 mL methanol in 50-mL polypropylene centrifuge tubes. Mixtures were shaken briefly by hand and then on a Burrell wrist-action shaker for 30 min. Solids were extracted a second time with 4 mL of ammonium citrate and 16 mL methanol, and a third time using 20 mL acetone. All extracts were combined and fortified with internal standards (doxycycline and roxithromycin, 40 ng each) and then concentrated on a Labconco RapidVap N_2 sample concentrator (Labconco Corporation, Kansas City, MO) at 30 °C (90% rotation speed) until the volume was reduced by half. Purified reagent water was added to bring the extract volume to 100 mL and the resulting aqueous solutions were extracted using 200 mg Oasis HLB SPE cartridges. SPE cartridges were eluted into borosilicate test tubes using 3 mL of 130 mM ammonium citrate in methanol. The solvent was reduced in volume to approximately 200 μ L under a stream of dry nitrogen and transferred to an autosampler vial with silane-treated insert and then mixed with 200 μ L reagent water.

All sample extracts were analyzed on a Waters 2695 high pressure liquid chromatograph (HPLC) interfaced with a Waters Quattro Micro triple quadrupole mass spectrometer (Govaerts et al., 2003; Snow et al., 2003). Analytes were separated on a reverse phase (HyPurity C18, 250 mm \times 2.1 mm, 5 μ m particle size) column at 50 °C with a 50- μ L injection volume. A gradient mobile phase (0.2 mL min^{-1}) was used consisting of A) 1 mM aqueous citric acid and methanol (97:3, v/v) and B) methanol and 1 mM aqueous citric acid (97:3, v/v). Initial gradient conditions (95% A) were held for 2 min, ramped to 5% A and held for 16 min, and then returned to 95% A for 5 min to equilibrate the column. Analytes were detected using Multiple Reaction Monitoring (MRM) mode with positive electrospray ionization. The most intense MRM transitions were determined by infusion and monitored for each analyte and linear calibration curves were generated for all analytes and surrogates.

Measuring the low concentrations of antibiotics in swine slurry requires specialized equipment, is labor intensive, and relatively expensive. As a result, antimicrobial analyses were performed for samples collected on days 1, 5, 14, 21, and 40 following introductions of additives or disinfectants.

The antibiotics that were detected in the swine slurry were chlortetracycline, lincomycin, and tiamulin which were from the antibiotic classes tetracyclines, lincosamides, and pleuromutines, respectively. Chlortetracycline is used to control ileitis, protects swine from bacterial enteritis, and is administered for the treatment of bacterial pneumonia and cervical jowl abscesses. Chlortetracycline is not sorbed or metabolized during animal digestion and more than 85% of the chlortetracycline that is fed can be excreted in bioactive forms (Dewey et al., 1999). Lincomycin is used in the treatment of infectious forms of arthritis and mycoplasma pneumonia. Tiamulin is administered to control swine dysentery and ileitis.

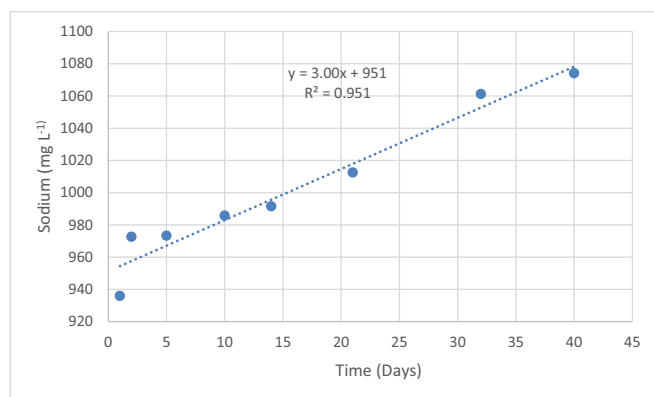


Fig. 2. Sodium content of slurry as affected by time for the Control treatment of the additives study.

2.7. Data analyses

The concentrations of chemical constituents resulting from the addition of both additives and disinfectants were found to increase in a linear fashion with time. The increase in sodium concentrations for the control treatment in the additive study was characteristic of the results obtained for the other chemical constituents (Fig. 2). It was hypothesized that evaporation from the experimental reactors contributed to the increase in constituent concentrations, independent of the addition of additives or disinfectants. Figures showing changes in the concentrations of the inert constituents potassium oxide, sodium, and zinc over time for the Control treatments were like graphs obtained for the treatments where additives and disinfectants were added. Thus, the hypothesis that evaporation contributed to the increase in concentrations appeared to be valid.

Regression equations were developed from data collected on the Control plots showing the percent volume reduction (y) in sodium concentrations over time in days (x) required to account for

evaporation. For the additive portion of the experiment, the regression equation was:

$$y = -0.243x + 99.8 \quad (1)$$

The equation derived for disinfectants was:

$$y = -0.364x + 101 \quad (2)$$

Physical, chemical, and antimicrobial measurements were multiplied by values obtained from equation (1) or 2 to obtain corrected values due to evaporation. The corrected values were used in the statistical analyses.

2.8. Statistical analyses

Additives, disinfectants, and time following initiation of the experiment were the treatment factors with additives and disinfectants assigned to bioreactors using a completely randomized design. A split-plot-in-time analysis of variance (ANOVA) was performed to determine the effects of the experimental treatments on physical properties, chemical characteristics, and antibiotic concentrations corrected for evaporation. If a significant difference was identified, the least significant difference (LSD) test was used to identify differences among experimental treatments. A probability level $p < 0.05$ was considered significant. Additional information concerning the experimental and statistical procedures is provided by Duerschner (2018).

3. Results and discussion

3.1. Physical properties for additive experiment

ANOVA indicated significant temporal changes in COD, TVS, TDS, TS, EC, and pH during the additive portion of the experiment (Tables 2a and 2b). Measurements of EC and pH decreased from 28.4 to 23.4 dS m^{-1} and 7.70 to 6.95, respectively, during the study period. No treatment by time interactive effects were found for any of the measured physical properties.

Table 2a

Additive effects on the physical characteristics of swine slurry.

Variable	COD (mg L^{-1})	TVS (mg L^{-1})	TSS (mg L^{-1})	TDS (mg L^{-1})	TS (mg L^{-1})
Baseline	66,200	33,800	35,400	24,000	57,200
Treatment					
Coban® 90	48,500 cd ^a	23,700 b	27,200 b	22,900	41,800 b
Control	45,500 d	22,000 b	23,500 b	24,100	40,300 b
Manure Magic®	51,700 c	23,000 b	25,700 b	23,600	41,300 b
MOC-7	63,900 b	32,400 a	39,200 a	24,400	56,800 a
More Than Manure®	68,400 a	32,400 a	42,000 a	23,900	56,500 a
Sludge Away	66,300 ab	32,300 a	40,500 a	24,200	56,200 a
Sulfi-Doxx	67,400 ab	32,200 a	42,500 a	24,300	56,000 a
Days					
1	67,000	25,100	33,600	24,118	43,900
2	70,200	23,900	29,600	19,424	41,900
5	56,600	31,000	35,200	23,375	53,100
10	55,800	30,300	34,800	25,944	53,200
14	57,800	27,700	34,700	15,570	50,000
21	55,500	30,200	39,100	28,291	53,400
32	55,200	30,000	35,700	29,254	53,000
40	51,700	28,100	32,200	25,325	49,800
ANOVA (P > F)					
Treatment	0.001	0.001	0.001	0.992	0.001
Time	0.001	0.001	0.073	0.001	0.001
Treatment x Time	0.064	0.512	0.668	0.792	0.329

COD = chemical oxygen demand, TVS = total volatile solids, TSS = total suspended solids.

TDS = total dissolved solids, TS = total solids.

^a Means without common letters differ significantly ($p < 0.05$).

Table 2b
Additive effects on the physical characteristics of swine slurry.

Variable	EC (dS m ⁻¹)	pH	DM (mg L ⁻¹)
Baseline	28.2	7.80	5.47
Treatment			
Coban® 90	28.1	7.51	4.27 b ^a
Control	26.8	7.67	3.55 c
Manure Magic®	27.8	7.48	4.22 b
MOC-7	25.5	7.47	5.79 a
More Than Manure®	26.2	7.46	5.97 a
Sludge Away	26.2	7.44	5.77 a
Sulfi-Doxx	26.2	7.44	6.11 a
Days			
1	28.2	7.70	5.18
2	28.4	7.68	5.11
5	28.0	7.61	5.23
10	27.6	7.53	5.24
14	27.2	7.49	5.16
21	26.6	7.39	5.17
32	24.0	6.99	4.88
40	23.4	6.95	4.94
ANOVA (P > F)			
Treatment	0.326	0.638	0.002
Time	0.001	0.001	0.553
Treatment x Time	0.390	0.532	0.502

EC = electrical conductivity, DM = dry matter.

^a Means without common letters differ significantly ($p < 0.05$).

A linear decrease in EC was observed over time (Fig. 3). The dominant ions that contribute to EC of manures are NH₄-N, Na, Ca, Mg, K, chloride, sulfate, and bicarbonate. The general decline in NH₄-N concentrations in the swine slurry over time due to volatilization (Table 3a) is thought to have contributed to the reduction in EC.

Two of the factors which may influence the pH of swine slurry are concentrations of volatile fatty acids and NH₄-N. Volatile fatty acids are formed during the storage of swine slurry which causes pH to decrease. The presence of NH₄-N serves to buffer the reduction in pH resulting from the increase in volatile fatty acids. However, as NH₄-N concentrations decrease due to volatilization, the neutralizing factor is also reduced resulting in lower pH values (Table 2b).

Concentrations of COD, TVS, TSS, TS, and DM were significantly greater in the reactors containing MOC-7, More Than Manure®, Sludge Away, and Sulfi-Doxx than the other experimental treatments (Tables 2a and 2b). No significant differences in concentrations of TVS, TSS, and TS were found among the Coban® 90, Control, and Manure Magic® treatments. The DM measurement of

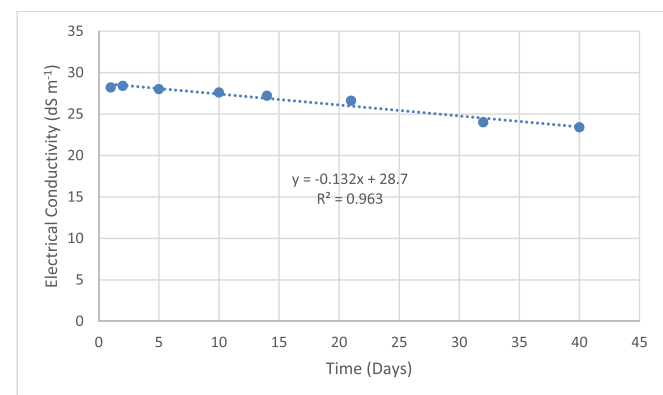


Fig. 3. Electrical conductivity of slurry as affected by time following introduction of additives.

3.55 mg L⁻¹ obtained for the Control treatment was significantly less than values obtained on the reactors where additives were introduced (Table 2b). Measurements of TDS, EC, and pH were not significantly affected by the introduction of additives. When compared to the control treatment, the introduction of additives did not significantly reduce measurements of COD, TVS, TSS, TDS, TS, EC, pH, or DM.

3.2. Chemical characteristics for additive experiment

The chemical constituents in the additive portion of the study for which significant temporal changes were found included TN and Ca (Tables 3a and 3b). Concentrations of TN decreased in a linear fashion from 5920 to 5370 mg L⁻¹ during the study period (Fig. 4). Although significant temporal changes in Ca concentrations were measured, the differences in Ca values were small varying from 1850 to 1990 mg L⁻¹ (Table 3b). Interactive treatment by time effects were not found for any of the chemical constituents.

The introduction of additives did not significantly reduce concentrations of the measured chemical constituents when compared to the Control treatment. The reactors containing MOC-7, More Than Manure®, Sludge Away, and Sulfi-Doxx had significantly greater concentrations of TN, P₂O₅, Ca, Mg, Zn, Fe, Mn, and Cu than the other experimental treatments (Tables 3a, 3b, and 3c). More Than Manure® consists of a copolymer that includes calcium and ammonium. The concentrations of Ca in the reactors containing More Than Manure® was among the largest of the experimental treatments. The P₂O₅, Ca, Mg, Fe, and Cu measurements of 5050, 1430, 1320, 165, and 29.2 mg L⁻¹ obtained for the Control treatment were significantly less than values found for the reactors where additives were introduced.

The ingredients contained in many of the additives are proprietary. One explanation for the increased concentrations in chemical constituents in selected reactors was that the additives themselves contained substantial quantities of certain chemical constituents.

3.3. Antibiotic concentrations for additive experiment

The antibiotics in the additive portion of the study for which significant temporal changes were found included chlortetracycline and tiamulin (Table 4). No distinct trend characterizing changes in antibiotic concentrations over time was apparent. Treatment by time interactions were found for tiamulin.

Significantly greater concentrations of chlortetracycline were found in the reactors containing MOC-7, More Than Manure®, Sludge Away, and Sulfi-Doxx (Table 4). No significant differences in chlortetracycline measurements were found among the Coban® 90, Control, and Manure Magic® treatments. The 324 ng g⁻¹ of lincomycin measured in the reactors containing Coban® 90 was significantly greater than values obtained for the other reactors. No significant differences in lincomycin concentrations were found among the other experimental treatments. When compared to the Control treatment, the introduction of additives did not significantly reduce antibiotic concentrations in the swine slurry.

3.4. Physical properties for disinfectant experiment

Significant temporal differences in COD, TVS, TDS, TS, EC, pH, and DM were found for the disinfectant portion of the experiment (Tables 5a and 5b). Consistent reductions in TVS, TS, EC, and pH were measured during the study period. Treatment by time interactive effects for the disinfectant study were found for TDS, EC, pH, and DM.

The concentration of TS consistently decreased in a linear

Table 3a
Additive effects on the chemical characteristics of swine slurry.

Variable	Org N (mg L ⁻¹)	NH ₄ -N (mg L ⁻¹)	NO ₃ -N (mg L ⁻¹)	Total N (mg L ⁻¹)	P ₂ O ₅ (mg L ⁻¹)
Baseline	2250	3630	1.10	5890	7490
Treatment					
Coban® 90	2740	2350	2.45	5090 e ^a	5510 d
Control	2280	2770	1.44	5050 e	5050 e
Manure Magic®	2710	2600	1.11	5290 d	5560 d
MOC-7	3400	2600	1.08	5960 c	8080 c
More Than Manure®	3010	3100	1.72	6110 b	8450 b
Sludge Away	3160	3010	1.23	6170 ab	8440 b
Sulfi-Doxx	3280	2920	1.30	6200 a	8850 a
Days					
1	2930	2980	1.40	5920	6950
2	3130	2780	1.39	5910	7090
5	3170	2670	1.55	5840	7210
10	2900	2910	1.43	5810	7200
14	2890	2790	2.08	5690	7100
21	2820	2760	1.56	5570	7160
32	2790	2660	1.26	5450	7060
40	2870	2500	1.15	5370	7320
ANOVA (P > F)					
Treatment	0.153	0.159	0.056	0.001	0.001
Time	0.174	0.067	0.406	0.001	0.065
Treatment x Time	0.811	0.755	0.721	0.813	0.323

Org N = organic nitrogen.

^a Means without common letters differ significantly ($p < 0.05$).

Table 3b
Additive effects on the chemical characteristics of swine slurry.

Variable	K ₂ O (mg L ⁻¹)	S (mg L ⁻¹)	Ca (mg L ⁻¹)	Mg (mg L ⁻¹)	Na (mg L ⁻¹)
Baseline	3050	816	1980	1930	973
Treatment					
Coban® 90	3433	620	1520 c ^a	1510 c	978
Control	3450	720	1430 d	1320 d	961
Manure Magic®	3370	630	1530 c	1520 c	951
MOC-7	3380	785	2140 b	2330 b	934
More Than Manure®	3430	805	2200 ab	2400 b	968
Sludge Away	3430	813	2220 a	2400 b	956
Sulfi-Doxx	3430	824	2270 a	2520 a	978
Days					
1	3360	703	1850	1970	938
2	3380	715	1850	1980	954
5	3400	729	1890	2000	972
10	3420	740	1930	2010	968
14	3420	726	1900	2000	958
21	3440	734	1900	2020	967
32	3400	769	1890	1980	954
40	3500	824	1990	2050	977
ANOVA (P > F)					
Treatment	0.943	0.104	0.001	0.001	0.427
Time	0.244	0.211	0.008	0.583	0.243
Treatment x Time	0.446	0.409	0.826	0.630	0.364

^a Means without common letters differ significantly ($p < 0.05$).

fashion with time (Fig. 5). Much of this decrease can be attributed to a reduction in TVS over time resulting from microbiological activities. Zhu et al. (2000) examined the dynamic changes in solids composition of swine manure obtained in southern Minnesota during a 30-day storage period. The TS content of the swine slurry analyzed in the present investigation decreased in a similar fashion over time.

Concentrations of TVS and TSS were significantly less in the reactors containing chlorine and Virkon™ (Tables 5a and 5b). The TDS measurement of 26,500 mg L⁻¹ and pH value of 7.27 obtained for the reactors containing Tek-Trol were significantly greater than measurements obtained for the other treatments. The introduction of disinfectants did not significantly affect measurements of COD or DM.

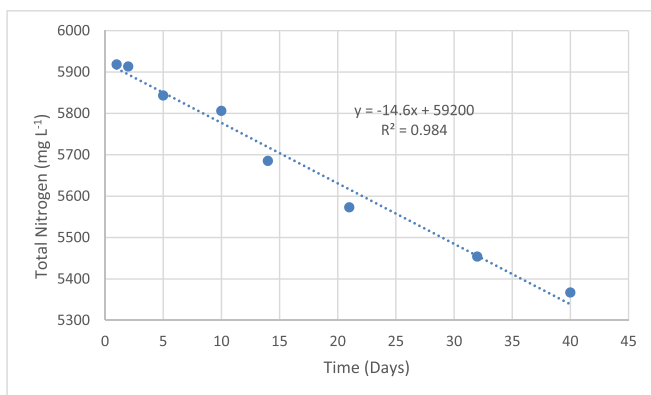
Deep pits located beneath swine production facilities usually have a large enough capacity to store materials generated during two to three swine wean to finish production cycles. A significant period may have expired before crops or forage have been harvested, soils have thawed, and manure application equipment can enter the land application site. As a result, swine slurry characteristics may vary substantially during the storage period. The slurry used during the additive study (Tables 2a and 2b) had less suspended and dissolved constituents than the slurry collected during the disinfectant investigation (Tables 5a and 5b).

Barret et al. (2013) collected swine slurry at shallow depths and from the bottom of outdoor concrete swine storage tanks. The contents of COD, TVS, TSS, and TS were found to vary from 27 to 194, 8–104, 5–136 and 13–142 g/L, respectively, which were

Table 3c
Additive effects on the chemical characteristics of swine slurry.

Variable	Zn (mg L ⁻¹)	Fe (mg L ⁻¹)	Mn (mg L ⁻¹)	Cu (mg L ⁻¹)	B (mg L ⁻¹)
Baseline	200	260	45.6	47.7	5.10
Treatment					
Coban® 90	142 c ^a	194 d	33.2 c	34.6 b	4.73
Control	142 c	165 e	32.0 c	29.2 c	7.31
Manure Magic®	142 c	194 d	33.2 c	34.7 b	4.63
MOC-7	201 b	264 c	50.6 b	49.0 a	4.85
More Than Manure®	206 ab	286 ab	51.0 ab	50.0 a	5.03
Sludge Away	206 ab	274 bc	50.7 b	50.2 a	5.05
Sulfi-Doxx	212 a	294 a	52.2 a	51.6 a	5.08
Days					
1	173	234	42.1	42.2	4.74
2	176	237	42.6	42.5	4.83
5	179	245	43.5	43.5	4.84
10	181	243	43.9	43.9	4.84
14	177	241	43.0	43.0	4.82
21	179	243	43.5	43.5	4.91
32	180	230	43.0	40.8	6.53
40	186	237	44.7	42.8	6.40
ANOVA (P > F)					
Treatment	0.002	0.002	0.001	0.001	0.610
Time	0.153	0.732	0.078	0.646	0.381
Treatment x Time	0.320	0.748	0.671	0.629	0.487

^a Means without common letters differ significantly ($p < 0.05$).

**Fig. 4.** Total nitrogen content of slurry as affected by time following introduction of additives.**Table 4**
Additive effects on concentrations of antibiotics in swine slurry (dry weight).

Variable	Chlortetracycline (ng g ⁻¹)	Lincomycin (ng g ⁻¹)	Tiamulin (ng g ⁻¹)
Baseline	9880	111	371
Treatment			
Coban® 90	8460 b ^a	324 a	207
Control	5600 b	96 b	180
Manure Magic®	7140 b	143 b	172
MOC-7	15,300 a	128 b	317
More Than Manure®	15,700 a	183 b	291
Sludge Away	14,300 a	127 b	265
Sulfi-Doxx	17,100 a	124 b	252
Days			
1	12,300	118	277
2			
5	12,700	169	251
10			
14	11,000	126	95
21	9320	232	130
32			
40	14,400	158	449
ANOVA (P > F)			
Treatment	0.003	0.029	0.243
Time	0.001	0.167	0.001
Treatment x Time	0.784	0.103	0.001

^a Means without common letters differ significantly ($p < 0.05$).

within the range of measurements reported in the present study for these constituents.

3.5. Chemical properties for disinfectant experiment

The chemical constituents in the disinfectant portion of the study for which significant temporal changes were found included NO₃-N, TN, P₂O₅, K₂O, S, Na, Zn, Mn, and Cu (Tables 6a, 6b, and 6c). Interactive treatment by time effects were found for NO₃-N and TN.

The only chemical constituent for which significant treatment effects were found was Na (Tables 6a, 6b, and 6c). The Na concentration of 1070 mg L⁻¹ measured in the reactors containing Clorox® was significantly greater than values for the other treatments. Sodium hypochlorite is an ingredient in Clorox® which could have contributed to the larger Na concentration in the reactors containing Clorox®. No significant differences in Na concentrations

Table 5a
Disinfectant effects on the physical characteristics of swine slurry.

Variable	COD (mg L ⁻¹)	TVS (mg L ⁻¹)	TSS (mg L ⁻¹)	TDS (mg L ⁻¹)	TS (mg L ⁻¹)
Baseline	82,600	56,100	54,400	31,500	85,900
Treatment					
Clorox®	83,622	48,500 c ^a	56,100 c	24,200 b	76,300 b
Control	80,553	49,500 b	58,400 b	25,300 b	77,000 a
Pi-Quat	84,213	51,500 a	64,600 a	24,000 b	78700a
Tek-Trol	81,078	52,100 a	61,800 b	26,500 a	79,600 a
Virkon™	78,265	47,700 c	52,600 c	23,100 c	75,300 b
Days					
1	80,400	53,400	57,600	31,200	82,100
2	81,200	52,000	61,600	27,800	79,800
5	94,000	51,900	63,500	30,000	79,700
10	84,800	51,300	56,500	20,800	79,200
14	75,000	50,700	57,800	21,200	78,200
21	85,400	48,000	56,100	19,700	74,500
32	71,000	46,800	57,600	22,200	74,000
40	80,400	44,900	58,900	24,100	71,600
ANOVA (P > F)					
Treatment	0.258	0.008	0.001	0.008	0.029
Time	0.004	0.001	0.217	0.001	0.001
Treatment x Time	0.213	0.150	0.117	0.001	0.448

COD = chemical oxygen demand, TVS = total volatile solids, TSS = total suspended solids, TDS = total dissolved solids, TS = total solids.

^a Means without common letters differ significantly ($p < 0.05$).

Table 5b
Disinfectant effects on the physical characteristics of swine slurry.

Variable	EC (dS m ⁻¹)	pH	DM (mg L ⁻¹)
Baseline	27.8	7.60	7.72
Treatment			
Clorox®	25.0 a ^a	7.17 b	7.30
Control	24.8 a	7.17 b	7.43
Pi-Quat	24.3 b	7.17 b	7.53
Tek-Trol	24.2 b	7.27 a	7.64
Virkon™	25.0 a	7.14 b	7.35
Days			
1	26.3	7.60	7.65
2	26.1	7.55	7.56
5	25.9	7.51	7.63
10	25.5	7.37	7.45
14	24.9	7.20	7.40
21	23.8	6.97	7.41
32	22.9	6.73	7.21
40	21.9	6.54	7.30
ANOVA (P > F)			
Treatment	0.019	0.014	0.070
Time	0.001	0.001	0.001
Treatment x Time	0.009	0.021	0.001

EC = electrical conductivity, DM = dry matter.

^a Means without common letters differ significantly ($p < 0.05$).

were measured among the Control, Pi-Quat, and Virkon™ treatments.

The effect of biological additives on nutrient reduction from liquid swine manure under aeration conditions was examined by Zhu et al. (2006). A finishing barn in southern Minnesota was the source of the manure which had pH, NH₄-N, and TN values of 7.77, 3190 mg L⁻¹, and 6240 mg L⁻¹, respectively. These measurements were similar to values obtained for the swine slurry used in the present investigation.

3.6. Antibiotic concentrations for disinfectant experiment

The antibiotic constituents in the disinfectant portion of the study for which significant temporal changes were found included chlortetracycline and tiamulin (Table 7). No distinct trend

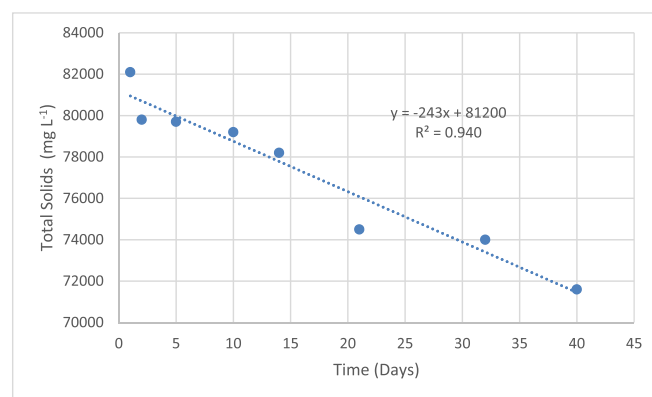


Fig. 5. Total solids content of slurry as affected by time following introduction of disinfectants.

characterizing changes in antibiotic concentrations over time was apparent. Treatment by time interactions were not found for any of the three antibiotics.

The baseline concentrations for lincomycin and tiamulin were less than values obtained for selected dates following addition of disinfectants (Table 7). The breakdown of dry matter during the study may have increased the quantities of these antibiotics contained in the liquid fraction of the slurry.

The chlortetracycline concentration of 8840 ng g⁻¹ measured for Tek-Trol was significantly less than values obtained for the other disinfectants (Table 7). No significant differences in chlortetracycline concentrations were found between the Control and treatments where Clorox®, Pi-Quat, and Virkon™ were added. The tiamulin concentration of 263 ng g⁻¹ measured in the reactors containing Pi-Quat was significantly larger than the other experimental treatments. The reactors containing Tek-Trol had a tiamulin concentration of 28.8 ng g⁻¹ which was significantly less than the other treatments including the Control which had a concentration of 151 ng g⁻¹. The introduction of disinfectants did not significantly affect concentrations of lincomycin.

Table 6a
Disinfectant effects on the chemical characteristics of swine slurry.

Variable	Org N (mg L ⁻¹)	NH ₄ -N (mg L ⁻¹)	NO ₃ -N (mg L ⁻¹)	Total N (mg L ⁻¹)	P ₂ O ₅ (mg L ⁻¹)
Baseline	2800	4420	0.90	7220	9580
Treatment					
Clorox®	3540	3320	1.23	6850	9200
Control	3700	3200	2.62	6900	9270
Pi-Quat	3480	3250	2.57	6730	9080
Tek-Trol	3270	3400	1.99	6670	9140
Virkon™	4000	2870	1.02	6880	9260
Days					
1	3640	3480	1.92	7120	9320
2	3810	3360	1.34	7170	9220
5	3900	3230	2.27	7130	9180
10	3670	3170	1.70	6840	9130
14	3390	3370	2.00	6760	9110
21	3620	3070	1.93	6690	9130
32	3380	3070	1.89	6460	9110
40	3380	2870	1.85	6269	9330
ANOVA (P > F)					
Treatment	0.623	0.798	0.077	0.228	0.717
Time	0.439	0.505	0.001	0.001	0.001
Treatment x Time	0.782	0.859	0.001	0.001	0.307

Org N = organic nitrogen.

Table 6b
Disinfectant effects on the chemical characteristics of swine slurry.

Variable	K ₂ O (mg L ⁻¹)	S (mg L ⁻¹)	Ca (mg L ⁻¹)	Mg (mg L ⁻¹)	Na (mg L ⁻¹)
Baseline	3710	792	2580	2600	983
Treatment					
Clorox®	3640	781	2450	2350	1070 a ^a
Control	3630	791	2450	2280	950 c
Pi-Quat	3620	760	2410	2300	950 c
Tek-Trol	3690	800	2420	2340	1010 b
Virkon™	3620	786	2440	2340	950 c
Days					
1	3680	794	2450	2380	1000
2	3660	782	2440	2350	990
5	3650	792	2400	2300	980
10	3630	775	2390	2300	980
14	3600	768	2420	2340	970
21	3600	779	2450	2310	980
32	3620	766	2420	2260	979
40	3680	811	2490	2330	998
ANOVA (P > F)					
Treatment	0.163	0.139	0.877	0.573	0.001
Time	0.001	0.001	0.087	0.439	0.004
Treatment x Time	0.509	0.009	0.179	0.322	0.341

^a Means without common letters differ significantly ($p < 0.05$).

Jacobsen and Halling-Sorensen (2006) reported chlortetracycline concentrations ranging from 1.1 to 16 mg kg⁻¹ (dw) from six swine production facilities in Denmark. Concentrations of chlortetracycline in 30 liquid manure samples from swine operations in Austria varied from 0.1 to 46 mg kg⁻¹ dry weight (dw) (Martinez-Carballo et al., 2007). Chlortetracycline concentrations in swine production facilities in China ranged from 0.4 to 27, 0.7 to 22, 0.2 to 21, and 5.0–98 mg kg⁻¹ (dw) (Hu et al., 2010; Li et al., 2013; Pan et al., 2011, Zhou et al., 2013). Concentrations of chlortetracycline in liquid manure samples from farms containing swine in Lower Saxony, Germany varied from 1.7 to 55 mg kg⁻¹ (dw) (Widyasari-Mehta et al., 2016). In the present study, chlortetracycline concentrations varied from 8.8 to 12.3 mg kg⁻¹ (dw) (Table 7) which are within the range of values reported by others.

Hu et al. (2010) reported the concentrations of lincomycin in liquid manure from four farms containing swine in northern China varied from 0.12 to 3.8 mg kg⁻¹ (dw) during winter sampling.

Lincomycin concentrations in feces samples from piglets raised on two farms in southern China varied from 0.36 to 17 mg kg⁻¹ (dw) (Zhou et al., 2013). In the present study, lincomycin concentrations ranged from 0.42 to 0.92 mg kg⁻¹ (dw) (Table 7).

Maximum tiamulin concentration in liquid manure from swine farms in Germany were 0.04 and 1.4 mg kg⁻¹ (dw) (Schlusner et al., 2003; Widyasari-Mehta et al., 2016). Concentrations of tiamulin in swine production facilities in the Shandong Province of China varied from 0.08 to 0.17 mg kg⁻¹ (dw) (Pan et al., 2011). Tiamulin concentrations ranged from 0.10 to 0.26 mg kg⁻¹ (dw) (Table 7) in the present study.

4. Conclusions

When compared to the Control treatment, the introduction of additives did not significantly reduce measurements of the physical properties, chemical characteristics, or antibiotic concentrations of

Table 6c
Disinfectant effects on the chemical characteristics of swine slurry.

Variable	Zn (mg L ⁻¹)	Fe (mg L ⁻¹)	Mn (mg L ⁻¹)	Cu (mg L ⁻¹)	B (mg L ⁻¹)
Baseline	224	286	58.0	49.2	3.10
Treatment					
Clorox®	216	265	55.9	47.4	3.70
Control	215	269	56.1	47.5	4.00
Pi-Quat	212	270	54.8	46.5	3.74
Tek-Tron	214	272	55.1	46.6	3.78
Virkon™	216	263	56.5	47.8	3.91
Days					
1	217	273	56.4	47.7	3.97
2	216	274	55.8	47.2	3.62
5	215	264	55.8	47.5	3.89
10	213	264	55.1	46.5	3.81
14	212	267	55.2	46.6	3.65
21	215	263	55.5	47.3	3.75
32	210	265	55.1	46.6	4.05
40	219	271	56.5	48.0	3.87
ANOVA (P > F)					
Treatment	0.823	0.446	0.318	0.301	0.272
Time	0.008	0.159	0.005	0.006	0.544
Treatment x Time	0.125	0.301	0.312	0.219	0.746

Table 7
Disinfectant effects on concentrations of antibiotics in swine slurry (dry weight).

Variable	Chlortetracycline (ng g ⁻¹)	Lincomycin (ng g ⁻¹)	Tiamulin (ng g ⁻¹)
Baseline	12,300	133	61.4
Treatment			
Clorox®	12,300 a ^a	416	103 c
Control	11,200 a	585	151 b
Pi-Quat	10,900 a	916	263 a
Tek-Trol	8840 b	794	28.8 d
Virkon™	10,900 a	557	137 bc
Days			
1	12,300	356	113
2			
5	8210	616	82.8
10			
14	10,300	824	100
21	11,600	715	203
32			
40	11,700	756	184
ANOVA (P > F)			
Treatment	0.024	0.217	0.005
Time	0.003	0.191	0.001
Treatment x Time	0.512	0.463	0.540

^a Means without common letters differ significantly ($p < 0.05$).

swine slurry. The P₂O₅, Ca, Mg, Fe, and Cu values of 5050, 1430, 1320, 165, and 29.2 mg L⁻¹ obtained for the Control treatment were significantly less than values obtained for the reactors where additives were introduced. Measurements of TDS, EC, pH, organic N, NH₄-N, NO₃-N, K₂O, S, Na, B, and tiamulin were not significantly affected by the introduction of additives. No significant differences in values for TVS, TSS, TS, Zn, Mn, and chlortetracycline were found among the Coban® 90, Control, and Manure Magic® treatments.

The introduction of disinfectants did not significantly affect measurements of COD, DM, organic N, NH₃-N, NO₃-N, TN, P₂O₅, K₂O, S, Ca, Mg, Zn, Fe, Mn, Cu, B, and lincomycin. Consistent reductions in TVS, TS, EC, and pH over time were measured for the disinfectant portion of the study. No significant differences in values of TDS and TS were found between the Control and Pi-Quat treatments. Physical properties, chemical characteristics, and antibiotic concentrations of swine slurry may be affected by the introduction of selected additives or disinfectants.

Declaration of competing interest

The authors have no conflicts of interest associated with this study or manuscript.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at <https://doi.org/10.1016/j.envpol.2020.114058>.

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