ACIDIFICATION OF DAIRY SLURRY: EFFECTIVE STRATEGY FOR LOWER GHG EMISSION ON MANURE MANAGEMENT?

MOHD SAUFI, B.[1*], MUHAMMAD SYAHREN, A.[2], MOHD GHAZALI, R.[1], SYAROL NIZAM, A. B.[1], MUHAMMAD AKMAL, H.[1], NUR ALYANI, S.[1], MOHD ALIF OMAR, M.[1], SYUHAIDAH, A. B.[1], RASHIDAH, A. M.[1], MUHAMAD FADZLY, M.P.[1]

1 Climate Change Programme, Agrobiodiversity and Environmental Research Centre, Malaysian Agricultural Research & Development Institute (MARDI), MARDI Headquarters, Persiaran MARDI-UPM, 43400 Serdang, Selangor.

2 Water and Fertilizer Science Research Center, Malaysian Agricultural Research & Development Institute (MARDI), MARDI Headquarters, Persiaran MARDI-UPM, 43400 Serdang, Selangor.

[*]*Corresponding author:* msaufi@mardi.gov.my

ABSTRACT. Dairy farms often use anaerobic lagoons or store slurry, which significantly contribute to greenhouse gas emissions. These storage methods release methane (CH_4), nitrous oxide (N_2O), and ammonia (NH_3) into the atmosphere, intensifying global warming. This study aims to evaluate the efficacy of a single acidification treatment on dairy cattle slurry as a potential method for reducing greenhouse gas emissions from stored slurry. The acidification process was carried out on the high-density polyethylene (HDPE) barrels in controlled environments, and simulated slurry storage in the anaerobic lagoon for over 90 days storage. Slurries were acidified to pH 5.0 using strong and weak acids (HCl and H_2SO_4; HNO_3 and $C_3H_6O_3$). Methane gas release was found inhibited at 36%, and nitrogen loss in the form of NH_3 was inhibited by 47% with the use HCl. The findings of this study provide an important approach towards a better carbon cycle and lower GHG emissions from dairy farms. Dairy manure management is not only a process of decomposing and transforming waste elements to reduce unpleasant odours but to provide a better environment without reducing yield and profits.

***Keywords*:** methane emission, slurry manure, ammonia volatilization, greenhouse gas, acidification

INTRODUCTION

Ruminant livestock is crucial for sustainable food systems as their manures serve as valuable organic fertiliser and utilising them as draught animals can enhance production in areas with limited mechanisation. The livestock sector significantly contributes to the emission of methane (CH_4), nitrous oxide (N_2O), and ammonia (NH_3), which are powerful greenhouse gases (GHGs) that lead to global warming. Methane in ruminants is produced via enteric fermentation through methanogenesis during anaerobic fermentation and from manures by the biological breakdown of animal waste organic materials. While NH_3 undergoes volatilization from the surface of a slurry and contributes to the acidification of watercourses and soil, as well as the eutrophication of nitrogen-depleted ecosystems (Hou *et al*., 2015). The dairy industry in Malaysia has experienced expansion in recent years, evident via the rise in the dairy cattle population and milk product demand. As a result, dairy activity will indirectly contribute to an increase in manure production and greenhouse gas emissions especially from the anaerobic lagoon. Utilising slurry-based manure management in dairy cattle is the most common method, facilitating the simple processing and storage of animal waste in the lagoon. Simultaneously, methanogenesis takes place and emits significant quantities of CH_4 (Jensen & Sommer, 2013).

Methane gas, emitted by methanogens, has a global warming potential (GWP) 28 times higher than CO_2 over a century (Myhre *et al.,* 2013). In Malaysia, CH_4 emissions may have been overlooked since the immediate effect on farm profitability or local ecosystems, animals, or farmers were less visible and due to lack of environmental effect. On the contrary, NH_3 volatilization pertains to the phenomenon wherein nitrogen gaseous in nature escapes from animal excrement into the troposphere, leading to the loss of plant-usable nitrogen fertiliser in manure. Anthropogenic NH_3 emissions indirectly harm the environment by causing eutrophication of water sources and soil acidification (Portejoie *et al*., 2003; Petersen *et al*., 2012). Ammonia's global warming potential is estimated to be 265 times greater than CO_2, contributing to the greenhouse effect and depleting the ozone layer (Sepperer *et al*., 2020). In 2022, Malaysia reported significant CH_4 emissions: 1224.5 Gg CO_2 eq from enteric fermentation and 660.1 Gg CO_2 eq from manure management. An additional 541.9 Gg CO_2 eq of indirect nitrous oxide (N_2O) emissions were originated from livestock manures (Ministry of Natural Resources, 2022). Addressing these emissions is a new challenge in Malaysia, driven by profit-driven livestock practices, with a focus on effective and affordable GHG reduction strategies and technologies

Several strategies for mitigating greenhouse gas emissions have been documented, including the use of inhibitors, dietary modifications, handling of animals, manure management, and technological advancements. Many developed countries use slurry acidification (Overmeyer *et al*., 2023; Regueiro *et al.*, 2016; Santonja *et al.*, 2017). According to Petersen *et al*. (2012) and Fangueiro *et al*. (2015), this technology has demonstrated effectiveness in reducing CH_4 emissions by 67 to 87% and NH_3 emissions by 50 to 88% when manure management and soil application is implemented. This implementation helps in increasing the soil carbon and nitrogen content by slurries application by surface broadcast or partial incorporation into soil (Fangueiro *et al.*, 2016).

The effect of the acidification of dairy cattle slurry during the storage period in reducing GHG and NH_3 emissions in Malaysian climate is unclear. This paper aims to provide a preliminary assessment of the ability of acids to prevent NH_3 volatilization and CH_4 emission from stored slurry. The experiment's specific goal is to ascertain whether acidification with various acids might effectively reduce CH_4 emissions and NH_3 volatilization in the Malaysian climate from dairy cattle slurry.

MATERIALS AND METHODS

Slurry Acidification

Fresh dairy slurry (FS) was collected from a holding pit and slurry handling pond on a private farm near MARDI headquarters. The slurry was obtained from various dairy cattle breeds mainly Australian Friesian Sahiwal with a range age from one to five years old. These animals were fed a total mixed ration (TMR) for dairy cattle at 3% dry matter (DM) basis of bodyweight, which included 60% concentrates and 40% fresh Guinea grass/pastures (% dry matter base). The concentrates are primarily composed of palm kernel expeller (PKE), grinded corn, soya bean meal, soya bean hull, grinded rice hull, crude palm oil (CPO), molasses, and limestone, with less than 0.002% minerals and trace elements added to meet animal growth requirements.

The slurry obtained was kept in a 1,200 L high-density polyethylene (HDPE) poly tank and stored under cover for 48-92 hours prior to use. The slurry physicochemical composition (pH;

oxidation redox potential, ORP; dry matter, DM; volatile solid, VS; carbon and nitrogen was characterised before the experimental design was carried out. Initial slurry characteristics were 2.52 ±0.2% dry matter kg^{-1} slurry (DM), 90.6 ±0.7% volatile solid kg^{-1} DM (VS), total carbon (C) 57.5 ±14.4 mg kg^{-1} slurry, total nitrogen (N) 7.1 ±1.1 mg kg^{-1} slurry and pH 6.5±0.04.

Approximately 60 kg of cattle slurry was transferred into 130 L HDPE barrels where the slurries were then acidified to pH 5.0 using concentrated hydrochloric acid (HCl, Merck, Germany), concentrated sulphuric acid (H_2SO_4, R & M Chemicals Essex, UK), lactic acid ($C_3H_6O_3$, Fisher Scientific, UK) and nitric acid (HNO_3, Fisher Scientific, UK). The acidification took a couple of hours until the pH stabilised and the slurries were stored for over 90 days for observation. During the storage period, NH_3 and GHG sampling was conducted at 12 sampling points periodically. The samples and treatments involved are summarized in Table 1.

Table 1. Summary of treatments used in the study

No.	Abbreviations	Treatments
1	**Ctrl**	Control, no acidification
2	**HCl**	Acidified using HCl
3	**$C_3H_6O_3$**	Acidified using lactic acid
4	**HNO_3**	Acidified using nitric acid
5	**H_2SO_4**	Acidified using sulfuric acid

Water Loss (Moisture), Temperature, pH and Oxidation Redox Potential (ORP)

Water evaporation and loss in the slurry was measured with a digital scale FX5000i manufactured by AND Company Limited in Japan. The oxidation reduction potential (ORP), temperature, and pH of the slurry were all assessed through the utilisation of a Hanna pH electrode instrument (model HI 991003; Hanna Instrument, USA). The humidity and temperature of the surrounding environment were monitored and recorded utilising an EasyLog EL-USB-2-LCD (Lascar Electronic, UK).

Slurry Dry Matter (DM) and Volatile Solids (VS) Content

The volatile solids (VS) and slurry dry matter (DM) were ascertained by subjecting 10 g slurry samples to drying at a temperature range of 80 °-105 °C for 16 hours at a constant weight and as loss-on ignition at 450 °C for 16 hours in a muffle furnace Carbolite CWF 1200 (Carbolite Ltd, UK).

Total Carbon (C) and Nitrogen (N)

The total C and N content of fresh slurry were measured using the Elementar Analyzer (CHNOS) type Vario Macro Cube (Elementar, Germany).

Greenhouse Gases Estimation

Methane Emission

Greenhouse gas fluxes were monitored on the barrel headspace through a butyl rubber septum in a closed system. Headspace gas samples were collected immediately (T0) after securing the lid in place, at 30 minutes (T30) and 60 minutes (T60). Gas samples were collected in 20 mL pre-evacuated gas vials and analysed using an Agilent 7890B gas chromatograph. The GC was equipped with J&W Scientific CS- Gaspro 45 m X 0.320 µm capillary columns and a flame ionised detector (FID). Gas fluxes were computed using the linear rise in gas concentration between the T0 and T60 samples over a one-hour period, as well as the headspace volume and slurry weight. Cumulative gas emissions over the storage period were determined by interpolating the values between adjacent sample sites using the trapezoidal rule (Cardenas *et al*., 2010).

Relative Ammonia Volatilisation

Concurrent NH_3 sampling was carried out during the gas sampling period. A 0.02 M orthophosphoric acid (H_3PO_4) passive trap was used to measure relative NH_3 volatilisation in a non-ventilated environment (Misselbrook & Powell, 2005). The acid trap was carefully suspended inside the headspace, 10-20 cm from the lid. Following the one-hour 'incubation', the traps were removed and the ammonium-N (NH_4^+N) concentration in the H_3PO_4 acid was determined as described by Mulvaney (1996). Prior to incubation at 30 °C, 15 µL of 6% disodium salt of ethylenediaminetetraacetic acid (Na_2EDTA), 60 µL of Na-Salicylate-nitroprusside and 30 µL of hypochlorite solution were added. Na-Salicylate-nitroprusside solution consisted of 7.8% ($^w/_v$) Na-Salicylate and 0.125% ($^w/_v$) Na-nitroprusside, while hypochlorite solution (pH 13) contained 2.96% ($^w/_v$) sodium hydroxide (NaOH), 9.96% dipotassium phosphate (K_2HPO_4) ($^w/_v$) and 10% ($^v/_v$) Na-hypochlorite. Absorbance measurements were taken 30 minutes after incubation using a SpectraMax ABS microplate reader at a 667 nm wavelength and processed with SoftMax Pro 7 (Molecular Devices, USA).

RESULTS AND DISCUSSION

General Physiochemical Observation

The characteristic of the slurry taken from the reception pit in this study was obviously at the lower end (Table 2) (Agriculture and Horticulture Development Board, 2023). The results showed that the Ctrl slurry used had 2.52% Fwt DM with 90.6% VS DM^{-1} content, and insignificant change after over 90 days storage period. Low dry matter content was subjected to the uses of solid-liquid separators at the same collection pond. There were no significant changes between treatments on DM and VS content (data not shown). Thus, other perimeters were subjected to the effect of the low dry matter content available. The slurry pH was near neutral at pH 6.5 and rose to pH 7.6 on the end of storage period. Lower C:N ratio at 7.5:1 led to rapid mineralization and release of nitrogen, which makes available for a plant uptake. The slurry temperature recorded is following the ambient temperature. After over 91 days of storage, the decrease in total C and the increase in NH_4 content indicated mineralization or biodegradation of organic matter in the slurry. In addition, acidification resulted in higher NH_4^+ content (Table 3). This NH_4^+ content is represented as available N for root uptake.

Table 2. Slurry characteristic and physiochemical properties

Slurry Characteristics	Start (Day 0)	End (Day 91)
Dry Matter (% Fwt)	2.52 ±0.18	2.76 ±0.39
Volatile solid (% VS /DM)	90.61 ±0.74	81.44 ±1.33
NH_4^+N Content (mg/kg slurry)	19.5 ±1.02	48.1 ±2.16
pH	6.5 ±0.04	7.6 ±0.03
ORP (mV)	-169 ± 7.6	-136.6 ±49.5
Total C (mg/kg)	57.8 ±14.4	14.7 ±0.02
Total N (mg/kg)	7.7 ±1.0	234.6 ±0.65
Slurry temperature (°C)	29.7 ±0.15	27.8 ±0.23

Table 3. Slurry ammonium content during storage period, n = 5

Treatments	NH_4^+N Content (mg/kg slurry, ±SEM)	
	Day 0	Day 91
Ctrl	19.5 ± 1.02^a	48.1 ± 2.16^a
HCl	$51.1\ 3.00^b$	64.3 ± 4.39^b
$C_3H_6O_3$	46.9 ± 3.91^b	58.2 ± 4.18^b
HNO_3	54.8 ± 2.61^b	53.4 ± 4.83^b
H_2SO_4	47.4 ± 3.54^b	67.3 ± 4.58^b

*Values represent mean ± SEM (n = 5). Different superscript letters between rows and columns indicate statistical significance

Moisture Loss and Temperatures

The experiment was conducted at open space, free air and wind flow with constant ambient temperature and humidity (Figure 1-i). The average ambient temperature and humidity were at 28.5 °C and 79.9% Rh. Meanwhile, the slurry temperature fluctuated during the entire storage period between 25.7 °C and 29.7 °C which is similar to the ambient temperature. There were no changes of slurry temperatures following acidification and changes from an anaerobic fermentation. The moisture emission from slurry surface was relatively constant among the treatments (Figure 1-ii, iii). The loss from slurry surfaces was passive with influence of surface area, the ambient temperature, and humidity level as well as the wind disturbance. Total moisture loss from all treatments ranged between 25.5 to 28.7%. This emission is expected to be higher if the slurries stored in open space without shed which exposed to direct sun.

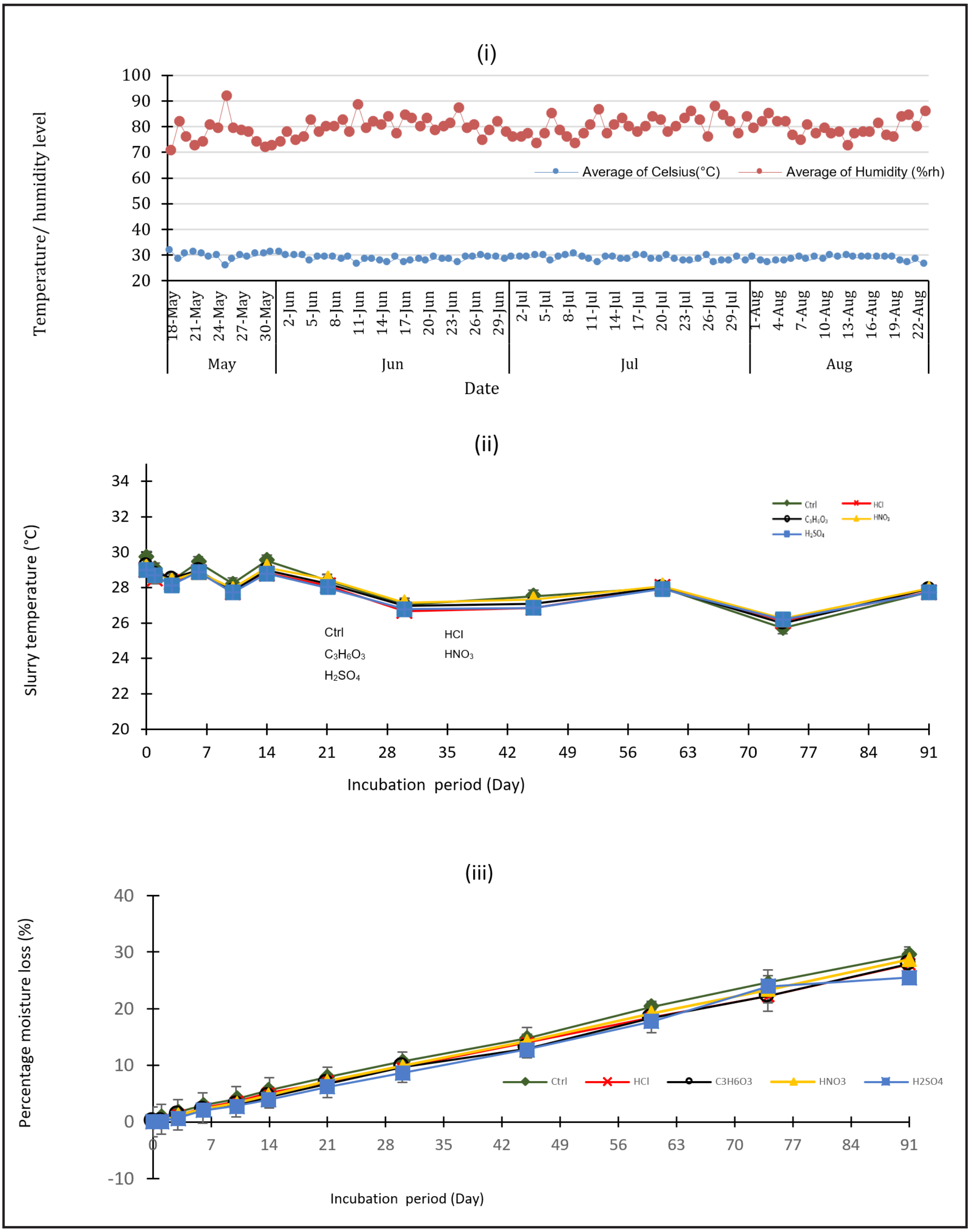


Figure 1. The (i) ambient temperature and humidity, (ii) slurry temperatures and (iii) moisture loss during storage period

The Oxidation Redox Potential (ORP) and pH Dynamic

The slurry ORP indicated the oxidation reduction level in the slurry and showed near to positive values after acidification processes (Figure 2-i). However, the ORP values gradually decreased to between -183 and -250 mV, except H_2SO_4 which was lower than -300 mV. This is possibly due to factors such as temperature, dissolved oxygen, and reactive elements within slurry which may affect the ORP-pH relationship. Meanwhile, Figure 2-ii shows that the acidification processes allowed the slurries pH remain acidic (below pH 6.0) for 10 days using weak acid (HNO_3 and $C_3H_6O_3$) and longer up to 14 and 30 days by using strong acids (HCl and H_2SO_4). The rise of slurries pH resulted from anaerobic microbial fermentation on organic matter. While the slurries pH near to pH 8.0 occurred as the solubility of CSO_2 is 200x lower than that of NH_3, a greater quantity of CO_2 is lost than NH_3 (Portejoie *et al*., 2003).

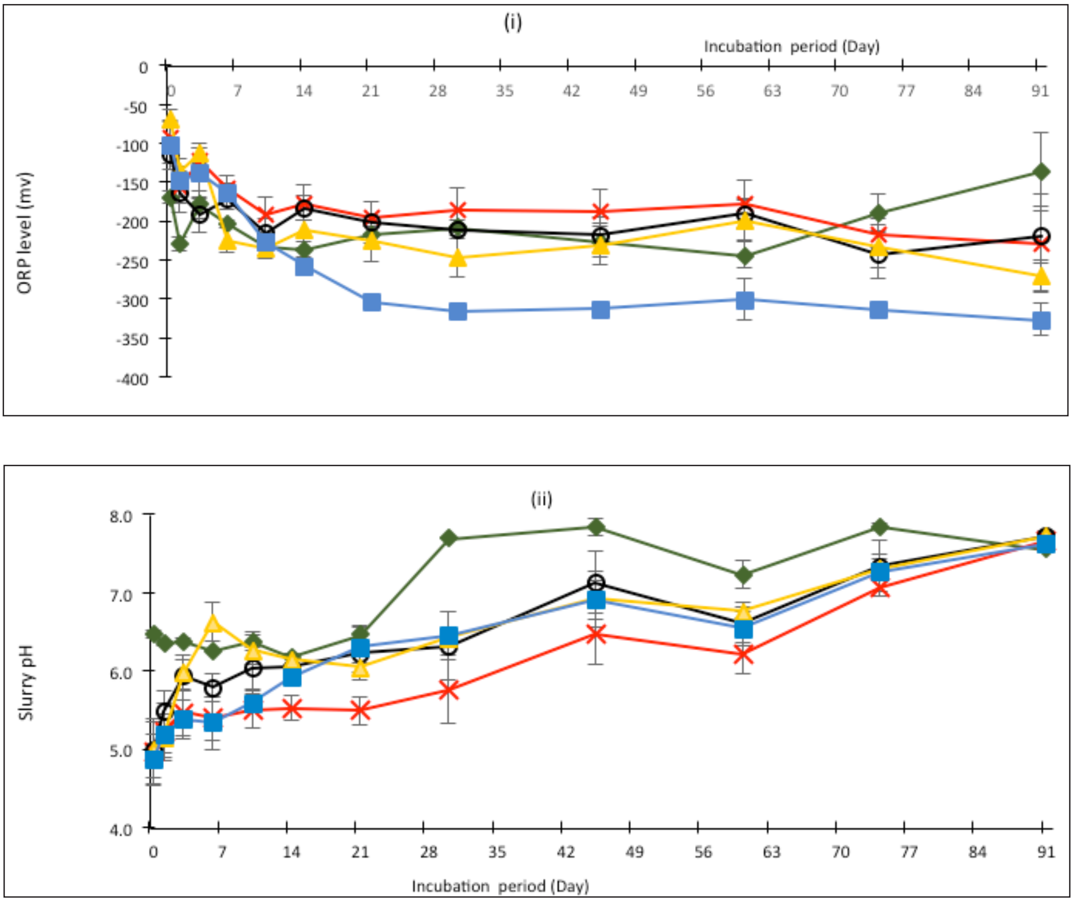


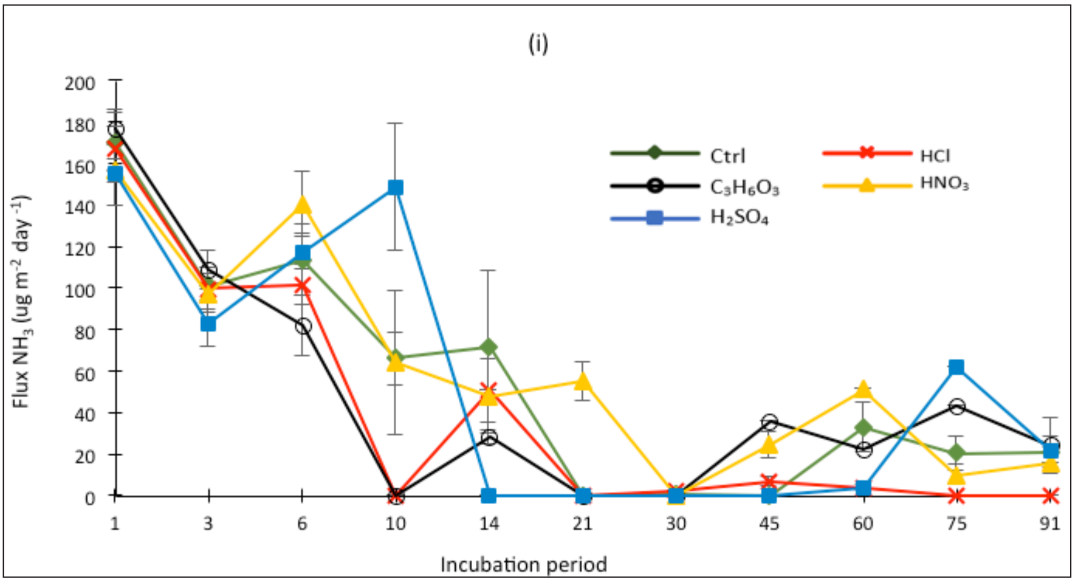
Figure 2. Slurry (i) ORP and (ii) pH during storage period

Slurry Acidification and GHG Emission

In this experiment, acidifications were carried on the HDPE barrels, controlled environments, and to simulate slurry storage in anaerobic lagoon. Early acidification such as at the beginning of the manure management chain reduced larger amounts of emissions in animal housing, slurry storage and after field application. Acidification of slurry can occur at many points in the manure management process. Acidification in the animal housing was done by pumping acidified slurry into the storage area under the slatted floors. On the other hand, if acidification was done in enclosed structures like barns, it could endanger human health and cause injury to animals.

Ammonia Volatilisation

Ammonia volatilisation is considered a nitrogen loss from the slurry. In this observation, except HNO_3 treated slurries, all slurries showed lower relative NH_3 loss during storage period compared to Ctrl. Although volatilisation fluxes indicated inconsistent rate during observations. The acidification resulted in inhibition of NH_3 volatilization at 47% by HCl, 27% by H_2SO_4 and 21.9% by $C_3H_6O_3$ during the storage period. The use of HNO_3 led to higher NH_3 loss by 9.1% compared to Ctrl. The NH_3 volatilisation was influenced by the aquas-gas equilibrium and acid base equilibrium of the slurry.



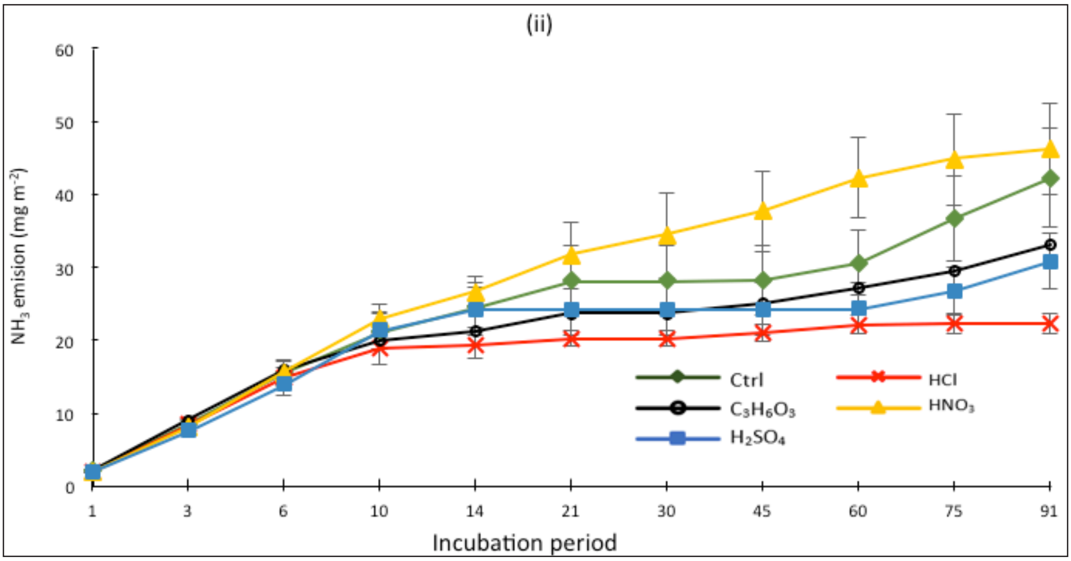
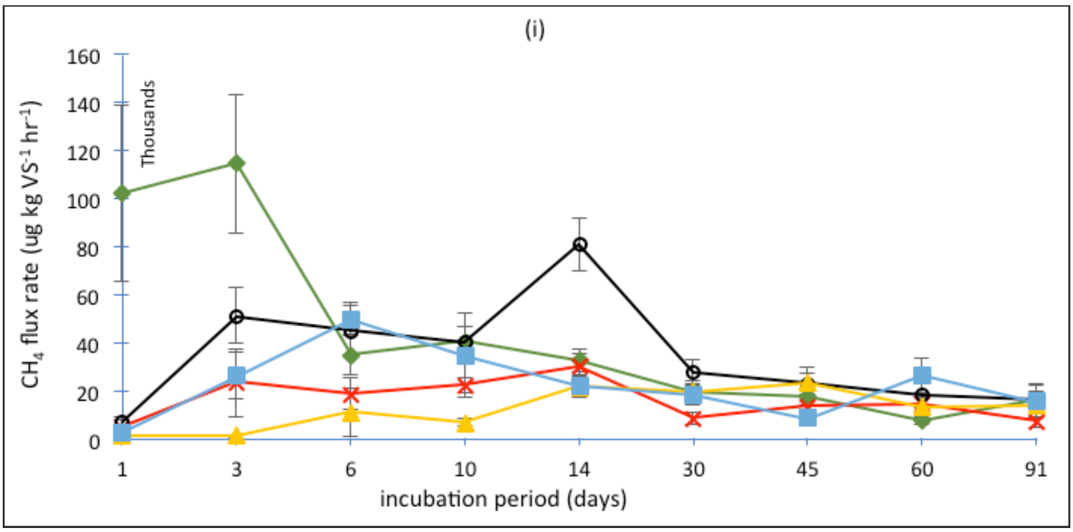


Figure 3. Observation on ammonia volatilisation, i) Ammonia fluxes rate, ii) Cumulative ammonia volatilisation loss during storage period

Methane Carbon Emission

In this study, Ctrl slurry had shown higher fluxes rate compared to acidified slurries (HCl, HNO_3, $C_3H_6O_3$, H_2SO_4) (Figure 4-i) especially during the first 30 days, except $C_3H_6O_3$ on day 14 showed two times higher than Ctrl fluxes. This is unknown and possible due to disturbance during sampling resulting in releases in CH_4 ebullition. The acidification reduced CH_4 emission by inhibiting the microbial activities and the breakdown of organic material was retarded. It had shown that the HCl indicated lowest cumulative emission at



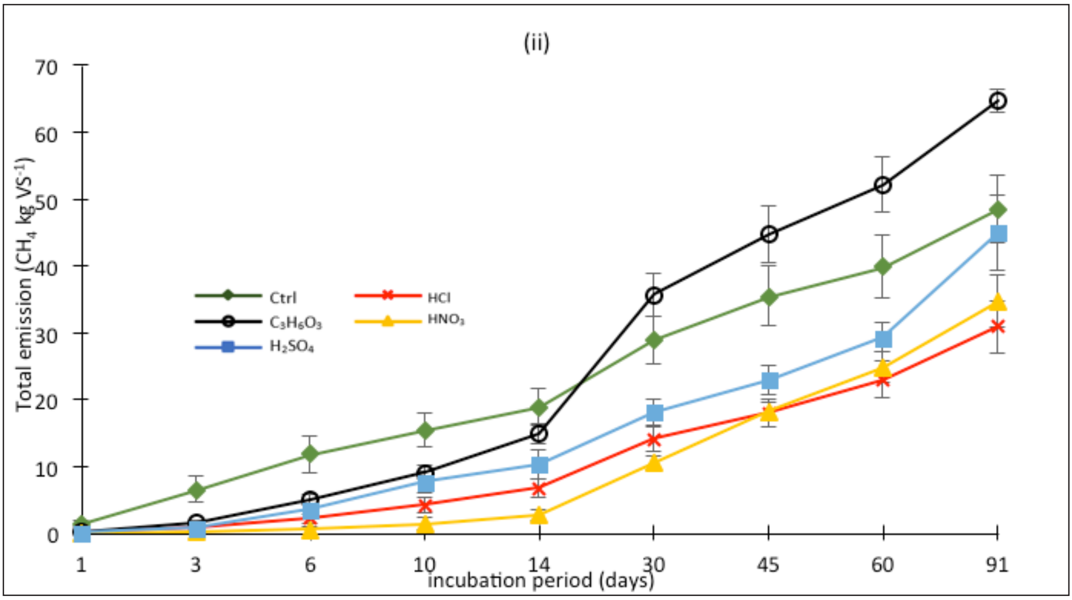


Figure 4. Observation on methane emission, i) Methane fluxes rate, ii) Cumulative methane emission during storage period.

30.9 CH_4 kg VS^{-1} over half that of Ctrl (64.6 CH_4 kg VS^{-1}). As stronger acid (HCl and H_2SO_4) can retain the slurries pH acidic longer than HNO_3 and lactic acid, thus CH_4 emitted was higher than HCl and H_2SO_4. However, this study showed that HNO_3 has a better inhibition on CH_4 emission than H_2SO_4. There is unclear reason on this matter probably due to the low dry matter content as the slurries were collected from a pit with a solid separation system.

Greenhouse Gases Emissions

Slurry acidification has been researched and tested over 3 decades and is now being used at the farms in a restricted number of countries (Fangueiro *et al*., 2015). However, there is lack of studies conducted in Malaysian farms. A reviewed study has shown a significant reduction on NH_3 and CH_4 emissions with a range of inhibition of 27-88% with the uses of various acids and 40 to 65% on CH_4 emission with the uses of HCl (Fangueiro *et al*., 2015). Further reduction could be attained by acidifying the manure at the location where it is produced, as opposed to after it is applied to pastures in the field. The current study determined that the most effective use of HCl to reduce greenhouse gas emissions was to inhibit the volatilization of CH_4 and NH_3 at 36%, and 47% respectively. Choosing the right chemical is crucial because using H_2SO_4 can result in increased production of hydrogen sulphide (H_2S) gas and higher sulphur content in the slurry (Wang *et al*., 2014). Conversely, HNO_3 results in substantial N_2O production (Berg *et al*., 2006). Greater volume of acid is needed if slurries were acidified by weak acid such as lactic acid (pKa 3.86) (Berg *et al.* 2006). Regeneration of CH_4 emission may occur when the pH of the slurry returns to the normal range, unless the low pH slurry is maintained for an extended period of time. (Petersen *et al*., 2012). The incremental rise was comparable to the results reported in Petersen's (2012) study on a 3-month storage trial. Therefore, periodic acid addition or re-acidification is necessary until all slurry is removed from storage (Haeussermann *et al*., 2006). Even though acidification increases farming operations costs, there are benefits for the farm, such as increased N availability for plant uptake (Sørensen & Eriksen, 2009). Acidification may result in increased organic matter retention than non-acidified slurry due to reduced biodegradation activity (Sørensen & Eriksen, 2009).

Table 4. Summary of CH_4 emission and NH_3 loss after 90 days of incubation

No.	Sample	CH_4 Emission		NH_3 Loss	
		Emission CH_4 (mg/kg VS)	of inhibition%	Loss NH_3 (mg/m^2)	of inhibition%
1	**Ctrl**	48,450.5	-	42.24	-
2	**HCl**	30,851.4	36.3	22.32	47.1
3	**H_2SO_4**	29,205.0	39.7	30.82	27.0
4	**HNO_3**	34,581.0	-28.6	46.09	-9.1
5	**$C_3H_6O_3$**	64,587.9	33.3	32.98	21.9

CONCLUSION

This study concludes that the acidification of dairy slurry is able to reduce CH_4 emission and NH_3 loss. The CH_4 emission was inhibited by 36%, and nitrogen loss in the form of NH_3 can be inhibited by 47% the use HCl. Other acids showed lesser percentages with various rates. Thus, acidification is a useful and effective approach in reducing the GHG emission from slurry manure. However, larger scale experiment or field observation is needed in evaluating the uses of these acids in their mitigation potential as this will add significant cost to the farmers management practices. The need for animal protein rises along with the global population, making rapid mitigation across the border necessary to maintain sustainable livestock production without increasing the anthropogenic gases in the atmosphere.

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