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Research Article

Combined Effects of Acidification, Zeolite, and Biochar on Ammonia Emission and Nitrate Leaching from Pig Slurry

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ABSTRACT

This study aimed to evaluate the efficiency of combining acidification with adsorbents (zeolite and biochar) to mitigate the environmental impacts of pig slurry, focusing on ammonia (NH_3) emission and nitrate (NO_3^-) leaching. The four treatments were applied: 1) pig slurry (PS) alone as a control, 2) acidified PS (AP), 3) acidified pig slurry with zeolite (APZ), and 4) acidified pig slurry with biochar (APB). The AP mitigates NH_3 emission and NO_3^- leaching compared to PS alone. Acidification reduced the cumulative NH_3 emission and its emission factor by 35.9% and 12.5%, respectively. The APZ and APB increased NH_4^+-N concentration, with the highest level in APB, compared to AP. The NH_4^+ adsorption capacity of APB (0.90 mg g^{-1}) was higher than that of APZ (0.63 mg g^{-1}). The APB and APZ treatments induced less NH_3 emission compared to AP. The cumulative NH_3 emission was reduced by 12.2% and 27.6% in APZ and APB, respectively, compared to AP treatment. NO_3^- leaching began to appear on days 12 and 13, and its peak reached on days 16 and 17, which were later than AP. The cumulative NO_3^- leaching decreased by 17.7% and 25.0% in APZ and APB, respectively, compared to AP treatment. These results suggest that combining biochar or zeolite with acidified pig slurry is an effective method to mitigate NH_3 emission and NO_3^- leaching, with biochar being particularly effective.

(**Key words**: Acidification, Ammonia emission, Biochar, Nitrate leaching, Pig slurry, Zeolite)

Ⅰ. INTRODUCTION

The global demand for meat products is steadily expanding due to the growing world population. Pork production has grown considerably over the past 50 years in response to the increasing demand (Lassaletta et al., 2019). This increase in pork production causes a rapid increase in manure production, which results in environmental problems due to limited cropland for recycling all manure produced. In particular, ammonia (NH_3) emission from pig slurry lead to pollution, order, and imbalance in the ecosystem. To mitigate the environmental impact of pig slurry, various solutions were investigated such as spreading and injection techniques (Tóth et al., 2022), storage treatments (Chen et al., 2024), biologic treatment (El bied et al., 2023), acidification (Park et al., 2018; Lee et al., 2022) and adsorption (Park et al., 2024). Among these approaches, the acidification of pig slurry and the adsorption methods have gained notable attention.

Acidification of pig slurry is known to be an effective method for the inhibition of NH_3 emission associated with agricultural waste processing. Various acids such as acetic acid (Regueiro et al., 2022), hydrochloric acid (Overmeyer et al., 2021), and sulfuric acid (Park et al., 2018; Lee et al., 2022) have been investigated. Sulfuric acid is the most popular method for the mitigation of NH_3 emission because of its low cost and additional sulfur fertilizer effect. In our previous study, acidified pig slurry with sulfuric acid reduced NH_3 emission throughout the experiment (Park et al., 2018). However, the acidification treatment alone has limitations in reducing NH_3 emission, raising the need for additional treatment methods.

Recently, adsorption such as biochar and zeolites has been regarded as one of the most effective and competitive methods

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for the mitigation of NH_3 emission due to its high cation-absorbing capacity with high surface area, negatively charged, and porous structure (Fahad et al., 2018). Zeolite application mitigates NH_3 emission from pig slurry-applied fields (Park et al., 2024) and in a laboratory incubation (Ferretti et al., 2017). The application of zeolite or combined biochar and zeolite to stored duck manure significantly reduces NH_3 emission (Banik et al., 2023). Similarly, Wang et al. (2017) observed that the combined application of biochar and zeolite effectively mitigated NH_3 emission from pig slurry. However, methods combining acidification treatment and cation adsorbents have not yet been sufficiently studied.

The present study aimed to investigate the effects of biochar or zeolite on reduction of NH_3 emission in acidified PS. Additionally, this study evaluates the potential N losses through nitrate leaching from the soil. This complex approach is expected to provide a more effective NH_3 reduction method by combining the advantages of each treatment method.

Ⅱ. MATERIALS AND METHODS

1. Experimental design

This study was conducted at Chonnam National University in Gwangju, South Korea. The experimental treatments consisted of four different pig slurry applications with three replicates: 1) pig slurry (PS) alone as a control, 2) acidified PS (AP), 3) acidified pig slurry with zeolite (APZ), and 4) acidified pig slurry with biochar (APB). The PS was obtained from the ECOBIO farming/agricultural association corporation (Namwon, Korea) and acidified slowly (to avoid foaming) by adding 1.5 M sulfuric acid until pH 6.0 (Table 1). The PS and acidified PS were applied at a rate of 200 kg N ha^{-1}. The pot and column experiments were conducted to evaluate the ammonia (NH_3) emission and nitrate (NO_3^-) leaching, respectively. The pot (0.32 m × 0.25 m × 0.3 m) and column (8 cm diameter × 25 cm) were filled with 21 kg and 1 kg soil mixed with zeolite (10%, w/w) or biochar (5%, w/w), respectively. The physiological characteristics of soil, zeolite, and biochar are presented in Table 2. The soil was moderately coarse-textured sandy loam (clay 10.4%, silt 27.3%, and sand 62.3%) and was collected from an agricultural field at Chonnam National University.

2. Soil, gas, and leachate sampling

The soil sampling was conducted in each treatment pot and column using a 3 cm diameter tube auger to collect soil cores at 0–5 cm depth randomly. The collected soil samples were air-dried, ground, and sieved to a particle size of <0.15 mm. The soil samples were stored in dry conditions for chemical analysis. Airtight acrylic chambers (8.2 cm diameter × 20 cm depth) were inserted to a depth of 5 cm in the pot soil for NH_3 gas sampling. To collect NH_3 emission, the acid trap system method described by Ndegwa et al. (2009) was adopted with minor modifications. Each chamber was connected (via a septum located in the lid of the chamber) to NH_3-N trapping bottles containing 150 mL of 0.2 mol L^{-1} H_2SO_4 (equivalent to 0.03 moles of acid). The other glass tube was connected to the vacuum

Table 1. Chemical properties of soil and pig slurry

	pH (1:5 w/s)	EC (ds m^{-2})	Available P (mg kg^{-1})	OM (%)	Nitrogen (mg N kg^{-1})			Exchangeable cation ($cmol^+$ kg^{-1})		
					NH_4^+	NO_3^-	Total N	K	Ca	Mg
Soil	7.11	0.71	255	2.19	24.4	13.4	150	0.21	2.67	1.74
PS	7.45	0.76	700	0.75	170.8	7.4	860	-	-	-

PS, pig slurry; EC, electric conductivity; OM, organic matter.

Table 2. Physiological characteristics of zeolite and biochar

	pH (1:5 w/s)	EC (ds m^{-2})	CEC ($cmol^+$ kg^{-1})	Total N (%)	Specific surface area (m^2 g^{-1})
Zeolite	7.5	0.71	2.64	0.28	47.2
Biochar	7.4	0.76	7.32	0.24	237.8

EC, electric conductivity; CEC, cation exchange capacity.

system that created an airflow through the chambers at a constant rate of 1.5 L per min to exhaust the NH_3-scrubbed air. Each chamber was closed and clamped with attached silicon sealing for 24 h. Potential NH_3 emission was determined daily for 40 days. The NO_3-leaching samples were collected from the soil column with PVC end caps on the bottoms. A hole was drilled through the end caps, and drain tubes (3 mm i.d.) were attached to the bottom of each column. The columns were incubated in a constant temperature room (25°C and 80 % relative humidity). The leachate from each column was collected in a 250 mL polyethylene bottle for ~24 h after the start of a leaching event. The amount of leachate collected daily for the 40 days in each column was determined gravimetrically.

3. Measurement and chemical analysis

The Kjeldahl procedure was performed to determine the total N content (Bremner, 1996). For inorganic N, extraction was carried out using 2 M KCl, and the NH_4^+-N was measured by distillation in an alkaline medium (MgO). The NH_4^+ adsorption was calculated using the method described by Phuong et al. (2021). The same method was applied to determine NO_3^--N after reducing it with Devarda's alloy (Lu, 2000). The NH_3 concentration in the acid trap solution, such as $(NH_4)_2SO_4$, was determined using Nessler's ammonium color reagent (Nessler's reagent, Sigma, St. Louis, MO, USA) after microdiffusion in a Conway dish (Kim and Kim, 1996). It was expressed as the NH_3-N content emitted per hectare. The concentration of NO_3^--N in leachates was determined using the Cataldo reagent, as previously described by Cataldo et al. (2021)

4. Statistical analysis

A completely randomized design was used with three replications per treatment. Duncan's multiple range test was used with the means of separate replicates. Statistical significance was postulated at $p<0.05$. Statistical analysis in all measurements was performed using SAS 9.1.3 software (SAS Institute, Cary, NC, USA).

Ⅲ. RESULTS

1. Ammonium-N (NH_4^+-N) concentration and NH_4^+ adsorption capacity

The NH_4^+-N concentration and NH_4^+ adsorption capacity were measured in PS, acidified PS (AP), acidified pig slurry with zeolite (APZ), and acidified pig slurry with biochar (APB) on days 0 and 40 (Fig. 1). The application of biochar and zeolite to acidified PS resulted in higher NH_4^+-N concentration compared to PS and AP. The highest level was observed in APB (Fig. 1A). NH_4^+ adsorption capacity was assessed for the APZ

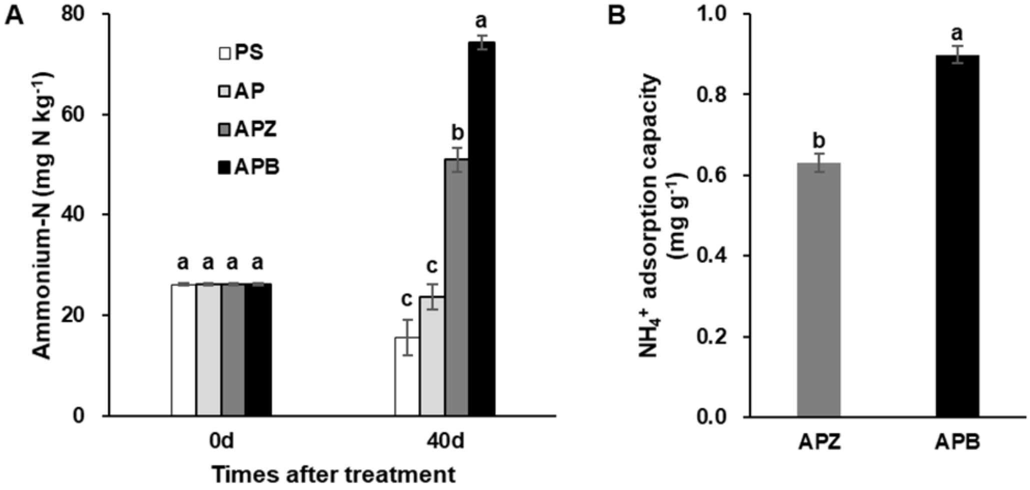

Fig. 1. Amount of (A) soil ammonium−N (NH_4^+−N) and (B) NH_4^+ adsorption capacity in pig slurry (PS, white), acidified pig slurry (AP, light gray), acidified pig slurry with zeolite (APZ, dark gray), and acidified pig slurry with biochar (APB, black). The values are presented as means ± s.d. (n = 3). The different letters at each sampling date present significant differences at $p<0.05$ by Duncan's multiple range test.

and APB treatments. NH_4^+ adsorption was higher in APB (0.90 mg g^{-1}) than in APZ (0.63 mg g^{-1}) (Fig. 1B). These results suggest that both biochar and zeolite enhance NH_4^+ adsorption more effectively, with biochar demonstrating superior performance in acidified conditions.

2. Ammonia (NH_3) emission

The daily NH_3 emission was highest during the first 12 days after PS application, with or without acidification (Fig. 2). Subsequently, the NH_3 emission slowly decreased to the end of the experimental period. The acidification with PS with biochar or zeolite mitigated NH_3 emission by less than 50% compared to PS alone. The lowest level was observed in APB throughout the experimental period. The cumulative NH_3 emission from PS was 43.5 ± 0.1 kg ha^{-1} and showed the highest level (Table 3). The cumulative NH_3 emission from AP, APZ, and APB was reduced by 35.9%, 43.7%, and 53.6%, respectively, compared to PS alone. The NH_3 emission factor was significantly higher in PS than in acidified PS. APB showed the lowest NH_3 emission factor. These results indicate that acidification of PS mitigates NH_3 emission, with biochar being particularly effective.

3. Nitrate (NO_3^-) leaching

NO_3^- leaching profiles revealed distinct patterns among the treatments (Fig. 3). NO_3^- leaching in PS and AP was first observed on 9 days after treatment and peaked on 14 days, with PS showing higher levels than AP. In APZ and APB, NO_3^- leaching occurred from days 12 and 13, and peaked on days 16 and 17, respectively. The peak height was lower in APZ and APB than in AP and PS. NO_3^- leaching decreased rapidly after peaking and maintained a consistent level after day 25 of treatment. The cumulative NO_3^- leaching level in PS was the highest but it was reduced by 12.3%, 27.9%, and 34.3% in AP, APZ, and APB, respectively, compared to PS. Similarly, the NO_3^-

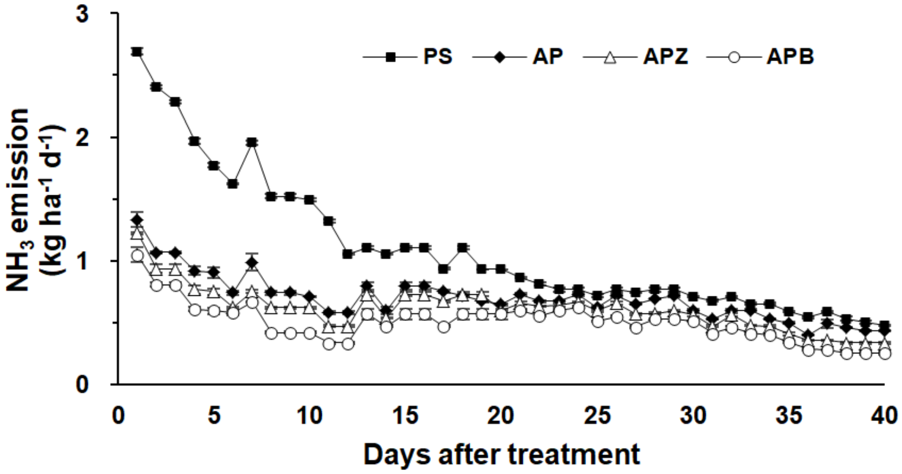

Fig. 2. Amount of daily ammonia (NH_3) emission from pig slurry (PS), acidified PS (AP), acidified pig slurry with zeolite (APZ), and acidified pig slurry with biochar (APB). The values are presented as means ± s.d. (n = 3).

Table 3. Total cumulative ammonia (NH_3) and nitrate (NO_3^-) leaching and their emission factor (EF) from pig slurry (PS), acidified pig slurry (AP), acidified pig slurry with zeolite (APZ), and acidified pig slurry with biochar (APB)

	Total NH_3 (kg ha^{-1})	EF for NH_3	Total NO_3^- leaching (μM)	EF for NO_3^-
Pig slurry (PS)	43.5±0.1^a	14.5±0.0^a	218.3±3.2^a	72.8±1.1^a
Acidified PS (AP)	27.9±0.2^b	9.3±0.1^b	191.0±4.1^b	63.7±1.4^b
AP + Zeolite (APZ)	24.5±0.0^c	8.2±0.0^c	157.3±1.7^c	52.4±0.6^c
AP + Biochar (APB)	20.2±0.4^d	6.7±0.1^d	143.5±4.6^d	47.8±1.5^d

The values are presented as means ± s.d. (n=3).
The different letters between treatments indicate significant differences at $p<0.05$ by Duncan's multiple range test.

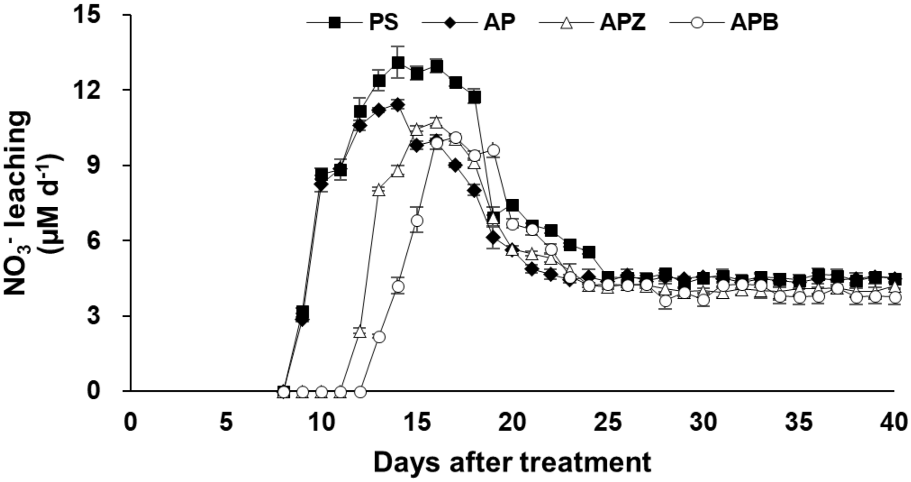

Fig. 3. Amount of daily nitrate (NO_3^-) leaching from pig slurry (PS), acidified pig slurry (AP), acidified pig slurry with zeolite (APZ), and acidified pig slurry with biochar (APB). The values are presented as means ± s.d. (n = 3).

leaching emission factor, which was highest in PS, was significantly mitigated with the addition of zeolite and biochar with acidified PS (Table 3). These findings suggest that biochar and zeolite are effective in reducing NO_3^- leaching under acidified PS conditions.

Ⅳ. DISCUSSION

Acidification of PS has been widely reported to mitigation of NH_3 emission. As shown in this study, AP mitigated daily NH_3 emission by 50.6% on day 1, compared to PS, which was confirmed 35.8% lower in cumulative NH_3 emission (Fig. 2; Table 3), which is similar to the result observed by Fangueiro et al. (2015). In our previous study, we found that the lower pH of the PS is directly associated with low NH_3 emission following slurry application to soil (Park et al., 2018). Given that conversion of NH_4^+ to NH_3 occurs at high pH, acidification of PS inhibits this process by reducing the pH of PS, resulting in mitigation of NH_3 emission. The difference in daily NH_3 emission between PS and AP was greatest for the first 12 days of the experiment and gradually decreased thereafter (Fig. 2). Acidification led to significant reductions in the soil NO_3^- leaching, indicating that nitrification of NH_4^+-N may delay or inhibit, in accordance with previous findings (Kai et al., 2008; Fangueiro et al., 2015). These results suggest that acidification of PS effectively mitigates NH_3 emission, consequently reducing NO_3-leaching.

The application of zeolite and biochar in acidified PS additionally reduced NH_3 emission (Fig. 2). Biochar with acidified PS (APB) showed the lowest daily NH_3 emission and reduced by 53.5% cumulative NH_3 emission compared to PS (Fig. 2; Table 3). These results are similar to those of Baral et al. (2023) who reported a reduction of NH_3 emission by 37-51% in the month of storage by acid-activated biochars. In addition, a 16-25% reduction in NH_3 emission was observed in biochar-applied PS (Meiirkhanuly et al., 2020). Biochar has porosity and surface area that are effective for NH_4^+ adsorption. It has been reported that acid activation increases biochar pore volume, biochar surface area, and functional groups (Fahad et al., 2016), which is pH-dependent, with the highest adsorption efficiency in the pH range of 4-8 (Kizito et al., 2015). These were confirmed in the present study that APB showed the highest NH_4^+-N in soil (Fig. 1A). Similarly, zeolite also mitigated daily NH_3 emission, consequently reducing by 43.7% cumulative NH_3 emission compared to PS (Fig. 2; Table 3). Waldrip et al. (2015) reported that manure amended with zeolite reduced the cumulative NH_3 emission by 42% compared to manure alone. The positive effect of zeolite in mitigating NH_3 emission is primarily due to NH_4^+ adsorption on the outer surface of zeolite (Sangeetha and Baskar, 2016) and may be attributed to increased retention of NH_4^+ on the ion-exchange sites (Bernal and Lopez-Real, 1993).

Positive effects on NO_3^- leaching were presented in APZ and APB treatments (Fig. 3). NO_3^- leaching began to appear on days 16 and 17, respectively, which is later than day 9 in the

PS and AP treatments. Additionally, the cumulative NO_3^- leaching amount was reduced by 27.9% and 34.3% in APZ and APB, respectively, compared to PS. NO_3^- leaching reduction following biochar application has been previously reported in field study (Major et al., 2012). These results suggest that biochar and zeolite effectively adsorb NH_4^+, delaying its conversion to NO_3^-, and consequently reducing NO_3^- leaching. Overall, biochar is more effective than zeolite for reducing NH_3 emission and NO_3^- leaching, as shown by the significant decrease from the first day of NH_3 emission and delaying and lowering the peak of NO_3^- leaching (Figs. 2 and 3). These results were consistent with a higher level of NH_4^+ adsorption capacity in APB than APZ (Fig.1), which is due to the porosity and high surface of biochar.

In conclusion, the combined acidification and adsorbent amendments, particularly biochar, offers an approach to reducing nitrogen losses from animal manure, thereby mitigating environmental impacts associated with NH_3 emission and NO_3^- leaching. These findings contribute to the growing knowledge of sustainable manure management strategies and provide practical solutions for reducing the environmental performance of intensive animal production systems.

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