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Comparative effectiveness of zeolite and rice husk biochar in mitigating NH_3 and N_2O emissions as linked to the nitrogen use efficiency of pig slurry during the vegetative growth of *Brassica napus*

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Abstract

Ammonia (NH_3) and nitrous oxide (N_2O) emitted from the pig slurry (PS)-applied soil lead not only to the risk of environmental pollution, but also to the loss of plant nutritional nitrogen (N). This study aimed to compare the effectiveness of zeolite and rice husk biochar (RHB) in mitigating the N losses via NH_3 and N_2O emissions from PS-amended soils, and N use efficiency (NUE) for vegetative growth of oilseed rape (*Brassica napus* L.). Both zeolite and RHB application significantly reduced NH_3 and N_2O emissions from the PS-applied soil and improved the NUE for the vegetative growth. When compared the effectiveness of two materials, RHB application has more positive effectiveness in soil pH buffering, urease activity, organic acid content and NH_4^+ content in soil throughout the experimental period, resulting in a higher mitigation of NH_3 and N_2O emissions as well as NUE for dry matter (DM) and crude protein (CP) production. The enhanced NH_4^+ content in soil closely related to the reduction of NH_3 and N_2O emissions, and with improved NUE for DM and CP production. A superior performance of RHB relative to zeolite is attributed mainly to a stronger capacity of NH_4^+ adsorption by higher oxygen-containing functional groups on its surface, and in part to the supply of bioavailable carbon derived from RHB leading to an increase in urease activity and soil organic acid content. Therefore, RHB can be considered a more promising amendment than zeolite for simultaneously mitigating gaseous N losses and enhancing NUE in oilseed rape cultivation.

Keywords N losses, Nitrogen use efficiency, Oilseed rape, Pig slurry, Rice husk biochar, Zeolite

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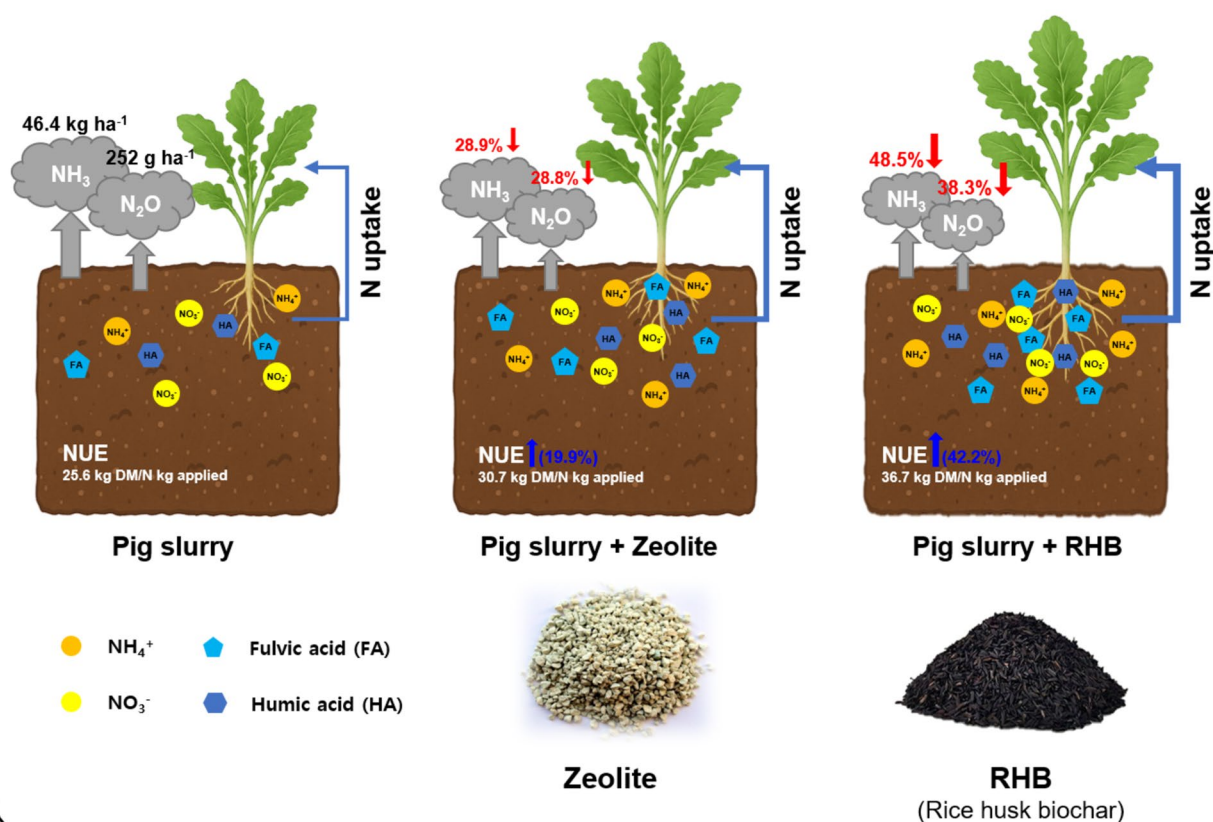
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Graphical abstract

Lower NH_3 and N_2O emissions and higher NUE with RHB application relative to zeolite

Background

The use of pig slurry (PS) as an alternative organic fertilizer has become the most viable recycling options, accounting for more than 80% of recycled animal manure in Korea [1]. When PS is applied to the soil, the loss of volatile NH_3 occurs by the hydrolysis of urea in the urine and the decomposition of organic N in the feces. The NH_4^+ decomposed by urease is readily converted to NH_3 , which is readily lost through volatilization—particularly following PS application, where surface-applied slurry without incorporation facilitates rapid NH_3 emission, making volatilization the dominant pathway of N loss [2]. The NH_3 released into the air rapidly adsorbs to surfaces and significant deposition (up to 20%) may occur within a few hundred meters of the source. The remaining atmospheric NH_3 can rapidly react with a number of acidic compounds (such as nitric acid or sulfuric acid) to form very small secondary aerosol particles (i.e., particulate matter, PM). NH_3 contributes to 50% (EU) and 30%

(US) of $\text{PM}_{2.5}$ air pollution [3]. NH_3 itself and PMs pose a risk to human and animal health and cause environmental degradation. A proportion of NH_4^+ is oxidized to NO_2^- and further NO_3^- , and anaerobic microbial denitrification, in which NO_3^- is reduced to gaseous nitrogen compounds, can generate N_2O , which is a long-lasting greenhouse gas (GHG). Therefore, the emission of NH_3 and N_2O from the PS-applied to agricultural fields is not only of environmental concern, but also represents N losses of organic fertilizer. In this context, numerous practices such as acidification of PS [4, 5] urease and nitrification inhibitors [6] have been attempted to minimizing NH_3 and N_2O emission, while improving the use efficiency of animal manure-derived N.

Materials that adsorb NH_4^+ can retain it in the soil and mitigate NH_3 and N_2O emissions [7, 8]. Zeolites (aluminosilicate minerals) are commonly used for these purposes as their surfaces are negatively charged and have high cation exchange capacity (CEC), enabling strong

electrostatic attraction and selective binding of small hydrated cations, such as NH_4^+ and K^+ [9, 10]. Their NH_4^+ adsorption capacity ranges from approximately 0.7 to 65.3 mg NH_4^+ g $^{-1}$, depending on the mineral purity, particle size, and ionic composition of the solution [11]. Zeolite rapidly adsorbs NH_4^+ onto its exchange sites, and this reversible sorption allows bound NH_4^+ to desorb as solution concentrations decline, thereby serving as a slow-release reservoir [12]. In this context, zeolite has been used to mitigate NH_3 and CH_4 emissions from duck manure [13] and composted food waste [14]. Recently, Manu et al. [15] reported that applying 5–10% zeolite could reduce soil N loss by 34–39%, thereby favoring N conservation. In our previous study [16], we confirmed that zeolite application in PS-amended fields mitigated NH_3 and N_2O emissions and improved nitrogen use efficiency (NUE) and forage production. Similarly, recent researches have more underlined the use of biochars in the perspective of mitigation of N loss to atmosphere and improvement of NUE by crops. It is widely known that biochars, carbon-rich material produced through the pyrolysis of biomass, have high porous structure and large surface area with carrying negative charges which enable the electrostatic adsorption of positively charged NH_4^+ ions [7, 17]. In addition, surface functional groups such as carboxyl ($-\text{COOH}$), hydroxyl ($-\text{OH}$), and phenolic groups can complex with NH_4^+ or contribute to CEC, further enhancing NH_4^+ retention in the soil matrix. Their NH_4^+ adsorption capacity varies widely (2.8–137.3 mg NH_4^+-N g $^{-1}$) depending of feedstock type, pyrolysis temperature, and surface chemistry [18]. These physicochemical properties mitigate the N losses via NH_3 volatilization and N_2O emissions by retaining NH_4^+ in soil [7, 19]. In addition, their carbon-rich porous structure supports microbial colonization, thereby enhancing nutrient cycling and overall soil health [20]. Biochar derived from residues has substantial environmental and economic advantages. Rice husk biochar (RHB) is produced via slow pyrolysis of rice husk at low to moderate temperatures [21]. Converting rice husk to biochar supports a circular economy by transforming agricultural waste into a high-value soil amendment. RHB is a cost-effective material and a readily available carbon-rich organic resource [22]. The positive effects of RHB application have been widely reported on physicochemical properties and C sequestration [23], nutrient leaching [18], greenhouse gas (GHG) emissions [24], and crop productivity [25].

Taken together, an individual effectiveness of zeolite or biochar application has been widely studied in achieving sustainable agriculture and environment. Although comparative studies on biochar and zeolite

have been conducted under controlled conditions such as soil column experiments, conventionally fertilized soils, or during composting, few have directly compared their relative effectiveness in enhancing nitrogen use efficiency (NUE) and mitigating gaseous nitrogen losses (e.g., NH_3 and N_2O) under PS-amended soils during the vegetative growth stage of field crops. Furthermore, such comparisons are especially limited in the context of Korean agricultural systems using RHB, a locally abundant but underutilized resource. In this context, we hypothesized that (1) RHB would be more effective than zeolite in enhancing soil water status and biological activity; (2) RHB and zeolite would exhibit different capacities to retain mineral N (NH_4^+ and NO_3^-); (3) their application would differentially reduce NH_3 and N_2O emissions from PS-amended soils; and (4) RHB would result in higher NUE for dry matter and crude protein production in oilseed rape (*Brassica napus*). To test this hypothesis, we assessed soil water status, enzymatic activity, inorganic N pools, and humic substance formation. These parameters were integrated with NH_3 and N_2O emissions to evaluate NUE during the vegetative growth stage of oilseed rape. This comparative approach offers new insights into how each amendment influences N stabilization and retention in PS-amended soils. These findings support their broader adoption as sustainable soil amendments, offering co-benefits for agricultural productivity and environmental management in intensive manure-recycling systems.

Materials and methods

Material preparation and characteristics

Soil was collected from the experimental field of faculty of agriculture of Chonnam National University (Gwangju, South Korea). The soil was a black-brown type (inceptisol) that had never been fertilized with either pig slurry (PS) or chemical fertilizer. The soil used in this study was classified as sandy loam with a relatively coarse texture (9.4% Clay, 26.3% silt, and 64.3% sand), with chemical properties (g kg $^{-1}$) comprising 0.150 ± 0.01 total N, 0.024 ± 0.003 NH_4^+ , 0.013 ± 0.001 NO_3^- and 0.255 ± 0.012 available P, and a pH_{water} (1:5) of 7.45 ± 0.05 . The PS used in this study was obtained from a local farm and contained on average (g L $^{-1}$): 0.86 ± 0.13 total N, 0.171 ± 0.015 NH_4^+ , 0.075 ± 0.01 NO_3^- and 0.70 ± 0.02 available P with a pH_{water} (1:5) of 7.45 ± 0.05 . The zeolite and rice husk biochar (RHB) used herein were provided by Gumok Inc. (Gyeongju, Gyeongbuk, Korea) and Yougi Ind. Co. (Gochang, Jeonbuk, Korea), respectively. The main physicochemical properties of RHB and zeolite used in the experiment are listed in Table 1.

Table 1 Chemical and physiological properties of zeolite and rice husk biochar (RHB)

Physicochemical property	Zeolite	RHB
pH (1:10, H ₂ O)	7.50	8.72
Specific surface area (m ² g ⁻¹)	47.2	123.2
CEC (cmol kg ⁻¹)	79.0	40.4
K (cmol kg ⁻¹)	21.5	32.5
Ca (cmol kg ⁻¹)	31.2	5.2
Mg (cmol kg ⁻¹)	11.9	2.1
Na (cmol kg ⁻¹)	14.4	0.6

Physicochemical analyses of soil, pig slurry, and amendments

The pH (1:5 or 1:10 material-to-water ratio) was measured using a pH meter (AB33PH, Ohaus Corp., NJ, USA). Specific surface area was determined using the Brunauer–Emmett–Teller (BET) method [26]. Available phosphorus (P) was extracted using the Bray-1 method, with molybdenum blue colorimetry for detection [27]. Soil ammonium (NH₄⁺–N) and soil nitrate (NO₃[–]–N) were extracted with 2 M KCl and measured by distillation. Magnesium oxide (MgO) and Devarda's alloy (mixture of Al, Cu, and Zn) were used to reduce the NH₄⁺–N and the NO₃[–]–N, respectively [28]. Exchangeable potassium (K), calcium (Ca), magnesium (Mg), and sodium (Na), were extracted using 1 M ammonium acetate and analyzed using measured by an inductively coupled plasma optical emission spectrometry (ICP–OES, Thermo Elemental–IRIS Advantage, USA) [29]. Cation exchange capacity (CEC) was calculated as the sum of the total exchangeable cations (K, Ca, Mg and Na).

Experimental design

Seeds of *Brassica napus* L. (cv. Mosa) were germinated in soil trays. Seedlings were then raised in a greenhouse under a 16-h light/8-h dark photoperiod, an average temperature of 27 °C day/20 °C night, and relative humidity (RH) in the range of 60–70%. At the four-leaf stage, the seedlings were transplanted into square pots (502 mm × 315 mm × 230 mm) and maintained under greenhouse conditions. Each pot was filled with 21 kg of soil sieved through a 2 mm mesh and mixed either with clinoptilolite zeolite or with RHB. Zeolite and RHB were each applied at a rate of 10% (w/w) based on dry weight. The treatments used included PS alone, PS combined with 10% (w/w) zeolite (PS + Z), and PS combined with 10% (w/w) rice husk biochar (PS + RHB). The PS was applied at a rate of 200 kg N

ha⁻¹. A corresponding amount of chemical fertilizer was added to each pot to adjust the P₂O₅:K₂O application ratio to 150:150 (kg ha⁻¹). Each treatment was performed in triplicate. All pots were irrigated twice daily and monitored daily with a soil moisture meter (WT-1000H, Mirae E&I, Seoul, Korea) to maintain ~80% field capacity.

Soil water retention

A soil moisture meter was inserted into the pots to 10 cm depth to control soil field capacity at the root development level. Field capacity for each treatment was determined gravimetrically from fully saturated and oven-dried soils. A regression equation was established using the values from 20 to 80% field capacity and the readings from the portable soil moisture meter. This equation was then used to maintain the soil at 80% field capacity throughout the experimental period. Similarly, regression equations were derived between the portable soil moisture meter readings and soil water content or soil moisture. The soil water condition was measured using portable soil moisture meter at 08:00 daily before soil evaporation occurred.

Forage, soil, and gas sampling

Forage samples were collected on day 43 after treatment. Shoots were cut into 20-mm segments, lyophilized, ground, and stored in a vacuum desiccator until analysis. On days 0, 7, 17, 22, 32, 39, and 43, soil cores ranging from 0 to 15 cm were collected using an auger 2.5 cm in diameter. The soil samples were air-dried and stored approximately 15–20 °C in a shaded environment. For the gas sampling to estimate NH₃ and N₂O emissions from PS-applied soils, two air-tight acrylic chambers (15 cm in diameter, 25 cm in height) were vertically inserted separately to a 5 cm depth on the side of plant cultivation pot (Supplementary Fig. S1). The NH₃ emitted from the NH₃ collection chamber was sampled by the modified system of acid trap method by connecting the sampling chamber to NH₃–N trapping bottles filled with 10 mL of 0.2 M H₂SO₄ in a vacuum source operating at a flow rate of 1.5 L min⁻¹ [16]. Chambers were secured in air-tight with clamps for a 24-h collection period. The gas sampling for N₂O emission was conducted through an air-tight septa installed on the headspace of chamber. The collected gas sample was stored in a Vacutainer tube. Gas sampling was performed daily at 09:00 to minimize diurnal variation.

Analytical procedures

Functional groups were obtained using Fourier transfer infrared (FTIR) spectroscopy (Spectrum 400, PerkinElmer, USA), with 32 scans in the wavenumber range of

4000–500 cm^{-1} at a resolution of 4 cm^{-1} with the standard instrument settings to the manufacturer's protocol. Soil urease activity was quantified by colorimetry [30]. Soils were incubated with 0.08 M aqueous or buffered urea solution (pH 10.0) at 37 °C for 2 h. The accumulated ammonium was subsequently extracted with a solution that contained 1 N KCl and 0.01 N HCl. Finally, the ammonium was quantified by spectrophotometry at 690 nm. Humic and fulvic acid fractions were isolated using the procedure developed by the International Humic Substances Society [31]. Humic substances were extracted with 0.1 M NaOH until the supernatant is a Light color. The extracted humic substances were then separated into humic acid and fulvic acid fractions by acidifying the extract to pH 1 with 10 NH_2SO_4 . The precipitate (humic acid) and the supernatant (fulvic acid) were separated by centrifugation. The humic fraction was suspended in 1 M NaOH solution. The carbon contents of humic acid and fulvic acid were determined through the $\text{K}_2\text{Cr}_2\text{O}_7$ oxidation method. Samples were collected to measure shoot dry matter (DM) and crude protein (CP) content for *Brassica napus* at harvest. CP was quantitated following the methodology of the Association of Official Analytical Chemists, which was determined using kjeldahl method [32].

Nitrogen use efficiency (NUE)

NUE was assessed based on *Brassica napus* shoot DM and CP content. DM-based NUE was calculated by dividing the total DM by the amount of N supplied during treatment (kg DM/N applied). CP-based NUE was calculated by dividing CP by the amount of N supplied during treatment (kg CP/N applied).

Gas emissions

NH_3 in the acid trap solution, such as $(\text{NH}_4)_2\text{SO}_4$, was microdiffused in a Conway dish [33], quantified with Nessler's ammonium color reagent (Sigma-Aldrich Corp., St. Louis, MO, USA), and expressed as $\text{NH}_3\text{-N}$ emitted per hectare. N_2O was measured by gas chromatography (GC; No. 7890A, Agilent Technologies, Santa Clara, CA, USA) fitted with an electron capture detector (ECD). Separation occurred in an HP-Plot 5A column (30 $\text{m} \times 0.53 \text{ mm} \times 25 \mu\text{m}$). Helium (He) was the carrier gas and its flow rate was 2 mL min^{-1} . N_2O fluxes were calculated following the method of Guo et al. [34]. Cumulative NH_3 and N_2O emissions over the entire experimental period were calculated by summing the daily measurements. Emission factors (EF) for NH_3 and N_2O were calculated as follows [16]:

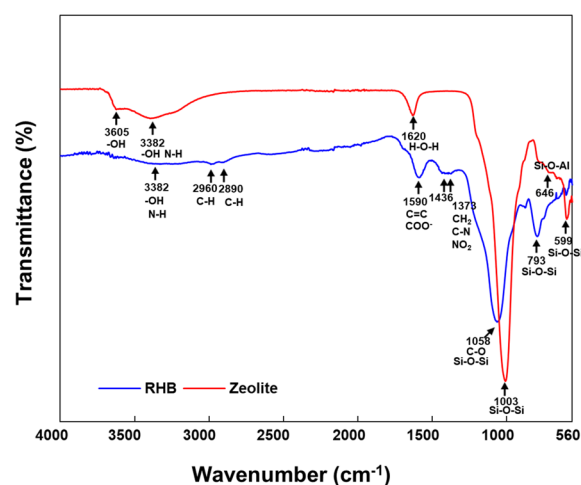


Fig. 1 Fourier-transform infrared (FTIR) spectroscopy spectra of zeolite (red) and rice husk biochar (RHB, blue)

Statistical analyses

A completely randomized design with three replications per treatment was used in the present study. Means were compared by Duncan's multiple range test ($P < 0.05$). Statistical analyses were conducted in SAS 9.1.3 (SAS Institute, Cary, NC, USA). Correlation analysis and principal component analysis (PCA) were performed with SRplot (<http://www.bioinformatics.com.cn/srplot>).

Results

Surface functional groups of zeolite and RHB

Fourier transform infrared (FTIR) spectroscopy was performed to identify surface functional groups present in zeolite and RHB (Fig. 1). In zeolite, the major peaks at 3605 and 3382 cm^{-1} corresponded to -OH stretching from structural hydroxyls and adsorbed water, while a peak at 1620 cm^{-1} indicated H-O-H bending vibrations from retained moisture. A strong Si-O-Si asymmetric stretch was observed at 1003 and 599 cm^{-1} . These features indicate the aluminosilicate framework typical of natural zeolites, which are essential for ion exchange and NH_4^+ adsorption. In contrast, RHB exhibited a broader and more complex spectrum, reflecting its diverse organic-inorganic composition. Notably, the peak at 1590 cm^{-1} was attributed to aromatic C=C and COO^- stretching, while peak at 1436 and 1373 cm^{-1} was attributed to CH_2 bending. The presence of peaks at 1058 and 793 cm^{-1} suggested C-O and Si-O-Si stretching,

$$\text{EF} = (\text{NH}_3 \text{ and } \text{N}_2\text{O}) \text{ in PS alone, PS - combined with zeolite or RHB/N mineral applied} \times 100$$

supporting the residual silicate and carbonaceous structure derived from rice husk. These functional group are involved in facilitation of NH_4^+ retention through both electrostatic attraction and hydrogen bonding.

Water status, urease activity, fulvic and humic acid content in soil

Water management was conducted to maintain the field capacity in a range of 60% and 80% throughout the experimental period (Fig. 2A). RHB and zeolite applications significantly influenced soil water status under controlled field capacity. Soil moisture content, which reflects the total amount of water retained in the soil, ranged from 32.8% to 47.0% in PS+RHB, 27.8–37.0% in PS+Z, and 29.4–36.7% in PS, with consistently higher values in the

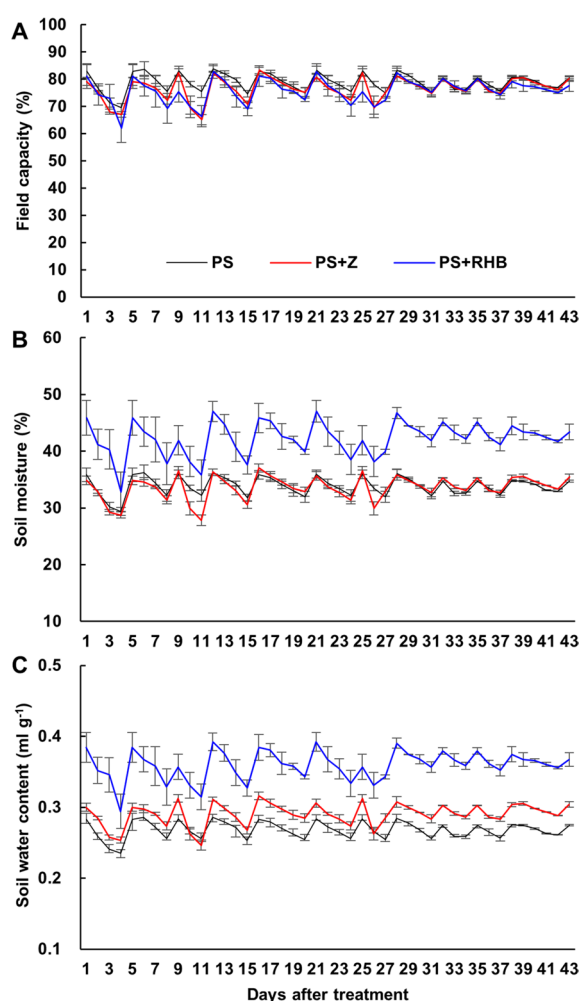


Fig. 2 Changes in field capacity (A), soil moisture (B), and soil water content (C) in pig slurry (PS), PS combined with zeolite (PS+Z), and PS combined with rice husk biochar (PS+RHB) during the vegetative growth of *Brassica napus* over 43 days. Data are means $\pm$ standard error ($n=3$)

RHB treatment (Fig. 2B). In parallel, soil water content, which indicates the portion of water readily available for plant uptake during the growth period, was significantly higher in PS+RHB ranged (0.31–0.39 ml g^{-1}), followed by PS+Z and PS alone (Fig. 2C). These values indicate that RHB, due to its higher water retention capacity, consistently improved soil moisture status compared to other treatments. The soil pH in the PS+RHB treatment was significantly higher than that in PS+Z and PS alone during the first 32 days, whereas PS+Z and PS alone maintained similar pH levels throughout the experimental period (Fig. 3A). The urease activity in PS-alone treatment peaked at day 7, then declined thereafter. In contrast, it continued to increase throughout the experiment both in PS+RHB- and PS+Z-treated soils, with a significantly higher levels in PS+RHB treatment (Fig. 3B). The fulvic acid content was consistently highest in PS+RHB-treated soils throughout the experiment, reaching a maximum of 1.91 g C kg^{-1} soil at day 22, compared to 1.46 g C kg^{-1} in PS+Z and PS alone (Fig. 3C). In PS+Z, a significant increase became evident only after day 32. Humic acid content in PS+RHB peaked early at day 17 (1.06 g C kg^{-1}), remaining stable thereafter. In contrast, PS+Z showed a gradual increase from 0.56 to 0.89 g C kg^{-1} , while PS alone remained relatively unchanged, ranging between 0.56 and 0.80 g C kg^{-1} throughout the period (Fig. 3D). The differences between treatments became more apparent after day 17.

Changes in NH_4^+ -N and NO_3^- -N in soil

During this earlier 7 days, the highest NH_4^+ -N was observed in PS-alone treatment. From day 17, the highest NH_4^+ -N content was recorded in PS+RHB, followed by PS+Z and PS-alone treatment. At day 43 (the final measurement), the NH_4^+ -N content was 29.1, 37.4 and 42.4 mg N kg^{-1} , respectively, in PS alone, PS+Z and PS+RHB treatment (Fig. 3E), demonstrating the superior N-retention capacity of RHB. The soil NO_3^- -N content peaked at day 17. From days 17 to 32, zeolite and RHB application tended to decrease NO_3^- -N compared to PS alone. The NO_3^- -N content at day 43 was 30.9, 32.1, and 34.2 mg N kg^{-1} , respectively, in PS alone, PS+Z and PS+RHB treatment (Fig. 3F). These results indicate that zeolite and RHB regulate the nitrification pathway, minimizing early stage N losses while maintaining N availability for plant uptake in later growth stages.

NH_3 and N_2O emissions

Both RHB and zeolite applications reduced NH_3 and N_2O emissions compared to PS alone, with RHB showing the greatest mitigation effect (Fig. 4). Daily NH_3 emissions decreased over time in all treatments, with PS+RHB consistently exhibiting the lowest emissions

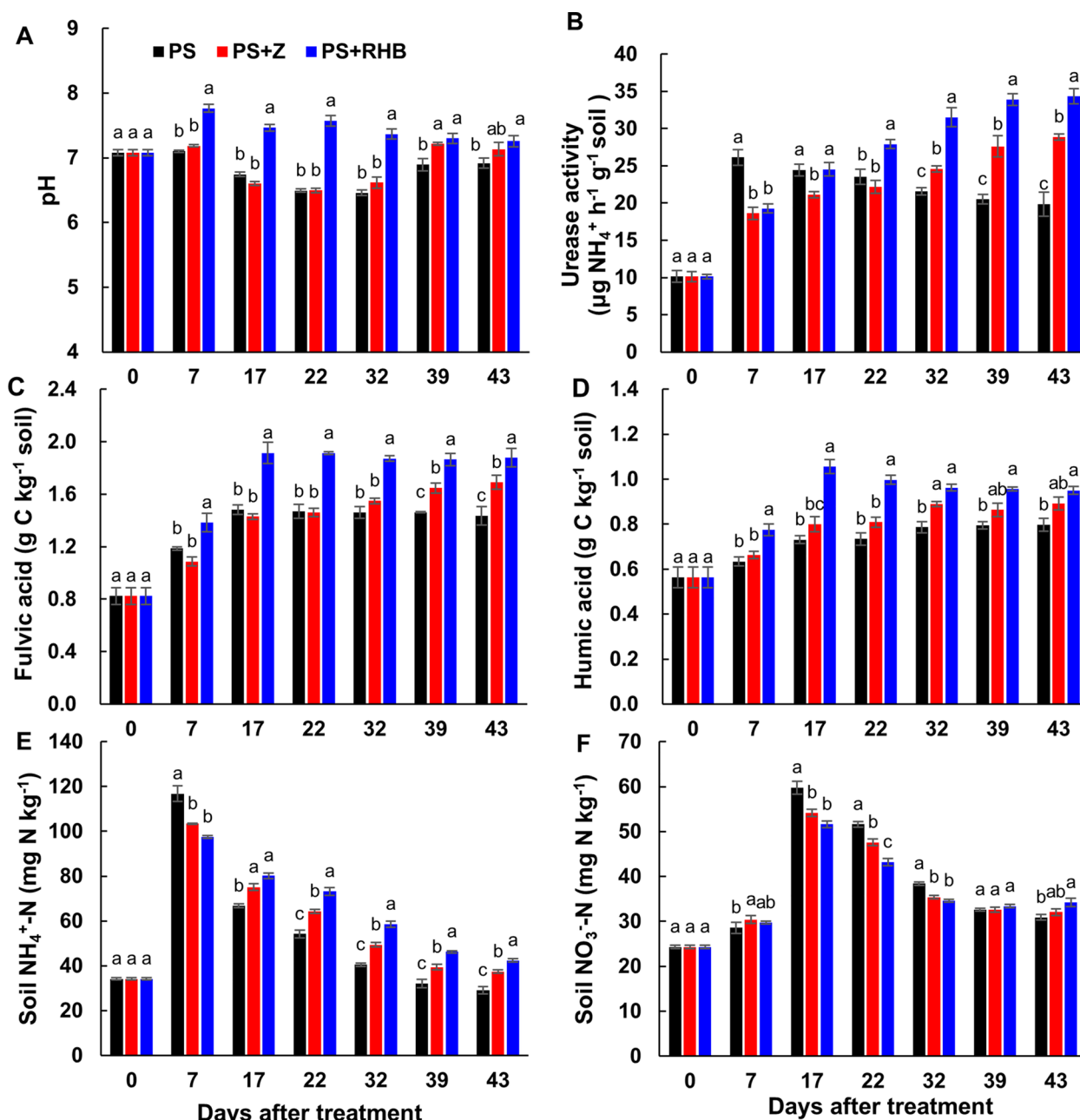


Fig. 3 Changes in pH (A), urease activity (B), content of fulvic acid (C), humic acid (D), ammonium-N (NH_4^+-N , E), and nitrate-N (NO_3^--N , F) in soil amended with pig slurry (PS), PS combined with zeolite (PS + Z), and PS combined with rice husk biochar (PS + RHB) during the vegetative growth of *Brassica napus* over 43 days. Data are means $\pm$ standard error ($n=3$)

(Fig. 4A). Daily N_2O emissions peaked sharply in PS alone during the first few days and then declined markedly. PS + Z showed a lower peak than PS alone but exhibited moderate fluctuations in the early phase, and its emission levels became similar to PS alone after 23 days. In contrast, PS + RHB maintained consistently

low N_2O emissions throughout the experiment (Fig. 4B). The cumulative NH_3 and N_2O emissions and their emission factor (EF) were highest for PS alone followed by PS + Z and PS + RHB, indicating the superior emission-reducing capacity of RHB (Table 2).

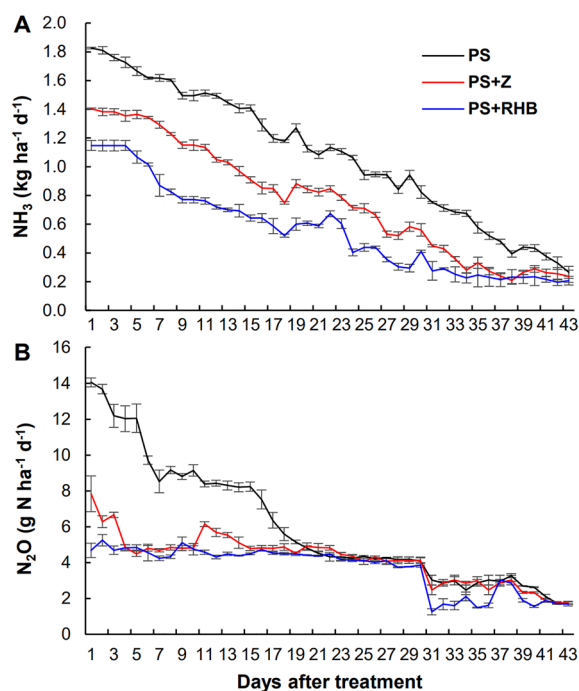


Fig. 4 Daily ammonia (NH_3) and nitrous oxide (N_2O) emissions from pig slurry (PS), PS combined with zeolite (PS+Z), and PS combined with rice husk biochar (PS+RHB) during the vegetative growth of *Brassica napus* over 43 days. Values with different letters at each sampling date significantly differ ($P < 0.05$) by Duncan's multiple range test. Data are means $\pm$ standard error ($n = 3$)

Shoot dry matter, crude protein, and nitrogen use efficiency of forage yield

Among the treatments, PS+RHB showed the most pronounced improvement in plant growth and nitrogen uptake (Table 3). Shoot dry matter (DM) in the PS+RHB treatment was significantly higher than in PS+Z and PS alone, with increase of 18.6% compared to PS+Z. Total shoot crude protein (CP) was highest for PS+RHB ($1475.6 \text{ kg N ha}^{-1}$), representing a 22.8% and 76.6% increase compared to PS+Z and PS alone, respectively. The nitrogen use efficiency (NUE) for shoot DM and CP were significantly enhanced PS+RHB relative to the other treatments, indicating superior N utilization efficiency with RHB application.

Pearson's correlation analysis

Pearson's correlation analysis was performed to examine the relationships among soil water content, nitrogen-related variables, cumulative NH_3 and N_2O emissions, and NUE for DM and CP (Fig. 5). NH_3 and N_2O emissions were negatively correlated with soil water content, NH_4^+-N , mineral N ($\text{NH}_4^+ + \text{NO}_3^-$), fulvic acid, humic acid, and NUE for both DM and CP. In contrast, soil NH_4^+-N showed positive correlations with fulvic acid, humic acid, and NUE for DM and CP.

Discussion

Effects of rice husk biochar and zeolite on physical and biological activity in PS-amended soil

In previous studies, biochar and zeolite have been shown to improve soil water retention even at relatively low application rates. For example, 1–5% biochar increased soil moisture by 3–57.8% [35, 36], while 1–5% zeolite improved water retention by 5–24% [37]. In contrast, this study applied 10% rice husk biochar (RHB) and zeolite, resulting in a markedly higher increase in soil moisture (12–40% for RHB, 1–10% for zeolite) (Fig. 2B, C). These results align with Guo et al. [38], who emphasized the value of direct comparisons at fixed rates (5–10%) despite material differences. Moreover, zeolite at 5–10% has been shown to reduce nitrogen loss by up to 39% [15], supporting the broader benefits of these amendments. RHB application was more effective than zeolite

Table 3 Dry matter (DM), crude protein (CP), and nitrogen use efficiency (NUE) for shoots harvested from pig slurry (PS), PS combined with zeolite (PS+Z), and PS combined rice husk biochar (PS+RHB) after 43 days of the vegetative growth of the *Brassica napus*

Treatment	DM (kg ha^{-1})	NUE for DM (kg DM/N applied)	CP (kg ha^{-1})	NUE for CP (kg CP/N applied)
PS	5113 ± 309^c	25.6 ± 1.6^c	835.7 ± 11.6^c	4.60 ± 0.31^c
PS+Z	6136 ± 213^b	30.7 ± 0.4^b	1201.3 ± 67.3^b	6.01 ± 0.34^b
PS+RHB	7275 ± 244^a	36.4 ± 1.2^a	1475.6 ± 67.5^a	7.38 ± 0.34^a

Values are means $\pm$ standard error ($n = 3$). Different superscript letters in vertical columns denote significant differences at $P < 0.05$ by Duncan's multiple range test

Table 2 Total cumulative NH_3 and N_2O emissions and their emission factors (EF) for pig slurry (PS), PS combined with zeolite (PS+Z), and PS combined with rice husk biochar (PS+RHB) after 43 days of the vegetative growth of the *Brassica napus*

Treatment	Total NH_3 (kg ha^{-1})	EF	Total N_2O (g N ha^{-1})	EF
PS	46.4 ± 0.13^a	23.2 ± 0.06^a	252.0 ± 1.8^a	0.126 ± 0.0010^a
PS+Z	33.0 ± 0.25^b	16.5 ± 0.12^b	179.5 ± 1.1^b	0.090 ± 0.0004^b
PS+RHB	23.9 ± 0.17^c	12.0 ± 0.08^c	155.6 ± 0.7^c	0.078 ± 0.0003^c

Values are means $\pm$ standard error ($n = 3$). Different superscript letters in vertical columns denote significant differences at $P < 0.05$ by Duncan's multiple range test

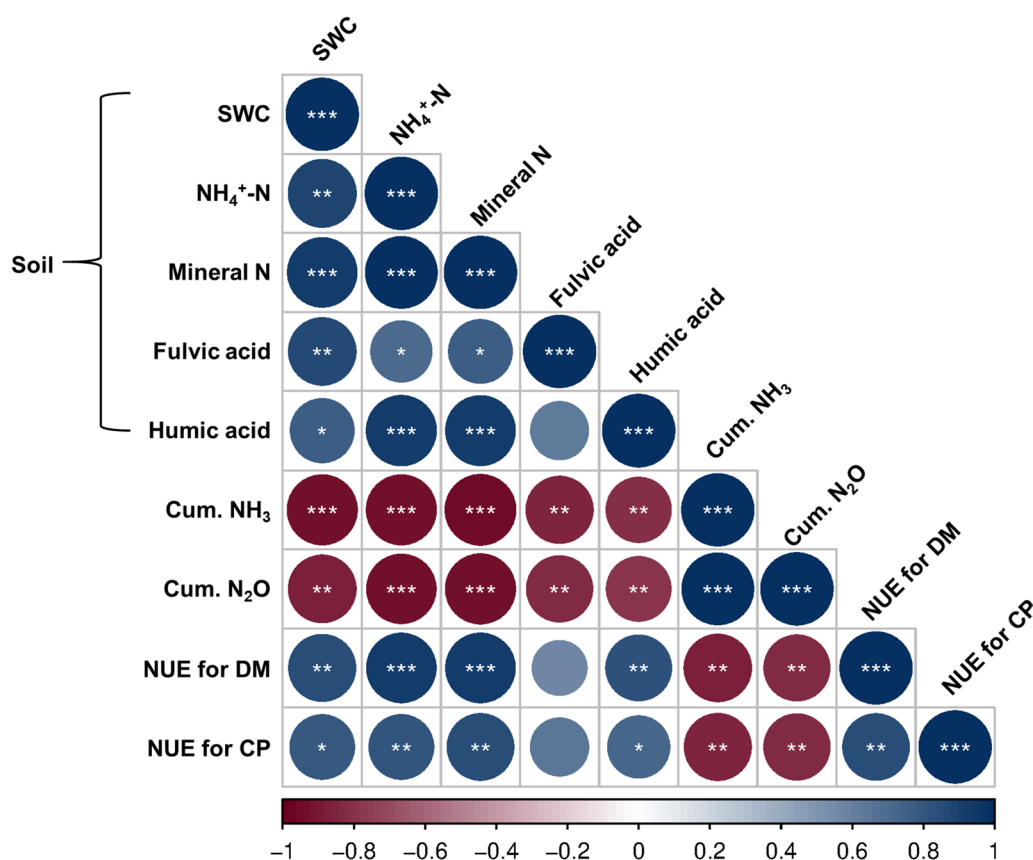


Fig. 5 Pearson's correlation coefficients of variables measured in pig slurry (PS), PS combined with zeolite (PS+Z), and PS combined with rice husk biochar (PS+RHB) during the vegetative growth of *Brassica napus* at 43 days. Blue and red indicate positive and negative effects, respectively. Color intensity increases with magnitude of correlation coefficient. Asterisks indicate statistical differences among correlations: * $P < 0.05$, ** $P < 0.01$, and *** $P < 0.001$. Cum. NH₃, cumulated NH₃; Cum. N₂O, cumulated N₂O; Mineral N, NH₄⁺ + NO₃⁻; SWC, soil water content

application at improving these properties (Fig. 2B, C). This substantial range observed with RHB likely reflects not only its superior water-holding capacity but also natural variability in plant water uptake and soil water dynamics during the 43-day period, despite the greenhouse conditions being environmentally controlled. The micropore and mesopore structures of RHB enable it to retain water effectively and provide plant-available moisture for extended periods [39]. In contrast, zeolite is predominantly macroporous, which enhances its hydraulic conductivity but limits its ability to retain water effectively [40]. Hence, zeolite improves short-term water availability, whereas RHB prolongs water retention. RHB application also buffered soil pH more strongly than zeolite application (Fig. 3A). RHB application consistently maintained pH throughout the experiment (Fig. 3A), as explained by the fact that biochar neutralizes soil acidity by releasing alkaline cations and introducing basic functional groups [41]. Zeolite application moderately increased pH only for the later period (e.g., from day 39) when compared to PS alone-treated soil (Fig. 3A)

as it has only a limited ability to release alkaline cations [40, 42]. Soil pH significantly influences microbial activity and nutrient cycling. Thus, the strong buffering capacity of RHB may have enabled it to maintain soil pH within a favorable range, thereby supporting microbial growth and enzyme activity and ultimately enhancing soil biological activity [43]. Urease activity is a critical indicator of nitrogen transformation in soils amended with urea- or manure-based fertilizers, as it governs the hydrolysis of urea into NH₄⁺ (i.e., ammonification), which subsequently affects NH₄⁺ retention in soil, NH₃ volatilization and nitrification processes [44]. In this study, urease activity increased continuously throughout the experimental period in both the PS+RHB and PS+Z treatments, with consistently higher levels observed in the PS+RHB treatment. In contrast, PS alone exhibited an early peak in urease activity at day 7, likely reflecting rapid urea hydrolysis. The enhanced and sustained urease activity observed in the PS+RHB may be attributed to the input of bioavailable carbon which is derived from RHB and promotes microbial proliferation and activity

[45] and the higher surface area with a porous structure which provides favorable microsites for microbial colonization [46]. These enhanced microbial processes may also contribute to the accumulation of humic substances in soil. Indeed, the content of fulvic and humic acids in soil was consistently higher in PS + RHB treatment, followed by PS + Z and PS-alone (Fig. 3C, D). Several studies have reported that biochars enhance humic substance formation by stimulating microbial activity and accelerating the decomposition of organic matter [47, 48]. For instance, Zhang et al. [47] demonstrated that biochar promotes compost humification by regulating bacterial and fungal communities and dominating the Maillard reaction with reducing sugars as the substrate. Song et al. [48] observed that biochar improves humification effectively during chicken manure composting by enhancing pH. Humic substances, end-products derived from the microbial transformation of organic matter, play a critical role in regulating nutrient dynamics in the soil. Their functional groups such as carboxyl, phenolic, and quinone moieties interact with soil minerals and enzymes, thereby enhancing nutrient retention and nitrogen cycling [49]. Among these, fulvic acid, a more labile-soluble fraction, is particularly effective in stimulating enzyme activity and microbial metabolism by enhancing nutrient availability and chelating micronutrients [50].

Effects of rice husk biochar and zeolite on mineral N in PS-amended soil

Zeolites and biochars have been known to adsorb NH_4^+ on their surface, enabling a slower release into the soil matrix and prolonging microbial access to the substrate for nitrification and denitrification [7, 9]. Both RHB and zeolite application significantly enhanced the NH_4^+ -N content in soil compared to that of PS alone, except during the early 7 days (Fig. 3E). This transient reduction likely resulted from delayed urea hydrolysis due to urease inhibition. Previous studies have demonstrated that biochar can suppress urease activity through surface-mediated oxidative reactions [51], while zeolite can adsorb urease enzymes, thereby reducing their catalytic activity [52]. In contrast, PS alone promoted rapid NH_4^+ formation via active urease activity [2]. The influence of RHB application on NH_4^+ -N enhancement was superior to that of zeolite during the latter period (Fig. 3E), suggesting that RHB consistently retained more NH_4^+ -N than zeolite. The enhanced and sustained NH_4^+ -N observed in the PS + RHB treatment would be attributed to higher specific surface area (123.2 in RHB vs. 47.2 $\text{m}^2 \text{g}^{-1}$ in zeolite) (Table 1) which may provide more accessible sorption sites with strong affinity for cations, such as NH_4^+ and K^+ [7, 9]. Hai et al. [53] demonstrated that activated biochar derived from sugarcane waste exhibited high

NH_4^+ adsorption capacity due to increased surface area and abundance of carboxylic and hydroxyl groups. In addition, the FTIR spectrum of zeolite exhibited prominent absorption bands corresponding to the stretching vibrations of hydroxyl groups associated with structural -OH and adsorbed water. Strong peaks in zeolite were assigned to Si-O-Si asymmetric stretching and bending vibrations, respectively, which are characteristic of the aluminosilicate framework (Fig. 1). In contrast, the FTIR spectrum of RHB exhibited more prominent oxygen-containing functional groups, including a broader -OH stretching band and additional peaks assigned to C=O, C=C, COO^- , C-O and Si-O-Si. These functional groups are known to facilitate strong electrostatic and chemical interactions with NH_4^+ and NH_3 . Trazzi et al. [54] confirmed that such functional groups in biochar play a critical role in NH_4^+ adsorption, especially under varying pH and temperature conditions. Specifically, hydroxyl and carboxyl groups strongly adsorb NH_4^+ via cation exchange [55]. Several studies have reported an increase in CEC of the soils amended with RHB [18, 23]. Moreover, NH_3 can be retained on RHB through weak van der Waals interactions or chemisorbed as amine-type complexes via surface functional groups [56]. In addition, the siliceous nature of RHB as evidenced by peaks at 1058 and 793 cm^{-1} (Fig. 1) leads possibly to more NH_4^+ retention through organo-mineral complex formation. In this study, during 17–32 days after treatment, the soil NO_3^- -N content was higher in PS alone than in PS + Z and PS + RHB treatments. This suggests that strong initial NH_4^+ adsorption by zeolite and RHB may have temporarily limited microbial access to the nitrification substrate, delaying NO_3^- formation. However, the subsequent increase in NO_3^- -N after day 39 indicates delayed but sustained nitrification driven by prolonged NH_4^+ availability in zeolite- and RHB-amended soils (Fig. 3E, F).

Effects of rice husk biochar and zeolite on NH_3 and N_2O emissions from PS-amended soil

The agriculture sector is the principal source of NH_3 emissions, responsible for more than 80% of emissions [57]. Among these emissions, approximately 30–40% are due to manure application to soil [58], which largely depends on manure and soil properties, environmental conditions, and application methods. Accordingly, highly enhanced value in a daily NH_3 flux from the PS-applied soil has been observed in various cropping systems [5, 16] and a laboratory incubation study [59]. Numerous studies have shown that the majority of NH_3 emissions are released within the first 2–7 days following the application of synthetic N fertilizers or organic manure, due to rapid hydrolysis and volatilization of the applied nitrogen [5, 6]. In the present study, 51.4% of total NH_3

emitted during vegetative growth period was lost via NH_3 volatilization at the early 14 days after PS application (Fig. 4A), suggesting that when PS is applied to the soil, the NH_4^+ in PS is rapidly converted to NH_3 , which is readily lost via NH_3 volatilization [2]. During this early period, the cumulative NH_3 amount emitted from PS alone-treated soil (22.5 kg ha^{-1}) significantly decreased by 23.3% and 43.2%, respectively, in PS + Z and PS + RHB treatment (Fig. 4A), confirming the capacity of zeolites [10] and biochars [7, 19] in capturing NH_3 , with stronger adsorption in RHB than zeolite. Consistently, in the latter period (from day 15), the daily NH_3 emission was lowest in PS + RHB, followed by PS + Z and PS-alone treatment (Fig. 4A). The NH_3 emission in three treatments during this period showed a reversed pattern of soil NH_4^+ content (Fig. 3E), indicating that lower NH_3 emission in PS + Z and PS + RHB treatment would be due to the enhanced and retained NH_4^+ -N in the soils (Fig. 3E) as evidenced by a close negative correlation (Fig. 5). Higher efficiency of RHB in mitigating NH_3 emission relative to zeolite would be explained by more compatible physicochemical properties of RHB in NH_4^+ adsorption on its surface [e.g., higher specific surface area (Table 1), stronger electrostatic and chemical interactions of functional groups (Fig. 1)], as shown in Kohira et al. [60], who emphasized that surface chemistry and specific surface area in biochar are key to NH_3 emission.

A portion of soil NH_4^+ is oxidized to NO_2^- and then to NO_3^- via aerobic nitrification, and then NO_3^- is reduced to N_2O through microbial denitrification. The extent of these progresses largely depends on the amount of NH_4^+ available for nitrification and the amount of soluble organic carbon available for denitrification, both of which are key determinants of N_2O emissions [6]. In the present study, approximately half of total N_2O emitted in PS alone-treated soil ($252.0 \text{ g N ha}^{-1}$) occurred within first 17 days (Fig. 4B). RHB and zeolite application reduced the total cumulative N_2O emissions by 38.3% and 28.8%, respectively, compared to PS alone (Table 2), with RHB showing greater mitigation potential. These findings are in line with a meta-analysis showing that biochar can reduce N_2O emissions by an average of 38% during the first year of application [24], as well as our previous study, where zeolite application reduced N_2O emissions by 15.6% and 31.5% in the Italian ryegrass and maize growing season, respectively [16]. A superior effectiveness of RHB can be explained by its higher NH_4^+ retention capacity, which lowers substrates (mainly NH_4^+) availability for the microbial nitrification and denitrification (Figs. 3E and 4B), as reflected in the close negative correlation between retained N_2O emissions

and cumulative N_2O emissions (Fig. 5). This is consistent with the findings of He et al. [61], who reported that biochar combined with N fertilizer significantly reduced cumulative N_2O emissions in Brassica crops while also influencing N-fixing bacteria spread. Ren et al. [62] highlighted role of biochar in regulating microbial functional genes and soil pH to suppress N_2O emissions. Zeolite reduces N_2O emissions by inhibiting microbial nitrification and denitrification, partly because soil bacteria have only limited access to its pores [63].

Effects of rice husk biochar and zeolite on NUE for DM and CP production

The availability of mineral N ($\text{NH}_4^+ + \text{NO}_3^-$) for the crop growth would be determined by the N retained in soil after N losses via NH_3 and N_2O emission, and NO_3^- leaching. It thus takes a notion that reducing NH_3 and N_2O emissions enhances N availability, which in turn improves NUE and crop productivity, even though yield-scaled response varies with the climate, soil texture, and N application rate [64]. In this study, content of mineral N retained in soil at day 43 was 60.0, 69.5 and 78.6 mg N kg^{-1} , respectively, in PS alone, PS + Z and PS + RHB treatment (Fig. 3E, F). Confirming the conceptual hypothesis-above mentioned, close negative correlations of soil mineral N with NH_3 emission and N_2O emission, close positive correlations with NUE for DM and NUE for CP yield were established (Fig. 5). In addition, NH_3 and N_2O emissions are negatively correlated with shoot DM and CP (Fig. 5). These results ensemble indicate that the mitigation of N losses via NH_3 and N_2O emissions by both zeolite and RHB applications conferred a benefit on the oilseed rape DM and CP production, resulting from the enhanced availability of mineral N in soil. In addition, it suggested that both zeolite and RHB have a role in slow releasing mineral N by retaining mineral N in soil in consistent with previous studies [12, 19]. Therefore, when compared the effectiveness of zeolite and RHB, higher efficiency of RHB in NUE for DM and CP production relative to zeolite would be mainly attributed to its stronger capacity NH_4^+ adsorption by higher oxygen-containing functional groups (Fig. 1), leading to higher mineral N retention in soil (e.g., higher availability) (Fig. 3E,F). In addition, its higher specific surface area (Fig. 1) provides more favorable microsites [46] and the input of bioavailable carbon derived from RHB promotes microbial proliferation and activity [45], thereby more facilitating microbial organic matter decomposition. This is evidenced by more enhanced fulvic acid (Fig. 3C) and humic acid (Fig. 3D). These compounds, as microbial transformation products of organic matter, play vital roles in

regulating nutrient cycling by stimulating enzyme activity and microbial metabolism in soil [52]. Indeed, the elevated humic acid contributed significantly to NUE for DM and CP production, while fulvic acid showed a positive but non-significant correlation (Fig. 5).

Conclusion

Materials that adsorb NH_4^+ has been widely studied as a soil amending matter for improving water and nutrient retention in soils, as well as slowing the release of nutrients to the soil solution. The present study to evaluate comparatively the effectiveness of zeolite and RHB, which are commercially available materials, in mitigating NH_3 and N_2O from PS-applied soils and in the use efficiency of N in PS-applied. Significantly positive effectiveness of both zeolite and RHB applications were estimated in mitigating NH_3 and N_2O emissions as well as in improving NUE for vegetative growth of oilseed rape when compared to PS-alone treatment. In a comparison between zeolite and RHB, the efficiency of RHB application was relatively higher in the buffering of soil pH and water, the availability of mineral N in soil, the mitigation of NH_3 and N_2O emissions as well as in the use efficiency of N for DM and CP production which was mainly due to a stronger capacity of NH_4^+ adsorption, and possibly to the supply of bioavailable carbon derived from RHB. Based on the data obtained from this study, we can conclude that RHB application a promising approach in improving NUE for crop growth while minimizing NH_3 and N_2O emissions from the PS-applied soil. In-depth studies on long-term field applications of RHB is further required to establish more efficient application practices. Further researches for developing proper methods of physical or chemical modification of rice husk-derived biochar would be also interesting to improve the active sites for adsorption and the functional groups for specific adsorption functions.

Supplementary Information

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Additional file 1.

Author contributions

Bok-Rye Lee: conceptualization, data curation, investigation, methodology, formal analysis, writing—original draft, and writing—review and editing. Sang-Hyun Park: conceptualization, investigation, formal analysis, funding acquisition, data curation, and writing—review and editing. Muchamad Muchlas: conceptualization, methodology, formal analysis, data curation, and writing—review and editing. Tae-Hwan Kim: conceptualization, investigation, methodology, supervision, validation, and writing—review and editing.

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Data availability

No datasets were generated or analysed during the current study.

Declarations

Competing interests

The authors declare no competing interests.

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