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Pyrolysis temperature and time of rice husk biochar potentially control ammonia emissions and Chinese cabbage yield from urea-fertilized soils

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Current agricultural practices are increasingly favoring the biochar application to sequester carbon, enhance crop growth, and mitigate various environmental pollutants resulting from nitrogen (N) loss. However, since biochar's characteristics can vary depending on pyrolysis conditions, it is essential to determine the optimal standard, as they can have different effects on soil health. In this study, we categorized rice husk biochars basis on their pH levels and investigated the role of each rice husk biochar in reducing ammonia (NH_3) emissions and promoting the growth of Chinese cabbage in urea-fertilized fields. The findings of this study revealed that the variation in pyrolysis conditions of rice husk biochars and N rates affected both the NH_3 emissions and crop growth. The neutral (pH 7.10) biochar exhibited effective NH_3 volatilization reduction, attributed to its high surface area (6.49 m^2 g^{-1}), outperforming the acidic (pH 6.10) and basic (pH 11.01) biochars, particularly under high N rates (640 kg N ha^{-1}). Chinese cabbage yield was highest, reaching 4.00 kg $plant^{-1}$, with the basic biochar application with high N rates. Therefore, the neutral rice husk biochar effectively mitigate the NH_3 emissions from urea-treated fields, while the agronomic performance of Chinese cabbage enhanced in all biochar amendments.

Given the increasing focus on sustainable ecosystems and eco-friendly agriculture, contemporary agricultural practices encounter several challenges[1]. These challenges include the necessity to reduce the use of chemical fertilizers and pesticides, adopt minimal tillage techniques, incorporate organic amendments (e.g., organic fertilizer, manure compost, and biochar), and effectively manage nutrient losses[1,2]. Specifically, the continuous and excessive application of nutrients, such as nitrogen (N) and phosphorus (P), through chemical fertilization can result in various environmental contaminations[2]. These contaminations involve the release of particulate matter (PM), greenhouse gases (GHGs), eutrophication, and algal bloom in both the atmosphere and aquatic ecosystems[3–5]. Ammonia (NH_3) volatilization stands out as a prominent source of N losses and contributes to the formation of secondary PM ($PM_{2.5}$) and nitrous oxide (N_2O)[2,4,5]. Furthermore, NH_3 emissions has detrimental effects on air quality[5], human health[4,6], and the Earth's radiative balance[2]. These pollutants further exacerbate the impacts of global warming and climate change[5].

Numerous studies have been dedicated to the development of sustainable and eco-friendly agricultural practices with the aim of reducing N losses, particularly NH_3, while simultaneously enhancing crop productivity[7–10]. These practices encompass a range of approaches, including the application of natural urease inhibitors[2,3], the introduction of elemental sulfur and polymers[9], the use of organic fertilizer[4], and the incorporation of biochar amendments[7,9,10]. Biochar, a carbon-rich material, is obtained through the pyrolysis of agricultural residues, biomass, and organic waste ingredients under relatively high temperatures and oxygen-limited conditions[7,9–12]. It

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has garnered attention for its distinctive characteristics, such as carbon (C) sequestration[7,13], promotion of plant growth[14,15], enhancement of soil pH[4], optimization of soil health[12], provision of a habitat for microorganisms[13], and the adsorption of heavy metals and nutrient contents[4,16]. Furthermore, biochar has the ability to absorb organic N, ammonium ions (NH_4^+), and gaseous NH_3 through its functional groups and microspores, resulting in reduced N losses[15]. These properties of biochar are evident in the reduced N loss observed in agricultural soils treated with N fertilizers in the presence of biochar[17]. Unfortunately, many experiments have focused on the combined effects of several substitutes, such as urease inhibitor[2,18,19], wood vinegar[20], zeolite[21], and compost[22,23], or have explored the influence of biochar's formulation[24] and feedstock sources[25] on NH_3 emissions in agricultural soil. This variation in results may be attributed to the diverse characteristics of biochar produced under different pyrolysis conditions. Therefore, further studies are necessary to assess the efficiency of NH_3 emission reduction by biochar, taking into account biochar characteristics such as pH and surface area, which are related to N adsorption capacity.

We hypothesize that (1) higher pH levels in rice husk biochar might increase soil pH, hypothetically affecting NH_3 mitigation efficiency, and (2) excessive N rates could disturb Chinese cabbage yield. To assess these hypotheses, this study evaluated NH_3 volatilization and crop yield in a Chinese cabbage field treated with different rates of N fertilizer and three types of rice husk biochar classified based on their pH levels. The rice husk biochars were categorized as acidic (AB, pH 6.10), neutral (NB, pH 7.10), and basic (BB, pH 11.01), while N rates applied as urea were designed as $N_{0.5}$ (160 kg N ha^{-1}), $N_{1.0}$ (320 kg N ha^{-1}, the recommended N rate), and $N_{2.0}$ (640 kg N ha^{-1}), respectively. Results revealed that both NH_3 mitigation efficiency by the rice husk biochars and Chinese cabbage yield increased with rising N rates from 160 to 640 kg N ha^{-1}. Interestingly, NH_3 emissions from N fertilization were lowest in the soil treated with NB, which had the highest surface area compared to AB and BB. Due to the conflicting influences between BB's alkali effect and urea's pH-reducing impact, the $N_{0.5}$ treatment exhibited higher soil pH than the $N_{2.0}$ treatment, and soil chemical properties except for soil pH did not reach negative levels in the $N_{2.0}$ treatment. These unexpected findings suggest that the NH_3 mitigation rate primarily depended on the rice husk biochar's surface area rather than their pH values. Moreover, there was no negative effect in crop yield caused by excessive N supply owing to higher initial soil pH and the increased NH_3 emissions.

Results

Pyrolysis conditions affect the characteristics of the rice husk biochar

Table 1 presents the chemical properties of the rice husk biochars and their corresponding pyrolysis conditions. The variations in pyrolysis temperature and time had a significant impact on the chemical properties of the rice husk biochar. The pH of the rice husk biochar exhibited a sharp increase as the pyrolysis temperature and time were raised from 400 to 600 °C and from 15 to 30 min, respectively. In contrast, the electrical conductivity (EC) values of AB, NB, and BB gradually decreased with the increase in their pyrolysis conditions. The surface area (SA) of the rice husk biochars was the highest in NB at 6.49 $m^2\ g^{-1}$, while AB and BB were observed at 2.55 and 5.30 $m^2\ g^{-1}$, respectively. The total carbon (TC) content of BB was significantly higher at 54.90% compared to 41.30% of AB and 44.10% of NB, while the total nitrogen (TN) content did not show a statistically significant difference among AB, NB, and BB. Conversely, the total hydrogen (TH) and total oxygen (TO) contents decreased with the increase in pyrolysis conditions and were the highest values in AB at 5.39 and 34.61%, respectively. Inorganic contents of the rice husk biochar gradually increased with the increased in pyrolysis conditions. The H:C and O:C ratio, which represent the aromaticity and polarity of the rice husk biochar, were higher at lower temperatures and shorter times.

The results of the analysis of functional groups on the surface of the rice husk biochar using Fourier transform infrared spectroscopy (FT-IR) were presented in Fig. 1. The secondary amide group, indicated by the –NH bond in the range of 3300–3325 cm^{-1}, was observed in NB and BB but not in AB. The C=C, $-CH_3$, and –C–CN bonds in the range of 1640–1660 cm^{-1}, 1000–1050 cm^{-1}, and 400–420 cm^{-1}, respectively, were strongly formed with the increased pyrolysis conditions.

Ammonia volatilization reduce effectively by the neutral rice husk biochar

Figure 2 displays the daily NH_3 volatilization resulting from different N rates and rice husk biochar amendments. The NH_3 emissions peaked within 7 days after N application, with the first top-dressing fertilization leading to the maximum NH_3 release compared to the basal and other top-dressing fertilizations. Furthermore, the NH_3 peaks were higher with increasing the N rates (Fig. 3). The AB + $N_{2.0}$ treatment recorded the highest peak value at 20,127.94 g ha^{-1} day^{-1} (20.13 kg ha^{-1} day^{-1}), while the NB + $N_{2.0}$ and BB + $N_{2.0}$ treatments reached 16,300.87 (16.30 kg ha^{-1} day^{-1}) and 13,847.16 g ha^{-1} day^{-1} (13.85 kg ha^{-1} day^{-1}), respectively. After reaching the highest peak, the NH_3 volatilization sharply decreased and became similar to the control with non-N fertilization.

Figure 4 illustrates the total NH_3 emissions during the Chinese cabbage cropping season. The total NH_3 emissions were influenced by the N rates, and the reduction efficiency on NH_3 emission varied depending on the pH of the rice husk biochar (Supplementary Table S1). Cumulative NH_3 emissions were the lowest in NB treatments, such as NB + $N_{0.5}$, NB + $N_{1.0}$, and NB + $N_{2.0}$, at 28.42, 42.99, and 108.54 kg ha^{-1}, respectively. In contrast, the only-urea treatments (i.e., $N_{0.5}$, $N_{1.0}$, and $N_{2.0}$) had the highest values at 38.64, 66.70, and 142.42 kg ha^{-1}, respectively. In comparison to the soil treated with basic rice husk biochar, the soil treated with acidic rice husk biochar exhibited lower NH_3 emissions, resulting in reductions of total NH_3 emissions by 6, 8, and 7% with varying N rates ($N_{0.5}$, $N_{1.0}$, and $N_{2.0}$). Moreover, the reductions in the total NH_3 emissions attributed to the rice husk biochar amendments were more pronounced with higher N rates, from $N_{0.5}$ to $N_{2.0}$, effectively mitigating the N losses. The highest reduction efficiency by the rice husk biochar was shown in the NB + $N_{1.0}$ treatment at 36% compared to the $N_{1.0}$ treatment.

Samples	Pyrolysis conditions		pH (1:10, H_2O)	EC (dS m^{-1})	Surface area (m^2 g^{-1})	TC (%)	TN (%)	TH (%)	TO (%)	TP (%)	CaO (%)	K_2O (%)	MgO (%)	Na_2O (%)	H:C ratio (%)	O:C ratio (%)
	Temp. (°C)	Time (min)														
AB	330	15	6.10 ± 0.01^{c}	11.49 ± 1.62^{a}	2.55 ± 0.01^{c}	41.30 ± 0.01^{b}	0.40 ± 0.02^{a}	5.39 ± 0.11^{a}	34.61 ± 0.59^{a}	0.14 ± 0.03^{a}	0.08 ± 0.02^{a}	0.36 ± 0.12^{b}	0.04 ± 0.02^{a}	0.03 ± 0.01^{a}	1.55 ± 0.06^{a}	0.63 ± 0.32^{a}
NB	400	15	7.10 ± 0.02^{b}	9.50 ± 0.83^{b}	6.49 ± 0.03^{a}	44.10 ± 0.02^{b}	0.40 ± 0.02^{a}	5.32 ± 0.03^{a}	32.50 ± 1.33^{a}	0.16 ± 0.02^{a}	0.09 ± 0.03^{a}	0.47 ± 0.07^{b}	0.04 ± 0.01^{a}	0.03 ± 0.01^{a}	1.44 ± 0.02^{b}	0.55 ± 0.21^{a}
BB	600	30	11.01 ± 0.05^{a}	6.59 ± 0.13^{c}	5.30 ± 0.05^{b}	54.90 ± 0.19^{a}	0.60 ± 0.01^{a}	2.11 ± 0.03^{b}	5.88 ± 1.98^{b}	0.21 ± 0.01^{a}	0.16 ± 0.05^{a}	0.78 ± 0.09^{a}	0.07 ± 0.03^{a}	0.04 ± 0.01^{a}	0.46 ± 0.08^{c}	0.08 ± 0.09^{b}
p-value			**	***	**	**	***	**	***	*	*	**	***	***	**	***

Table 1. Chemical characteristics of rice husk biochar produced from different pyrolysis conditions. *AB* acidic (pH 6.1) rice husk biochar; *NB* neutral (pH 7.1) rice husk biochar; *BB* basic (pH 11.0) rice husk biochar; *EC* electrical conductivity, *TC* total carbon, *TN* total nitrogen, *TH* total hydrogen, *TO* total oxygen, *T-P* total phosphorus.

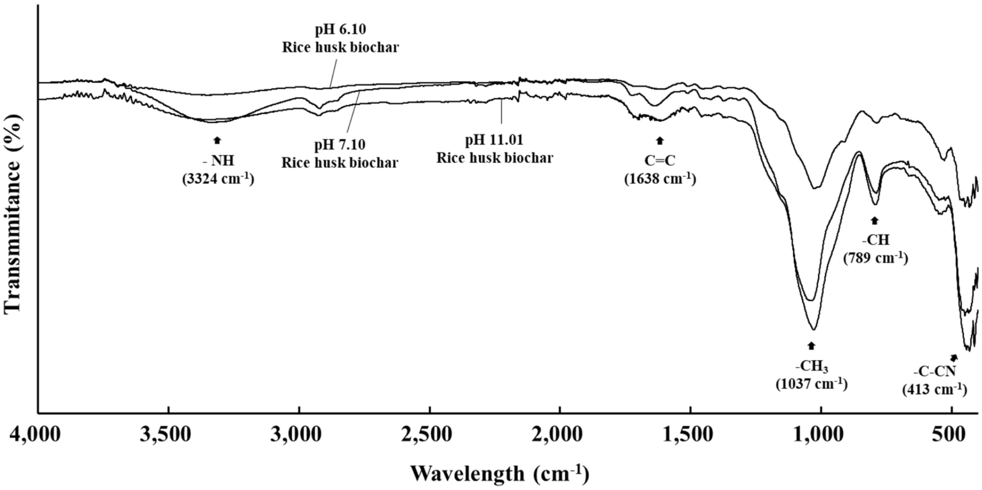

Figure 1. FT-IR spectrum of rice husk biochars categorized by their pH values.

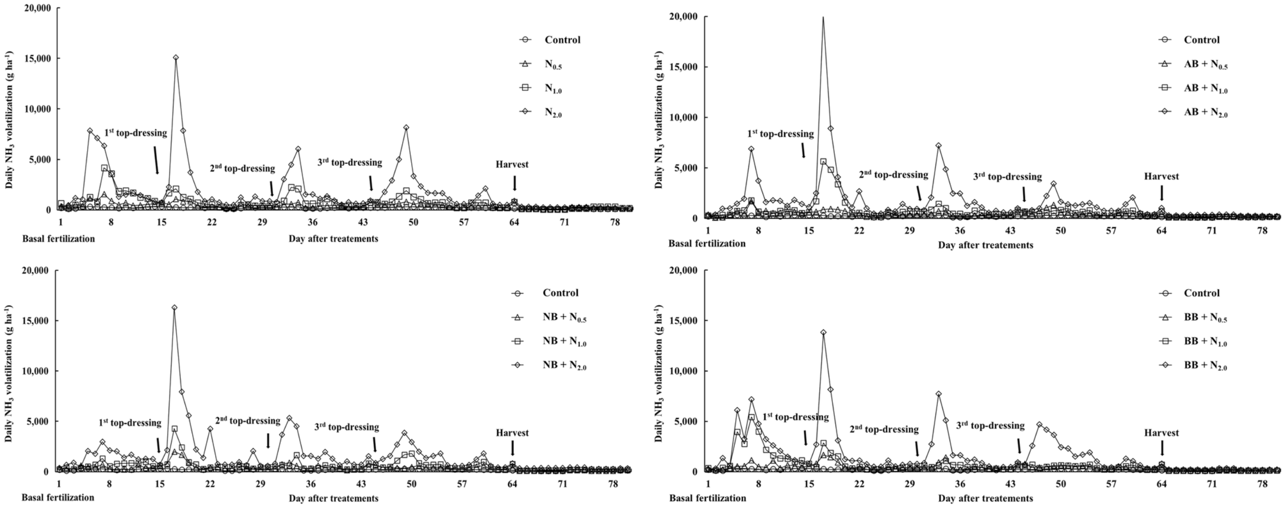

Figure 2. Daily NH_3 volatilization effected by different nitrogen rates and three types of rice husk biochar during the Chinese cabbage cropping period. $N_{0.5}$, $N_{1.0}$, and $N_{2.0}$ exhibited nitrogen application rates equivalent to 160 kg N ha^{-1}, 320 kg N ha^{-1}, and 640 kg N ha^{-1}, respectively, while AB, NB, and BB donated the acidic (pH 6.1), neutral (pH 7.1), and basic (pH 11.0) rice husk biochars.

Growth of Chinese cabbage increases the N rates and the pH of the rice husk biochar

Table 3 presents the growth characteristics of Chinese cabbage influenced by the varying N rates and rice husk biochar amendments. The BB + $N_{2.0}$ treatment achieved the highest fresh weight at 4.00 kg $plant^{-1}$, while the $N_{2.0}$, AB + $N_{2.0}$, and NB + $N_{2.0}$ treatments yielded 3.40, 3.63, and 3.89 kg $plant^{-1}$, respectively. Additionally, fresh weight increased with the rising N rates and the pH of the rice husk biochar from $N_{0.5}$ to $N_{2.0}$ and from pH 6.10 to pH 11.01, respectively (Supplementary Table S2). However, the moisture contents of each treatment did not exhibit statistical significant difference. Head height and width were the highest in the BB + $N_{2.0}$ treatment, measuring 25.87 and 16.70 cm, respectively. Head growth increased with the increase in the N rates and the pH of the rice husk biochar. Furthermore, leaf length and width were higher with increasing the N rates and the pH of rice husk biochar, but statistically significant differences were observed only in the control treatment. The chlorophyll and TN content of Chinese cabbage were the highest in NB + $N_{0.5}$ and AB + $N_{1.0}$, with SPAD values of 35.19 and a TN content of 3.71%, respectively, although they exhibited a non-specific trend.

Soil chemical properties change the N rates and the properties of the rice husk biochar

The soil chemical properties were influenced by both the N rates and the pH of the rice husk biochar (Table 2). Soil pH decreased as the N rates increased from 160 kg N ha^{-1} ($N_{0.5}$) to 640 kg N ha^{-1} ($N_{2.0}$), while soil EC increased. Furthermore, among the rice husk biochar amendments, soil pH increased with the rise in the pH of rice husk biochar. The EC values of the soil treated with AB, NB, and BB were lower than those of treatments

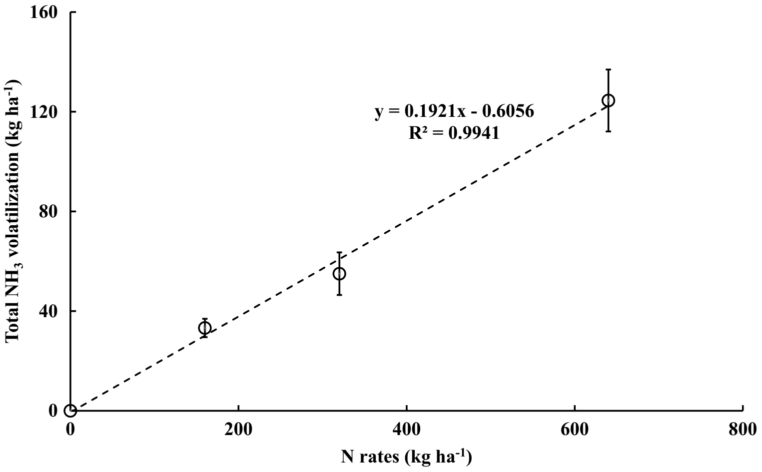

Figure 3. Correlation between nitrogen rates and total NH_3 volatilization.

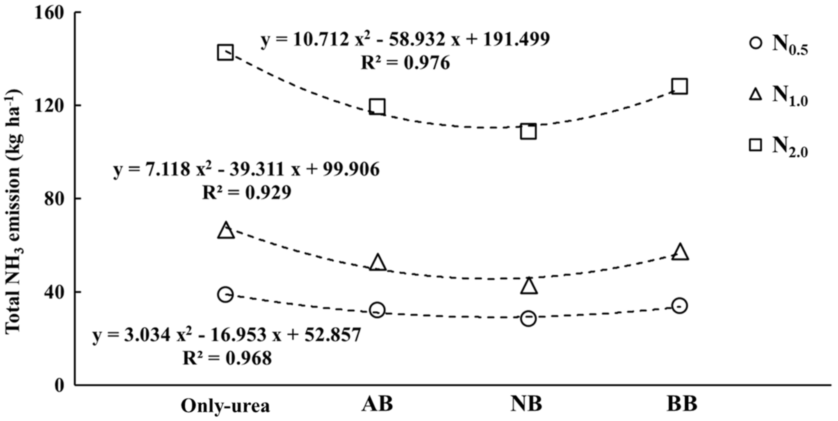

Figure 4. Total NH_3 emissions effected by three types of rice husk biochar and different nitrogen rates. $N_{0.5}$, $N_{1.0}$, and $N_{2.0}$ exhibited nitrogen application rates equivalent to 160 kg N ha^{-1}, 320 kg N ha^{-1}, and 640 kg N ha^{-1}, respectively, while AB, NB, and BB donated the acidic (pH 6.1), neutral (pH 7.1), and basic (pH 11.0) rice husk biochars.

with only urea (i.e., $N_{0.5}$, $N_{1.0}$, and $N_{2.0}$). The highest soil pH and EC were observed at pH 7.48 in BB + $N_{0.5}$ and 1.26 dS m^{-1} in $N_{2.0}$, respectively. The rice husk biochar amendments effectively increased soil TC and TN contents compared to treatments with only urea. For instance, the co-application of BB and $N_{2.0}$ yielded the highest TC content at 2.36%, while the individual treatments of $N_{0.5}$, $N_{1.0}$, and $N_{2.0}$ decreased from the initial soil pH value of 0.71% to 0.61, 0.66, and 0.64%, respectively. In contrast, soil TN content increased with N fertilization, although no statistically significant difference was observed. Soil available nitrogen (Avail. N) content increased with the rice husk biochar amendment, with NB effectively increasing the Avail. N content under the same N rates conditions. In contrast, there were no significant differences observed in available phosphorus (Avail. P) content of N-treated soil (e.g., $N_{0.5}$, $N_{1.0}$, AB + $N_{0.5}$, NB + $N_{1.0}$, and BB + $N_{2.0}$). The highest Avail. P content was recorded in BB + $N_{1.0}$ at 125.05 mg kg^{-1}, while the Avail. P content of initial soil and control was 94.10 and 89.26 mg kg^{-1}, respectively. After rice husk biochar amendment and N fertilization, the content of exchangeable cations, such as Ca^{2+}, K^+, Mg^{2+}, and Na^+, increased, but no statistically significant difference was observed.

Discussion

Numerous prior studies have consistently shown that an increase in pyrolysis temperature results in heightened parameters such as pH, surface area, cation exchange capacity, and carbon content of biochar[26,27]. Particularly, the escalation in biochar pH predominantly arises from carbonate formation and the elevation in inorganic alkali contents[28,29]. Furthermore, the pH of biochar increases owing to presence of ash content and oxygen functional groups[30]. However, the composition of cellulose and hemicellulose in plant-based ingredients occurs at relatively low temperature (between 200 and 300 °C) and generate various organic acids and phenolic substances that decrease the pH of the material[30]. This implies that biochar produced at lower temperature might exhibit a lower pH compared to the initial raw material. On the other hand, the TH, and TO contents of rice husk biochars

Treatments		pH (1:5, H_2O)	EC (dS m^{-1})	TC (%)	TN (%)	OM (%)	Avail. N (mg kg^{-1})	Avail. P (mg kg^{-1})	Exchangeable cations			
									Ca^{2+} ($cmol_c$ kg^{-1})	K^{+} ($cmol_c$ kg^{-1})	Mg^{2+} ($cmol_c$ kg^{-1})	Na^{+} ($cmol_c$ kg^{-1})
Initial soil		7.00 ± 0.20^{c}	0.35 ± 0.05^{d}	0.71 ± 0.19^{d}	0.11 ± 0.05^{b}	1.22 ± 0.33^{d}	24.77 ± 0.76f.	94.10 ± 21.08^{b}	4.50 ± 0.21^{ab}	0.21 ± 0.03^{c}	1.24 ± 0.11^{b}	0.19 ± 0.00^{a}
Control		7.45 ± 0.16^{a}	0.40 ± 0.06^{d}	0.44 ± 0.23^{e}	0.04 ± 0.01^{c}	0.76 ± 0.40^{e}	48.11 ± 9.34^{e}	89.26 ± 4.73^{b}	4.48 ± 0.14^{ab}	0.23 ± 0.04^{c}	1.20 ± 0.10^{b}	0.19 ± 0.01^{a}
$N_{0.5}$		7.16 ± 0.36^{bc}	0.75 ± 0.39^{b}	0.61 ± 0.19^{d}	0.16 ± 0.02^{a}	1.05 ± 0.33^{d}	74.51 ± 11.07^{d}	113.38 ± 3.90^{a}	4.59 ± 0.10^{ab}	0.23 ± 0.04^{c}	1.46 ± 0.05^{a}	0.18 ± 0.02^{a}
$N_{1.0}$		6.80 ± 0.17^{cd}	1.12 ± 0.29^{a}	0.66 ± 0.13^{d}	0.19 ± 0.03^{a}	1.14 ± 0.22^{d}	88.75 ± 17.32^{c}	117.40 ± 17.30^{a}	4.62 ± 0.05^{ab}	0.27 ± 0.05^{bc}	1.50 ± 0.01^{a}	0.20 ± 0.01^{a}
$N_{2.0}$		6.59 ± 0.18^{e}	1.26 ± 0.49^{a}	0.64 ± 0.18^{d}	0.21 ± 0.03^{a}	1.10 ± 0.31^{d}	104.34 ± 12.46^{bc}	112.74 ± 9.97^{a}	4.69 ± 0.30^{b}	0.29 ± 0.08^{bc}	1.48 ± 0.18^{a}	0.21 ± 0.02^{a}
AB	$N_{0.5}$	7.19 ± 0.18^{bc}	0.52 ± 0.09^{c}	1.03 ± 0.12^{c}	0.17 ± 0.02^{a}	1.78 ± 0.21^{c}	94.51 ± 16.07^{cd}	120.69 ± 19.97^{a}	4.60 ± 0.13^{ab}	0.23 ± 0.05^{c}	1.54 ± 0.12^{a}	0.21 ± 0.05^{a}
	$N_{1.0}$	7.19 ± 0.49^{bc}	0.58 ± 0.34^{c}	1.19 ± 0.06^{c}	0.18 ± 0.02^{a}	2.05 ± 0.10^{c}	101.33 ± 14.02^{bc}	119.69 ± 19.63^{a}	4.63 ± 1.05^{ab}	0.33 ± 0.03^{b}	1.56 ± 0.09^{a}	0.22 ± 0.04^{a}
	$N_{2.0}$	7.12 ± 0.43^{bc}	1.09 ± 0.38^{a}	1.14 ± 0.17^{c}	0.20 ± 0.03^{a}	1.97 ± 0.29^{c}	124.98 ± 25.78^{a}	115.08 ± 11.34^{a}	4.90 ± 0.37^{a}	0.48 ± 0.04^{a}	1.59 ± 0.08^{a}	0.22 ± 0.02^{a}
NB	$N_{0.5}$	7.26 ± 0.22^{b}	0.64 ± 0.37^{c}	1.64 ± 0.37^{b}	0.17 ± 0.01^{a}	2.83 ± 0.64^{b}	97.90 ± 18.08^{c}	118.01 ± 17.98^{a}	4.54 ± 0.57^{ab}	0.31 ± 0.09^{b}	1.52 ± 0.03^{a}	0.20 ± 0.01^{a}
	$N_{1.0}$	7.14 ± 0.36^{bc}	0.69 ± 0.34^{c}	1.78 ± 0.68^{b}	0.18 ± 0.01^{a}	3.07 ± 1.17^{b}	115.80 ± 14.48^{b}	120.56 ± 11.74^{a}	4.66 ± 0.21^{ab}	0.37 ± 0.03^{ab}	1.57 ± 0.25^{a}	0.22 ± 0.04^{a}
	$N_{2.0}$	7.13 ± 0.69^{bc}	1.16 ± 0.40^{a}	1.62 ± 0.49^{b}	0.21 ± 0.01^{a}	2.79 ± 0.84^{b}	143.31 ± 23.44^{a}	123.44 ± 5.80^{a}	4.96 ± 0.52^{a}	0.44 ± 0.09^{a}	1.56 ± 0.08^{a}	0.25 ± 0.09^{a}
BB	$N_{0.5}$	7.48 ± 0.21^{a}	0.67 ± 0.16^{c}	2.09 ± 0.58^{a}	0.20 ± 0.01^{a}	3.60 ± 1.00^{a}	93.87 ± 17.01^{cd}	113.42 ± 6.22^{a}	4.33 ± 0.07^{b}	0.32 ± 0.04^{b}	1.55 ± 0.02^{a}	0.21 ± 0.02^{a}
	$N_{1.0}$	7.44 ± 0.22^{a}	0.88 ± 0.04^{b}	2.06 ± 0.53^{a}	0.21 ± 0.01^{a}	3.55 ± 0.91^{a}	104.33 ± 12.46^{bc}	125.05 ± 9.92^{a}	4.61 ± 0.34^{ab}	0.41 ± 0.07^{a}	1.58 ± 0.11^{a}	0.26 ± 0.08^{a}
	$N_{2.0}$	7.24 ± 0.64^{ab}	1.18 ± 0.24^{a}	2.36 ± 0.45^{a}	0.23 ± 0.01^{a}	4.07 ± 0.76^{a}	138.61 ± 19.82^{a}	117.17 ± 34.87^{a}	4.97 ± 0.55^{a}	0.43 ± 0.06^{a}	1.57 ± 0.08^{a}	0.20 ± 0.02^{a}
p-value		*	**	***	**	***	*	***	**	*	***	***

Table 2. Changes in soil chemical properties affected by different nitrogen rates and biochar amendments. $N_{0.5}$, 160 kg N ha^{-1}; $N_{1.0}$, 320 kg N ha^{-1}; $N_{2.0}$, 640 kg N ha^{-1}. *AB* acidic (pH 6.1) rice husk biochar, *NB* neutral (pH 7.1) rice husk biochar, *BB* basic (pH 11.0) rice husk biochar, *EC* electrical conductivity, *TC* total carbon, *TN* total nitrogen, *OM* organic matter, *Avail. N* available nitrogen, *Avail. P* available phosphorus. *, **, and *** are used to indicate statistically significant differences at the $p < 0.05$, $p < 0.01$, and $p < 0.001$, respectively. $^{a–f}$Each value with different letters within a column are significantly different from each other as determined by Duncan's multiple range test ($p < 0.05$).

Treatments		Fresh weight (kg $plant^{-1}$)	Moisture content (%)	Head		Leaf			TN (%)
				Height (cm)	Width (cm)	Length (cm)	Width (cm)	Chlorophyll content (SPAD)	
Control		0.70 ± 0.14f.	90.51 ± 2.21^{a}	15.10 ± 2.43^{b}	6.93 ± 1.43^{c}	24.91 ± 0.68^{c}	15.34 ± 0.17^{b}	19.32 ± 3.01^{c}	2.62 ± 0.08^{b}
$N_{0.5}$		2.41 ± 0.10^{e}	91.20 ± 0.81^{a}	22.63 ± 2.80^{a}	12.30 ± 0.36^{b}	33.26 ± 0.57^{ab}	22.59 ± 0.81^{a}	25.15 ± 1.11^{b}	2.51 ± 0.19^{b}
$N_{1.0}$		3.08 ± 0.73^{d}	92.80 ± 2.12^{a}	23.53 ± 4.18^{a}	13.73 ± 0.56^{b}	33.20 ± 2.16^{ab}	23.02 ± 2.54^{a}	29.95 ± 4.90^{ab}	2.52 ± 0.26^{b}
$N_{2.0}$		3.40 ± 0.25^{c}	93.11 ± 0.62^{a}	24.97 ± 2.25^{a}	15.33 ± 1.33^{a}	34.81 ± 1.15^{ab}	24.90 ± 0.16^{a}	30.51 ± 1.41^{ab}	2.55 ± 0.03^{b}
AB	$N_{0.5}$	2.63 ± 0.24^{e}	92.11 ± 1.52^{a}	22.47 ± 0.70^{a}	12.27 ± 0.59^{b}	31.52 ± 0.93^{b}	22.93 ± 0.37^{a}	32.37 ± 0.73^{a}	3.68 ± 0.01^{a}
	$N_{1.0}$	3.17 ± 0.09^{cd}	92.44 ± 0.82^{a}	23.03 ± 0.83^{a}	13.73 ± 0.67^{b}	33.23 ± 1.81^{ab}	23.08 ± 1.49^{a}	33.71 ± 1.82^{a}	3.71 ± 0.04^{a}
	$N_{2.0}$	3.63 ± 0.23^{ab}	92.08 ± 1.26^{a}	24.53 ± 1.50^{a}	15.70 ± 0.87^{a}	33.91 ± 1.30^{ab}	24.98 ± 2.10^{a}	30.37 ± 1.44^{ab}	3.70 ± 0.02^{a}
NB	$N_{0.5}$	2.67 ± 0.18^{e}	91.16 ± 0.87^{a}	22.40 ± 2.10^{a}	12.77 ± 0.78^{b}	32.32 ± 1.50^{ab}	22.38 ± 0.73^{a}	35.19 ± 5.76^{a}	3.69 ± 0.05^{a}
	$N_{1.0}$	3.30 ± 0.37^{c}	91.90 ± 1.32^{a}	24.07 ± 2.19^{a}	13.87 ± 1.96^{b}	33.27 ± 2.40^{ab}	24.09 ± 2.96^{a}	34.76 ± 3.08^{a}	3.69 ± 0.06^{a}
	$N_{2.0}$	3.89 ± 0.24^{a}	91.32 ± 0.69^{a}	25.63 ± 2.00^{a}	16.13 ± 0.83^{a}	35.28 ± 0.97^{a}	24.65 ± 1.25^{a}	32.43 ± 4.56^{a}	3.65 ± 0.04^{a}
BB	$N_{0.5}$	2.73 ± 0.11^{e}	90.09 ± 0.75^{a}	23.20 ± 2.08^{a}	12.63 ± 1.45^{b}	32.63 ± 1.30^{ab}	22.21 ± 0.95^{a}	31.28 ± 2.73^{ab}	3.67 ± 0.11^{a}
	$N_{1.0}$	3.52 ± 0.17^{b}	91.42 ± 1.15^{a}	24.93 ± 1.81^{a}	15.17 ± 0.21^{a}	35.72 ± 0.67^{a}	24.37 ± 0.68^{a}	30.55 ± 1.30^{ab}	3.65 ± 0.14^{a}
	$N_{2.0}$	4.00 ± 0.54^{a}	91.34 ± 0.47^{a}	25.87 ± 0.42^{a}	16.70 ± 0.87^{a}	36.08 ± 0.75^{a}	24.49 ± 1.21^{a}	34.84 ± 3.10^{a}	3.66 ± 0.04^{a}
p-value		**	***	**	**	***	**	**	***

Table 3. Growth characteristics of Chinese cabbage affected by the pH of rice husk biochar and different nitrogen rates. $N_{0.5}$, 160 kg N ha^{-1}; $N_{1.0}$, 320 kg N ha^{-1}; $N_{2.0}$, 640 kg N ha^{-1}. *AB* acidic (pH 6.1) rice husk biochar, *NB* neutral (pH 7.1) rice husk biochar, *BB* basic (pH 11.0) rice husk biochar, *TN* total nitrogen. *, **, and *** are used to indicate statistically significant differences at the $p < 0.05$, $p < 0.01$, and $p < 0.001$, respectively. $^{a–f}$Each value with different letters within a column are significantly different from each other as determined by Duncan's multiple range test ($p < 0.05$).

decreased with the increasing the pyrolysis conditions, resulting in a sequential reduction of H:C and O:C ratio. These findings indicate that pyrolysis conditions play a crucial role in regulating the element composition of the rice husk biochar, thereby influencing its quality in terms of stability and aromaticity. The stability and aromaticity of rice husk biochar are reflected in the H:C and O:C ratio, respectively, with lower values considered superior. Previous studies have reported that higher pyrolysis conditions lead to an increase in the proportion of non-volatile compounds, particularly aromatic substances[31,32]. As the content of aromatic substances rises, the fixed carbon content and non-volatile compounds in rice husk biochar increase, contributing to the enhancement of its stability and aromaticity[32]. Furthermore, the aforementioned parameters were also decreased by the TC content of rice husk biochar, showing a positive (+) correlation with pyrolysis conditions.

The NH_3 emissions from agricultural soils are potentially depended on several factors such as the presence of soil amendments, the pH and moisture content of agricultural soil, method of nitrogen fertilizer application, and various agricultural practice (e.g., tillage, irrigation duration, and soil mulching)[4,33,34]. The PCA results by Liu et al.[35] were indicated that NH_3 volatilization varied in a descending order as follow: soil type, N source, soil pH, soil environmental conditions (e.g., temperature and moisture content). The application of biochar can adjust soil pH and enhance soil drainage, thereby improving the soil environment, which may influence NH_3 emissions[10,36]. Previous studies reported results indicating that biochar amendments promote the NH_3 emissions from agricultural soil owing to their alkali effects, which increase the soil pH[36–38]. In particular, high soil pH leads to higher rates of NH_3 volatilization because it raises the NH_3 concentrations dissolved in soil moisture[39]. Furthermore, another study documented that total NH_3 emissions increased by 10 to 71% with higher application rates of biochar[40]. These studies primarily focused on changes in soil pH influenced by the pH of biochar, and the NH_3 losses were found to be more pronounced in soil pH levels between 7 and 8[41]. To effectively manage the NH_3 emissions from agricultural land, it is necessary to maintain the soil pH below 7.0.

Conversely, several previous studies, which yielded conflicting results compared to the aforementioned studies, indicated that biochar amendments can effectively reduce the NH_3 volatilization from urea-treated soil under various conditions[42,43]. They demonstrated that the functional groups on the surface, adsorption ability, and cation exchange capacity of biochar contribute to decreasing NH_3 emissions from N-fertilized agricultural soils[44–46]. In this study, the soil amended with AB, NB, and BB exhibited lower NH_3 emissions compared to the solely urea-treated soil, which had the lowest soil pH values. These findings suggest that the reduction efficiency of NH_3 emissions by rice husk biochars, attributed to their functional groups, microspores, and adsorption ability, outweighs the increase in NH_3 emissions associated with elevated soil pH values. Furthermore, the reduction efficiency of rice husk biochars varied based on their pH, with BB amendment exhibiting higher NH_3 emissions compared to AB and NB amendments. As the pyrolysis conditions increased, the functional groups of BB decreased (Fig. 1), indicating a potential decrease in the NH_3 reduction efficiency of BB. This reduction could lead to a relatively higher NH_3 emissions, particularly when compared to AB or NB amendments, emphasizing the impact of pyrolysis conditions on the ammonia reduction efficiency of the biochar.

The application of rice husk biochars has been proven to enhance the growth and N uptake of Chinese cabbage, as shown in Table 3. This is supported by several previous studies that have examined the relationship between plant growth and biochar amendment[47]. Crop growth is primarily influenced by soil health, and biochar amendments are one of the factors that improve soil properties, fertility, and quality[48]. For instance, Munoz et al.[47] illustrated that biochar amendments can reduce both soil bulk density and particle density, while Peake et al.[48] demonstrated that the application of biochar improves soil compaction by more than 10%. Additionally, biochar application enhances soil fertility as it supplies essential elements such as N, P, K, Ca, Mg, Fe, and Si[48]. The findings of this study also support the notion that soil nutrient contents (e.g., Avail. N and Avail. P) were increased by rice husk biochar amendments (Table 2). The application of rice husk biochars increased the soil Avail. N content by capturing gaseous NH_3 and NH_4^+ through their functional groups. Nitrogen fixation by biochar was achieved through the surface characteristics of the biochar, primarily characterized by a negative charge[10]. The biochar absorbed N in cationic from (i.e., NH_4^+), and it exhibited superiority with a large surface area. In this study, the application of NB (6.49 $m^2\ g^{-1}$), which had a larger surface area compared to AB (2.55 $m^2\ g^{-1}$) and BB (5.30 $m^2\ g^{-1}$), resulted in the highest Avail. N content in the N-fertilized soil under the same N rates condition (Table 2). However, since the ionic bond between the biochar surface and the cationic form of N needs to be disconnected for N uptake by plants, the fixed N was not immediately utilized by plants in the short term. These reasons supported our findings, which demonstrated the highest fresh weight of Chinese cabbage in the short-term cultivation experiment with BB amendment, not NB amendment, attributed to the higher soil OM content. Although, not showing statistically significant differences among AB, NB, and BB amendments, the NB application may still improve soil fertility over the long term, resulting in better crop yields.

Conclusions

This study demonstrates the significant impact of rice husk biochar amendments in mitigating the NH_3 emissions during the Chinese cabbage cropping period. The NH_3 emissions resulting from chemical fertilization in agricultural soil decreased in the presence of rice husk biochar. Notably, the neutral (pH 7.10) rice husk biochar amendment exhibited the most substantial reduction in the NH_3 volatilization compared to the acidic (pH 6.10) and basic (pH 11.01) rice husk biochars. Furthermore, biochar amendments improved the Chinese cabbage yield, and this improvement was more pronounced with an increase in the pH of rice husk biochar. The highest agronomic performance of Chinese cabbage was observed in the basic rice husk biochar treatment with the 640 kg N ha^{-1} ($N_{2.0}$). Therefore, the application of neutral rice husk biochar can effectively reduce the NH_3 emissions from N-fertilized agricultural soil, while basic rice husk biochar leads to the highest agronomic performance and yield of Chinese cabbage.

Materials and methods

Experimental site

This study was conducted at the experimental field located in Chungnam National University, Daejeon, South Korea (35° 14′ 12.8″ N, 139° 7′ 0.5″ E). The experimental area experiences a humid continental and subtropical climate, both of which are influenced by the East Asian Monsoon[4]. During the summer season, which typically begins in June or July, the area receives high precipitation and is occasionally affected by typhoons. Detailed meteorological conditions during the cultivation period are presented in Fig. 5. The experimental field had been conventionally used for cultivating Chinese cabbage for approximately 5-year. The soil in the experimental field is classified as sandy loam, consisting of 12.8% clay, 41.4% silt, and 45.8% sand, and it belongs to the Inceptisols order.

Preparation of rice husk biochar

The rice husk biochars were prepared under different pyrolysis conditions using an electrical furnace (1100 °C Box Furnace, Thermo Scientific Inc., Waltham, Massachusetts, USA). Initially, rice husks sourced from rice paddy at Chungnam National University underwent thorough washing with deionized water to eliminate several impurities (e.g., bird poop, insect corpse, soil, and crop residue). Subsequently, the damp rice husks were stored in a glass greenhouse for 2-week to remove their moisture content. Following this, the dried samples were placed in a stainless-steel barrel (Ø 260 × 140 mm) with and aluminum packing, and subjected to pyrolysis using an electrical furnace. In this study, the aluminum packing was used to block the oxygen (O_2) inflow. Finally, the rice husk biochars were categorized based on their pH values, specifically pH 6.1 (AB), pH 7.1 (NB), and pH 11.0 (BB). AB was produced at 350 °C for 15 min, while NB and BB were manufactured at 450 °C for 15 min and 600 °C for 30 min, respectively. The selected pyrolysis conditions were established based on prior studies[15], that delineated the chemical properties of rice husk biochar under varying pyrolysis conditions, and preliminary experiment (Supplementary Table S3). In this study, AB exhibited the relatively minor differences from NB, likely attributed to the initial pH of the rice husk (pH 6.27). However, BB showed discernible differences from NB with increasing pyrolysis conditions. Therefore, we extended the pyrolysis time for BB from 15 to 30 min to observed the effect of stark pH differences.

Cultivation experiment

The cultivation experiment spanned a duration of 80 days, from April 12 to Jun 30, 2021, and followed a randomized complete block design with three replications. The 'Chunkwang' variety of Chinese cabbage (*Brassica rapa* L.) was sown in each plot, covering an area of 2.5 m × 3.0 m (7.5 m^2) with two rows. This study comprised thirteen treatments, including the following: control (non-fertilization), N fertilizer applied at recommended rate (320 kg N ha^{-1}, $N_{1.0}$), N fertilizer applied at half the recommended rate (160 kg N ha^{-1}, $N_{0.5}$), and N fertilizer applied at double the recommended rate (640 kg N ha^{-1}, $N_{2.0}$), as well as combined applications of the rice husk biochars (i.e., AB, NB, and BB) with N fertilizers (i.e., $AB + N_{1.0}$, $AB + N_{0.5}$, $AB + N_{2.0}$, $NB + N_{1.0}$, $NB + N_{0.5}$, $NB + N_{2.0}$, $BB + N_{1.0}$, $BB + N_{0.5}$, and $BB + N_{2.0}$). The rice husk biochars were applied to the agricultural soil at a rate of 1% (w w^{-1}), which was recommended by previous studies[49], and a mechanical tiller was used to incorporate the rice husk biochars with the soil. Before transplanting, 78 kg P_2O_5 ha^{-1}, and 60 kg K_2O ha^{-1}, in the form of fused phosphate and potassium chloride, respectively, were applied as basal fertilizer. Additionally, 46 kg K_2O ha^{-1} of potassium chloride was applied at 15, 30, and 45 days after transplanting. Similarly, 55, 110, and 220 kg N ha^{-1}, in the form of urea, were applied as basal fertilizer, with 35, 70, and 140 kg N ha^{-1} applied in three installments during the cultivation period. The plots were irrigated every 2 days and after each fertilizer application to prevent water stress.

Ammonia measurement and analysis

The measurement of daily and total NH_3 emissions during the Chinese cabbage cultivation period was conducted using a static chamber made of acrylic material (h: 30 × Ø: 12 cm, 0.011 m^2)[24]. To capture the released NH_3, a sponge soaked in a glycerol-phosphoric acid solution was placed inside the chamber for 24 h. Collection of

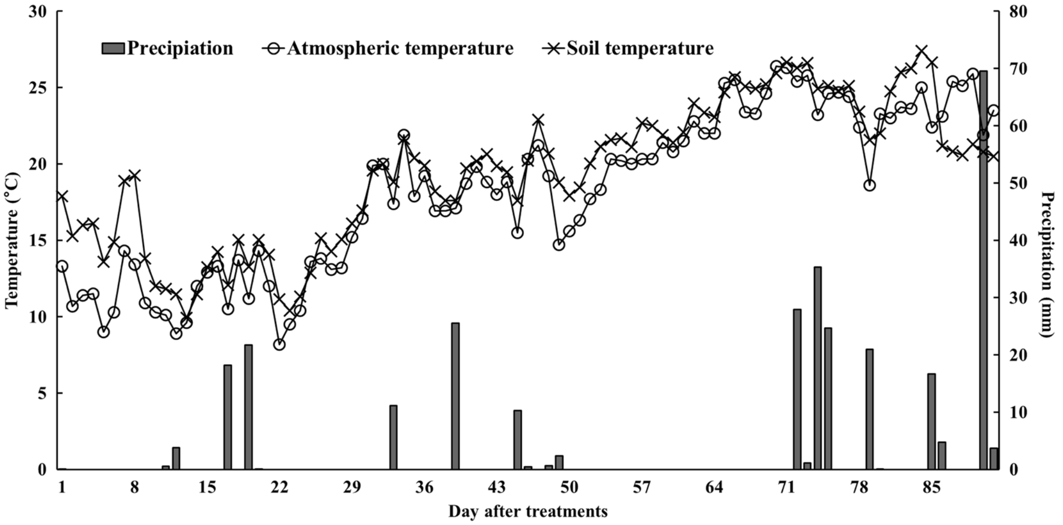

Figure 5. The meteorological data during the Chinses cabbage cultivation.

gaseous NH_3 was performed daily throughout the Chinese cabbage cultivation period, and after harvest, it was conducted twice a week until NH_3 volatilization resulting from N fertilization was no longer observed. The collected NH_3 samples were subsequently extracted using an excess of 2 M potassium chloride solution and quantified using a UV/Vis-spectrophotometer (Genesys 50, Thermo Scientific Inc., Waltham, Massachusetts, USA) following the Indophenol Blue method. Furthermore, another sponge was placed at the top end of the chamber before tightly sealing it. This top sponge served to isolate and absorb air or foreign substances, preventing their interference with the measurements. The daily and total NH_3 emissions during the Chinese cabbage cultivation period were calculated using the following equations[4,24].

$$Daily\ NH_3\ emission = \frac{(C \times V)}{(t \times A)} \tag{1}$$

$$Total\ NH_3\ emission = \sum_{i=0}^{n} (N_i \times D_i) \tag{2}$$

In Eq. (1), C represents the NH_4^+ concentration in sponge (mg L^{-1}), V denotes the volume of NH_4^+ solution obtained by sponge squeeze (L), t indicates the time to capture gaseous NH_3 samples (day), and A is the surface area of chamber (0.011 m^2). In Eq. (2), N_i represents the rate of daily NH_3 emissions in the *i*th sampling interval, D_i denotes the number of days in the *i*th sampling interval, and *n* represents the number of sampling intervals.

Soil, biochar, and plant analysis

Soil sample analysis involved the selection of ten random sampling points within each treatment. Soil texture was determined using the hydrometer method. Soil pH and EC were measured in soil slurry, where 1 g of soil was mixed with 5 mL of distilled water, using a pH and EC meter (ORION™ Versa Star Pro™, Thermo Scientific Inc., Waltham, Massachusetts, USA). The TC and TN contents were analyzed using an elemental analyzer (TruSpec Micro, Leco, Michigan, USA), while the OM content was calculated based on the TC content. The Avail. P and Avail. N contents were determined using a UV/Vis-spectrophotometer following the Lancaster method (for Avail. P content), Indophenol Blue method (for NH_4^+ content), and Brucine method (for NO_3^- content), respectively. Additionally, the Avail. N content was calculated as the sum of NH_4^+ and NO_3^- contents. Soil exchangeable cations were extracted using a neutral 1 M ammonium acetate solution and analyzed using a ICP-OES (ICAP 7000series ICP spectrometer, Thermo Scientific Inc., Waltham, Massachusetts, USA).

The pH of EC of the rice husk biochars were measured in a biochar slurry, where 1 g of biochar was mixed with 10 mL of distilled water, using a pH and EC meter. The BET surface area of the rice husk biochars was determined using a surface area analyzer (ASAP 2420, Micromeritics Inc., Norcross, Georgia, USA). Surface area was assessed using a N gas-adsorption method, and the sorption curves of N gas were analyzed to determine the biochar's surface area. The TC, TN, TH, and TO contents were analyzed by an elemental analyzer. The TP content was determined using the vanadate molybdate method with a UV/Vis-spectrophotometer. The inorganic contents (i.e., K_2O, CaO, MgO, and Na_2O) were analyzed using a FT-IR (Spectrum Two, Perkin Elmer, Waltham, Massachusetts, USA).

The growth parameters of Chinese cabbage were assessed as follows: fresh weight, moisture content, head height, head width, leaf length, leaf width, and chlorophyll content. Fresh weight was measured after harvest. Head height and width were estimated by measuring the half of Chinese cabbage, after measuring the length and width of the top three leaves with a ruler. Chlorophyll content was determined using a chlorophyll meter (SPAD-502 plus, Konica Minolta, Tokyo, Japan).

Statistical analysis

Each dataset was subjected to statistical analysis using multivariate analysis of variance (MANOVA) followed by Duncan's multiple range test to determine significant differences at a significant level $(p) < 0.05$. The statistical analysis was performed using the statistical software SPSS version 4.10.6 (SPSS Inc., Chicago, State of Illinois, USA).

Ethics approval and consent to participate

The seeds of Chinese cabbage were obtained from Sakata Korea (Seoul, South Korea). This study was conducted by complying with the Agricultural Life Resource Management Guidelines of Rural Development Administration, South Korea, the IUCN Policy Statement on Research Involving Species at Risk of Extinction, and the Convention on the Trade in Endangered Species of Wild Fauna and Flora.

Data availability

All data generated or analyzed during this study are included in this published article [and its supplementary information files].

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Author contributions

Y.G.K., J.H.C., and T.K.O. conceived and planned the experiments. Y.G.K., J.H.C., J.Y.L., and Y.U.Y. carried out the cultivation experiments. Y.G.K. and J.Y.L. contributed to the rice husk biochar sample preparation. J.H.C. and Y.U.Y. characterized the rice husk biochar samples. Y.G.K., Y.U.Y., and J.K.S. verified the analytical method. Y.G.K., J.H.C., and J.K.S. wrote the initial manuscript. Y.G.K., Y.U.Y., and T.K.O. revised the manuscript. Y.U.Y. and J.Y.L. prepared figure files. Y.G.K. and Y.U.Y. prepared table files. J.K.S. and T.K.O. supervised the project. All authors reviewed the final manuscript.

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Competing interests

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