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Reducing Ammonia Volatilization from Urea Fertilizer Applied in a Waterlogged Tropical Acid Soil *via* Mixture of Rice Straw and Rice Husk Biochars

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ABSTRACT
Nitrogen deficiency frequently occurs in agricultural soil because of ammonia volatilization to the environment, resulting in low urea-N use efficiency by plants. A laboratory incubation experiment was conducted to assess the effect of rice straw and rice husk biochar's on ammonia volatilization, soil pH, exchangeable ammonium, and available nitrate in comparison to the urea without additives under waterlogged conditions. Application of rice straw and rice husk biochar's mixture at application rate 5-10 t ha^{-1} had significantly minimized ammonia volatilization by 30.86% - 38.61% over T1 (175 kg ha^{-1} urea). T2 (5 t ha^{-1}) and T3 (10 t ha^{-1}) also had significantly increased retention of ammonium by 79% - 95% and nitrate ions by 49% - 51% over control. The treatments amended with biochar had successfully improved soil pH compared to T0 (soil only) and T1. Hence, the findings suggest that urea amended with rice straw and rice husk biochar's altered the nutrients level in the soil by minimizing ammonia loss to enhance nitrogen availability in waterlogged conditions.

Keywords: Ammonia volatilization, Biochar, Nitrogen, Urea, Ammonium

1. Introduction

Nitrogen (N) is a vital soil nutrient essential for good and abundant plant growth (Hajdu 2020). The main source of N for the plant comes from the external input application. Currently, urea (46% N) is being used in rice field as a main N contributor due to its abundant availability and relatively low price compared to other N fertilizers. However, there is a major concern in using urea as a N source because it is easily hydrolyzed and volatilized to the environment (Soares et al. 2012; Sunderlage & Cook 2018). Urea hydrolyzes upon contact with water and forms ammonia gas (NH_3) which is susceptible to atmospheric loss via volatilization process. Around 60% of urea volatilized in the form of NH_3 to the environment (Sommer et al. 2004; Rochette et al. 2009). The emission of NH_3 gas to the environment triggers air pollution, which harms living things. Besides, during the urea hydrolysis process, the formation of inorganic ammonium ion (NH_4^+) speeds up. Retention of NH_4^+ ion in the soil is relatively very poor due to the lack of binding-adsorptive agent. The NH_3 volatilization and poor retention of inorganic-N ions become very problematic for both farmers and plants. Deficient of N to plants cause farmers to increase the application of urea fertilizer, whereby this practice is not economical, efficient and reliable for long term use since it creates an environmental problem and costly.

Hence, an organic amendment such as biochar is necessary to minimize NH_3 loss to the environment. Biochar is a porous carbonaceous solid produced by charring or pyrolysis method of organic materials under oxygen-depleted environment (Lehmann & Joseph 2015; Ding et al. 2016). Biochar can be produced by utilizing agricultural wastes that are easily accessible and abundant. Mansor et al. (2018) stated that rice residues such as rice straw and rice husk are being produced annually more than 7,518,073 tonnes and 926,886 tonnes, respectively. The wastes are abundant and being burnt continuously. Burning wastes is hazardous for both the environment and human. So, turning the rice residues waste into biochar could be a promising approach to achieve sustainable waste management and benefits agronomy.

Biochar has a huge potential to improve soil fertility either by direct supply of nutrients or by fixing nutrients from the external source followed by slow release of the adsorbed nutrients (Unger 2008). The porosity and larger surface area of the biochar helps in nutrients absorption from the soil, which directly improves soil fertility (Lehmann & Joseph 2015). Biochar's surface area plays an essential role in binding cations, and anions (Atkinson et al. 2010; Chan & Xu 2009) which directly increases nutrient retention in biochar amended soil (Gai et al. 2014). Biochar capable to adsorb ammonium (NH_4^+) and (nitrate) NO_3^- onto its surface, thus increase the presence of these ions in the soil for plant uptake. Besides, biochar has been said to increase the formation of NH_4^+ and NO_3^- over NH_3. Eventually, this reduces NH_3 volatilization from applied urea fertilizer.

Yeboah et al. (2009) reported that an increase in soil nutrient retention due to the application of biochar results in the reduction of total fertilizer requirements in agricultural soil. Moreover, the alkaline nature of biochar modifies the soil acidity. Yuan et al. (2011), stated that biochar could act as an alternative liming agent to modify the soil pH in a way that it fits the crop growth.

Previous researchers demonstrated that biochar comes in different properties based on feedstock, charring condition, and activation. This agrees with Spokas et al. (2012) who also stressed the need for further research on biochar's economic and agronomic benefits. Additionally, there is also a scarcity of information on green feedstock biochar in amending soil fertility by preventing N loss either in rice, cash crops or other agricultural fields. It is crucial to know the properties of rice husk biochar and its ability to retain nutrients. It is hypothesized that the use of biochar produced from rice straw and rice husk wastes can create a pool of negative charges to retain and chelate the positively-charged NH_4^+ ions to prevent it from loss through NH_3 volatilization. Over time, the sorbed NH_4^+ ions on biochar will be gradually released and become available to plants and microorganisms. Biochar can also induce microbial immobilization of N into the soil. Hence, this study was carried out to determine the effect of mixing urea with mixtures of rice straw and rice husk biochar on NH_3 volatilization, exchangeable soil NH_4^+, and available NO_3^- as compared to the application of urea alone under a waterlogged condition.

2. Material and Methods

2.1. Soil sampling, preparation and characterization

The soil used in this study was Renggam sandy clay loam (*Typic Paleudult*). It was sampled at 0-30 cm from a land at the Agro Techno Park in University Malaysia Kelantan Jeli Campus, Malaysia (5^0 44' 69.55" N latitude and 101^0 51' 83.89" E longitude) has not been cultivated since 2007. The collected soil was air-dried, crushed, and sieved to pass through a 2-mm sieve for initial soil characterization. Soil pH was measured in a ratio of 1:10 (soil:water) using a digital pH meter (Peech 1965). Soil texture was determined using the hydrometer method (Bouyoucos 1962). Total organic matter content, ash content, and total organic carbon were determined using the loss-on ignition method (Tan 2005).

Total N was determined using the Kjeldahl method (Bremner 1965). The double acid method described by Mehlich (1953) was used to extract soil available P and exchangeable cations (Ca, Mg, K, and Na), after which the cations were determined using an Atomic Absorption Spectrophotometer (AAS) (Analyst 800, Perkin Elmer, Norwalk, USA). Soil available P was determined using molybdenum blue method (Murphy & Riley 1962). The developed blue color was analyzed using a UV-VIS spectrophotometer (Thermo Scientific Genesys 20, USA) at 882 nm wavelengths. Soil cation-exchange capacity (CEC) was determined by the ammonium acetate leaching method (Cotteinie 1980). The exchangeable acidity and exchangeable aluminum (Al^{3+}) were determined by the acid-base titration method described by Rowell (1994). The method described by Keeney & Nelson (1982) was used to extract exchangeable NH_4^+ and available NO_3^-, after which the ions were determined *via* steam distillation (Tan 2005).

2.2. Biochar production, activation and characterization

Rice husk was collected from Pasir Puteh Rice Mill whilst rice straw was collected from Kemubu granary area, Kota Bharu, Malaysia. Two cylindrical kilns, a 200 L drum with removable chimney caps and an airtight 110 L drum were constructed for biochar production. The rice husk and rice straw were bulked separately inside the 110 L drum then, closed and placed in the middle of the 200 L drum, where the fire was kindled starting from the bottom of the drum. The residence time was 4 hours with the temperature ranging from 300 - 400 °C and left for cooling for 2 hours. The temperature inside the kiln was measured using Extech TM100 K/J (Single Input Thermometer, Waltham, Massachusetts, United States). Later, the pile of biochar sample was spread out for cooling. After this, the enrichment of biochar was carried out by soaking with 5% chicken slurry for 7 days which later was dried and stored in a big container for further use. The enrichment of biochar with chicken slurry was crucial to further increase the nutrient content, alter the surface area, and increase the pore size (Selvarajh et al. 2021a). The enriched biochars were analyzed for pH (Peech 1965), CEC and total N (Bremner 1965). The single dry ashing method (Tan 2005) was used to extract nutrients from rice husk and rice straw biochar for analysis of Ca, Mg, Na, P, and K using an AAS (Analyst 800, Perkin Elmer, Norwalk, USA), while total P content was determined using the molybdenum blue method (Murphy and Riley 1962), after which the blue color developed was analyzed using a UV-VIS Spectrophotometer (Thermo Scientific Genesys 20, USA) (Murphy and Riley 1962). Total C was determined using the loss on ignition method (Tan 2005). Additionally, microanalysis through Scanning Electron Microscopy-attached with Energy Dispersive X-ray Spectroscopy analysis (SEM-EDX JEOL JSM-6400) was carried out analyze the surface morphology of enriched rice husk and rice straw biochar.

2.3. Ammonia volatilization laboratory incubation study

For laboratory-scale NH_3 volatilization study, the actual amount of urea applied was 0.7 g, scaled down from the 175 kg ha^{-1} application rate. The rice husk and rice straw biochar (1:1 ratio) actual application for 100 g of soil, scaled-down from 5, 10, 15, and 20 t ha^{-1} was 2.8 g, 5.5 g, 8.3 g, and 11.1 g, respectively. The treatments evaluated were listed in Table 1. Soil, urea, and biochar were mixed well before deposited into 250 mL conical flask, after which water was added to create a waterlogged condition. The water level was maintained 3 cm above the soil throughout the study. This set up was done to depict the waterlogged condition in the actual rice field. The system was set to be a closed dynamic airflow system, and the NH_3 loss from urea was measured daily (Siva et al. 1999; Ahmed et al. 2006a, 2006b). The system includes a 250 mL conical flask exchange chamber containing soil mixture and a trap 250 mL conical flask chamber containing 75 mL of boric acid, which were stoppered and fit with inlet/outlet pipes. The chamber inlet containing the water was connected with an aquarium air pump and outlet connected with pipe tubing to the trap containing boric acid solution. Air was passed through the chambers at a rate of 2.75 L^{-1} min^{-1} $chamber^{-1}$. This setup was done to create soil aeration and trap NH_3 loss *via* volatilization process. The released NH_3 was captured in the trapping solution containing 75 mL of boric acid with colour indicator. The incubation chambers Boric acid-indicator traps were replaced every 24 h and back, titrated with a size of 0.01 M HCl, to estimate the NH_3 released. Measurement was continued until the loss declined to 1% of the N added with urea (Ahmed et al. 2008) After the NH_3 volatilization was evaluated, the soil samples were used for pH, exchangeable NH_4^+ and available NO_3^- determinations.

Table 1- Treatments evaluated in ammonia volatilization incubation study

Treatments	***Description***
T0	Soil only
T1	Soil + 175 kg ha^{-1} urea
T2	Soil + 175 kg ha^{-1} urea + 2.5 t ha^{-1} rice husk biochar + 2.5 t ha^{-1} rice straw biochar
T3	Soil + 175 kg ha^{-1} urea + 5 t ha^{-1} rice husk biochar + 5 t ha^{-1} rice straw biochar
T4	Soil + 175 kg ha^{-1} urea + 7.5 t ha^{-1} rice husk biochar + 7.5 t ha^{-1} rice straw biochar
T5	Soil + 175 kg ha^{-1} urea + 10 t ha^{-1} rice husk biochar + 10 t ha^{-1} rice straw biochar

2.4. Statistical analysis

The experiments were arranged in a completely randomized design with three replicates. The effect of different rice husk and rice straw biochar addition rates was subjected to one-way analysis of variance (ANOVA). Statistical analysis for all the data was performed using SPSS software version 24.0 (SPSS Inc, US). Significant differences among treatments were separated by Tukey's HSD test and considered significant at $P \leq 0.05$.

3. Result and Discussion

3.1. Selected soil physico-chemical properties

The selected soil physico-chemical properties used in this study are presented in Table 2. The soil showed a sandy clay loam texture with pH of 5.5. The soil showed relatively high Al (1.14 $cmol_c\ kg^{-1}$) concentrations and Fe (0.091 $cmol_c\ kg^{-1}$) due to low soil pH. This condition correlates to the lesser P availability (0.385 ppm) in soil because of P fixation by Al and Fe. Exchangeable K, Ca, Mg, and Na was low in the soil due to the soil's lower CEC (5.4 $cmol_c\ kg^{-1}$). Lower CEC of the soil leads to inefficient nutrient holding and retention capacity of basic cations. The content of N (0.07 %), NH_4^+ (89 ppm), and NO_3^- (30 ppm) in the soil were low because of soil acidity, which slows down the mineralization process. Khalil et al. (2005) stated that acidic soil causes N immobilization instead of N mineralization. The soil used in this study needs amelioration to improve the soil quality and fits for crop growing.

Table 2- Selected soil physico-chemical properties

Property	***Value obtained***
pH	5.5
EC (dS m^{-1})	0.022
Texture	Sandy clay loam
Soil organic matter (%)	6.24
Total Carbon (%)	3.62
Ash content (%)	6.4
Cation exchange capacity ($cmol_c\ kg^{-1}$)	5.4
Ammonium (ppm)	89
Nitrate (ppm)	30
Total N (%)	0.07
Available P (ppm)	0.385
Exchangeable K ($cmol_c\ kg^{-1}$)	0.084
Exchangeable Ca ($cmol_c\ kg^{-1}$)	0.10
Exchangeable Mg ($cmol_c\ kg^{-1}$)	0.082
Exchangeable Fe ($cmol_c\ kg^{-1}$)	0.091
Exchangeable acidity ($cmol_c\ kg^{-1}$)	0.7
Exchangeable Al ($cmol_c\ kg^{-1}$)	1.14

3.2. Characterization of rice husk and rice straw biochars

The surface morphological characteristics of rice husk biochar and rice straw biochar are shown in Figure 1 and Figure 2. Both rice straw and rice husk biochar composed of numerous pores and comes with a high surface area. This property is highly beneficial for agronomical practices in terms of increasing nutrient retention and boosting crop growth. Biochar's porous structure and bigger surface area help in binding ions from soil and external inputs. Lin et al. (2012) stated that biochar's high porous structure might have extractable humic-like and fluvic-like substances that act as chelators. Humic and fluvic acid restrict toxins in the soil by reducing harmful substances to reach the crop roots. Besides, biochar has a high surface area with a strong affinity to attract inorganic ions (Schmidt et al. 2015). This would be a great advantage in the agriculture field to bind nutrients from the soil and release it slowly as it degrades. The capability of biochar to bind nutrients also related to the higher CEC value, where rice straw biochar and rice husk biochar CEC is 75.6 $cmol_c$ kg^{-1} and 66.6 $cmol_c$ kg^{-1}, respectively (Table 3). Biochar with higher CEC value adsorb more nutrients onto its surface and minimize volatilization. Not only that, the alkaline nature of both biochars (pH>9) can act as natural liming agent to reduce the acidity of soil at a certain application rate. The biochar also inherently packed with nutrients such as N, P, K, Ca, Mg, and Na. Eventually, the nutrients in biochar will be released to the soil for effective utilization by plants.

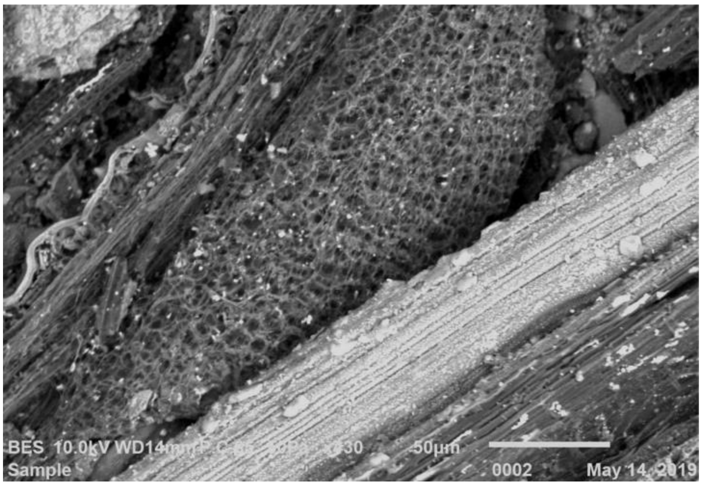

Figure 1- Rice husk biochar surface at 550x, magnification under SEM

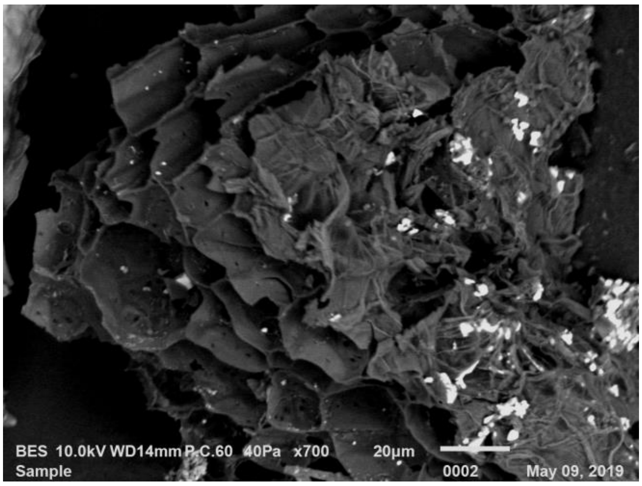

Figure 2- Rice straw biochar surface at 730x, magnification under SEM

Table 3- Selected physico-chemical properties of rice straw and rice husk biochar

Property	*Rice straw biochar*	*Rice husk biochar*
pH (water)	9.2	9.1
CEC (cmol kg^{-1})	75.6	66.6
Total Nitrogen (%)	0.45	0.33
Available P (ppm)	14.3	14.3
Exchangeable Ca ($cmol_c$ kg^{-1})	0.98	0.21
Exchangeable Mg ($cmol_c$ kg^{-1})	0.58	0.27
Exchangeable K ($cmol_c$ kg^{-1})	7.68	2.51
Exchangeable Na ($cmol_c$ kg^{-1})	0.04	0.05

3.3. Combined effect of rice husk and rice straw biochars on NH_3 volatilization

The daily NH_3 volatilization from urea fertilizer during the incubation study over 28 days is presented in Figure 3. NH_3 loss started on day 2 of incubation in treatment T1, on day 7 for T2 and T3, day 6 for T4 and T5, while no loss was found for T0. The delayed loss upon urea application shows the efficacy of added rice straw and rice husk biochars as an organic amendment in minimizing NH_3 formation. The NH_3 loss from urea can be delayed for 3-6 days with the addition of organic materials (Omar et al. 2010). The maximum NH_3 loss for T1 occurred on day 5, T2 on day 12, T3 on day 13, T4 on day 11, and T5 on day 13. The graph shows that NH_3 loss peaks up and reduces gradually up to 28th day until N added as urea ceases up to 1%. Rapid NH_3 loss in T1 was probably due to increased soil pH as urea hydrolysis leads to hydrogen ions (H^+) from the soil solution. However, in treatment amended with rice straw and rice husk biochar, the NH_3 loss was minimal due to the increased formation of NH_4^+ over NH_3 in the soil. Besides, Dougherty et al. (2017) stated that biochar's addition minimizes NH_3 volatilization by increasing the NH_3 adsorption at the oxygen-containing surface functional group or biochar micropores.

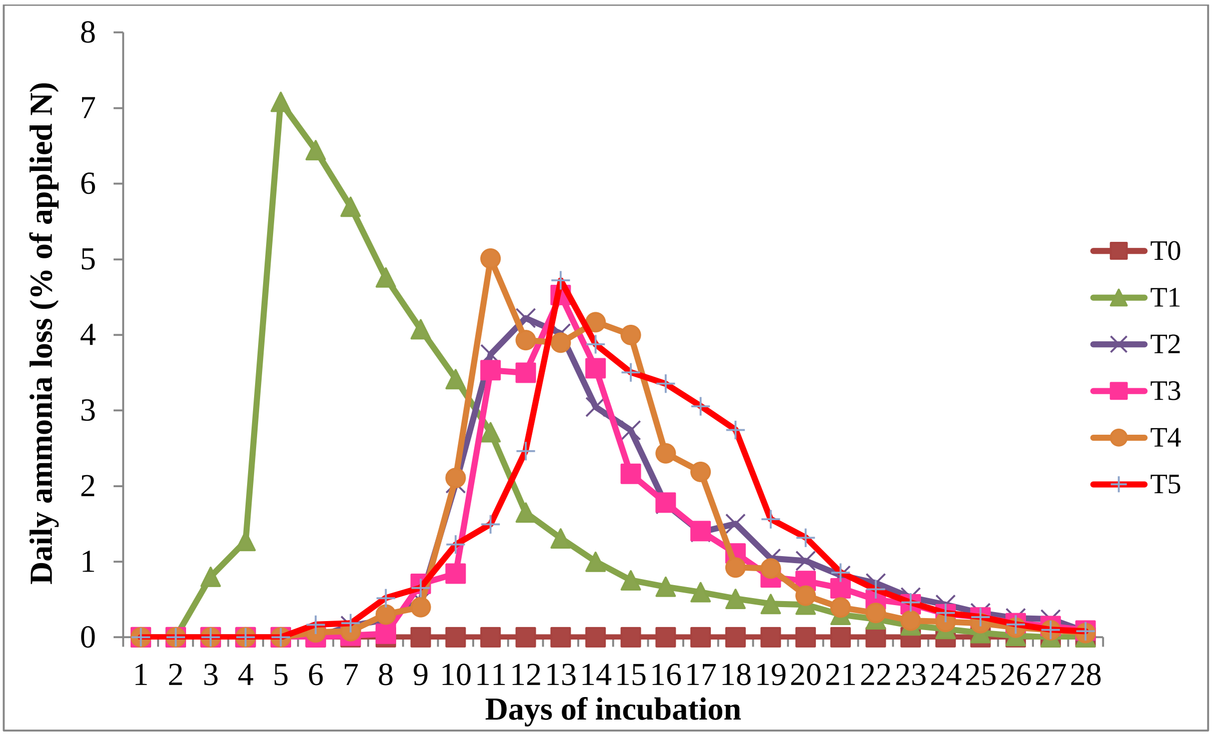

Figure 3- Ammonia volatilization over 28 days of incubation under waterlogged condition

The treatments with biochar as an additive (T2, T3, T4, and T5) had significantly minimized NH_3 loss compared to urea without additives (T1) (Figure 4). The total amounts of NH_3 lost at the end of the incubation period as a percentage of urea-N added were 0, 44.52, 30.79, 27.33, 32.62, and 33.66% for T0, T1, T2, T3, T4, and T5, respectively. Noticeably, T2 and T3 were significantly effective in minimizing NH_3 loss over T1. Irrespective of the application rate, all the treatments with biochar as an additive had effectively reduced NH_3 loss compared to T1. Addition of porous biochar with larger surface area delayed and minimized NH_3 loss due to its capability to bind more NH_4^+ and NO_3^- ions (Figures 6 and 7). This was in agreement with a study conducted by Chen et al. (2013). Besides, biochar increases the soil volume and pore size and stabilize the soil aggregate (Burrell et al. 2016). Since the volume of soil increased with which urea is mixed, it will also increase the time required for complete urea hydrolysis (Fan & Mackenzie 1993). Delays in urea hydrolysis due to the biochar application can minimize N loss, which will benefit plants in the agricultural field.

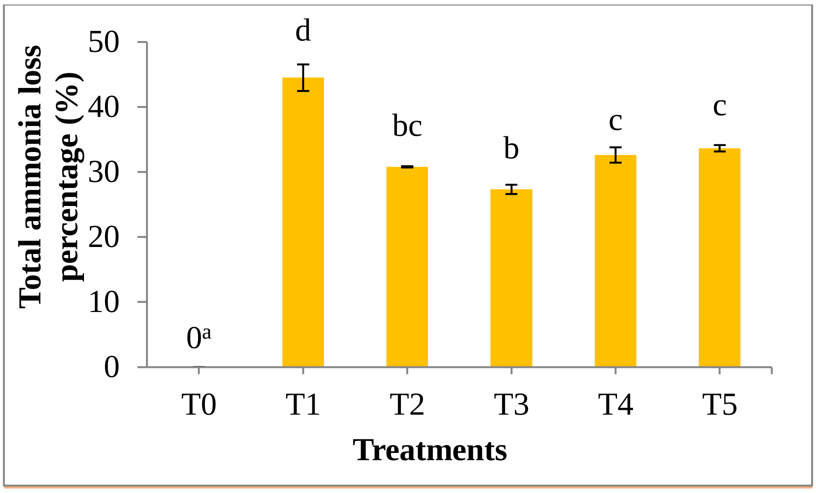

Figure 4- Total NH_3 losses from incubation study under waterlogged conditions.

Besides, biochar has alkaline nature where it can act as a liming agent to reduce soil acidity. Treatments with biochar had improved soil pH (Figure 5). Ch'ng et al. (2016) and Tang et al. (1999) stated that an increase in the soil pH was due to the rapid proton exchange between soil and biochars. The increase in soil pH is also related to the release of anions from rice straw and rice husk biochar, where anions undergo decarboxylation and exchange of protons in soil. In the previous study, it has been reported that NH_3 volatilization speeds up in soil with higher pH (Sun et al. 2019), but contrastingly in this study, the added biochars minimized the volatilization. This is due to the nutrients' adsorptive capability of the rice straw and rice husk biochars. Selvarajh et al. (2021b) also stated that increased soil pH due to biochar's addition does not significantly trigger ammonia loss.

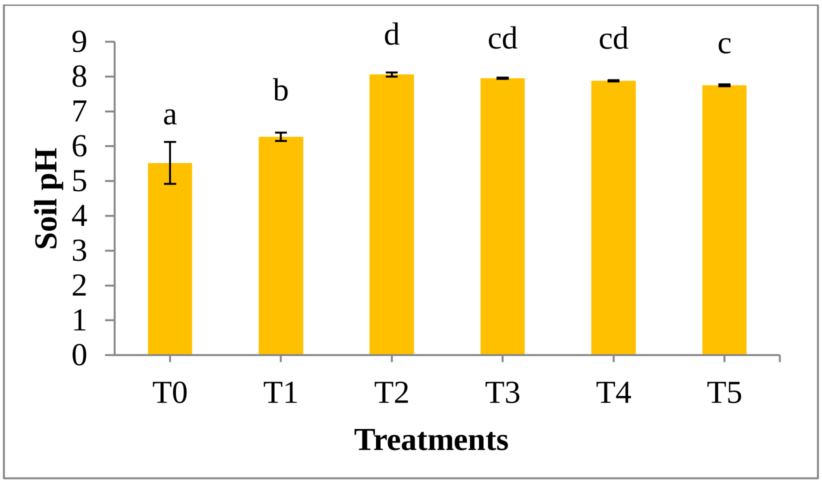

Figure 5- Soil pH after incubation study

Additionally, biochar had successfully sorb nutrients from the soil. T2, T3 and T4 had shown significant NH_4^+ retention in soil over T0, T1, and T5 (Figure 6). T2 and T3 had retained the highest amount of NH_4^+ by 95% and 79% respectively over T1, followed by T4 and T5, which is 54% and 12%. This shows that the biochar had increased the formation of NH_4^+ ions over NH_3. Besides, the nitrate ions in the soil are found to be higher. The T2 and T3 had retained more amount of NO_3^- by 51% and 49%, respectively, compared to T1, followed by T4 and T5 which is 23% and 9% (Figure 7). Biochar retains more charged ions because it has zwitterions properties that bind ions on its surface (Waters et al. 2010). Another reason for the higher retention of NH_4^+ could be associated with the higher CEC of rice husk and rice straw biochar, 66.6 $cmol_c$ kg^{-1} and 75.6 $cmol_c$ kg^{-1}, respectively absorbs the ions and releases it slowly. This was in agreement with Omar et al. (2010). The higher content of NH_4^+ and NO_3^- suggest that the inclusion of combined biochar of rice husk and rice straw had improved the presence of nutrients in the soil for uptake by plants.

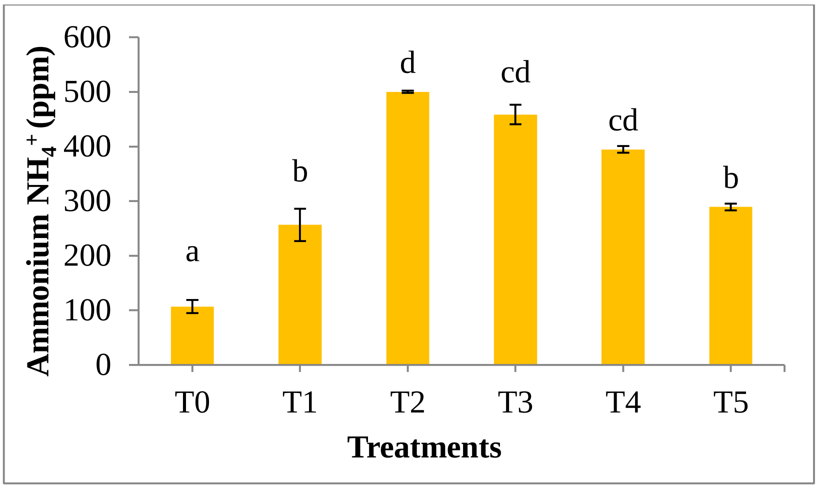

Figure 6- Ammonium (NH_4^+) retention in soil after incubation study.

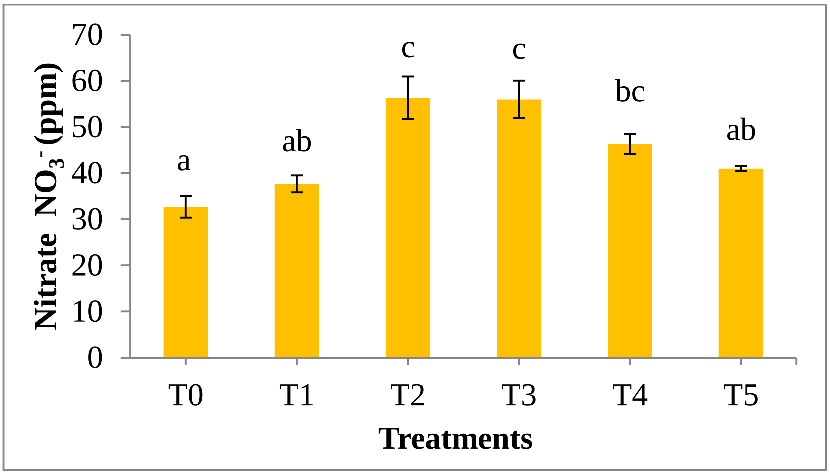

Figure 7- Nitrate (NO_3^-) retention in soil after incubation study.

Note: Mean values with different letter(s) indicate significant difference between treatments by Tukey's test at P≤0.05. Bars represent the mean values ± SE.

4. Conclusions

The result of this study suggests that the application of urea with a mixture of rice straw and rice husk biochars at the rates of 5 t ha^{-1} and 10 t ha^{-1} offers a significant advantage over urea alone. The biochar mixtures have effectively retained more NH_4^+ and NO_3^- ions in the soil by minimizing conversion to NH_3 even at increased soil pH levels. This leads to a significant reduction in NH_3 released into the atmosphere. The addition of a mixture of rice straw and rice husk biochars retained more inorganic N in the soil. Eventually, this will lead to sustainable N management in rice production and prevent greenhouse NH_3 gas emissions.

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