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EFFECT OF BIOCHARS PRODUCED AT DIFFERENT PYROLYSIS TEMPERATURES ON AMMONIUM (NH_4^+) AND NITRATE (NO_3^-) LEACHING: COLUMN EXPERIMENT

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Abstract: Nitrogen (N) leaching from agricultural soils is a global problem with negative effects on both human health and the environment. Efforts should be made to increase the efficiency of use of plant nutrients and minimize N losses from terrestrial ecosystems to aquatic ecosystems. In this study, the effects of different doses (%0, %1 and %2) of biochar obtained from corn cob and rice husk biomass, which are agricultural production residues, at three different temperatures, on ammonium (NH_4^+) and nitrate (NO_3^-) leaching in a coarse-textured soil were investigated. Polyethylene (PE) columns with a diameter of 70 mm and a height of 20 cm were used in the study, which was carried out in three replications according to the randomized plots experimental design. Total nitrogen (27 kg N da^{-1}) and water amounts (969 mm) used for 6 tons da^{-1} yield of sugar beet were applied. Total water was given to each column in equal volume using drip adjustment sets at one-week intervals, simulating 6 irrigation periods, and the leaked water was collected in each irrigation period and NO_3^- and NH_4^+ concentrations were determined. Applications of 1 and 2 doses of corn and rice biochars obtained at three different pyrolysis temperatures caused a significant decrease in NH_4^+ concentrations leaching from the column. Similarly, biochar applications (especially 2% dose) caused a significant decrease in NO_3^- concentrations leaching from the column. While the total NO_3^- concentration leaching from the control columns was 149.23 mg kg^{-1}, 2% dose of rice husk biochars at 300, 400 and 500 °C temperature applications caused a decrease in the total NO_3^- concentrations washed from the column by 51%, 55% and 51%, respectively. The results revealed that biochar applications significantly reduced nitrogen leaching from the soil.

Keywords: Ammonium, Biochar, Fertilizer, Leaching, Nitrate, Soil

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1. Introduction

The leaching of nutrients from agricultural lands, due to its adverse effects on ecosystems, is recognized globally as a significant environmental issue (Rashmi et al., 2017). Nitrogen, one of the most commonly used plant nutrients in agricultural production, occupies a central position in this issue (Yahaya et al., 2023). Farmers tend to apply excessive nitrogen in order to achieve higher yields when cultivating high-income crops. This situation exacerbates the issue of nitrogen leaching, particularly in the form of nitrate, further threatening soil fertility. The amount of leaching nitrogen increases proportionally with the quantity of nitrogen-based fertilizers applied (Abascal et al., 2022). Nitrate, known as a mobile ion, particularly enhances the leaching potential in low clay content soils (Forde and Zhang, 1998; Köhler et al., 2006). Sustainable agricultural practices and proper fertilization techniques are of critical importance in overcoming this issue, increasing crop productivity, and reducing environmental adverse effects. As a potential solution, biochar has the capacity to reduce nitrogen leaching from agricultural production areas (Borchard et al., 2019).

Biochar, a carbon-rich material obtained through the pyrolysis of organic matter, has garnered significant attention due to its potential for carbon (C) sequestration (Teutscherova et al., 2018; Tepecik et al., 2024) and mitigating climate change through the reduction of greenhouse gas emissions.

Biochar can enhance the cation exchange capacity of soil, thereby preventing leaching of nutrients and harmful chemicals (Elkhlifi et al., 2023). Additionally, biochar improves the physical and chemical structure of soil, increasing its water retention capacity and promoting microbial activity (Banik et al., 2023). Due to its porous structure and surface charge, biochar emerges as a promising material for reducing N leaching (Laird et al., 2010). An increasing number of studies demonstrate that biochar's extensive surface area and surface charge can reduce N leaching (Ding et al., 2010). Indeed, the cation exchange capacity (CEC) is likely responsible for the retention of ammonium (NH_4^+-N) by biochar (Jellali et al., 2022). However, the sorption properties of biochar depend on the feedstock and pyrolysis temperature. For instance, Yao et al. (2012) reported that biochars produced at temperatures of 600 °C or higher exhibited the highest nitrate (NO_3^--N) adsorption. Consequently, nitrogen management and biochar applications are significant strategic practices to enhance the sustainability of agricultural lands and leave healthier soils for future generations. This approach and its implementations not only reduce environmental impacts but also increase productivity and profitability in agricultural production. In this study, the effect of biochar applications derived from agricultural residues, such as corn cob and rice husk biomass, obtained at three different temperatures, applied at control, 1%, and 2% doses, on ammonium and nitrate leaching in a coarse-textured soil, was investigated.

2. Materials and Methods

2.1. Soil and Biochar Materials

Corn cob and rice husk, agricultural residues abundantly available as waste, were used as raw materials for biochar in the experiment. Biomasses collected from the field were brought to the laboratory and initially dried at 70 °C until reaching a constant weight. While corn cobs were ground using a fodder grinder due to their large particle size, rice husks were left untreated without any grinding process. Biochar was produced from corn cob and rice husk biomass through slow pyrolysis in a specially made biochar production chamber (40x25x20 cm^3) located in the Department of Soil Science and Plant Nutrition at Tokat Gaziosmanpaşa University, Türkiye. The biomass was subjected to pyrolysis at temperatures of 300 °C, 400 °C, and 500 °C individually in a muffle furnace, resulting in the production of biochar. During production, syngas and tar generated were not stored. Some physical and chemical analysis results of the obtained biochars are provided in Table 1.

The soil material used in the study was collected from the Agricultural Research and Application Field of Tokat Gaziosmanpaşa University, Tokat, at a depth of 0-30 cm, and air-dried after passing through a 4 mm sieve. The soil used in the study is slightly alkaline (pH: 7.83), non-saline (EC: 0.74 µS/cm), low in organic matter content (%1.46), and characterized by a sandy loam texture (%88.40 sand, %7.91 clay, %3.69 silt).

2.2. Column Leaching Experiment and Laboratory Analyses

For the leaching experiment, PE columns with a diameter of 70 mm and a height of 20 cm were utilized. Coarse filter papers were placed at the bottom of the columns to prevent the mixture of soil+biochar from spilling, and to protect the filter paper from damage, a fine porous fiber mesh was used to cover the top of the column, secured with a PE clamp (Figure 1).

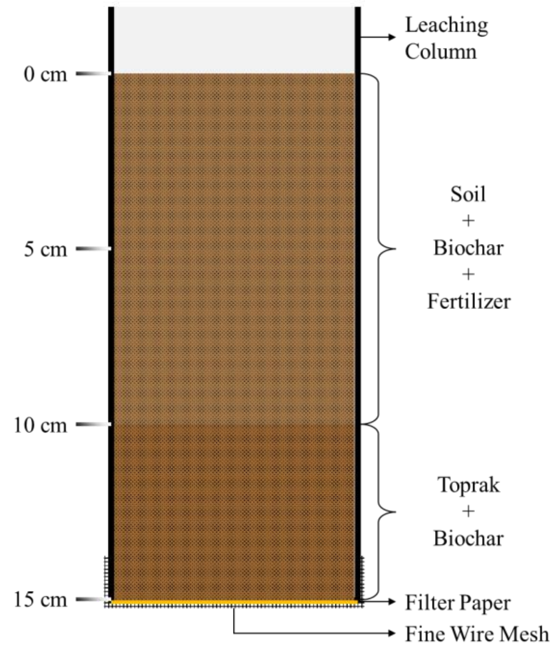

Figure 1. General structure of the column model.

The amount of fertilizer used in the leaching experiments was determined based on the nitrogen requirement of sugar beet plants. According to a study, it was determined that 4-5 kg/ha of pure nitrogen is removed from the soil for the production of 1 ton of sugar beet (İlbaş et al., 2016). The column experiment, conducted in accordance with a randomized complete block design with three replications, involved biochar applications derived from rice husk and corn cob produced at different pyrolysis temperatures (300, 400, and 500 °C) at three different doses (control, 1%, and 2% BC).

Table 1. Some physical and chemical properties of the biochars used in the study

Parameter	Corn Cob			Rice Husk		
	300 °C	400 °C	500 °C	300 °C	400 °C	500 °C
C (%)	43.8	58.6	62.7	48.5	51.7	54.7
N (%)	0.54	0.38	0.29	0.71	0.76	0.58
C/N	81.2	154.2	216.3	68.2	68.1	94.3
P (%)	1.94	2.69	4.12	0.09	0.13	0.18
K (mg kg^{-1})	60.1	96.3	172.7	76.0	82.6	98.6
Mg (mg kg^{-1})	16.3	20.6	26.4	13.6	20.0	22.2
Specific Surface Area (m^2 g^{-1})	156	221	320	68	79	127
pH (1/20)	8.40	8.60	9.72	7.94	8.43	10.4
EC (1/20) (µS cm^{-1})	214	685	1251	176	274	615

Before adding soil (650 g soil) to each column, biochar doses were thoroughly mixed with soil to achieve homogeneity. Subsequently, the soil+biochar mixture was added to the 10-15 cm interval of each column, while a mixture of soil+biochar+fertilizer was added to the 0-10 cm interval. Accordingly, considering a sugar beet yield of 6 tons/ha, a fertilizer rate of 27 kg da^{-1} was applied in the form of NH_4NO_3. In the experiment, to simulate the irrigation during the sugar beet plant's growing season, the total rainfall received by Tokat province during the sugar beet production season, reported as 969 mm (TAGEM, 2017), was divided into 6 irrigation periods, and a total of 3.73 L of water was applied to each column using drip irrigation sets with one-week intervals (Figure 2). Water applications were conducted weekly, equally to each column, and the experiment was terminated when no further water leakage was observed from the column at the end of the 6th week. At the end of each week, the concentrations of NH_4^+ and NO_3^- in the leachate obtained from each column were determined. Nitrate was measured by spectrophotometry based on the yellow color complex formed by nitrate with sodium salicylate (Fabig et al., 1978), while ammonium was determined based on the green color complex formed by ammonium with nitroprusside salicylate (DEZWAS, 1983).

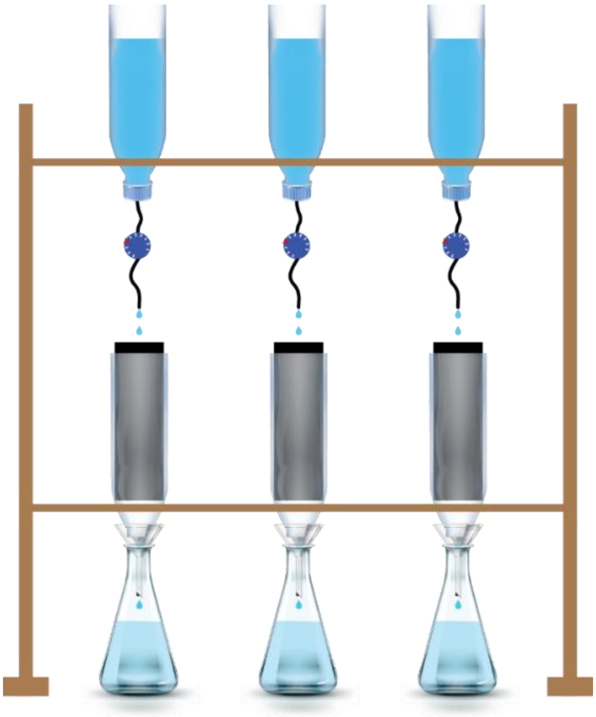

Figure 2. Schematic of the column assembly.

2.3. Statistical Analysis

Descriptive statistical data for the concentrations of NH_4^+ and NO_3^- in the leachates obtained from different doses of biochar applications were determined, and the differences in the effects of different biochar applications on ammonium and nitrate leaching were assessed using one-way analysis of variance (ANOVA) to determine if they were statistically significant or not. Homogeneity tests for the applications were determined by Duncan grouping. These statistical analyses were conducted using the SPSS 27.0 software package (Genc and Soysal, 2018).

3. Results and Discussion

The values of ammonium (NH_4^+) concentrations in the solutions leached from columns with applications of corn and rice biochars obtained at different pyrolysis temperatures at doses of 1% (BC1) and 2% (BC2) are presented in Figure 3. For all pyrolysis temperatures of corn and rice biochars (except BC2 for CC500), the variation in NH_4^+ concentrations leached from the columns in both 1% and 2% dose applications was found to be statistically significant (P<0.05) (Figure 3 a,b,c; Table 2). Comparing with the control columns, it was observed that the NH_4^+ concentrations leached from the columns with biochar applications were lower (Figure 3abc; Table 2).

Table 2. Total NH_4^+ and NO_3^- concentrations leaking from the columns

Application	NH_4^+ (mg L^{-1})	NO_3^- (mg L^{-1})
Control	53.27^{m}	149.23^{l}
RH300-%1	29.92^{k}	91.83^{i}
RH300-%2	27.86^{j}	72.49^{e}
RH400-%1	18.39^{g}	86.91^{h}
RH400-%2	13.12^{e}	67.37^{c}
RH500-%1	4.01^{c}	108.84^{k}
RH500-%2	3.70^{b}	72.72^{e}
CC300-%1	6.13^{d}	79.18^{f}
CC300-%2	0.66^{a}	83.00^{g}
CC400-%1	19.89^{h}	69.24^{d}
CC400-%2	16.04^{f}	59.22^{a}
CC500-%1	23.73^{i}	89.02^{i}
CC500-%2	32.28^{l}	60.86^{b}

*There is no statistical difference between the averages shown with the same letter (P<0.05) (Each column is lettered within itself).

When evaluating the columns under rice husk biochar applications, it was observed that the total NH_4^+ concentration leached from the control application was 53.27 mg L^{-1}. However, under RH500 BC1 dose, it decreased by 92.5% to 4.01 mg L^{-1}, and under RH500 BC2 dose, the NH_4^+ concentration decreased by 93% to 3.70 mg L^{-1} (Table 2). When evaluating the weekly leached NH_4^+ concentrations, it was found that both the control application and the applications of rice husk and corn cob at all doses and temperatures had higher NH_4^+ concentrations leached in the 3rd week compared to other weeks (Figure 3 a,b,c). In contrast, NH_4^+ concentrations leached from the columns were lower in the 1st and 6th weeks (Figure 3 a,b,c). Similar to our findings, Ding et al. (2010) reported that bamboo biochar adsorbed NH_4^+ ions through cation exchange, protecting against leaching and significantly delaying the vertical movement of NH_4^+ to deeper soil layers during a 70-day experimental period. Laird et al. (2010) reported that biochar produced from a mixture of oak and walnut sawdust reduced total N and P leaching by 11% and 69%, respectively, with applications ranging from 0%, 0.5%, 1%, and 2%.

Among biochars produced from two different feedstocks, the leaching of NH_4^+ was observed to be the lowest in the RH500 BC2 dose of rice husk biochar and the CC300 BC2 dose of corn biochar (Table 2). In previous studies, it has been reported that biochar reduces N leaching due to its extensive surface area and surface charge (Ding et al., 2010; Yao et al., 2012). Researchers have reported that biochars protect against leaching by increasing the retention of ammonium (NH_4^+-N) due to their high cation exchange capacity (CEC) (Alkharabsheh et al., 2021). In a study where pine biochar was mixed with soil at rates of 0.5%, 2.5%, and 10%, and ammonium nitrate was mixed with the soil to achieve a rate of 10 kg/ha, weekly leachate was conducted for 6 weeks. In the study, the application of 10% biochar reduced ammonium leaching by 86% and nitrate by 96% (Sika & Hardie, 2014). According to the biochar application dose, the total nitrogen (N) content of the soil increased according to the application doses (Şenay and Tepecik, 2024).

Due to nitrate being a highly mobile anion, it tends to leach more in soils with low clay content and coarse to medium texture (Ferretti et al., 2023). Consistent with the literature, it was found that the leached NO_3^- concentrations from columns without biochar applications were higher than those from columns with biochar applications (Figure 4 a,b,c). The applications of both corn and rice biochars at doses of 1% and 2% for all pyrolysis temperatures significantly reduced the NO_3^- concentrations leached from the columns ($P<0.05$) (Figure 4 a,b,c; Table 2). Compared to the control applications, especially the rice husk biochar applications at 2% doses led to a significant decrease in NO_3^- concentrations leached from the columns. While the total NO_3^- concentration leached from the control columns was 149.23 mg L^{-1}, this value decreased to 72.49 mg L^{-1} (51% reduction) in the RH300 BC2 dose, 67.37 mg L^{-1} (55% reduction) in the RH400 BC2 application, and 72.72 mg L^{-1} (51% reduction) in the RH500 BC2 dose (Table 2).

Control (mg L^{-1})	1. Week	2. Week	3. Week	4. Week	5. Week	6. Week
	3.88	10.96	17.08	8.52	7.48	5.36

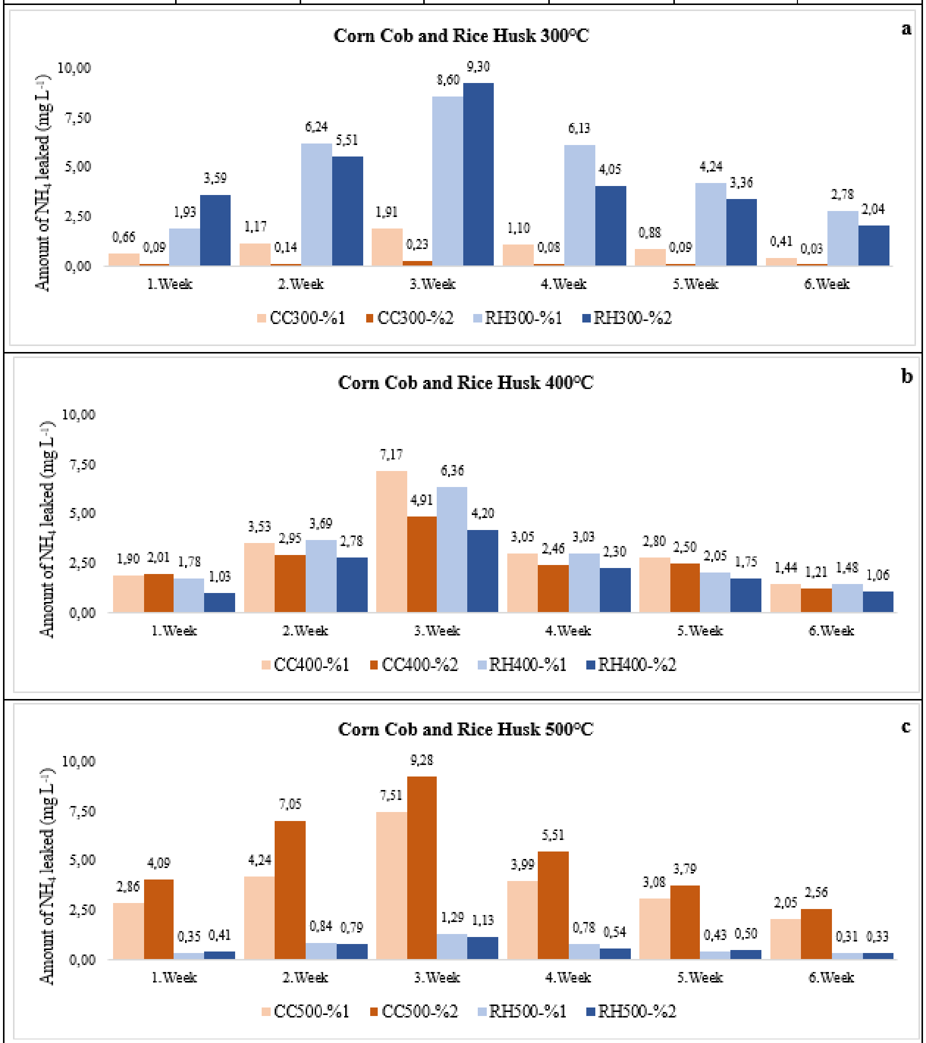

Figure 3. Weekly NH_4^+ change in leaking water after RH and CC biochar application.

A similar situation was observed for the corn cob biochar at 2% dose for pyrolysis temperatures of 400 (60% reduction) and 500 (59% reduction) (Figure 4 a,b,c; Table 2). The results showed that both corn and rice biochars at 2% dose (except for CC300) significantly reduced NO_3^- leaching at all temperatures. It has been reported that applications of acacia tree biochar obtained at different pyrolysis temperatures (300, 400, and 500°C) significantly reduced the leached NO_3^- concentration from the soil, with lower concentrations from biochar applications obtained at pyrolysis temperatures of 400 and 500°C compared to those obtained at 300°C (Uzoma et al., 2011). It has been stated that the properties of biochar change significantly depending on the pyrolysis temperature (Tepecik et al., 2022). Tomczyk et al. (2020) reported that with increasing pyrolysis temperature of the biomass, the amount of volatile organic compounds volatilized increased parallelly with the increase in the surface area of biochar. It has been reported that the application of biochar to sandy soils increases the cation exchange capacity of the soil and therefore the sorption capacity, thus reducing the leaching of nutrient elements such as NO_3^- and NH_4^+ (Lv et al., 2021). When evaluating the results obtained from the study, it can be concluded that rice husk and corn cob biochars produced at pyrolysis temperatures of 400 and 500°C are more effective against NO_3^- leaching. This effectiveness can be attributed to the higher specific surface areas of both materials at 400 and 500 degrees compared to 300°C (Table 1).

When evaluating the results in terms of weekly leached NO_3^- concentrations, similar to NH_4^+ concentrations, it was observed that the NO_3^- concentrations leached in the 3rd week were higher than other weeks for the control, rice husk, and corn cob biochar applications at all doses and temperatures (Figure 4 a,b,c). However, in the 1st, 5th, and 6th weeks, the NO_3^- concentrations leached from the columns were lower (Figure 4 a,b,c).

Günal et al. (2017) investigated the effects of tomato harvest residues-derived biochar produced at 500°C and applied at different doses (Control, 1%, 3%, and 6%) on leached NO_3^- and NH_4^+ concentrations from a loamy soil. The researchers reported that more NO_3^- was leached in control applications compared to biochar applications, and the amount of leached nitrate increased rapidly after the second leaching (especially in the 2nd and 3rd leaching), while the NO_3^- concentration remained constant in subsequent leachings (4th and 5th).

Control (mg L^{-1})	1. Week	2. Week	3. Week	4. Week	5. Week	6. Week
	10.15	36.84	43.47	21.72	21.38	15.67

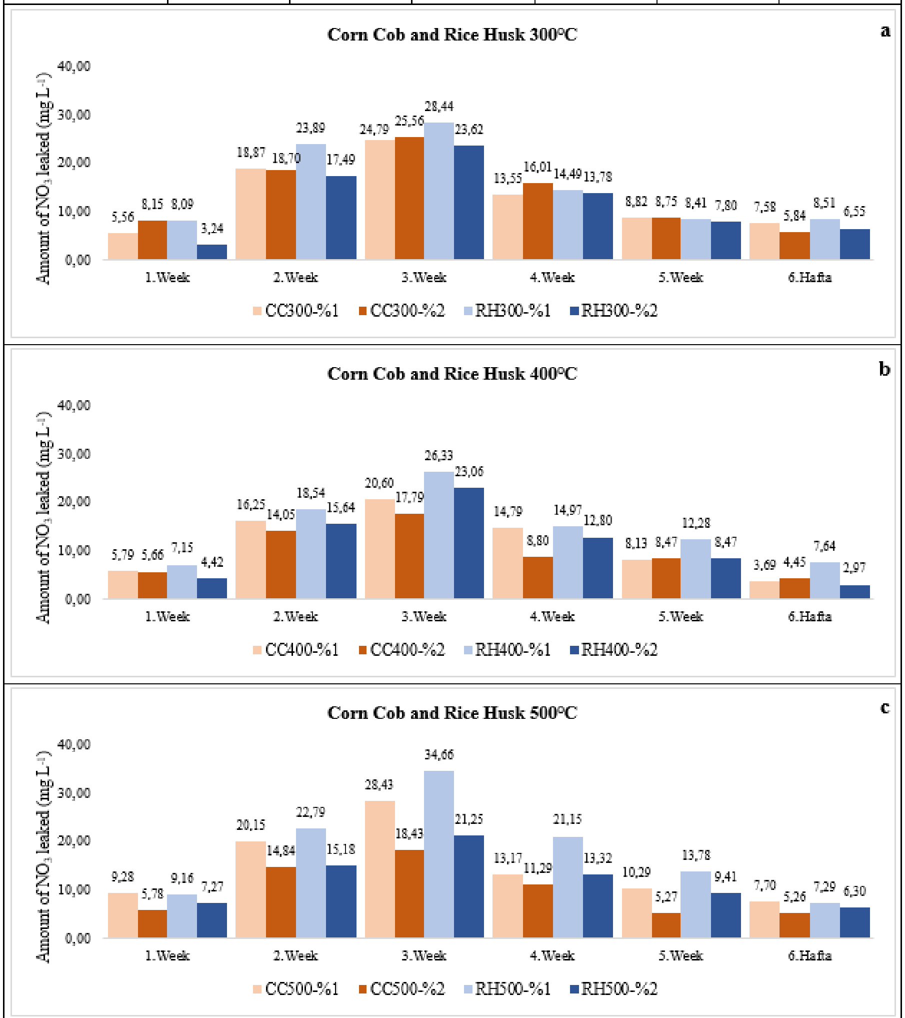

Figure 4. Weekly NH_3^- change in leaking water after RH and CC biochar application.

Bodur (2016) reported a significant reduction in leached NO_3^- concentrations from columns with the application of rice husk biochar at different doses (2.5%, 5%, and 10%) compared to the control application. The researcher detected NO_3^- concentrations of 11.3 mg L^{-1} and 3.7 mg L^{-1} in leachate from the 1st week with 2.5% and 5% biochar doses, respectively; however, no nitrate was detected in leachates from other weeks and with a 10% biochar dose.

4. Conclusion

The conversion of corn and rice crop residues, which are extensively produced in our country every year, into biochar and their application to a soil with loamy sand texture has been shown to retain a significant portion of NH_4^+ and NO_3^- in the soil. The variations in leached NH_4^+ concentrations from columns with the application of corn and rice biochars (except for BC2) at all pyrolysis temperatures and doses (1% and 2%) were statistically significant ($P<0.05$). The application of rice husk biochar resulted in a reduction of NH_4^+ concentrations by 92.5% in RH500 BC1 dose and 93% in RH500 BC2 dose.

Similarly, the application of corn and rice biochars at all pyrolysis temperatures and doses (1% and 2%) resulted in a statistically significant ($P<0.05$) reduction in leached NO_3^- concentrations from the columns. Particularly, the effectiveness of rice husk and corn cob biochars at 2% application level in reducing NO_3^- leaching was notable. Another significant finding from the study is that biochars with higher pyrolysis temperatures were more effective in reducing NO_3^- leaching. These results suggest that biochar applications can significantly reduce the leaching of nitrogen in both NO_3^- and NH_4^+ forms in agricultural soils. The substantial reduction in nitrogen leaching through biochar applications is important for preventing groundwater contamination and enhancing plant uptake of applied nitrogen fertilizers.

Author Contributions

The percentage of the author(s) contributions is presented below. All authors reviewed and approved the final version of the manuscript.

	C.Ç.G.	H.E.
C	50	50
D	70	30
S		100
DCP	80	20
DAI	40	60
L	50	50
W	30	70
CR	20	80
SR	20	80
PM	50	50
FA	50	50

C=Concept, D= design, S= supervision, DCP= data collection and/or processing, DAI= data analysis and/or interpretation, L= literature search, W= writing, CR= critical review, SR= submission and revision, PM= project management, FA= funding acquisition.

Conflict of Interest

The authors declared that there is no conflict of interest.

Ethical Consideration

Ethics committee approval was not required for this study because of there was no study on animals or humans.

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