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CHARACTERIZATION OF DIFFERENT TYPES OF BIOCHARS AND THEIR MODIFIED FORMS

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SUMMARY

The objective of this study was to synthesize, characterize, and modify rice and wheat straw biochar for various parameters (pH, EC, organic carbon and organic matter, N, P, and K). In this experiment, rice straw biochar (RSB) and wheat straw biochar (WSB) production applied temperature at 300 °C. These biochars' chemical modification with hydrochloric acid (HCl) and sulfuric acid (H_2SO_4) resulted in four variants: HCl-modified rice straw biochar (MRSB-1); H_2SO_4-modified rice straw biochar (MRSB-2); HCl-modified wheat straw biochar (MWSB-1); and H_2SO_4-modified wheat straw biochar (MWSB-2). Comprehensive characterization revealed that rice and wheat straw biochar, when modified with HCl and H_2SO_4 changed the values of pH (7.6 to 5.4), EC (1.51 to 0.28), organic carbon (53.65 to 48.25), organic matter (92.28 to 82.99), N (1.68 to 0.84), P (2.66 to 1.54), and K (3.85 to 1.98). However, the impact of H_2SO_4-modified biochars remained more prominent than the others.

Keywords: Biochar, rice straw, wheat straw, modified, H_2SO_4, HCl

Key findings: Modifications of biochars with acids proved fruitful in all characteristics, but the use of H_2SO_4, when assessed, emerged superior to HCl.

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INTRODUCTION

Addressing the challenges posed by saline-sodic soils initiated the development of several remediation strategies, like using chemical amendments, such as gypsum, thereby improving soil structure and permeability (Qadir *et al*., 2007). However, these methods often require large quantities of gypsum and rely on sufficient irrigation to leach out the displaced sodium, making them less effective in water-scarce regions (Minhas and Gupta, 2019). Organic amendments, such as compost and manure, also sought to enhance the soil organic matter, improve microbial activity, and promote better soil aggregation (Tejada *et al*., 2006).

Biochar, a carbon-rich material produced by pyrolysis of organic biomass in low-oxygen environments, has surfaced as a potential soil amendment for improving soil health and enhancing crop productivity (Verheijen *et al*., 2010; Lehmann and Joseph, 2015). The characteristics of biochar, including high surface area, porosity, and cation exchange capacity (CEC), are largely dependent on the type of feedstock used and the pyrolysis conditions, including temperature and duration (Mukherjee *et al*., 2021). The porous structure and wide surface area of biochar offer ample sites for water retention and nutrient absorption, making it highly beneficial for soil enhancement. Its high CEC allows it to retain essential nutrients, reducing leaching and enhancing soil fertility over time (Spokas *et al*., 2012). Additionally, biochars' alkaline nature can help neutralize soil acidity, making them a versatile amendment for various soil types (Abel *et al*., 2013).

Production of biochar entails thermal breakdown of organic materials in low-oxygen environments, a process referred to as pyrolysis. Pyrolysis temperature, which typically ranges from 300 °C to 700 °C has a substantial impact on the chemical and physical characteristics of the resulting biochar. Higher temperatures typically raise carbon (C) content and lower yield while improving stability and adsorption capacity of biochar (Issaka *et al*., 2022).

Acid modification of biochar is one such approach that has shown promising results. By treating biochar with acids like sulfuric, nitric, or phosphoric, its surface properties can undergo significant alteration, leading to increased surface area, enhanced pore structure, and improved nutrient adsorption capacities (Liang *et al*., 2022). This treatment boosts the number of functional groups on the surface of biochar, including carboxyl, hydroxyl, and sulfonic groups, which can improve cation exchange capacity and nutrient retention (Yang *et al*., 2019). Acid-modified biochar has demonstrated an ability to improve soil pH and nutrient availability and decrease sodium adsorption in saline-sodic soils, making it an exceptionally effective amendment (Yuan *et al*., 2021). This study's design sought to accomplish the objective of synthesis, characterization, and modification of rice and wheat straw biochar for various parameters, like pH, electrical conductivity (EC), organic carbon, organic matter, nitrogen (N), phosphorus (P), and potassium (K).

MATERIALS AND METHODS

The study commenced at the University of Sargodha, with an arid to semi-arid climate zone. This study comprised the synthesis, characterization, and modification of rice and wheat straw biochar with HCl (hydrochloric acid) and H_2SO_4 (sulfuric acid) at two rates.

Production of biochar

Biochar synthesis from agricultural waste comprised rice husks and wheat straws. The rice straw biochar (RSB) and wheat straw biochar (WSB) production by pyrolysis had the relevant feedstock at a temperature of 300 °C with a residence time of one hour.

Nature of study

This laboratory-based study focused on synthesizing, characterizing, and modifying rice and wheat straw biochars. The experiment involved producing biochar from rice and wheat straw through controlled pyrolysis and chemical modification using acid treatments.

Characterization of biochar

The final product of pyrolysis, as characterized by different parameters, included measuring pH, EC, organic carbon, organic matter, N, P, and K in the laboratory. For all these determinations, distilled water served as a solvent to make suspensions and extractions for different types of biochars.

Modification of biochar

After preparation, both kinds of biochar underwent modification using HCl and H_2SO_4, preparing a 1% solution of HCl and H_2SO_4 accordingly. Both types of biochar, when modified, used a 1:5 ratio of these solutions. Biochar modified with acids sustained drying in open air for final use.

Treatment plan

The experiment comprised six treatments: a) RSB = rice straw biochar; b) MRSB-1 = HCl-modified rice straw biochar; c) MRSB-2 = H_2SO_4-modified rice straw biochar; d) WSB = wheat straw biochar; e) MWSB-1 = HCl-modified wheat straw biochar; and f) MWSB-2 = H_2SO_4-modified wheat straw biochar.

Measurements

The pH, EC, organic C and OM, N, P, and K of different types of biochars entailed measurements and analysis using standard analytical techniques. Measuring pH, EC, organic C, P, and K applied the methods 21a, 4b, 24, 61, and 11a of the US. Salinity Laboratory Staff (1969), respectively. The determined organic carbon succeeded in multiplying by 1.72 to get the organic matter. For nitrogen determination, samples' digestion took place, with the digested samples subsequently filtered to obtain a clear filtrate (Wolf, 1982). Next, transferring 10 ml of the aliquot to a Kjeldahl tube continued. After distillation, an aliquot in the receiver flask reached titration with 0.01 N H_2SO_4 until the solution turned pink. Recording the initial and final titration readings helped determine the nitrogen content (Jackson, 1962).

RESULTS

The pH of different biochars

In this study, biochar produced from rice straw and wheat straw, with initial pH values of 7.6 and 7.1, respectively, experienced significant pH reduction after treatment with hydrochloric acid (HCl) and sulfuric acid (H_2SO_4). The results show that H_2SO_4 caused a greater reduction in pH than HCl (Figure 1). This can be due to the chemical nature of the acids used, where H_2SO_4, being a diprotic acid, donates two protons (H^+ ions) per molecule, compared with HCl, which is monoprotic, donating only one proton. This higher availability of protons in H_2SO_4 enhances its acidifying capacity, leading to a greater reduction in pH. Additionally, sulfuric acid introduces sulfate ions (SO_4^{2-}) into the biochar, which may further enhance the acidification process by binding to basic cations and promoting more H^+ release. The structural changes induced by sulfuric acid are also more pronounced, as it can oxidize the biochar surface, improving the concentration of acidic functional groups, such as carboxyl groups. The variation in pH reduction between rice straw biochar (1.45 with HCl, 1.92 with H_2SO_4) and wheat straw biochar (1.1 with HCl, 1.7 with H_2SO_4) may refer to differences in the feedstock properties. The rice straw biochar contains more mineral salts that provide less buffering capacity, resulting in a slightly higher final pH than the wheat straw biochar.

The EC of different biochars

Figure 2 shows the EC of six types of biochar, measured in decisiemens per meter (dSm^{-1}). In this study, the measured EC of untreated rice straw biochar was at 1.51 dSm^{-1}, which was significantly higher than the wheat straw biochar, with an EC of 0.75 dSm^{-1}. This

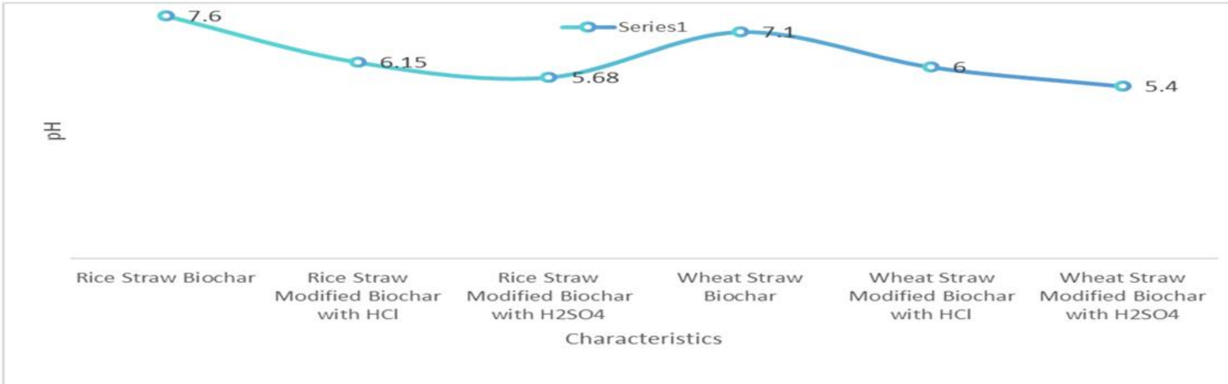

Figure 1: pH of the different types of biochars.

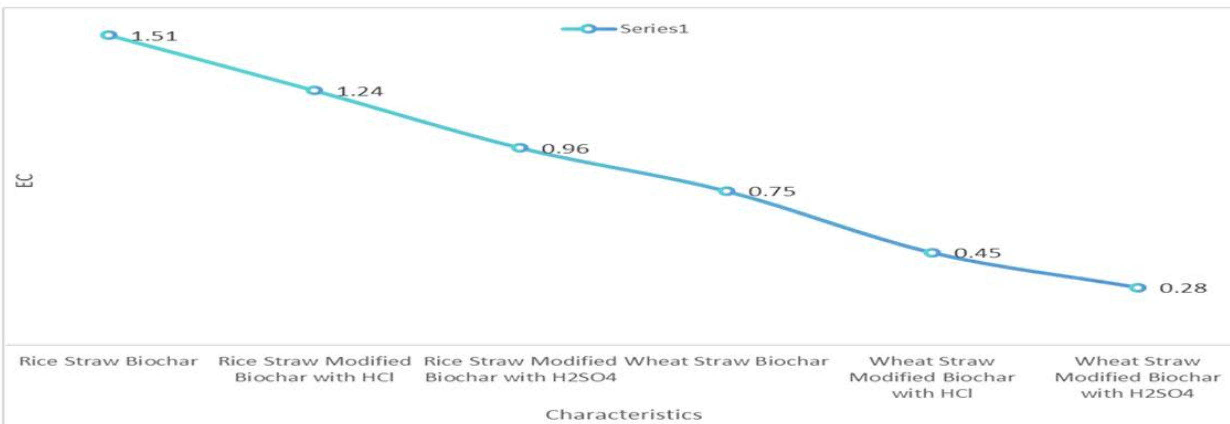

Figure 2: EC of the different types of biochars.

difference highlights the greater concentration of soluble ions, such as potassium (K^+), calcium (Ca^{2+}), and magnesium (Mg^{2+}), present in the rice straw biochar, making it potentially more influential in increasing soil salinity. Acid modification of biochar has a direct impact on its EC values. Treating rice straw biochar with HCl reduces its EC to 1.24 dSm^{-1}, indicating a slight reduction in the number of water-soluble ions. The mechanism behind this involves the exchange of H^+ ions from the acid with soluble cations in the biochar. HCl, being a monoprotic acid, may not be as effective in completely removing these ions, which explains the relatively modest decline in EC after HCl treatment. In contrast, treatment with H_2SO_4 lowers the EC of rice straw biochar more significantly to 0.96 dSm^{-1}. This more substantial reduction in EC is likely because H_2SO_4, being a diprotic acid, releases more protons into the biochar matrix, facilitating a greater exchange of cations and extraction of soluble ions. Wheat straw biochar, which initially has a lower EC (0.75 dSm^{-1}), gave a similar pattern of decline upon acid treatment. After modification with HCl, its EC drops to 0.45 dSm^{-1}, suggesting a moderate reduction in soluble ions. However, the most significant reduction in EC occurs when wheat straw biochar obtained the sulfuric acid treatment, resulting in an EC value of 0.28 dSm^{-1}. This considerable decrease is consistent with the findings for rice straw biochar and supports the idea that H_2SO_4 is more efficient in reducing the concentration of soluble ions than HCl.

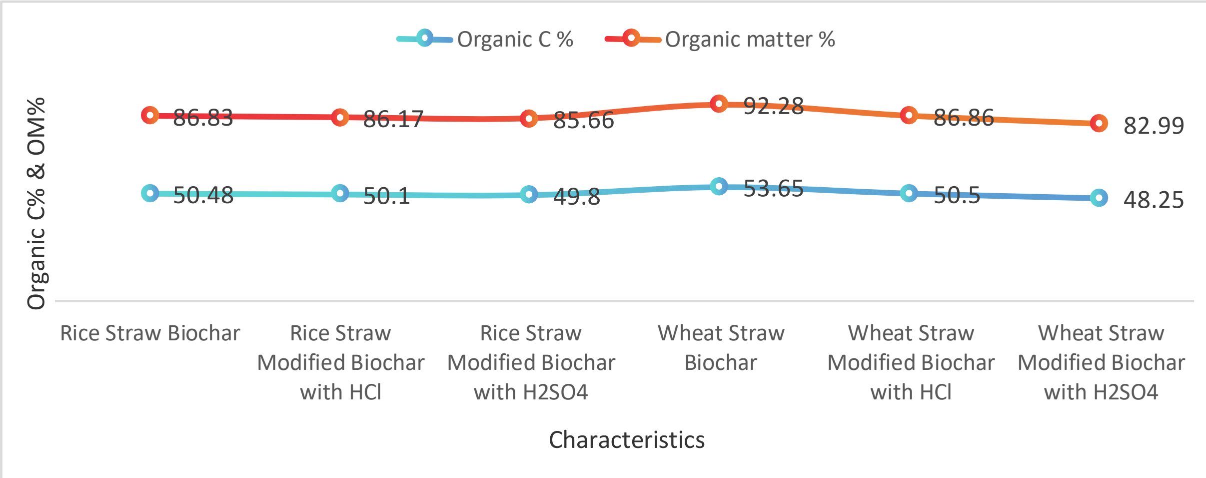

Figure 3: Organic carbon and organic matter in the different types of biochars.

Organic carbon and organic matter in biochars

Figure 3 presents the levels of organic carbon content (organic C %) and organic matter (OM %) in different types of biochar. The organic carbon content among different biochar types remains relatively stable, with RSB exhibiting slightly lower organic carbon levels (50.48%) than WSB (53.65%). The variation in organic carbon content between these two feedstocks can be ascribable to differences in the lignin, cellulose, and hemicellulose content of the biomass used to produce the biochar. Wheat straw typically contains a higher amount of lignin, which is more resistant to decomposition during pyrolysis, leading to higher organic carbon retention.

Acid modification has varied effects on the organic carbon content of rice straw and wheat straw biochar. For RSB, the organic carbon content remains fairly constant after HCl treatment (50.1%), but it decreases slightly (49.8%) following H_2SO_4 treatment. This slight decline in organic carbon after sulfuric acid treatment can be due to the more aggressive nature of H_2SO_4, which is capable of oxidizing some carbon compounds in the biochar. This oxidation can result in the loss of certain volatile organic compounds, thereby decreasing the organic carbon content. Conversely, HCl, being a weaker acid, induces less oxidation and structural modification of the biochar; hence, the minimal change in organic carbon levels. The WSB shows a more pronounced decline in organic carbon content after acid treatment, with reductions of 3.15% for HCl and 5.40% for H_2SO_4. The organic matter (OM) content follows a similar trend, with RSB exhibiting a significant reduction from 86.83% in its unaltered state to 86.17% after HCl treatment and 85.66% after H_2SO_4 treatment. Similarly, WSB experienced a more substantial drop in OM, from 92.28% to 86.86% with HCl and further to 82.99% with H_2SO_4.

Nitrogen in biochars

Figure 4 shows the nitrogen (N %) content in original and modified types of biochar. In this study, the nitrogen concentration in RSB appeared to be the highest at 1.68%, followed by the biochar modified with HCl at 1.60% and H_2SO_4 at 1.52%. In contrast, WSB exhibited a lower nitrogen content of 0.97%. The reduction in nitrogen content following acid modification is consistent with acidic treatments tending to remove or degrade nitrogenous compounds in biochar. When biochar sustains HCl or H_2SO_4, the acid interacts with nitrogen-containing functional groups, such as amines, amides, and nitriles, leading to the removal of volatile nitrogen compounds. Sulfuric acid, being a stronger acid than HCl, induces a greater nitrogen content reduction due to its more aggressive oxidation of nitrogenous compounds and better ability to leach soluble nitrogen.

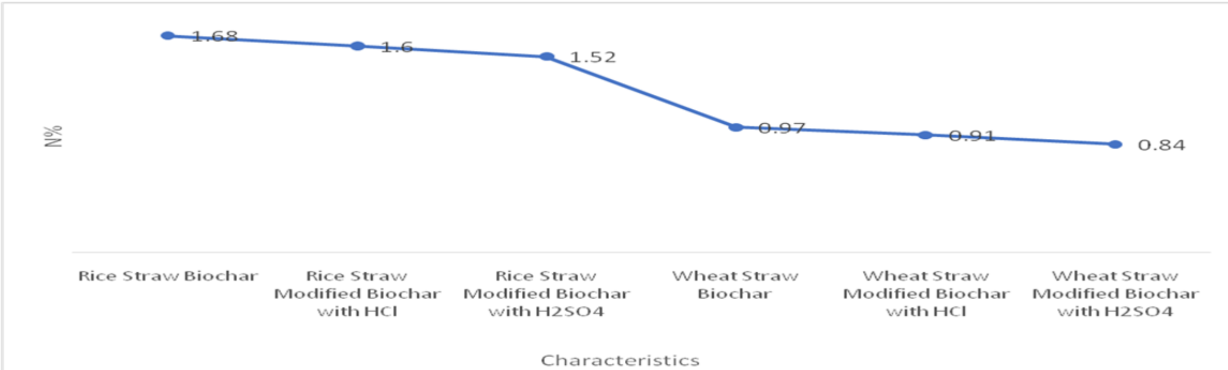

Figure 4: Nitrogen in the different types of biochars.

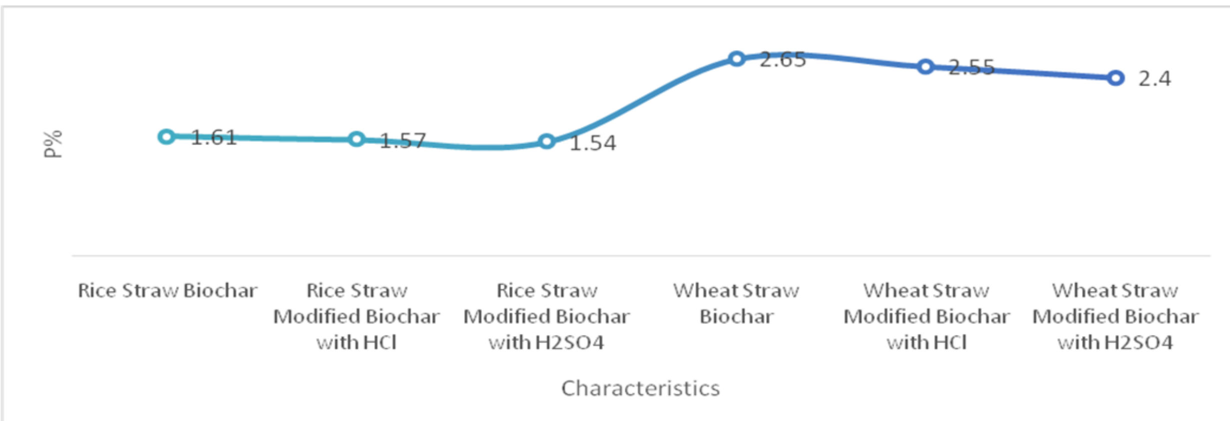

Figure 5: Phosphorus in the different types of Biochars.

The WSB has a lower initial nitrogen content (0.97%), which decreases even further with acid treatment. The lower nitrogen content in WSB than RSB can be because of the lesser nitrogen uptake in wheat crops, as well as the different composition of the two feedstocks. Moreover, the WSB's larger reduction in nitrogen content after acid treatment suggests that the nitrogenous compounds in WSB are more susceptible to removal during acid modification. This is possibly because they are less stable or even more readily leachable than those in RSB.

Phosphorus in biochars

Figure 5 depicts the phosphorus (P) percentages of various types of biochar and modified biochar employed to remediate saline-sodic soils. The results of this study indicate that WSB typically contains a higher phosphorus content than RSB. The WSB biochar exhibits the highest phosphorus content at 2.65%, followed by WSB treated with HCl (2.55%) and H_2SO_4 (2.4%). In contrast, the phosphorus content in unmodified RSB is 1.61%, which is better than its acid-modified forms (1.57% for HCl-treated and 1.54% for H_2SO_4-treated biochar). These results suggest that unmodified biochar, particularly from wheat straw, may be more effective in enriching soils with phosphorus due to its naturally superior phosphorus content.

The reduction in P content after acid treatment, observed in both wheat and rice straw biochars, is likely due to the

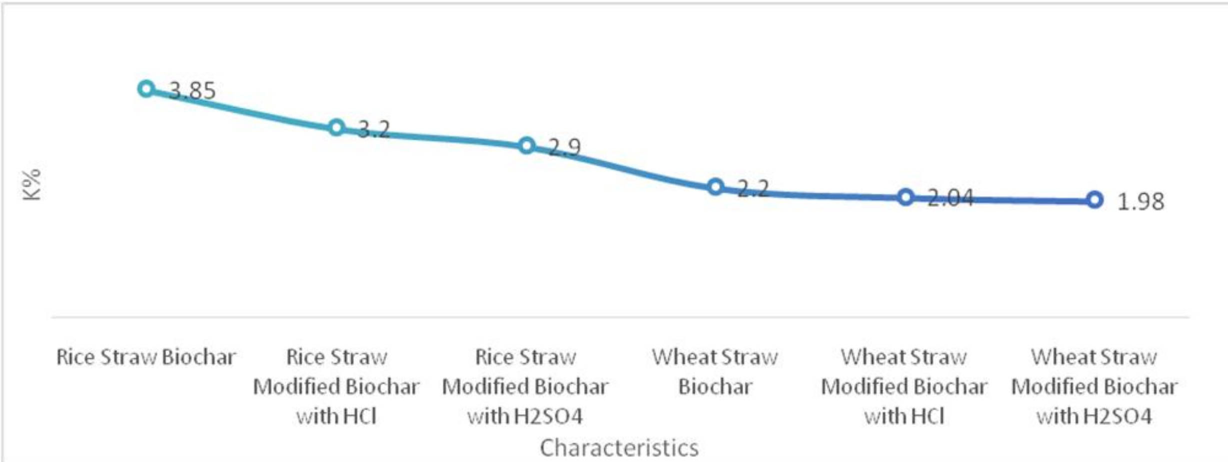

Figure 6: Potassium in the different types of biochars.

solubilization and leaching of phosphorus compounds during the acid modification process. When biochar gains treatment with hydrochloric acid or sulfuric acid, some P conversion into soluble forms occurs that can be washed away, thereby lowering the overall phosphorus content of the biochar.

Potassium in biochars

Figure 6 displays the potassium concentration (K %) in six types of biochar and modified biochar obtained from rice and wheat straws. The data indicated the unmodified RSB has the supreme K concentration at 3.85%, while wheat WSB contains a lower K concentration of 2.2%. Acid modification leads to a reduction in K levels for both types of biochar. After treatment with hydrochloric acid, K of RSB drops to 3.2%, and with sulfuric acid, it decreases further to 2.9%. Similarly, the K content of WSB decreases to 2.04% with HCl treatment and to 1.98% with H_2SO_4 modification. This demonstrates that the acid treatment, especially with sulfuric acid, generally reduces the K content of biochar, with such acid having a stronger leaching effect on K than HCl.

The greater initial potassium content in RSB, compared with WSB, can be ascribable to the variations in the mineral composition of the two feedstocks. Rice straw generally accumulates more K than wheat straw, as potassium is critical for rice plants in regulating water use and enzyme activation, crucial for their growth under paddy field conditions. This higher K content in rice straw remains during pyrolysis before incorporation into the biochar, making rice straw biochar a potentially more effective amendment for increasing K availability in soils.

DISCUSSION

Soil pH is an important parameter that controls all other chemical properties and the availability of nutrients to plants. This substantial reduction in biochar pH through acid treatment has critical implications for its use as a soil amendment, particularly in alkaline or calcareous soils. Acid-modified biochar can help neutralize soil alkalinity, improve nutrient availability, and enhance the bioavailability of micronutrients like iron and zinc. The stronger acidification effect of H_2SO_4-modified biochar could make it more effective in highly alkaline soils. These findings are consistent with other studies, such as Xu *et al*. (2012) and Tag *et al*. (2016), highlighting that sulfuric acid treatment results in more acidic biochar and improved functionality for soil amendments. The change in pH levels indicates a reduction in soil sodicity, as higher pH values generally have an association with increased availability of essential nutrients and improved soil structure, which can foster better root growth and enhance crop yield (Lehmann and Joseph, 2015). This variation can connect to the chemical composition and surface characteristics of different biochar types, which influence their ability to interact with soil constituents and release alkaline ions into the soil solution (Biederman and Harpole, 2013).

Electrical conductivity is a key factor reflecting the concentration of soluble ions in a solution, significantly affecting soil health, plant growth, and the productivity of agricultural systems (Amezketa, 1999). Use of biochar, particularly those modified with acids, can mitigate these effects by facilitating the leaching of excess salts and improving overall soil quality (Yuan *et al.,* 2019). Acid treatments can also alter the chemical structure of biochar, potentially increasing its alkalinity and changing nutrient availability, directly influencing EC levels (Kloss *et al.,* 2012). Studies by Hussain (2023) reinforced the potential of biochar to improve soil quality and lower salinity levels, emphasizing its role in sustainable agricultural practices. The observed decline in EC values across both types of biochar (rice straw and wheat straw) after acid modification indicates that the choice of feedstock and the type of acid used for modification significantly affect the biochar's ion content and, consequently, its EC. The reduction in EC after acid treatment has practical implications for biochar's use as and in soil amendment, especially in saline or sodic soils. Biochar with lower EC is less likely to exacerbate soil salinity, making acid-modified biochar, particularly H_2SO_4-treated biochar, more suitable for improving soil structure and fertility without increasing salt content. These findings align with research by Novak *et al*. (2009), which stated that acid-modified biochar can reduce the risk of soil salinization by lowering its soluble ion content, thereby improving its suitability for soil remediation efforts. Sulfuric acid treatment is more effective than hydrochloric acid in reducing the concentration of soluble ions in biochar, as evidenced by the lower EC values.

This capacity of biochar to retain nutrients and organic compounds has been documented by Gul *et al.* (2015), who found biochar amendments can considerably increase organic C in soils by promoting the stabilization of organic matter. Biochars with higher surface area and functional groups can improve the sorption of organic compounds, thus increasing the organic matter content in the soil (Kammann *et al.*, 2018). The decrease in organic matter content is indicative of the acids' ability to extract or degrade certain organic components of the biochar, particularly soluble organic fractions or volatile organic compounds. The H_2SO_4's stronger oxidizing capacity than HCl likely leads to a greater breakdown of organic material, as it facilitates ion exchange and oxidizes organic molecules, contributing to a larger decline in OM. The presence of a high proportion of stable organic carbon in biochar also contributes to long-term carbon sequestration, which is vital for mitigating climate change. These findings agree with research from Lehmann *et al*. (2015), which highlights the role of biochar in improving soil properties, particularly in challenging soil environments. Moreover, Novak *et al*. (2009) demonstrated that biochar with high organic carbon content can significantly improve soil fertility by enhancing soil organic matter levels and improving nutrient retention. Thus, both unmodified and acid-modified biochar have valuable applications for soil remediation, with the choice of treatment depending on the specific needs of the soil being treated.

The increased N content in rice straw biochar makes it a more effective amendment for enhancing nitrogen availability in saline-sodic soils, which are often deficient in essential nutrients like nitrogen. The reduction in nitrogen content after acid treatment can make biochar more porous and reactive, enhancing its capacity to adsorb nutrients and other soil amendments. Research conducted by Domingues *et al*. (2017) and Lehmann *et al*. (2015) has shown that biochar, even with reduced nitrogen levels, can enhance soil fertility by boosting cation exchange capacity (CEC), raising water retention, and stabilizing soil organic matter. In conclusion, the nitrogen content of biochar incurs effects from the type of feedstock used and the modification treatment applied. Acid treatments, such as those with HCl or H_2SO_4, can alter the chemical structure of biochar, enhancing its capacity to retain nutrients, including nitrogen (Kloss *et al.,* 2012). The presence of more functional groups on the biochar surface can facilitate better adsorption of ammonium (NH_4^+) and nitrate (NO_3^-) ions, thereby increasing their availability in the leachate (DeLuca *et al.,* 2006).

Applying biochar can alter the soil pH, which, in turn, affects P solubility. Studies by Zhang *et al.* (2022) demonstrated that biochar can alter soil pH, thereby reducing the formation of insoluble phosphorus compounds and enhancing phosphorus availability for plant uptake. The ability of biochar to stabilize nutrients, including phosphorus, has also received highlights from Bai *et al.* (2021), who found that biochar effectively improves nutrient cycling and retention in agricultural soils. The organic fraction is typically more susceptible to degradation during pyrolysis and acid treatment, while the inorganic fraction, such as phosphates, could remain more effectively in unmodified biochar. Despite the reduction in phosphorus content after acid treatment, biochar still retains significant levels of P, which can contribute to improving soil fertility. Research by Glaser *et al*. (2002) supports the idea that biochar can increase P availability in soils by enhancing phosphorus retention and reducing phosphorus leaching, especially in soils prone to nutrient loss. Additionally, biochar's porous structure and large surface area can help retain P in soils, reducing its mobility and preventing it from becoming unavailable to plants. Acid-modified biochar, although containing less P, may still be valuable for improving soil fertility, particularly when used in combination with other soil amendments or fertilizers.

Potassium in biochar is typically present in soluble forms, such as potassium carbonate (K_2CO_3) or potassium hydroxide (KOH), which are easily leachable under acidic conditions. When biochar bears treatment with acids like HCl or H_2SO_4, the acidic environment promotes the dissolution of these potassium compounds. Sulfuric acid interacts with the potassium in biochar to form this stable salt, which may precipitate out or become unavailable for plant uptake, leading to a greater reduction in K content in biochar modified with H_2SO_4. In contrast, HCl treatment primarily dissolves K into soluble chloride forms, which are more easily leached but less likely to precipitate as an insoluble compound. The presence of K in biochar can improve soil fertility, particularly in K-deficient soils, by providing a slow-release source of this nutrient. Research by Rajkovich *et al*. (2012) and Mukherjee and Zimmerman (2013) suggested that biochar can enhance the availability of potassium in soils and reduce the risk of K leaching, making it an effective amendment for improving soil nutrient retention and crop productivity. Research by Méndez *et al.* (2021) highlights that modified biochars have superior K retention and release capacities due to these enhanced properties. Biochar improves the retention of K in soils through its high CEC, which helps prevent leaching of K ions, making it more available to plants (Yang *et al.,* 2019).

CONCLUSIONS

Rice and wheat straw biochars underwent modifications with HCl and H_2SO_4, according to the treatment plan. Rice and wheat straw biochars, when modified with HCl and H_2SO_4 decreased the values of pH and EC. They also increased the contents of soil organic carbon, organic matter, N, P, and K. The decrease and increase in values of all these parameters remained statistically significant. However, the impact of H_2SO_4 modified biochars remained more prominent than with all the others. The study concluded that the presence of sulfate ions played its role significantly in increasing biochar efficiency.

RESEARCH GAPS, LIMITATION, AND FUTURE ASPECTS

Treatment of any biochar prepared from various feedstocks with any type of acid to enhance its efficiency still has a big research gap. This research gap has limited the practical outcome or beneficial efficiency of biochar.

Hence, the study recommends that to achieve the best performance of biochar application to salt-affected soils, it needs to be treated with various types of acids.

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