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Synthesis and Characterization of Biochar–Iron Complex Derived from Sugarcane Bagasse for Iron Nutrient Retention

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Abstract Iron deficiency in alkaline soils poses a significant challenge to crop productivity due to limited iron availability and poor nutrient retention. Conventional fertilizers often fail to address this effectively and sustainably. This research was geared towards utilizing biochar as a chelating agent in the synthesis of biochar-iron complexes. Biochar–iron complexes were synthesized via pyrolysis, where biochar derived from sugarcane bagasse was treated with $FeCl_3$ at different concentrations (0.25 M, 0.5 M and 0.75 M). FT-IR analysis confirmed the successful incorporation of iron into the biochar matrix across all $FeCl_3$ concentrations used. There were noticeable peak shifts in hydroxyl (–OH) and carboxyl (–COOH) functional groups, indicating strong chelation between iron ions and the biochar surface. The appearance of new bands at 671.23 cm^{-1} and 507.28 cm^{-1} emanating from Fe–O vibrations upon complexation is a clear indication of successful incorporation of iron into the biochar structure. XRF analysis quantitatively demonstrated enhanced iron retention. The study will therefore form a base for applying the biochar-iron complex synthesized as an alternative iron-rich fertilizer to enhance iron uptake in the spinach crop.

Keywords Biochar, Characterization, Complexation, Nutrient Retention, Synthesis

1. Introduction

Iron deficiency is a critical challenge in many agricultural systems, particularly in alkaline soils where iron becomes insoluble and unavailable to plants [1] [2]. Conventional iron fertilizers often suffer from rapid precipitation and immobilization, limiting their effectiveness. Biochar, produced via the pyrolysis of organic waste materials, has emerged as a promising soil amendment due to its high porosity and abundance of functional groups that facilitate nutrient retention and slow release [3] [4]. Sugarcane bagasse, an abundant agricultural by-product, is an ideal feedstock for biochar production. Converting bagasse into biochar not only reduces waste but also creates a value-added product that can enhance soil fertility. Despite these advantages, limited studies have explored the synthesis of biochar–iron complexes using sugarcane bagasse and their potential for improved nutrient retention. A review of existing studies shows that the majority of research has focused on the use of Fe-modified bagasse biochar for pollutant remediation such as phosphates, arsenic, antibiotics, pesticides [5] [6] rather than direct evaluation of its role as an iron nutrient source. The optimization of preparation methods remains another research frontier. The iron loading concentration can dramatically influence surface chemistry, pore structure, and the oxidation state of iron, all of which determine nutrient release dynamics [7]. This study addresses this gap by synthesizing and characterizing biochar–iron complexes with varying $FeCl_3$ concentrations. Iron deficiency is a prevalent issue in alkaline soils, leading to reduced crop yields, poor plant health, and diminished nutritional quality. Traditional iron fertilizers are often ineffective in such soils due to their rapid precipitation and immobilization, resulting in poor nutrient use efficiency and high environmental losses. Although biochar has emerged as a promising soil amendment, most studies have focused on its general nutrient retention and carbon sequestration benefits. A number of studies have demonstrated the potential of biochar to enhance nutrient retention through its porous structure and functional groups [8] [9] [10]. The biochar produced from biomass such as sugarcane bagasse is rich in hydroxyl (–OH), carboxyl (–COOH), and phenolic groups—functional moieties that play key roles in metal adsorption and chelation [3]. Research by [11] showed that these oxygenated groups facilitate the formation of stable complexes with iron, effectively reducing leaching in alkaline soils. Further, [4] reported that the efficiency of such complexation is significantly influenced

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by the concentration of the iron precursor.

In synthesis protocols, [12] optimized conditions for mixing and reacting biochar with $FeCl_3$, laying a strong foundation for subsequent studies aiming at enhanced iron loading. Together, these studies underscore the potential to improve iron availability in soil through strategic biochar modification, a concept that this work further investigates through detailed characterization. This study utilized biochar from sugarcane bagasse as a chelating agent to enhance iron availability and uptake in Spinacia oleracea. By investigating the complexation of iron with sugarcane bagasse–derived biochar, this study aimed to offer insights into an innovative, sustainable approach for improving iron nutrition in spinach crops grown in iron-deficient soils.

2. Materials and Methods

2.1. Biochar Preparation

Sugarcane bagasse was collected from Mtito Andei Market, washed thoroughly with distilled water, and oven-dried at 105°C for 24 hours [13]. The dried bagasse was pyrolyzed in a Thermo Scientific muffle furnace under conditions optimized from the literature [14] to produce biochar with high carbon content and well-preserved functional groups.

2.2. Preparation of Biochar–Iron Complexes

The produced biochar was sieved to ensure uniform particle size and divided into three portions. Each portion was mixed with an aqueous solution of $FeCl_3$ at concentrations of 0.25 M, 0.50 M, and 0.75 M, using weight-to-volume ratios of 1:5, 1:10, and 1:15, respectively. The mixtures were agitated at room temperature for 24 hours, filtered, washed with deionized water, and then dried at 105°C for 12 hours [12].

2.3. Characterization Techniques

2.3.1. Functional Group Analysis Using Fourier Transform Infrared Spectroscopy

FT-IR was performed on the raw biochar and each biochar–iron complex to identify functional groups. Samples were prepared as KBr pellets, and spectra were recorded over the range 4000–400 cm^{-1}. Shifts in characteristic peaks (e.g., –OH and –COOH) were used to confirm iron chelation [15].

2.3.2. X-Ray Fluorescence of Biochar–Iron Complexes

XRF analysis quantified the iron content retained in the biochar–iron complexes. Powdered samples were analyzed using an XRF spectrometer, and the iron content was reported as a percentage by weight with calculated standard deviations [16].

3. Results and Discussions

3.1. Characterization of Sugarcane Bagasse-Derived Biochar Using FT-IR

The FT-IR spectra provided key understanding into the chemical interactions between biochar and iron at varying concentrations. The absorption peaks in FT-IR analysis confirmed the presence of functional groups such as hydroxyl (–OH) and carboxyl (–COOH), which are vital for iron binding [11]. These results align with previous findings indicating that biochar's high surface functionality facilitates metal adsorption, improving its suitability for soil amendment [17]. The FT-IR spectra of the sugarcane bagasse-derived biochar revealed strong absorption bands corresponding to hydroxyl (–OH), carboxyl (–COOH), and phenolic (–PhOH) groups. These functional groups are essential for iron binding during complexation and play a role in nutrient retention, water adsorption, and buffering soil pH. Their presence demonstrates that pyrolysis preserved important surface functionalities critical for biochar's performance as a soil amendment. In terms of synthesis, this study aligns with the general methodologies reported in literature of pyrolysis of sugarcane bagasse followed by iron impregnation using $FeCl_3$ [18]. However, this study uniquely varies $FeCl_3$ concentration (0.25 M, 0.50 M, 0.75 M) to directly assess iron loading efficiency. Most other studies employ a single concentration or mass ratio optimized for adsorption performance, without systematically examining how metal salt concentration influences nutrient release profiles for agricultural uptake [18].

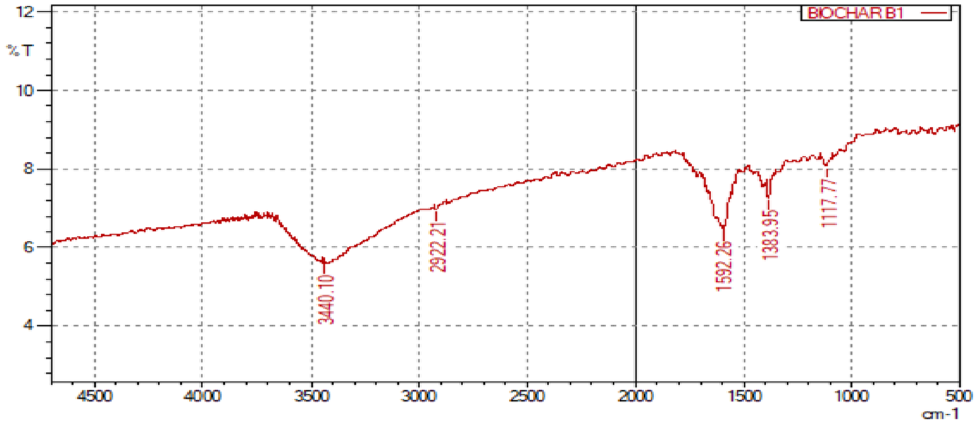

Figure 1. Characterization of Sugarcane Bagasse-Derived Biochar Using FT-IR

The FT-IR spectrum of sugarcane bagasse-derived biochar, highlights its functional groups that are critical for nutrient retention and metal chelation. The broad absorption at 3440.10 cm^{-1} corresponds to hydroxyl (–OH) stretching vibrations, primarily from residual cellulose, hemicellulose, and lignin. These hydroxyl groups enhance biochar's hydrophilicity and ability to form hydrogen bonds, improving water retention and facilitating metal adsorption. The peak at 1383.95 cm^{-1} is attributed to carboxyl (–COOH) groups, which provide negatively charged sites for binding metal ions like Fe^{3+} through electrostatic attraction or chelation. Similarly, the band near 1117.77 cm^{-1} corresponds to phenolic –OH or ether functionalities, further contributing to cation exchange capacity. The presence of these oxygen-containing groups confirms that pyrolysis preserved essential surface functionalities, which are vital for subsequent iron complexation. These observations agree with previous studies [11] [19] that report similar retention of functional groups in lignocellulosic biochars. Overall, Figure 1 validates the chemical suitability of the synthesized biochar for iron loading and soil fertility applications.

3.2. Characterization of the Biochar-Iron Complex Using FT-IR

FT-IR analysis confirmed successful iron incorporation into the biochar matrix across all $FeCl_3$ concentrations. Noticeable peak shifts in hydroxyl (–OH) and carboxyl (–COOH) functional groups indicated strong chelation between iron ions and the biochar surface. The formation of Fe–O bonds was also evident in the spectra, confirming direct chemical bonding and improved stability. These changes reflect the modification of biochar's surface chemistry without compromising its structural integrity, consistent with previous reports [4] [12].

The FT-IR spectrum of the biochar-iron complex treated with 0.25 M $FeCl_3$ shows a shift of the hydroxyl band from 3440.10 cm^{-1} (in raw biochar) to 3429.43 cm^{-1} suggests coordination between Fe^{3+} ions and –OH groups. The appearance of new bands at 671.23 cm^{-1} and 507.28 cm^{-1}, characteristic of Fe–O vibrations, confirms successful incorporation of iron into the biochar structure. Additionally, peaks at 1633.71 cm^{-1} and 1539.20 cm^{-1} correspond to aromatic C=C stretching, indicating the retention of the biochar's carbonized framework, which is critical for long-term stability. A weak band near 2358.94 cm^{-1} may indicate minor CO_2 adsorption, a common phenomenon during metal modification. Overall, the 0.25 M concentration provided efficient iron binding without over-saturation, suggesting it could be ideal for balanced iron enrichment and structural stability.

In the FT-IR spectrum of the 0.5 M Biochar-Iron Complex, a broad absorption band at 3406.29 cm^{-1} corresponds to hydroxyl (-OH) stretching, indicative of water molecules or surface hydroxyl groups (Figure 3). The peak at 2360.87 cm^{-1} suggests the presence of CO_2 or C≡C stretching vibrations, possibly linked to surface oxidation processes or adsorbed atmospheric CO_2. The peak at 1114.86 cm^{-1} corresponds to C-O stretching, likely from oxygen-containing functional groups such as phenols or carboxylates (Figure 3). These groups improve the complex's sorption capacity by providing active sites for iron coordination.

The FT-IR spectrum of the biochar-iron complex treated with 0.50 M $FeCl_3$ shows that hydroxyl peak appears at 3406.29 cm^{-1}, indicating continued strong interaction between Fe^{3+} ions and surface –OH groups. The presence of a band at 2360.87 cm^{-1} suggests increased CO_2 adsorption or surface carbonate formation, which often occurs at higher metal loading levels. Notably, a distinct band at 1114.86 cm^{-1} (C–O stretching) confirms the persistence of phenolic or ether groups, ensuring that reactive oxygen sites remain active despite modification. Compared to 0.25 M, this complex demonstrates greater iron binding, but potential pore blockage at higher loading could impact surface accessibility. These observations suggest 0.50 M is highly effective for iron enrichment while maintaining biochar functionality.

The FT-IR spectrum for the 0.75 M Biochar-Iron Complex, show key functional group interactions similar to the 0.5 M Biochar-Iron Complex, but with slight shifts in peak positions, which might indicate stronger or altered bonding at a higher iron concentration (Figure 4). In the 0.75 M Biochar-Iron Complex, the broad hydroxyl bond was at 3419.79 cm^{-1}. This peak is slightly shifted from 3406.29 cm^{-1} in the 0.5 M complex, suggesting possible changes in hydrogen bonding due to higher iron loading. The carboxyl group peak was at 1593.20 cm^{-1}, compared to the 1595.13 cm^{-1} peak in the 0.5 M complex (Figure 4). The 1118.71 cm^{-1} stretching from phenolic or ether groups, similar to the 1114.86 cm^{-1} in the 0.5 M complex, indicating that oxygen-containing functional groups remain significant in both complexes. Overall, the 0.75 M Biochar-Iron Complex retains the primary functional groups of biochar while exhibiting slight shifts that suggest enhanced interaction with iron. This could improve its metal-binding properties, making it potentially more effective for applications such as heavy metal adsorption or soil remediation.

The FT-IR spectrum of the biochar-iron complex prepared with 0.75 M $FeCl_3$, the highest precursor concentration was tested. The hydroxyl band appears at 3419.79 cm^{-1}, while the carboxyl peak is observed at 1593.20 cm^{-1}, both slightly shifted compared to lower concentrations. These shifts indicate stronger or more extensive iron coordination. Fe–O peaks remain present, confirming iron incorporation, but differences between 0.50 M and 0.75 M are minimal, suggesting that most reactive sites were already occupied at 0.50 M. The additional iron likely exists as surface precipitates rather than new coordination bonds, which could reduce porosity and limit functional performance. Therefore, while 0.75 M maximizes iron loading, the practical benefits beyond 0.50 M appear marginal, highlighting the importance of optimizing precursor concentration for cost-effectiveness and soil application performance.

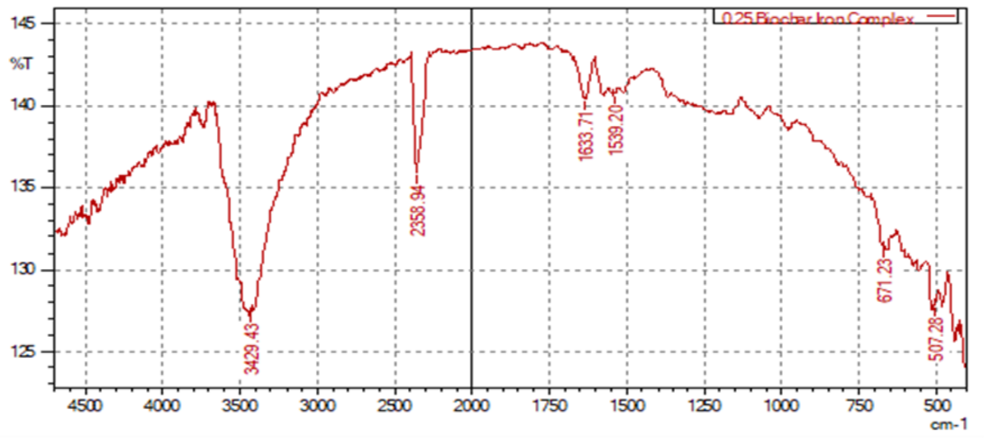

Figure 2. Characterization of Biochar-Iron Complex Using FT-IR (0.25 M Fecl3 (1:5w/V))

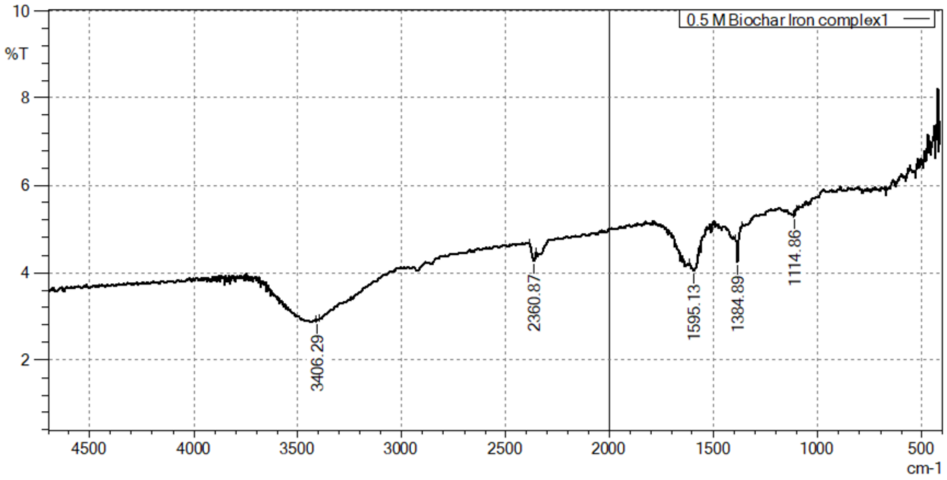

Figure 3. Characterization of Biochar-Iron Complex Using FT-IR (0.5 M Fecl3 (1:10w/V))

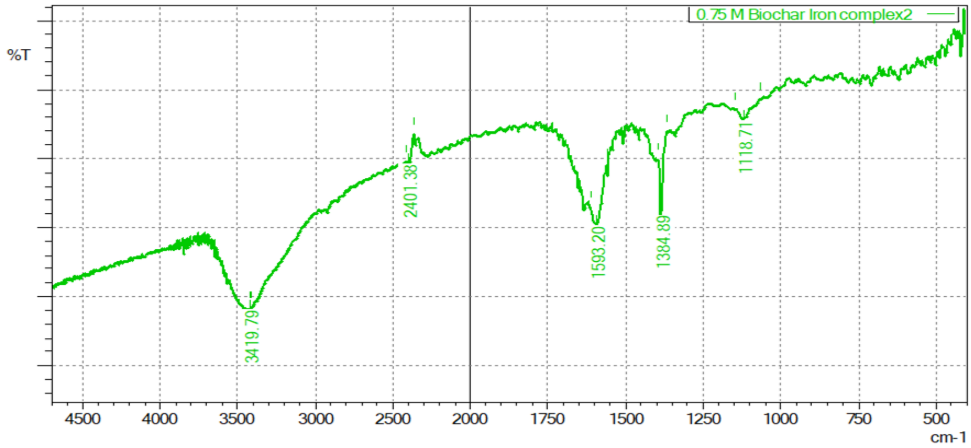

Figure 4. Characterization of Biochar-Iron Complex Using FT-IR (0.75 M Fecl3 (1:15w/V)

Overall, The FT-IR spectra of the sugarcane bagasse–derived biochar–iron complexes confirmed the presence of functional groups such as carboxyl (–COOH), hydroxyl (–OH), and Fe–O bonds. These findings are consistent with previous reports on Fe-modified biochars synthesized from sugarcane bagasse, where similar functional groups have been shown to contribute to high metal binding capacities [20] [21]. However, unlike most earlier studies that focus on pollutant removal [22] [23] [24], the functional group interactions in the present study are interpreted in the context of iron retention and potential bioavailability to crops.

3.3. XRF Analysis of Iron Levels in the Biochar-Iron Complex

XRF analysis quantified the incorporation of iron into the biochar matrix, showing a clear trend of increased iron retention with higher $FeCl_3$ concentrations. Specifically, iron content rose from 51.694% at 0.25 M to 65.754% at 0.5 M and reached 72.049% at 0.75 M (Table 1). Standard deviation values decreased slightly (from 1.318 to 1.159) as concentration increased, indicating more uniform iron deposition at higher molarities. This suggests that increasing $FeCl_3$ concentration enhances iron loading through improved ion-exchange interactions and surface complexation. However, the incremental gain between 0.5 M and 0.75 M (from 65.754% to 72.049%) was less pronounced than between 0.25 M and 0.5 M, indicating a potential saturation point or limited availability of reactive sites. These findings align with studies by [25], where excessive precursor concentrations yield diminishing returns due to steric hindrance or precipitation on the surface, and [26], which observed higher iron sorption capacities in biochar composites treated with elevated $FeCl_3$ levels. Therefore, while 0.75 M achieves maximum loading, 0.5 M may represent an optimal compromise between iron enrichment and material efficiency. These findings underscore the importance of dosage optimization for practical soil amendment use, balancing cost, performance, and environmental considerations.

Table 1. Determination of Iron Percentage in Different Biochar-Iron Complex Concentration Using XRF

Biochar-iron complex concentration	Iron Concentration (%)	Std Deviation
0.25M	51.694	1.318
0.5M	65.754	1.275
0.75M	72.049	1.159

The data demonstrate that the iron content increased with the concentration of $FeCl_3$ used, confirming that higher precursor concentrations lead to enhanced iron loading on the biochar. The reduction in standard deviation with increasing concentration also indicates more uniform iron distribution.

4. Conclusions

This study successfully synthesized sugarcane bagasse–derived biochar and complexed it with $FeCl_3$ to form a biochar–iron complex. FT-IR analysis confirmed the binding of iron ions to key functional groups on the biochar, and XRF analysis quantitatively demonstrated enhanced iron retention, particularly at the 0.75 M concentration. The biochar–iron complex shows significant potential as a sustainable soil amendment for improving nutrient retention and could contribute to enhanced soil fertility and crop productivity. Overall, these results reinforce the growing body of evidence that Fe-modified biochars are multifunctional materials capable of nutrient retention. However, whereas prior research has concentrated on macronutrient retention (particularly phosphorus) or contaminant immobilization, this study directly targets micronutrient enhancement in crop production. This represents a critical step toward addressing iron deficiency in leafy vegetables and developing biochar-based fertilizers tailored to micronutrient delivery.

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