Hue University Journal of Science: Natural Science
Vol. 134, No. 1S-3, 115–123, 2025 (Special Issue: CBA 2025)

pISSN 1859-1388
eISSN 2615-9678

Assessment of extractable carbon and nitrogen mineralization in paddy soil under Fenton reaction

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(*Received: 19 July 2025; Revised: 09 September 2025; Accepted: 16 September 2025*)

Abstract. This study aims to evaluate the effects of straw, Fenton reagent, and the combination of straw and Fenton treatments on the decomposition of organic matter and nitrogen mineralisation in soil. Extractable carbohydrate, total dissolved organic carbon (DOC), ammonium (NH_4^+) and nitrate (NO_3^-) contents, and total inorganic nitrogen were analysed. The results show that the Fenton treatment significantly increased the NH_4^+ content (23.65 mg/kg) and the inorganic-to-total-N ratio (20.30%), reflecting a strong mineralisation capacity. Meanwhile, the combined treatment resulted in the lowest NO_3^- concentration, indicating potential nitrogen retention or loss. Principal component analysis (PCA) reveals a clear grouping among treatments, with DOC and NH_4^+ being the key variables contributing to this grouping. These findings provide a scientific foundation for selecting appropriate treatment methods to enhance soil nutrient availability in agricultural systems.

Keywords: Fenton, nitrogen mineralization, extractable carbonhydrate, PCA, DOC

1 Introduction

Rice production plays a central role in national food security and the livelihoods of millions of Vietnamese farmers. According to the General Statistics Office, Vietnam has 7.1 million hectares of rice land in 2023, making it one of the world's largest rice-producing countries [1]. Along with the annual rice yield, a large amount of agricultural by-products, especially straw, are also produced, accounting for 6 tons of straw per hectare of harvested rice [2, 3]. In the Central region of Vietnam, where the wet rice farming system has been practised for years because of specific climate and soil conditions, straw is often left in the fields after harvest. However, post-harvest treatment of rice straw remains inappropriate: the most common practice is burning in the field – a method that both pollutes the air and loses valuable organic matter to the soil [4].

Meanwhile, rice straw is a carbon-rich source of raw materials, which can become an effective soil amendment if properly treated and decomposed. The addition of rice straw to the soil significantly changes biochemical processes, primarily the carbon (C) and nitrogen (N) cycles [4–6]. Under the anaerobic conditions typical of flooded rice fields, rice straw decomposes slowly because of its high content of lignin and cellulose, which are difficult for normal microorganisms to

DOI: 10.26459/hueunijns.v134i1S-3.7989

decompose. Incomplete decomposition not only reduces the efficiency of nutrient supply to plants but can also produce greenhouse gases such as CH_4 and N_2O, and affect the biological and chemical fertility of soil [7–10].

Gaining insight into and managing the decomposition of rice straw, along with the transformation of carbon and nitrogen in the soil, are essential for enhancing the utilisation of agricultural by-products, promoting soil health, and advancing sustainable agriculture. In that context, solutions to promote rice straw decomposition, especially under anaerobic conditions, are attracting considerable attention. The Fenton reaction (Fe^{2+} + H_2O_2) – an oxidation-reduction process that generates strong oxidising hydroxyl radicals (•OH) – has been shown to be capable of rapidly decomposing stable organic compounds such as lignin and cellulose in water and wastewater environments [11–13]. However, the application of Fenton in soil conditions, predominantly anaerobic rice soil, remains relatively limited and has not been studied in depth in Vietnam.

This study was conducted to evaluate the effectiveness of the Fenton reaction in promoting the anaerobic decomposition of rice straw. Perennial rice soils in Central Vietnam were used to establish an anaerobic composting model under laboratory conditions. The post-composting extracts were analysed for NH_4^+ and NO_3^- content, soluble carbohydrates, and total dissolved organic carbon (DOC) to evaluate the changes in soluble N and C compounds in the soil, thereby clarifying the promoting role of the Fenton reaction in the decomposition of rice straw, nitrogen mineralization, and improving the potential for recycling by-products in the rice-growing system.

2 Methods

2.1 Experimental design

The land used in this study is located in Hoa Chau commune, Hoa Vang district, Da Nang City, central Vietnam (38°15'N, 140°15'E), and has been cultivated with wet rice for over 100 years. According to the data from the General Statistics Office [1], this site has an average annual rainfall of 2,407 mm and an average temperature of 24.4 °C over the past two decades (2001–2020).

The Fenton solution was prepared as follows: First, 1.02 mL of a 30% H_2O_2 solution was mixed with 100 mL of distilled water to obtain solution A. Next, 1.39 g of $FeSO_4$ was dissolved in 100 mL of distilled water to form solution B. Then, 0.5 mL of solution A and 1 mL of solution B were added to 500 mL of distilled water to obtain solution C (Fenton solution). Solution C was used directly in the Fenton experiments under the influence of the strong oxidation reaction of the Fenton system (H_2O_2/Fe^{2+}).

10 g of soil was mixed with 30 mL of distilled water, and the mixture was incubated in a sealed jar in the dark at ambient temperature for 10 days to activate the microorganisms in the soil. Then, the Fenton solution was added according to the following scheme.

- Control: Soil (10 g) + 30 mL distilled water
- Straw: Soil (10 g) + 0.2 g straw + 30 mL distilled water
- Fenton: Soil (10 g) + 100 nM Fenton solution (30 mL C solution)
- Combination: Soil (10 g) + 0.2 g straw + 100 nM Fenton solution (30 mL C solution)

Three days after Fenton supplementation, the sealed vials were removed and filtered to measure the parameters related to C and N.

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eISSN 2615-9678

2.2 Analyzing indicators

In this study, important indicators, namely soluble carbohydrates (ECH), DOC, ammonium (NH_4^+) and nitrate (NO_3^-) contents in the soil extract, were analysed to evaluate the decomposition process of rice straw under the influence of the Fenton reaction. The carbohydrate content was determined with the Dubois method, which involves the reaction between sugar and a phenol-H_2SO_4 reagent to form an orange-yellow compound; the optical density was measured at a wavelength of 490 nm. DOC was analysed with a method that oxidises organic carbon to CO_2, and then measured with an infrared detector. The ammonium content was determined with the phenate method, in which NH_4^+ reacts with the reagent to form a blue compound, and the optical density of which was measured spectrophotometrically at a wavelength of about 630 nm. The nitrate content was analysed with the cadmium reduction method and the Griess reaction to form a pink-red compound, whose optical density was measured at 540 nm.

2.3 Statistical analysis

In this study, we used ANOVA to compare the differences in extractable carbohydrate, total organic carbon, and ammonium and nitrate contents using SPSS version 20 software (IBM Corp., Armonk, NY, USA). Soil samples were collected in triplicate. Pearson correlation analysis was also conducted to examine the relationships among measured soil parameters at a significance level of $p < 0.05$. In addition, Principal Component Analysis (PCA) was performed to examine the relationships between soil parameters and treatments.

3 Results and discussion

3.1 Changes in extracted carbon

The experimental results show that both straw and the Fenton reaction had a significant effect on the changes in the ECH and DOC content in the soil (Fig. 1, Table 1). The ECH content in the control soil was only 20.72 mg/kg soil, the lowest among the treatments. The addition of straw significantly increased ECH (89.19 mg/kg soil), reflecting the decomposition of cellulose and hemicellulose from straw under anaerobic conditions.

Table 1. ANOVA analysis of differences between treatments regarding nutritional parameters from soil extracts

Source of Variation	ECH	DOC	NH_4^+	NO_3^-	N inorganic
SS	8528	64777	586	0.982	605.80
df	3	3	3	3	3
MS	2843	21592	195	0.33	202
F	25.84	30.40	8.33	51.10	8.46
P-value	0.000	0.000	0.008	0.000	0.007

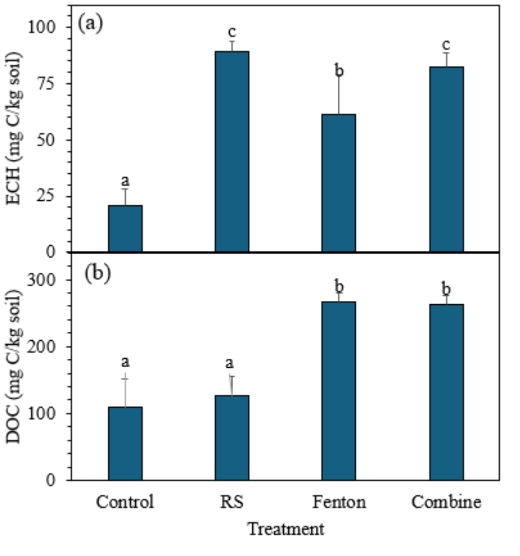

Fig. 1. Effect of straw and Fenton solution on ECH and DOC content in soil extract. Letters *a* and *b* in the column represent group differences according to Tukey's test, $p < 0.05$

Fenton treatment alone also increased ECH to 61.46 mg/kg soil, indicating that hydroxyl radicals generated from the Fenton reaction are capable of breaking down stable carbohydrate structures in soil. These results are consistent with those from previous studies [14–16]. In particular, the combined treatment of straw and Fenton reaction resulted in an ECH of 82.16 mg/kg soil, which is higher than that of the single Fenton treatment and practically equivalent to that of the straw treatment. This shows the synergetic effect of the carbon substrate source (straw) and the oxidizing agent (Fenton).

For DOC, the Fenton and combination treatments show significantly higher values than the control and straw treatments. Specifically, the DOC in the control and straw treatments was 110.24 and 126.24 mg/kg soil, respectively, with no statistically significant difference ($p > 0.05$). Meanwhile, the Fenton and combination treatments recorded DOCs of 266.57 and 262.88 mg/kg soil, respectively, which were more than twice that of the control value and showed a statistically significant difference ($p < 0.05$). This indicates that the Fenton reaction not only breaks down straw but also substantially releases soluble organic compounds from soil and organic materials, contributing to the enrichment of readily available carbon sources in anaerobic soil systems [13, 17].

3.2 Changes in nitrogen mineralisation

The concentrations of nitrogen mineralised forms (NH_4^+ and NO_3^-) in the treated soil show a statistically significant difference among treatments (Fig. 2, Table 1).

The highest NH_4^+ concentration was recorded in the Fenton treatment with a value of 23.65 mg/kg soil, more than three times that of the control (7.54 mg/kg soil). This indicates that the Fenton oxidation reaction significantly promotes the decomposition of organic compounds containing N, especially under anaerobic conditions where nitrogen transformation typically ceases in the form of ammonium. The straw (8.17 mg/kg) and straw + Fenton (6.92 mg/kg) treatments did not differ significantly from the control ($p > 0.05$), indicating that straw addition alone or together with Fenton was insufficient to increase NH_4^+ accumulation compared with the Fenton reaction alone. Similar studies on rice soil have also shown that Fenton affects the ammonium loss from rice fields [18] and also in wastewater [19]. The NO_3^- content in the straw (0.77 mg/kg) and Fenton (0.56 mg/kg) treatments was significantly higher than that in the control (0.21 mg/kg soil), indicating that some nitrate formation occurred, although anaerobic conditions are generally unfavourable for nitrification. This may result from the early reaction stage or the local presence of aerobic microorganisms that produced small amounts of NO_3^-. However, in the combined treatment, the NO_3^- concentration decreased sharply to only 0.04 mg/kg soil – the lowest among all treatments – reflecting the strong NO_3^- utilisation capacity of anaerobic microorganisms, or because of the NO_3^- reduction reaction to N_2/N_2O under anaerobic conditions with excess biodegradable carbon.

However, it can be seen that NO_3^- concentration in all treatments was low (<1 mg/kg soil). This is because we collected dry soil during the experiments; hence, the previous NO_3^- concentration in the field soil had been leached. The low NO_3^- concentration is also confirmed in similar studies that we have recently published [3, 20]. Overall, the results indicate that the Fenton reaction had a significant impact on N solubility and NH_4^+ accumulation. At the same time, straw addition increased the NO_3^- levels, but this effect could not be sustained under anaerobic conditions when Fenton and straw were combined

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pISSN 1859-1388
eISSN 2615-9678

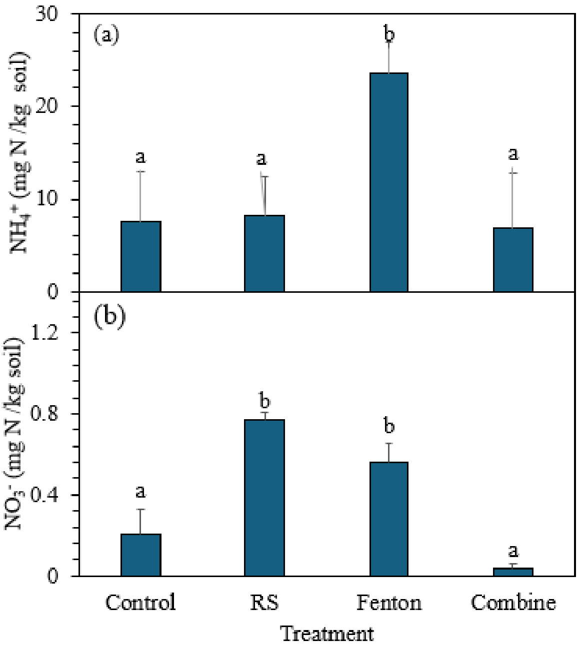

Fig. 2. Effect of straw and Fenton reaction on ammonium and nitrate contents in soil extract. Letters *a* and *b* in the column represent different groups according to Tukey's test, $p < 0.05$

3.3 Contribution of extracted carbon and minerilised nitrogen

Table 2 presents the contribution of decomposed carbon and minerilised nitrogen, reflecting the degree of mineralisation and nutrient supply potential in the treatments after anaerobic incubation and Fenton treatment. The straw treatment yielded an ECH-to-SOC ratio of 5.75% and a DOC-to-SOC ratio of 8.13%, significantly higher than that of the control (1.33% and 7.10%), indicating that the addition of straw increased the ECH and DOC contents. In particular, the ECH-to-DOC ratio reached 70.65%, reflecting that the majority of the soluble C after straw treatment was in easily degradable carbon compounds. The Fenton treatment exhibits superior efficiency in decomposing organic matter, yielding the highest DOC-to-SOC ratio (17.17%) and ECH-to-DOC ratio of 23.06%. However, the ECH-to-SOC ratio was only 3.96%. This suggests that Fenton produces more soluble organic compounds overall, but only a fraction of them is readily biodegradable.

Table 2. Contribution of extracted carbon and mineralised nitrogen after 2 weeks of anaerobic incubation

Treatment	Contribution of extracted C (%)			Contribution of mineralised N (%)		
	ECH/SOC	DOC/SOC	ECH/DOC	NH_4^+/Total N	NO_3^-/Total N	Inorganic N/Total N
Control	1.33	7.1	18.79	6.32	0.18	6.5
Straw	5.75	8.13	70.65	6.85	0.64	7.49
Fenton	3.96	17.17	23.06	19.83	0.47	20.3
Combined	5.29	16.93	31.25	5.8	0.03	5.83

In contrast, the combined treatment of straw and Fenton resulted in an intermediate ECH-to-SOC ratio of 5.29%, DOC-to-SOC ratio of 16.93%, and ECH-to-DOC ratio of 31.25%, indicating an interaction that reduces the efficiency of straw degradation when Fenton was used simultaneously, possibly because of the formation of more stable intermediate products or the effect on microorganisms [15, 17].

Regarding inorganic nitrogen, the Fenton treatment exhibits a significant conversion rate of organic nitrogen to NH_4^+ and NO_3^-, with the total inorganic nitrogen accounting for 20.30% of the total nitrogen, which is three times higher than that of the control (6.50%) and other treatments. Notably, the majority of mineral nitrogen in this treatment was in the form of NH_4^+ (19.83%), which is suitable for anaerobic conditions and has a strong degradation ability because of the Fenton reaction. In contrast, the combined treatment yielded the lowest conversion rate in inorganic nitrogen (5.83%), primarily because of NH_4^+ accounting for 5.80% of the total. In comparison, NO_3^- was almost absent (0.03%), possibly because of the strong reduction of nitrate under anaerobic conditions with excess degradable carbon from straw. The inorganic nitrogen extraction rate of the straw treatment alone was 7.49%, higher than that of the control, primarily because of the increase in NO_3^- (0.64%). Overall, the results show that Fenton significantly promoted the mineralisation of carbon and nitrogen, producing more inorganic nitrogen (NH_4^+). In addition, straw increased the amount of soluble carbon and nitrate ratio, but the overall nitrogen mineralisation efficiency was low. The combination of Fenton and straw did not exhibit a synergistic effect and, in fact, reduced the nitrogen mineralisation efficiency, possibly because of secondary reactions or microbial inhibition [14].

3.4 Correlation between analytical indicators

Principal component analysis was performed by measuring parameters to assess the differences between treatments (Fig. 3). The first two principal components (PC1 and PC2) explained 48.8 and 35.3% of the total variance, respectively, indicating that PCA effectively reflected the variation in the dataset. The score plot results show that the combined treatment (Straw + Fenton) was clearly separated from the remaining groups, indicating a significant difference in soil chemical properties after treatment. The single Fenton group was also clearly separated, while the Straw and Control groups were closer to each other, reflecting similarities in chemical composition. The loading plot shows that DOC, ECH, and NH_4^+ contributed the most to the PC1 component, especially DOC and NH_4^+. This indicates that the Fenton and Combined treatments had high levels of dissolved carbon and ammonium nitrogen, which were factors that contributed to a clear separation in the PCA. In contrast, NO_3^- had a lesser effect and was primarily related to PC2. Overall, the PCA clarified that Fenton treatment of soil, especially when combined with straw, resulted in significant changes in the dissolved organic matter and inorganic nitrogen contents of the soil [11].

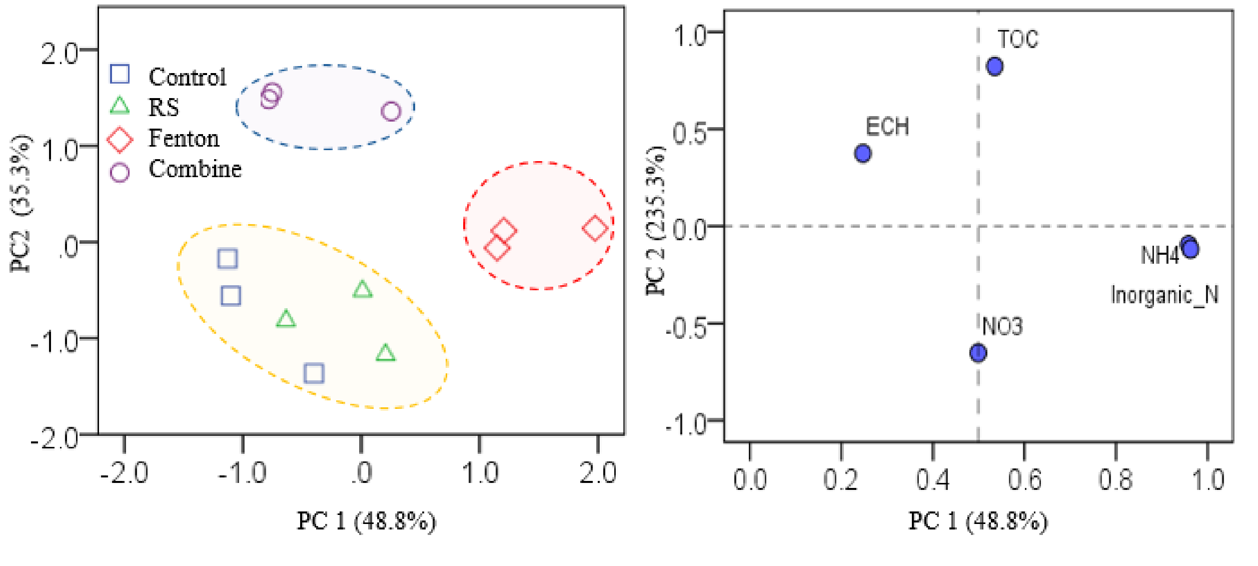

Fig. 3. Principal component analysis of parameters and treatments

pISSN 1859-1388
eISSN 2615-9678

Table 3 shows the Pearson correlation coefficients between soil nutrient parameters extracted, including dissolved carbon (ECH, DOC) and inorganic nitrogen (NH_4^+, NO_3^-, total inorganic nitrogen). The results show a robust correlation between NH_4^+ and total inorganic nitrogen (r = 0.999), indicating that NH_4^+ was the primary contributor to the inorganic nitrogen content in the soil after extraction.

Table 3. Pearson correlation analysis between nutritional parameters from soil extracts

	ECH	DOC	NH_4^+	NO_3^-	Inorganic N
ECH	1.00				
DOC	0.38	1.000			
NH_4^+	–0.01	0.43	1.000		
NO_3^-	0.33	–0.26	0.39	1.00	
Inorganic N	–0.00	0.411	0.99	0.42	1.00

DOC had a moderate correlation with NH_4^+ (r = 0.426) and total inorganic nitrogen (r = 0.411), indicating a relationship between dissolved carbon and nitrogen mineralisation. ECH, representing total soluble carbon, was only weakly correlated with DOC (r = 0.382) and exhibited no significant relationship with NH_4^+ or total inorganic nitrogen. In contrast, NO_3^- was positively correlated with NH_4^+ (r = 0.388) and total inorganic nitrogen (r = 0.418). However, the coefficient was small, reflecting the minor role of NO_3^- in total inorganic nitrogen. These results suggest that nitrogen mineralisation in post-treatment soils is significantly influenced by dissolved carbon content and biochemical reactions involving NH_4^+.

4 Conclusion

The results indicate that different treatments had a significant impact on the decomposition of organic matter and nitrogen mineralisation in soil. Fenton treatment significantly increased the NH_4^+ and inorganic nitrogen contents, indicating its effectiveness in promoting organic matter mineralisation. In contrast, the combination of Fenton and straw failed to increase mineral nitrogen forms. However, they significantly reduced the NO_3^- and total inorganic nitrogen contents, reflecting the ability to retain or lose nitrogen because of more complex biological/chemical processes. PCA analysis reveals that the treatments were clearly separated on the principal component plane, with DOC and ECH contributing the most to this separation. Pearson correlation analysis reveals that DOC had a moderate relationship with NH_4^+ and total inorganic nitrogen, whereas NH_4^+ completely dominated the amount of inorganic nitrogen in the soil (r = 0.999). Overall, the Fenton treatment alone significantly promoted decomposition and nutrient release. However, the combination of Fenton and straw should be considered further because of the possibility of decreasing the efficiency of mineral nitrogen release. These results provided a crucial scientific foundation for the application of organic and advanced oxidation treatments in soil management and agricultural nutrition.

Acknowledgements

This study was funded by the Ministry of Education and Training through project number B2024.DNA.10.

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