



## Article

# Impact of Crop Residue, Nutrients, and Soil Moisture on Methane Emissions from Soil under Long-Term Conservation Tillage

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**Abstract:** Greenhouse gas emissions from agricultural production systems are a major area of concern in mitigating climate change. Therefore, a study was conducted to investigate the effects of crop residue, nutrient management, and soil moisture on methane (CH<sub>4</sub>) emissions from maize, rice, soybean, and wheat production systems. In this study, incubation experiments were conducted with four residue types (maize, rice, soybean, wheat), seven nutrient management treatments {N0P0K0 (no nutrients), N0PK, N100PK, N150PK, N100PK + manure@ 5 Mg ha<sup>-1</sup>, N100PK + biochar@ 5 Mg ha<sup>-1</sup>, N150PK + biochar@ 5 Mg ha<sup>-1</sup>}, and two soil moisture levels (80% FC, and 60% FC). The results of this study indicated that interactive effects of residue type, nutrient management, and soil moisture significantly affected methane (CH<sub>4</sub>) fluxes. After 87 days of incubation, the treatment receiving rice residue with N100PK at 60% FC had the highest cumulative CH<sub>4</sub> mitigation of -19.4 μg C kg<sup>-1</sup> soil, and the highest emission of CH<sub>4</sub> was observed in wheat residue application with N0PK at 80% FC (+12.93 μg C kg<sup>-1</sup> soil). Nutrient management had mixed effects on CH<sub>4</sub> emissions across residue and soil moisture levels in the following order: N150PK > N0PK > N150PK + biochar > N0P0K0 > N100PK + manure > N100PK + biochar > N100PK. Decreasing soil moisture from 80% FC to 60% FC reduced methane emissions across all residue types and nutrient treatments. Wheat and maize residues exhibited the highest carbon mineralization rates, followed by rice and soybean residues. Nutrient inputs generally decreased residue carbon mineralization. The regression analysis indicated that soil moisture and residue C mineralization were the two dominant predictor variables that estimated 31% of soil methane fluxes in Vertisols. The results of this study show the complexity of methane dynamics and emphasize the importance of integrated crop, nutrient, and soil moisture (irrigation) management strategies that need to be developed to minimize methane emissions from agricultural production systems to mitigate climate change.

**Keywords:** methane; mitigation; crop residue; soil moisture; nutrient; residue mineralization



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## 1. Introduction

Methane (CH<sub>4</sub>) is a potent greenhouse gas (GHG) that significantly contributes to global warming. Thus, it is crucial to consider the role of CH<sub>4</sub> fluxes in the global carbon cycle. Methane, in particular, has seen a significant increase in atmospheric concentration, reaching 1.5 times the levels observed in pre-industrial times [1]. Methane contributes 18% of the global warming potential, making it the second-highest contributor to long-lived GHGs. Agriculture, forestry, and other land use (AFLOU) are responsible for approximately 22%

of global net anthropogenic emissions, with AFLOU-CH<sub>4</sub> accounting for almost 41% of the total net anthropogenic CH<sub>4</sub> emissions, with agriculture accounting for 88% of the AFOLU component [2]. When it comes to agricultural activities like managing residue and applying nutrients, it is important to comprehend how different types of residue, nutrient management, and soil moisture interact and influence methane emissions. That is because these factors have varying effects on the release of methane [3–7]. Different residue types, including maize, rice, soybean, and wheat, possess unique chemical compositions and decomposition rates, which can affect the soil's methane production and consumption processes [8–11]. Nutrient management practices, such as fertilization with nitrogen (N), phosphorus (P), and potassium (K), as well as the use of biochar and organic manure, can alter soil microbial activity and nutrient availability, influencing methane emissions [9,12–17]. Soil moisture content is another critical factor that regulates methane production and consumption, as it affects the availability of oxygen required for methane oxidation [4,18–20]. This study aims to develop effective mitigation strategies by examining the effect of crop residue type, nutrient management, and soil moisture with the underlying mechanisms on CH<sub>4</sub> emission.

Crop residues, commonly used to enhance soil fertility and soil health [21], can serve as both sources and sinks of atmospheric CO<sub>2</sub> and CH<sub>4</sub> [8]. Adding crop residues provides carbon substrates and nutrients that promote methanogenesis, increasing CH<sub>4</sub> production and consumption [16,22–24]. Residue incorporation can create anaerobic microsites, increasing soil moisture and favoring methanogenesis and CH<sub>4</sub> emissions [25]. On the other hand, improved aeration due to residue addition enhances CH<sub>4</sub> oxidation by promoting methanotroph activity [26,27]. Methanogenesis, carried out by methanogenic archaea in anaerobic environments, is stimulated by carbon-rich crop residues, leading to increased CH<sub>4</sub> emissions [28]. Conversely, methane oxidation, performed by methane-oxidizing bacteria in aerobic conditions, can be influenced by residue addition through changes in soil properties and oxygen availability [29]. Additionally, crop residues can indirectly influence methane oxidation by altering soil properties, such as oxygen availability, pH, and nutrient availability, which affect the activity and abundance of methanotrophs [29–31].

The effect of crop residue on CH<sub>4</sub> emissions is also driven by soil moisture and nutrient concentration. Soil moisture content is critical in regulating CH<sub>4</sub> emissions by influencing soil water and oxygen availability for microbial activity, carbon and nitrogen mineralization, and CO<sub>2</sub> respiration [18,20,32]. Decreased soil moisture levels can enhance CH<sub>4</sub> uptake under semi-arid conditions due to increased oxygen diffusivity, stimulating soil CH<sub>4</sub> oxidation [32–34]. Excess soil moisture through flood irrigation with straw incorporation resulted in the highest average CH<sub>4</sub> fluxes, leading to a total CH<sub>4</sub> emission of  $-0.94 \text{ kg ha}^{-1}$ . In the wheat–maize cropping system, straw incorporation (ca. straw removal) reduced CH<sub>4</sub> emission by 17.1% with surface drip irrigation and 14.0% with partial root-zone irrigation [26]. Du et al. [35] reported that limited irrigation and nitrogen management resulted in a relatively higher cumulative CH<sub>4</sub> uptake in the wheat season in the wheat–maize cropping system and reduced greenhouse gas intensity without additional cost. In a laboratory experiment, Korhonen et al. [36] demonstrated that excess soil moisture with fresh carbon input reduced the CH<sub>4</sub> oxidation potential of soil. A literature review reported a shift in the balance between methanogens and methanotrophs' activities and abundance influencing either an increase/decrease in soil CH<sub>4</sub> emission under different soil moisture content driven by soil organic input and nitrate nitrogen concentration [37].

Soil nutrient concentrations, particularly nitrogen and phosphorus, affect CH<sub>4</sub> emissions or oxidation, with excess nitrogen promoting methanogenesis and nutrient limitation enhancing methane oxidation [12,27]. Additionally, adding organic residues has increased CH<sub>4</sub> oxidation in clay soil [38]. Shaikat et al. [12] reported that incorporating a biochar amendment at a rate of 2% in conjunction with a nitrogen (N) application of 140 kg N per hectare can be a promising approach to mitigate CH<sub>4</sub> emissions from paddy rice cultivation in an Alfisol soil. Sainju et al. [7] reported that N rates did not affect soil CH<sub>4</sub> uptake but increased soil CO<sub>2</sub> fluxes in the northern Great Plains, USA. A meta-analysis concluded that CH<sub>4</sub> emissions were stimulated at low N application rates ( $<100 \text{ kg N ha}^{-1}$ ) but inhibited

at high N rates ( $>200$  kg N  $\text{ha}^{-1}$ ) as compared to no N fertilizer (control) [39]. Applying chemical NPK fertilizer (240 kg urea-N  $\text{ha}^{-1}$ , 90 kg  $\text{P}_2\text{O}_5$   $\text{ha}^{-1}$ , and 120 kg  $\text{K}_2\text{O}$   $\text{ha}^{-1}$ ), manure, and their combination increased seasonal mean  $\text{CH}_4$  emissions by 67.4%, 20.4%, and 101.2%, respectively, compared with PK (90 kg  $\text{P}_2\text{O}_5$   $\text{ha}^{-1}$  and 120 kg  $\text{K}_2\text{O}$   $\text{ha}^{-1}$ ) treatment without N fertilizer input in rice paddies of China [16]. Nutrient addition alters the soil elemental stoichiometry (C:N:P ratio) with residue C input, resulting in varied responses of GHG emission from residue return soils [1]. There is growing evidence of biochar as an amendment for soil carbon sequestration [40–42]; however, previous researchers have reported positive [43], negative [40], and uncertain [44] effects of biochar on mitigating  $\text{CH}_4$  emissions from agricultural soils. Several environmental [44], soil [40,44], and management [45,46] factors regulate the effectiveness of biochar, including the rate of N fertilization and crop residue type. Therefore, in this study, we aimed to assess the integrated application of synthetic fertilizers with manure/biochar as a nutrient management strategy for evaluating the responses to  $\text{CH}_4$  emissions.

By considering these factors (residue types, nutrient application, and soil moisture) and investigating the associated mechanisms, we can develop effective strategies to mitigate methane emissions while maintaining crop productivity. Furthermore, quantifying the impact of residue, nutrients, and soil moisture on residue carbon mineralization can improve our understanding of greenhouse gas inventories and enhance predictive models for assessing climate change impacts. In this study, we hypothesize the following: (1) residue types and nutrient management will significantly impact methane emissions, with variations observed among different residue types and nutrient treatments; (2) soil moisture content will interact with residue and nutrient management, leading to distinct methane flux patterns under different moisture conditions; and (3) specific soil properties and microbial processes, such as labile organic carbon, nutrient availability, and residue C mineralization, will significantly mediate methane emissions in response to residue, nutrient, and moisture conditions.

## 2. Materials and Methods

### 2.1. Study Site

The experiment was conducted in the ICAR-Indian Institute of Soil Science laboratory in Bhopal, India. The place is at  $23^\circ 15'$  N latitude and  $77^\circ 25'$  E longitude, with an elevation of 427 m above sea level and a humid subtropical climate. The soil is deep Vertisols (IsohyperthermicTypicHaplustert) with a clayey texture (54% clay). Its bulk density is  $1.34$   $\text{Mg m}^{-3}$  at  $0.27$   $\text{g g}^{-1}$  soil water content and has 0.99% total soil organic carbon content (0–15 cm depth). The soil is neutral to alkaline (pH—7.85) with an electrical conductivity of  $0.3$   $\text{ds m}^{-1}$ , and  $\text{Ca}^{2+}$  is the main exchangeable cation in the Ap horizon. The soil sample for incubation was collected from the top 0–15 cm of soil after harvesting wheat in 2020. It came from a 12-year conservation tillage experiment in a soybean–wheat system. The production method involved reduced tillage with 30% residue return plus fertilizer (30:60:30 kg N– $\text{P}_2\text{O}_5$ – $\text{K}_2\text{O}$   $\text{ha}^{-1}$  for soybean and 100:60:30 kg N– $\text{P}_2\text{O}_5$ – $\text{K}_2\text{O}$   $\text{ha}^{-1}$  for wheat).

### 2.2. Incubation Experimental Detail

The soil sample for incubation was collected from the top 0–15 cm of soil after harvesting wheat in 2020. It came from a 12-year conservation tillage experiment in a soybean–wheat system. The production method involved reduced tillage with 30% residue return plus fertilizer (30:60:30 kg N– $\text{P}_2\text{O}_5$ – $\text{K}_2\text{O}$   $\text{ha}^{-1}$  for soybean and 100:60:30 kg N– $\text{P}_2\text{O}_5$ – $\text{K}_2\text{O}$   $\text{ha}^{-1}$  for wheat). The soil samples were sieved to remove big fragments and stored at  $4^\circ\text{C}$  until further study. Various crop residues like rice, maize, soybean, and wheat were air-dried, then milled and sieved to 2 mm. A subsample of crop residues was dried for water content assessment, while others were analyzed chemically. An elemental analyzer (NC analyzer, Thermofisher Scientific, Rodano, Italy, Flash 2000 model) and the acid detergent fiber method were used to determine C and N concentrations and lignin and cellulose contents, respectively. The total carbon/nitrogen (TC:TN) ratio of the organic amendments

was 8:1 in manure, 45:1 (biochar), 76:1 (wheat straw), 50:1 (rice straw), 61:1 (soybean straw), and 65:1 (maize straw), respectively. The mean lignin content was 13.1% (*w/w*), 13.5% (*w/w*), 15.4% (*w/w*), and 9.0% (*w/w*) for wheat, rice, soybean, and maize straw, respectively. The mean cellulose content was 56.0% (*w/w*), 29.0% (*w/w*), 42.3% (*w/w*), and 49.7% (*w/w*) for wheat, rice, soybean, and maize straw, respectively. Reference is made to our previous work (Lenka et al. [5] and Raul et al. [47]) for more details on the properties of biochar and crop residues.

The soil had been pre-incubated for ten days at 70% of the two moisture levels (80% FC and 60% FC) and room temperature to kickstart microbial activity. Following pre-incubation, the crop residues (<2 mm) were completely mixed with soil (<2 mm) for incubation. A factorial experiment was set up with three replications to investigate the impact of crop residue type, nutrient levels, and soil moisture on CH<sub>4</sub> emission and carbon mineralization. The experiment consisted of five different levels of crop residue (wheat straw, maize straw, soybean straw, rice straw, and no residue), two levels of soil moisture content (80% FC and 60% FC), and seven nutrient treatments (N0P0K0, no nutrients; N0PK; N100PK; N150PK; N100PK + manure@ 5 Mg ha<sup>-1</sup>; N100PK + biochar@ 5 Mg ha<sup>-1</sup>; and N150PK+ biochar@ 5 Mg ha<sup>-1</sup>). The various degrees of nutrient management have been used to simulate the impact of synthetic fertilizer or the combined use of synthetic fertilizers and organic amendment (manure/biochar) on CH<sub>4</sub> emissions. Two treatments were set up in 460 mL glass jars: (a) 20 g soils (dry weight basis) combined with wheat, maize, soybean, or rice straw residues at a rate of 2.23 mg g<sup>-1</sup> soil, equaling 5 Mg ha<sup>-1</sup> residue incorporation; and (b) 20 g soil (dry weight basis) without crop residue (control). Seven nutrient treatments were applied to both the control soil and residue-incorporated soil: N0P0K0 (no nutrients), N0PK, N100PK, N150PK, N100PK + manure@ 5 Mg ha<sup>-1</sup>, N100PK + biochar@ 5 Mg ha<sup>-1</sup>, and N150PK+ biochar@ 5 Mg ha<sup>-1</sup>. The treatments N100 and N150 represented N application rates of 100 kg N ha<sup>-1</sup> and 150 kg N ha<sup>-1</sup>, respectively, using AR-grade ammonium nitrate. Besides the no-nutrient (N0P0K0) treatment, the same concentrations of phosphorus (P)@ 22 kg ha<sup>-1</sup> and potassium (K)@ 21 kg ha<sup>-1</sup> were added to the rest of the six nutrient treatments to assess the effect of increasing N levels. The application of phosphorus and potassium was made through AR-grade potassium dihydrogen phosphate to maintain a nutrient ratio of N: P<sub>2</sub>O<sub>5</sub>: K<sub>2</sub>O of 4:2:1, equivalent to 100 kg N ha<sup>-1</sup>. Nutrients were given in a solution made with distilled water containing NH<sub>4</sub>NO<sub>3</sub> + KH<sub>2</sub>PO<sub>4</sub> with pH adjusted to 7 using 1 M NaOH while maintaining incubation moisture levels at 80% FC and 60% FC. Field capacity was measured at matric potentials of -33 kPa using sieved (< 2 mm) soil samples in pressure plate extractors from Soil Moisture Equipment Corp., Santa Barbara, CA, with FC moisture content at 0.27 m<sup>3</sup> m<sup>-3</sup>. The two soil moisture levels, 80% FC and 60% FC, were selected for the incubation study to represent optimal and deficit moisture conditions, respectively. The 80% FC provides an environment conducive to microbial activity, supporting organic matter decomposition and nutrient cycling processes. This level mimics near-optimal conditions where microbial communities are most active. In contrast, 60% FC represents a moderate moisture deficit, which helps study how reduced water availability impacts microbial processes, particularly those involved in decomposing organisms and methane production and consumption. That allows for assessing microbial responses and greenhouse gas emissions under varying moisture regimes. A blank glass jar without soil or residue was included to consider atmospheric CO<sub>2</sub> and CH<sub>4</sub> concentration in the headspace of incubation jars for determining evolved gasses from the treatments. Evolved gasses (CO<sub>2</sub>/CH<sub>4</sub>) from the treatments were calculated alongside a blank jar without soil or residue, accounting for atmospheric CO<sub>2</sub>/CH<sub>4</sub> concentration at an incubation temperature of 30 °C based on the region's long-term average temperature over an incubation period spanning 87 days where soil moisture was maintained through regular weighing and water addition to make up for evaporation losses during gas sampling intervals.

### 2.3. Greenhouse Gas Sampling and Measurements

Headspace gasses were sampled with a syringe before being promptly transferred to an evacuated glass vial at set intervals on specific days and analyzed using gas chromatography (Agilent Technologies model 7890A, Santa Clara, CA, USA). These days included 0, 1, 4, 10, 17, 26, 33, 40, 47, 57, 67, 77, and 87 days of incubation. The purpose of these uneven intervals was to capture the usual asymptotic decrease observed in incubation experiments. All jars remained open for half an hour to replenish headspace oxygen and CO<sub>2</sub> to the normal concentration before being tightly sealed with aluminum caps on each sampling day. The flux rate (CH<sub>4</sub>/CO<sub>2</sub>) was determined by calculating the change in headspace concentration (µg C or mg C) per kg of soil (dry wt. equivalent) over a unit time of incubation using the ideal gas law. Cumulative CO<sub>2</sub> and CH<sub>4</sub> emissions were computed by integrating the fluxes from each measurement time. Apparent residue C mineralization was evaluated as the difference in CO<sub>2</sub> emission between soil amended with residue and control soil at the corresponding nutrient level [5].

### 2.4. Post Incubation Soil Analysis

During the incubation period of 87 days, soil samples were collected to analyze soil mineral N components—NO<sub>3</sub>, NO<sub>2</sub>, and NH<sub>4</sub>. Dehydrogenase (DHA) and labile SOC were also included in the analysis. The moisture content was determined gravimetrically using the oven dry method. For soil mineral nitrogen extraction, 2 M KCl was used, and the subsequent analysis employed standard methods [48]. Dehydrogenase activity assessment involved tracking the production rate of triphenylformazon (TPF) [49]. Labile SOC calculations utilized the potassium permanganate oxidation method [50,51]. More information on the incubation experiment and soil analysis can be found in our prior research work reference [5].

### 2.5. Statistical Analysis

Data underwent testing for normality and homogeneity of variance. In cases where a significant improvement was observed in normality variance, log transformation was utilized. Given the factorial design of our experiment setup, the statistical analysis was conducted using SPSS software (version 21.0, SPSS Inc., Chicago, IL, USA) through an analysis of variance under the generalized linear model. This analysis explored potential differences in the response variable (CH<sub>4</sub>/residue C mineralization) regarding residue types, nutrients, and soil moisture treatments. The significance level was established at  $\alpha = 0.05$ . Tukey's HSD multiple comparisons were employed to compare the main factor and interaction means and derive homogenous subsets. To identify predictor variables of soil CH<sub>4</sub> emissions, Pearson correlation (two-tailed significance) and a stepwise multiple regression analysis were conducted.

## 3. Results

### 3.1. Methane (CH<sub>4</sub>) Fluxes

Soil CH<sub>4</sub> fluxes were significantly influenced by the main factor effect of crop residue type, nutrient, and soil moisture, and the interactive effects of residue × nutrient, residue × moisture, nutrient × moisture, and residue × nutrient × moisture (Table 1). The results of two-way and three-way interactions are presented in this section because, while a three-way interaction indicates that the relationship between any two factors depends on the third, discussing two-way interactions helps clarify how these relationships behave in simpler contexts. That can guide the interpretation of the three-way interaction.

**Table 1.** Analysis of variance (significance  $p$  value) for methane ( $\text{CH}_4$ ) emissions to study the interactive effect of residue type and nutrient management at 80 and 60% FC soil moisture content after 87 days of incubation.

Source of Variation	Degrees of Freedom	$\text{CH}_4$ Emission	Degrees of Freedom	Residue C Mineralization
Residue	4	<0.001	3	<0.001
Nutrient	6	0.002	6	<0.001
Moisture	1	<0.001	1	<0.001
Residue $\times$ Nutrient	24	<0.001	18	0.066
Residue $\times$ Moisture	4	<0.001	3	0.002
Nutrient $\times$ Moisture	6	0.003	6	0.001
Residue $\times$ Nutrient $\times$ Moisture	24	<0.001	18	0.124
Error	140		112	
Total	210		168	
Corrected Total	209		167	

### 3.1.1. At 80% FC Interactive Influence of Residue $\times$ Nutrient

The magnitude and trend of temporal dynamics of  $\text{CH}_4$  fluxes differed with treatments (Figures S1 and S2). For 60% FC, the average  $\text{CH}_4$  fluxes ranged from  $-8.25 \mu\text{g C kg}^{-1} \text{ soil day}^{-1}$  (maize + N0P0K0) to  $5.41 \mu\text{g C kg}^{-1} \text{ soil day}^{-1}$  (soil without residue + N100PK + biochar@ 5 Mg/ha). Similarly, at 80% FC, the average  $\text{CH}_4$  fluxes ranged from  $-1.76 \mu\text{g C kg}^{-1} \text{ soil day}^{-1}$  (soybean + N100PK) to  $6.91 \mu\text{g C kg}^{-1} \text{ soil day}^{-1}$  (soybean + N0PK). Irrespective of nutrient management, the cumulative mean  $\text{CH}_4$  flux was negative and the lowest in soil amended with maize residue ( $-1.87 \mu\text{g C kg}^{-1} \text{ soil}$ ). The cumulative  $\text{CH}_4$  fluxes ( $\mu\text{g C kg}^{-1} \text{ soil}$ ) during 87 days of incubation followed the order maize ( $-1.87$ ) > rice ( $-0.33$ )  $\approx$  soybean (2.21) > wheat (6.88) > control soil (7.18) (Figure 1). Averaged across residue types, the trend of nutrient management on  $\text{CH}_4$  fluxes ( $\mu\text{g C kg}^{-1} \text{ soil}$ ) was N100PK (0.78) < N100PK + biochar (1.28) < N150PK + biochar (1.43) < N0P0K0 (2.76) < N100PK + manure (4.10) < N150PK (4.27) < N0PK (5.08). Mixed responses of nutrient application were observed on  $\text{CH}_4$  fluxes; e.g., N100PK + manure, N150PK, and N0PK nutrient treatments increased  $\text{CH}_4$  emission by 48–84% compared with minus nutrients, N0P0K0. On the contrary, N100PK, N100PK + biochar, N150PK + biochar decreased  $\text{CH}_4$  emission by 48 to 72% across residue. Further, the effect of nutrient management varied significantly with different residue treatments and soil without residue. In soil without residue, the nutrient effect followed the order N100PK + biochar < N0P0K0 < N150PK < N0PK < N100PK < N100PK + manure < N150PK + biochar. However, in soil amended with rice residue, the nutrient effect varied in the order N100PK + biochar < N100PK < N150PK < N0P0K0 < N100PK + manure < N150PK + biochar.

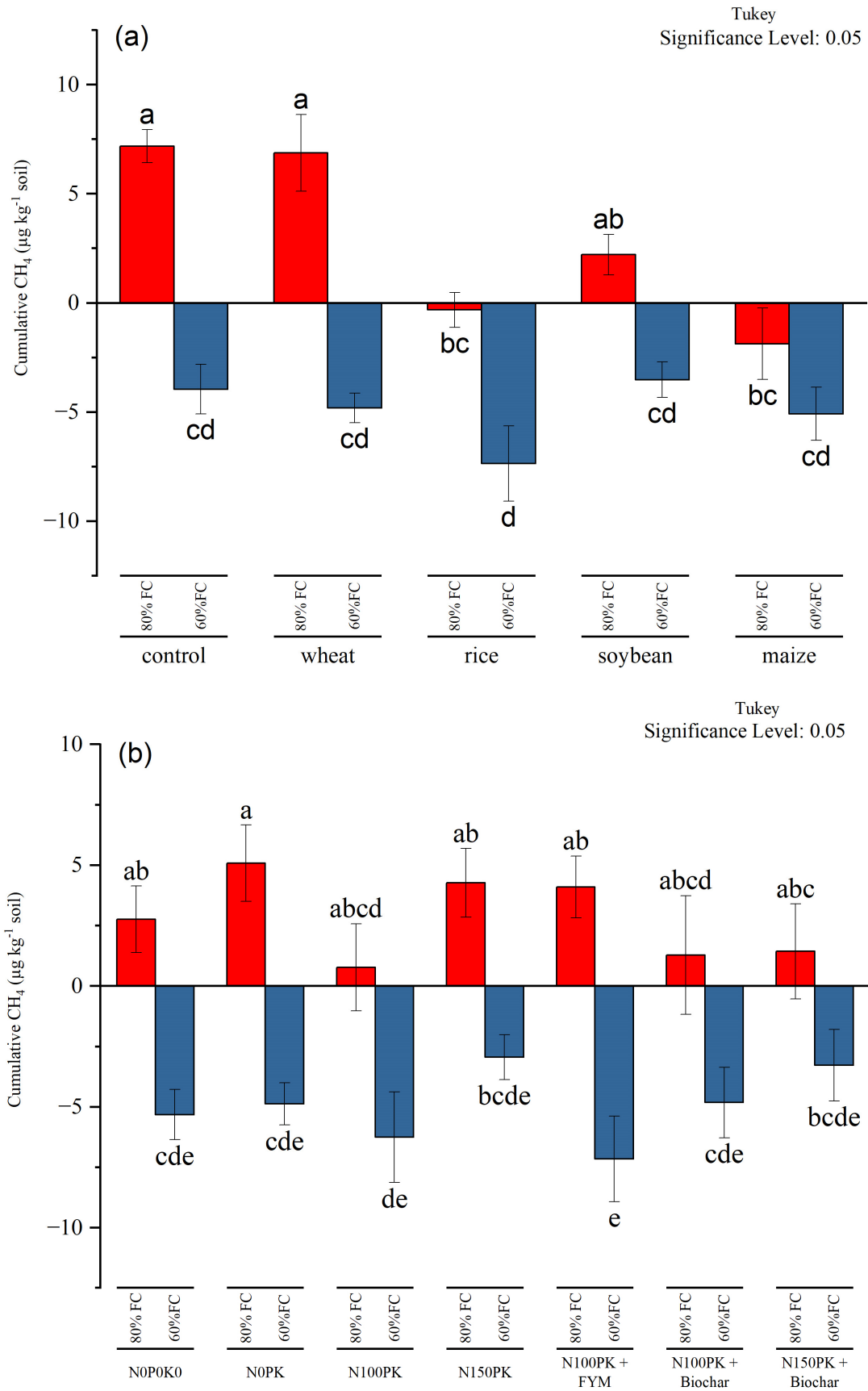
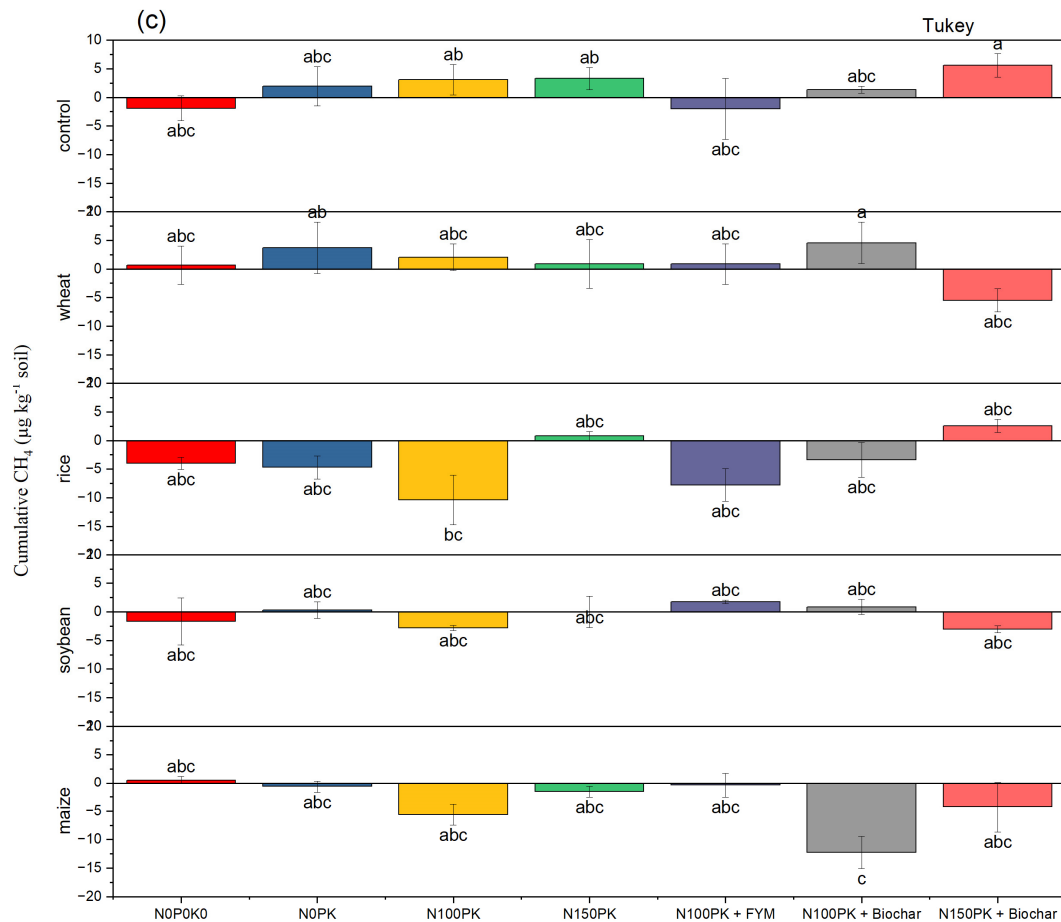


Figure 1. Cont.



**Figure 1.** Cumulative soil methane (CH<sub>4</sub>) flux (µg-C kg<sup>-1</sup> soil) (a) effect of residue types and soil moisture across nutrient management, (b) effect of nutrient management and soil moisture across residue treatment, and (c) effect of nutrient management and residue types across soil moisture treatment. Vertical bars represent mean ± standard error (n = 3). Different lower-case letters indicate significant differences among treatments at  $\alpha < 0.05$ .

### 3.1.2. At 60% FC Interactive Influence of Residue × Nutrient

Soil CH<sub>4</sub> flux was significantly influenced by soil moisture ( $p < 0.001$ ) (Table 1). The mean cumulative CH<sub>4</sub> flux was negative indicating methane oxidation after the end of the 87-day incubation period in all treatments at 60% FC. Similar to 80% FC, the interaction effect of the residue and nutrient was found to be significant ( $p < 0.001$ ) (Table 1). The highest impact of methane oxidation was observed in soil amended with maize residue + N100PK + biochar ( $-13.87 \mu\text{g C kg}^{-1} \text{ soil}$ ), and the lowest in wheat + N0PK ( $+12.93 \mu\text{g C kg}^{-1} \text{ soil}$ ) (Figure 1). Irrespective of nutrient management, the residue treatments followed the order of rice < maize < wheat < soil without residue < soybean. In rice, maize, and wheat residue-amended soils, the methane oxidation increased by 86, 29, and 22%, respectively, compared to soil without residue. In contrast, the soybean residue decreased methane oxidation by 11% across nutrient management. The nutrient management had an inconsistent effect on methane oxidation from soils amended with and without residue. Overall, across soils with and without residue, the nutrient effect on CH<sub>4</sub> fluxes ( $\mu\text{g C kg}^{-1} \text{ soil}$ ) followed the order N100PK + manure < N100PK < NOP0K0 < N0PK < N100PK + biochar < N150PK + biochar < N150PK. The treatments N100PK + manure, N100PK, N0PK, N100PK + biochar, N150PK + biochar, and N150PK increased the cumulative mean CH<sub>4</sub> consumption by 35.5%, 17.7%,  $-8.3\%$ ,  $-9.3\%$ ,  $-38.4\%$ , and  $-44.7\%$ , respectively, compared with the control treatment (NOP0K0), across with and without residue treatment. Similar to 80% FC, the effect of nutrient management varied with residue type and control soil without residue.



### 3.1.3. Interaction Effect

A significant influence of residue type, nutrient management, and soil moisture and their interactions on CH<sub>4</sub> fluxes was observed in the present study (Tables 1 and S1). The cumulative mean CH<sub>4</sub> flux was the highest in the treatment receiving wheat residue +80% FC + N0PK (+12.93 μg C kg<sup>-1</sup> soil) and the lowest in rice residue + 60% FC + N100PK (−19.24 μg C kg<sup>-1</sup> soil). On decreasing the soil moisture from 80% FC to 60% FC, the CH<sub>4</sub> fluxes decreased by −1.76 times, indicating CH<sub>4</sub> consumption across residue types and nutrient management. Across nutrients and soil moisture, residue application decreased the methane fluxes (μg C kg<sup>-1</sup> soil) in the order soil without residue (+1.61) > wheat (1.03) > soybean (−0.65) > maize (−3.48) > rice (−3.84). The increasing effect of different nutrient management across residue and soil moisture on CH<sub>4</sub> fluxes was N150PK > N0PK > N150PK + biochar > N0P0K0 > N100PK + manure > N100PK + biochar > N100PK. Overall, the nutrient treatments N100PK + manure, N100PK + biochar, and N100PK decreased methane fluxes by 20%, 39%, and 115%, respectively, compared to those without nutrients (N0P0K0), and N150PK, N0PK, and N150PK + biochar increased emissions by 152%, 108%, and 28%, across soil moisture and residue types. The results showed that the effects of nutrient inputs on CH<sub>4</sub> emission varied significantly with different nitrogen application rates, integrated use of nutrients, crop residue types, and soil moisture.

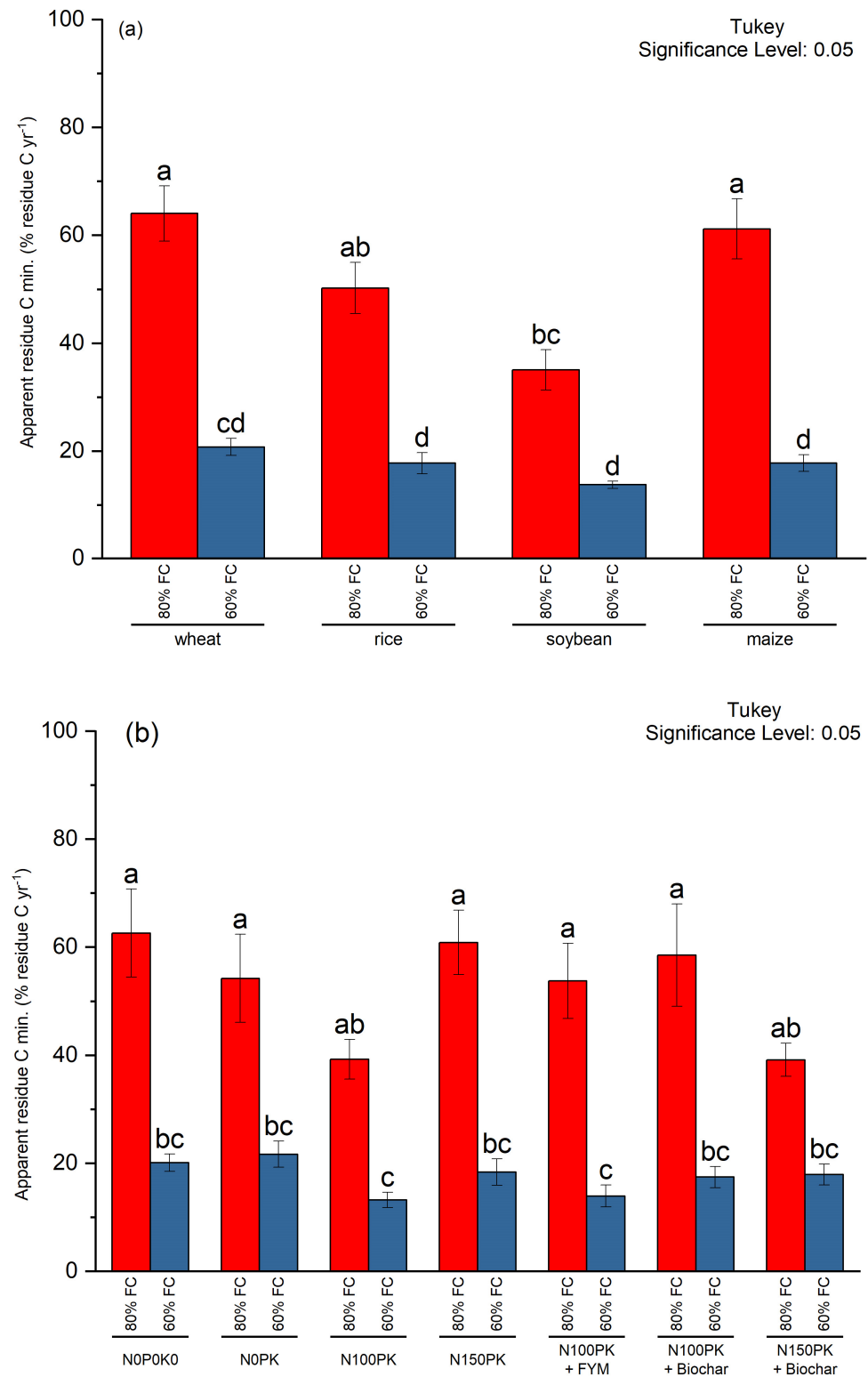
### 3.2. Apparent Residue C Mineralization

Apparent residue C mineralization (% residue C yr<sup>-1</sup>) was significantly influenced by residue type, nutrient management, soil moisture, and residue × nutrient × moisture interaction ( $p < 0.01$ ) (Table 1). The mineralization of total residue C was the highest in wheat (39–86%) and maize (40–94%), followed by rice (32–74%) and soybean (14–47%) residue, at 80% FC (Figure 2). The cumulative residue C mineralization was three times ( $p < 0.001$ ) higher in 80% FC than 60% FC soil moisture, suggesting that soil moisture affected the mineralization of residue C. Nutrient input decreased the mineralization of residue C, and the decreasing order observed was N0P0K0 > N150PK > N0PK > N100PK + biochar > N100PK + manure > N150PK + biochar > N100PK. Regardless of soil moisture, the residue C mineralization decreased by 8% (N0PK), 37% (N100PK), 4% (N150PK), 18% (N100PK + manure), 8% (N100PK + biochar), and 31% (N150PK + biochar) over N0P0K0 treatment. The treatment combination maize residue + N0P0K0 + 80% FC recorded the highest residue C mineralization (94.08%) compared with soybean + N100PK + 60% FC being the lowest (10.46%).

### 3.3. Correlation and Regression between CH<sub>4</sub> Emission, Residue C Mineralization, and Measured Variables

The partial correlation tests showed that the cumulative residue C mineralization was significantly correlated with soil and residue characteristics (Table 2). Among the soil properties, the correlation was positively robust for NH<sub>4</sub>-N, NO<sub>3</sub>-N, labile C, dehydrogenase activity, and soil moisture ( $p < 0.01$ ), and among the properties of residue, negative for lignin/TC but positive for cellulose/TC ( $p < 0.05$ ). Factors significantly influencing the residue C mineralization were chosen by the stepwise regression analysis, which showed that soil labile C, lignin, soil moisture content, and residue TC exerted powerful effects. The constant and each coefficient of variables were significant ( $p < 0.001$ ), including the R<sup>2</sup> (0.391) and adjusted R<sup>2</sup> (0.376) in Equation (1).

$$\text{Residue C mineralization (\% residue C yr}^{-1}\text{)} = -7.895 + 0.128 \text{ labile C (mg/kg)} - 2.457 \text{ lignin (\%)} + 1.155 \text{ soil moisture (\% FC)} - 1.878 \text{ residue TC (\%)} \quad (1)$$



**Figure 2.** The apparent residue C mineralization ( $\% \text{ residue C yr}^{-1}$ ) (a) effect of residue types and soil moisture across nutrient management, (b) effect of nutrient management and soil moisture across residue treatment, and the effect of nutrient management and residue types across soil moisture treatment were found to be nonsignificant; therefore, the figure is given in the Supplementary File as Figure S3. Vertical bars represent the mean  $\pm$  standard error ( $n = 3$ ). Different lower-case letters indicate significant differences among treatments at  $\alpha < 0.05$ .

**Table 2.** Pearson correlation coefficients (r) between measured variables during soil incubations and residue characteristics.

	CH <sub>4</sub>	NO <sub>3</sub>	NO <sub>2</sub>	NH <sub>4</sub>	DHA	Labile C	CO <sub>2</sub>	TC: TN	Soil Moisture	Lignin/TN	Cellulose/lignin	Cellulose/TC	Res C min	Res. TC	Res. TN	Lignin	Cellulose
CH <sub>4</sub>	1	0.083	0.160 *	0.330 **	0.143	0.222 **	0.450 **	0.270 **	0.511 **	0.274 **	−0.004	0.148	0.387 **	0.174 *	−0.254 **	0.131	0.186 *
NO <sub>3</sub>	0.083	1	0.511 **	0.270 **	0.642 **	0.276 **	0.199 **	−0.038	0.158 *	−0.029	0.047	−0.079	0.058	0.276 **	0.076	0.127	−0.026
NO <sub>2</sub>	0.160 *	0.511 **	1	0.269 **	0.599 **	0.368 **	0.437 **	0.110	0.369 **	0.028	0.016	0.171 *	0.332 **	0.182 *	−0.106	−0.077	0.190 *
NH <sub>4</sub>	0.330 **	0.270 **	0.269 **	1	0.370 **	0.722 **	0.739 **	−0.072	0.373 **	−0.054	−0.038	−0.072	0.601 **	−0.022	0.075	0.015	−0.073
DHA	0.143	0.642 **	0.599 **	0.370 **	1	0.380 **	0.410 **	0.083	0.270 **	0.098	0.005	0.039	0.298 **	−0.043	−0.088	0.028	0.035
Labile C	0.222 **	0.276 **	0.368 **	0.722 **	0.380 **	1	0.687 **	0.052	0.336 **	0.045	−0.101	0.046	0.501 **	−0.010	−0.056	−0.009	0.044
CO <sub>2</sub>	0.450 **	0.199 **	0.437 **	0.739 **	0.410 **	0.687 **	1	0.105	0.488 **	0.054	−0.041	0.156 *	0.866 **	−0.049	−0.125	−0.110	0.138
TC: TN	0.270 **	−0.038	0.110	−0.072	0.083	0.052	0.105	1	0.472 **	0.903 **	0.048	0.747 **	0.096	0.455 **	−0.989 **	0.169 *	0.828 **
Soil moisture	0.511 **	0.158 *	0.369 **	0.373 **	0.270 **	0.336 **	0.488 **	0.472 **	1	0.517 **	−0.057	0.182 *	0.332 **	0.450 **	−0.419 **	0.368 **	0.281 **
Lignin/TN	0.274 **	−0.029	0.028	−0.054	0.098	0.045	0.054	0.903 **	0.517 **	1	−0.012	0.396 **	0.031	0.425 **	−0.856 **	0.544 **	0.508 **
Cellulose/lignin	−0.004	0.047	0.016	−0.038	0.005	−0.101	−0.041	0.048	−0.057	−0.012	1	0.123	0.004	0.002	−0.062	−0.116	0.112
Cellulose/TC	0.148	−0.079	0.171 *	−0.072	0.039	0.046	0.156 *	0.747 **	0.182 *	0.396 **	0.123	1	0.186 *	0.202 **	−0.813 **	−0.517 **	0.979 **
Res C min	0.387 **	0.058	0.332 **	0.601 **	0.298 **	0.501 **	0.866 **	0.096	0.332 **	0.031	0.004	0.186 *	1	−0.155 *	−0.132	−0.185 *	0.146
Res. TC	0.174 *	0.276 **	0.182 *	−0.022	−0.043	−0.010	−0.049	0.455 **	0.450 **	0.425 **	0.002	0.202 **	−0.155 *	1	−0.346 **	0.438 **	0.390 **
Res. TN	−0.254 **	0.076	−0.106	0.075	−0.088	−0.056	−0.125	−0.989 **	−0.419 **	−0.856 **	−0.062	−0.813 **	−0.132	−0.346 **	1	−0.043	−0.865 **
Lignin	0.131	0.127	−0.077	0.015	0.028	−0.009	−0.110	0.169 *	0.368 **	0.544 **	−0.116	−0.517 **	−0.185 *	0.438 **	−0.043	1	−0.358 **
Cellulose	0.186 *	−0.026	0.190 *	−0.073	0.035	0.044	0.138	0.828 **	0.281 **	0.508 **	0.112	0.979 **	0.146	0.390 **	−0.865 **	−0.358 **	1

\*: Correlation is significant at the 0.05 level (2-tailed); \*\*: Correlation is significant at the 0.01 level (2-tailed). CH<sub>4</sub>: methane; NO<sub>3</sub>: nitrate N; NO<sub>2</sub>: nitrite N; NH<sub>4</sub>: ammoniacal N; DHA: dehydrogenase activity; CO<sub>2</sub>: carbon dioxide; Res C min: residue C mineralization; Res. TC: residue total carbon; Res. TN: residue total nitrogen.

The cumulative CH<sub>4</sub> emission was significant ( $p < 0.01$ ) and positively correlated with NH<sub>4</sub>-N, labile SOC, residue C mineralization, C: N ratio of crop residue, and lignin/N ratio of crop residue, and negatively correlated with plant total nitrogen (Table 2). The quantification of the variation partitioning analysis of the effects of studied soil and crop properties on CH<sub>4</sub> emissions was performed by the stepwise multiple regression analysis. Equation (2) describes CH<sub>4</sub> emissions as a function of studied soil and crop residue properties. The constant and each coefficient of variables were significant ( $p < 0.001$ ), including the R<sup>2</sup> (0.314) and adjusted R<sup>2</sup> (0.306) in Equation (2).

$$\text{CH}_4 (\mu\text{g C kg}^{-1} \text{ soil}) = -27.037 + 0.352 \text{ soil moisture (\% FC)} + 0.069 \text{ residue C mineralized (mg C kg}^{-1} \text{ soil)} \quad (2)$$

The regression analysis showed that the two dominant predictor variables within the studied variables were soil moisture and residue C mineralization, which estimated 31% of soil methane fluxes in Vertisols.

## 4. Discussion

### 4.1. Apparent Residue C Mineralization

The results indicate that apparent residue carbon (C) mineralization was significantly influenced by several factors, including residue type, nutrient management, soil moisture, and their interactions. The mineralization of total residue C varied among different crop residues, with wheat and maize residues showing the highest mineralization rates, followed by rice and soybean residues. Different crop residues have varying biochemical compositions, with wheat and maize residues often containing higher amounts of labile organic matter that decomposes faster than rice and soybean residues [52–54]. As a result, the higher mineralization rates observed in wheat and maize residues are attributed to their higher content of easily decomposable carbon compounds [55]. On the other hand, soybean residues typically have higher lignin content (cf. wheat, maize, and rice), which makes them more resistant to microbial degradation and, consequently, leads to slower carbon mineralization [5]. This study also found that soil moisture played a crucial role in residue C mineralization, with higher moisture levels (80% FC) leading to three times higher mineralization than lower moisture levels (60% FC). The role of soil moisture in regulating residue C mineralization is well established in the literature. Adequate soil moisture levels promote microbial activity and enhance the decomposition of organic matter, including crop residues [5,18,56,57]. Under conditions of waterlogged soils or high soil moisture, anaerobic conditions prevail, leading to a slowdown in residue decomposition and lower carbon mineralization rates [8]. The residue C mineralization was weakly correlated with residue TC: TN and total nitrogen (TN); however, it was negatively correlated to lignin/total carbon (TC) and positively with cellulose/TC at  $p < 0.05$  (Table 2). In our study, the application of nutrients (NPK/NPK + organic amendments) eliminated the nutrient stoichiometry imbalance from crop residue incorporation (cf. N0P0K0), which explains why residue TC: TN ratio/TN probably did not significantly affect the residue C mineralization. Furthermore, previous studies have indicated that the mechanism of residue C mineralization involves soluble residue C as the crucial factor during the initial stage, while lignin, cellulose, and hemicellulose are the primary drivers in the later stages [53]. Factors significantly influencing the residue C mineralization were chosen by the stepwise regression analysis, which showed that soil labile C, lignin, soil moisture content, and residue TC exerted powerful effects ( $p < 0.001$ ; Equation (1)).

Additionally, averaged across residue types and soil moisture, nutrient inputs decreased the mineralization of residue C, with the lowest mineralization observed in N100PK treatment (cf. N0P0K0). The findings align with previous studies that have reported the inconsistent influence of nutrient management on residue decomposition and carbon mineralization in agricultural soils [16,55,58,59]. The inverse relationship between nutrient inputs and residue C mineralization may be attributed to the priming effect [60]. Nutrient application, particularly nitrogen (N), can stimulate microbial activity and increase crop residues and soil organic matter decomposition. However, in the presence of an abundant

external carbon source (crop residue in this case), the microbes preferentially utilize the labile carbon from the residue, leading to a reduced decomposition of native soil organic matter [60]. Nutrient (NPK alone or in combination with organic amendments) application would retard the mineralization of crop residues when soil nutrient enrichment satisfies the microbial N and nutrient demand and thereby decreases the need for microbes to decompose crop residue for obtaining N and nutrients [61,62].

#### 4.2. Methane Fluxes

The results of this study clearly indicate a significant variation in soil methane fluxes based on residue types and nutrient management, aligning with prior research that has underscored the importance of these factors in greenhouse gas emissions from soils [14,15]. The negative cumulative mean CH<sub>4</sub> flux in soil amended with maize residue ( $-1.87 \mu\text{g C kg}^{-1} \text{ soil}$ ) is particularly noteworthy, as it suggests a methane oxidation process, turning the soil into a CH<sub>4</sub> sink rather than a source. This finding is consistent with previous research that has shown that different crop residues can have varying effects on methane emissions from soils [4,7,9,10,30]. Studies have reported that the type of crop residue added to the soil can influence methane emissions due to differences in the composition and decomposition rates of the residues. For example, rice residues are known to be a significant source of methane emissions due to their high carbon content and the presence of easily decomposable organic matter, which facilitates methanogenesis in flooded paddy soils [1]. Rice straw application significantly increased seasonal CH<sub>4</sub> flux by an average of 28–122% over no straw [13]. On the other hand, maize residues typically have a higher lignin content, resulting in slower decomposition and lower methane production [53,63]. Further crop residues provide a source of carbon for methanogenic microbes, which are microorganisms that produce methane. The availability of fresh organic carbon from crop residues can stimulate the growth and activity of these methanogenic microbes, leading to increased methane production [13,22,24], thereby accelerating soil C and N cycling. In contrast, wheat straw return and soil warming in northern China Plain increased the CH<sub>4</sub> uptake due to reduced decomposition and mineralization from soil warming [22].

The mixed responses of nutrient application on CH<sub>4</sub> fluxes highlight the complexity of these interactions, necessitating a nuanced approach to nutrient management in agroecosystems. For instance, the increase in CH<sub>4</sub> emissions with N100PK + manure, N150PK, and N0PK treatments could be due to the enhanced availability of labile organic carbon, stimulating methanogenic microbes [30]. In contrast, the decrease in CH<sub>4</sub> emissions with N100PK, N100PK + biochar, and N150PK + biochar treatments could be attributed to the stimulation of methane-oxidizing bacteria or changes in soil physicochemical properties that suppress methane production [12,16,64]. On the other hand, the nutrient management treatments show a clear trend, with N0PK inducing the highest CH<sub>4</sub> emissions, which could be associated with its potential to stimulate methanogenic microbial activities or suppress methane-oxidizing bacteria [8,55,65]. For instance, the addition of biochar has been reported to increase soil porosity, enhance soil microbial activity, and thus potentially augment CH<sub>4</sub> oxidation [21,43,66,67]. In contrast, high nitrogen rates have often been linked to higher CH<sub>4</sub> emissions, as excessive nitrogen can inhibit methane oxidation. Further, nitrogen (N) fertilization, in particular, has been shown to stimulate methane production in soil. Nitrogen fertilizer application can enhance microbial activity and organic matter decomposition, promote anoxic conditions, and be favorable for methanogens, leading to higher methane emissions [14]. However, the effect of other nutrients like phosphorus (P) and potassium (K) on methane emissions is less consistent and may vary depending on soil conditions and microbial activity [30,68]. Similar to our results, Yang et al. [55] reported that N fertilization significantly increased cumulative CH<sub>4</sub> emissions from maize straw incorporation during the spring season, and cumulative CH<sub>4</sub> absorption decreased with a higher N fertilization rate in autumn in dual maize cropping in China. The integration of biochar with N100PK significantly decreased CH<sub>4</sub> emissions, which could be due to enhanced porosity and nutrient availability, fostering methanotrophic activity. This reinforces

the argument that biochar application can significantly enhance methane oxidation in agricultural soils [15,24,30,67]. In contrast, the application of manure with N100PK increased CH<sub>4</sub> emissions, which might be due to increased organic matter providing substrates for methanogenesis [31,38].

The distinct influence of soil moisture on CH<sub>4</sub> flux is in line with the existing literature, as soil moisture levels are well known to significantly impact methane emissions and oxidation in soils [18,32,36,69–72]. The observed decrease in CH<sub>4</sub> flux during the incubation period suggested enhanced methane oxidation at lower soil moisture levels. This aligns with previous studies that have reported increased CH<sub>4</sub> oxidation in drier soil conditions [18,32,73–75]. The significant impact of residue and nutrient interaction at 80% and 60% FC further highlights the complexity of these relationships. The highest cumulative methane consumption in soil amended with rice residue + N100PK at 60% FC (−19.24 μg C kg<sup>−1</sup> soil) was comparable to maize residue + N100PK + biochar (−13.87 μg C kg<sup>−1</sup> soil) and is a promising result, indicating the potential of these combinations in mitigating CH<sub>4</sub> emissions from soils. This result highlights the effectiveness of these combinations in mitigating methane emissions from soils, demonstrating a promising strategy for reducing greenhouse gas emissions in agricultural practices. Soil moisture significantly influences methane flux optimal moisture levels (e.g., 60% FC) with the addition of nutrients (N100PK)/N100PK + biochar along with maize and rice residue possibly having supported methanotrophic bacteria while preventing conditions that favor methanogenesis [30]. In contrast, the highest cumulative CH<sub>4</sub> fluxes (production) were observed in treatment receiving wheat residue + N0PK at 80% FC (+12.93 μg C kg<sup>−1</sup> soil), suggesting that not all residue types contribute positively to CH<sub>4</sub> mitigation. The inconsistent effects of nutrient management on methane oxidation in soils amended with and without residue highlight the need for site-specific and residue-specific nutrient management strategies to mitigate CH<sub>4</sub> emissions effectively.

This study explored the relationship between methane (CH<sub>4</sub>) emissions and various soil and crop properties. The results revealed significant correlations between cumulative CH<sub>4</sub> emissions and specific parameters. Notably, CH<sub>4</sub> emissions were positively correlated with NH<sub>4</sub>-N (ammoniacal nitrogen), labile SOC (labile soil organic carbon), residue C mineralization, and the TC: TN ratio of crop residue. On the other hand, CH<sub>4</sub> emissions were negatively correlated with plant total nitrogen (N). The positive correlation between CH<sub>4</sub> emissions and NH<sub>4</sub>-N and labile SOC is consistent with previous research. Ammonium nitrogen is a precursor for methanogenesis, and its availability in the soil positively influences methane production by promoting the growth and activity of methanogenic microorganisms [55]. Similarly, labile SOC provides a readily available carbon source for methanogens, enhancing methane production in the soil [1]. The negative correlation between CH<sub>4</sub> emissions and plant total nitrogen is likely due to competition for nitrogen between methane-producing microbes and other heterotrophic microorganisms [1]. The positive correlation between CH<sub>4</sub> emissions and the TC: TN ratio of crop residue suggests that crop residues with higher TC: TN ratios (e.g., wheat) may contribute more to methane emissions than residues with low TC: TN ratios (e.g., rice). Residues with lower TC: TN ratios decompose more rapidly, releasing labile carbon that supports higher methane oxidation [30]. The regression analysis using Equation (2) provides insights into the relative contributions of soil moisture and residue C mineralization to CH<sub>4</sub> emissions. Soil moisture and residue C mineralization were identified as the dominant predictor variables, explaining 31% of the variation in soil methane fluxes.

While this study provides valuable insights into methane flux dynamics, the relationships between methane emissions, and various soil and crop properties, it is essential to acknowledge some limitations. This study was conducted under controlled laboratory conditions, and the results may not fully represent the complexities of methane emissions in actual field environments. Additionally, this study focused on short-term incubation experiments, and long-term field studies are needed to confirm the findings and assess the sustainability of the observed effects. However, the findings underscore the importance of

adopting sustainable soil management practices to mitigate greenhouse gas emissions and enhance soil carbon sequestration.

## 5. Conclusions

This research shows the importance of relationships between methane (CH<sub>4</sub>) fluxes, residue C mineralization, and agricultural management practices (nutrient and irrigation practices) to mitigate climate change. Key findings of this study emphasize that CH<sub>4</sub> emissions are significantly affected by residue type, nutrient management, and soil moisture levels, with rice and maize residue exhibiting the lowest CH<sub>4</sub> flux under specific nutrient practices. The effect of N100PK with biochar was found to be the best strategy for mitigating CH<sub>4</sub> emission. Additionally, soil moisture plays a pivotal role, and at lower soil moisture levels (60% FC), methane oxidation becomes evident across treatments. Residue C mineralization was significantly influenced by nutrient management and soil moisture levels. Nutrient inputs, particularly nitrogen, decreased residue C mineralization. The results of this study elucidate the intricate relationships between soil and residue characteristics, C mineralization, and methane emissions in agricultural fields. They underscore the significance of considering both intrinsic soil properties and residue quality in understanding and predicting organic matter decomposition and greenhouse gas emissions from soils. Two predictors, soil moisture and residue C mineralization, were identified in this study, and their relationships with CH<sub>4</sub> emissions are significant findings of this study, which will help develop more accurate models for mitigating greenhouse gas emissions from agricultural soils.

**Supplementary Materials:** The following supporting information can be downloaded at: <https://www.mdpi.com/article/10.3390/soilsystems8030088/s1>, Figure S1: Effect of crop residue types and nutrient management on temporal dynamics of soil methane (CH<sub>4</sub>) flux (μg-C kg<sup>-1</sup> soil day<sup>-1</sup>) at 80% FC during the incubation period of 87 days; Figure S2. Effect of crop residue types and nutrient management on temporal dynamics of soil methane (CH<sub>4</sub>) flux (μg-C kg<sup>-1</sup> soil day<sup>-1</sup>) at 60% FC during the incubation period of 87 days; Figure S3. Apparent residue C mineralization (% residue C yr<sup>-1</sup>) (c) effect of nutrient management and residue types across soil moisture treatment. Vertical bars represent the mean ± standard error (n = 3). Different lower-case letters indicate significant differences among treatments at α < 0.05; Table S1: Effect of crop residue type, nutrient management, and soil moisture on soil cumulative CH<sub>4</sub> flux (μg C/kg soil) over 87 days of incubation.

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