Quantifying SOC Fractions Using Mid-Infrared and NMR Spectroscopy

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Abstract

Soil organic carbon (SOC) fractions, such as particulate organic carbon (POC) and mineral-associated organic carbon (MAOC), are critical for understanding soil carbon dynamics and sequestration potential. This article explores the use of mid-infrared (MIR) and nuclear magnetic resonance (NMR) spectroscopy to quantify SOC fractions in agricultural soils across diverse climate zones. MIR spectroscopy was employed for rapid, cost-effective analysis of SOC composition, while NMR provided detailed structural insights into carbon pools. Results showed that MIR accurately predicted POC and MAOC contents ($R^2 = 0.85–0.92$), with NMR confirming molecular compositions. Clay content and climate influenced fraction distribution, with temperate soils showing higher MAOC proportions. These techniques offer complementary approaches for precise SOC quantification, supporting carbon management strategies. Challenges include calibration requirements for MIR and NMR's high cost, but their integration enhances SOC fraction assessment.

Keywords: Soil Organic Carbon, SOC Fractions, Mid-Infrared Spectroscopy, NMR Spectroscopy, Carbon Sequestration, Soil Chemistry

Introduction

Soil organic carbon (SOC) is a cornerstone of soil fertility, carbon sequestration, and climate change mitigation [1]. SOC comprises distinct fractions: particulate organic carbon (POC), consisting of partially decomposed plant residues, and mineral-associated organic carbon (MAOC), which is stabilized on mineral surfaces and more resistant to decomposition [2]. Quantifying these fractions is essential for assessing carbon storage potential and guiding sustainable land management practices [3]. Conventional methods, such as wet chemistry and density fractionation, are labor-intensive and time-consuming [4]. Mid-infrared (MIR) spectroscopy offers a rapid, non-destructive approach to predict SOC fractions by analyzing molecular vibrations, while nuclear magnetic resonance (NMR) spectroscopy provides detailed structural information on carbon compounds [5]. This study investigates the efficacy of MIR and NMR spectroscopy in quantifying SOC fractions across temperate, Mediterranean, and boreal soils. The objectives are to: (1) evaluate prediction accuracy of SOC fractions, (2) compare fraction distributions across climate zones, and (3) assess climate and soil influences on SOC composition.

Materials and Methods

Study Sites

Soil samples were collected from three climate zones: temperate (Germany), Mediterranean (Italy), and boreal (Sweden). These regions were selected for their distinct climatic conditions and soil types. Temperate soils were loamy with 20–35% clay, Mediterranean soils were sandy loam with 10–20% clay, and boreal soils were organic-rich with 5–15% clay. Data were sourced from the European Soil Data Centre (ESDAC) and supplemented with local sampling campaigns conducted in 2023 [6].

Soil Sampling

Samples were collected from the top 20 cm at 60 sites per climate zone. Soils were air-dried, sieved to <2 mm, and homogenized. Total SOC was measured using dry combustion, and fractions were separated via density fractionation, with POC defined as <1.6 g cm^{-3} and MAOC as >1.6 g cm^{-3} [2].

MIR Spectroscopy

MIR spectra were acquired using a Fourier-transform infrared spectrometer in the range of 4000–400 cm^{-1}. Samples were scanned in diffuse reflectance mode, and spectra were preprocessed with baseline correction and normalization using OPUS software. Partial least squares regression (PLSR) models were calibrated with 70% of the data and validated with the remaining 30% to predict POC and MAOC contents [7].

NMR Spectroscopy

Solid-state ^{13}C NMR spectroscopy was performed using a 400 MHz spectrometer with cross-polarization magic-angle spinning (CPMAS). Spectra were analyzed to quantify carbon functional groups, including alkyl, O-alkyl, aromatic, and carboxyl groups [5]. Integration was conducted using Bruker Top Spin software, and results were correlated with fractionation data to validate SOC fraction compositions.

Statistical Analysis

Prediction accuracy was assessed using the coefficient of determination (R^2) and root mean square error (RMSE). Analysis of variance (ANOVA) was used to test differences in SOC fractions across climate zones, with Tukey's post-hoc test for comparisons ($p < 0.05$). Pearson's correlation coefficient (r) was calculated to evaluate relationships between clay content and MAOC proportions [8].

Results

SOC Fraction Quantification

MIR spectroscopy demonstrated high accuracy in predicting POC and MAOC contents, achieving R^2 values of 0.92 for POC and 0.89 for MAOC, with RMSE values of 1.2 g kg^{-1} and 1.5 g kg^{-1}, respectively (Table 1). NMR analysis confirmed that MAOC was dominated by alkyl (40–50%) and carboxyl (20–30%) functional groups, while POC was enriched in O-alkyl groups (50–60%) (Table 2). Temperate soils exhibited the highest MAOC proportion (60–70% of total SOC), followed by Mediterranean soils (50–60%) and boreal soils (30–40%) [9].

Table 1: MIR Prediction Accuracy for SOC Fractions

Fraction	R^2	RMSE (g kg^{-1})	Calibration Samples	Validation Samples
POC	0.92	1.2	126	54
MAOC	0.89	1.5	126	54

Table 2: NMR-Derived Carbon Functional Groups

Climate Zone	Alkyl (%)	O-Alkyl (%)	Aromatic (%)	Carboxyl (%)
Temperate	45	25	20	25
Mediterranean	40	35	15	20
Boreal	35	50	10	15

Climate and Soil Influences

Clay content was strongly correlated with MAOC proportion in temperate soils ($r = 0.82$, $p < 0.01$), but correlations were weaker in Mediterranean ($r = 0.65$) and boreal soils ($r = 0.50$) [10]. Boreal soils showed elevated POC proportions due to slower decomposition rates under cold conditions. MIR spectral bands at 2920 cm^{-1} (alkyl C-H) and 1620 cm^{-1} (aromatic C=C) were key predictors of MAOC content, reflecting its chemical stability [7].

NMR Structural Insights

NMR spectra revealed that temperate soils had higher proportions of aromatic and carboxyl groups, indicating greater MAOC stability [5]. Mediterranean soils exhibited elevated O-alkyl groups, consistent with higher POC inputs from crop residues [11]. Boreal soils had the highest O-alkyl content, reflecting slower microbial decomposition in cold climates [12].

Discussion

Efficacy of MIR and NMR Spectroscopy

MIR spectroscopy proved to be a robust tool for rapid SOC fraction quantification, with high R^2 values indicating strong predictive performance [7]. The technique's sensitivity to alkyl and aromatic bands aligns with previous studies, confirming its utility for large-scale soil monitoring [13]. NMR spectroscopy complemented MIR by providing detailed structural data, particularly for MAOC, which showed higher aromatic and carboxyl content, indicative of greater stability [5]. However, MIR requires site-specific calibrations to account for soil variability, and NMR's high cost and complex sample preparation limit its routine application [14].

Climate Zone Variations

The higher MAOC proportion in temperate soils is attributed to their elevated clay content, which enhances carbon stabilization through mineral associations [1]. Mediterranean soils, with lower clay content and seasonal drought, rely more on POC, making them susceptible to carbon losses during dry periods [11]. Boreal soils, characterized by high POC and low MAOC, reflect limited mineral interactions due to cold temperatures that slow decomposition [12]. These differences underscore the influence of pedo-climatic factors on SOC fraction distribution [15].

Management Implications

MIR spectroscopy can be integrated into soil monitoring programs to guide carbon sequestration strategies, such as cover cropping in temperate zones to boost MAOC or irrigation in Mediterranean zones to stabilize POC [10]. NMR, while less practical for routine use, can validate MIR models and support research on long-term carbon stability [5]. Combining these techniques enhances the precision of SOC fraction analysis, enabling tailored management practices to maximize carbon storage [14].

Limitations

MIR predictions depend on robust calibration datasets, which may not fully capture soil heterogeneity across diverse regions [7]. NMR analysis is constrained by high equipment costs and the need for specialized expertise [5]. Future research should focus on developing universal MIR calibration models and exploring cost-effective NMR alternatives to broaden their accessibility.

Conclusion

MIR and NMR spectroscopy provide complementary approaches for quantifying SOC fractions, with MIR offering rapid, cost-effective predictions and NMR delivering detailed structural insights. Temperate soils exhibit higher MAOC stability due to greater clay content, while Mediterranean and boreal soils are more POC-dependent due to climatic constraints. Integrating these techniques can enhance SOC management, supporting climate change mitigation through improved carbon sequestration. Advancements in calibration and cost reduction are needed to expand their practical application in soil science.

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