

Tillage-residues affect mineral-associated organic matter on Vertisols in northern Mexico

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ABSTRACT

Managing croplands for increased storage of soil organic matter (SOM) contributes to the development of resilient farming systems in a changing climate. We examined SOM dynamics in a wheat (*Triticum durum* L.) – maize (*Zea mays* L.) irrigated bed planting system established near Ciudad Obregón, Sonora, Mexico. Soil samples (0–15 cm) were collected from conventionally tilled raised beds (CTB) with all crop residues incorporated (CTB-I) and permanent raised beds (PB) with crop residues burned (PB-B), removed (PB-R), partly retained (PB-P) or fully retained (PB-K) receiving 0, 150 or 300 kg N ha⁻¹, and analyzed for organic C (OC), total N (TN) and $\delta^{13}\text{C}$ in whole-soil, light fraction (LF) and coarse- (sand) and fine- (silt and clay) mineral-associated organic matter (MAOM). Results indicated that PB-K and PB-B increased soil OC ($p < 0.05$) in whole-soil relative to CTB-I, mainly through increases in sand- and silt-size MAOM, respectively. Similarly, N-fertilization increased soil OC and TN contents in whole-soil, coarse-MAOM and fine-MAOM, but not in the LF pool. Soil $\delta^{13}\text{C}$ was higher ($p < 0.05$) in PB-K (–20.18‰) relative to PB-B (–20.67‰), possibly due to the stabilization of partly decomposed maize-C in silt- and clay-size MAOM. Composition of SOM surveyed by CPMAS ^{13}C NMR was not affected by tillage-residue management, and roughly consisted of 35% O-alkyl-C, 31% alkyl-C, 24% aromatic-C and 10% carboxyl-C. Our results indicate that long-term PB-K and PB-B adoption increased surface soil OC contents relative to CTB-I, even though pathways of SOM stabilization differed between systems. Under PB-K, accumulation of fine-MAOM was mostly related to straw-C inputs, whereas in PB-B it was closely associated to black-C precursors. Fine-MAOM appeared responsive to crop residue management, and should be therefore considered when analyzing mechanisms of SOM stabilization in irrigated croplands.

1. Introduction

Vertisols occupy 8.3% of Mexico's territory, including the Yaqui Valley, an intensively cultivated area of nearly 250,000 ha devoted to irrigated crop production (Balbontín et al., 2009; McCullough and Matson, 2016). Most farmers cultivate durum wheat (*Triticum durum* L.), which is sown on top of raised-beds or ridges, furrow-irrigated and fertilized with rates >250 kg N ha⁻¹ (Limon-Ortega et al., 2000; Verhulst et al., 2011a). Under this regime, crop residues are typically incorporated (0–20 cm) by conventional tillage (CT), i.e., disk

ploughing, which forms new raised-beds (CTB) each season (Sayre et al., 2005; Erenstein et al., 2012). Widespread adoption of CTB facilitated weed, fertilizer and irrigation management, but also exposes local Vertisols, which are prone to rapid degradation, to severe wind and water erosion (Limon-Ortega et al., 2000; Sayre et al., 2005).

Conservation agriculture (CA) in the form of permanent-bed (PB) planting has emerged as a sustainable option for irrigated crop production in northern Mexico (McCullough and Matson, 2016). Fields under PB are not tilled, i.e., zero tillage (ZT), on the surface which retains crop residues after harvest (Verhulst et al., 2011b). Mechanical

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disturbance is limited to the furrows, which are only re-shaped as needed between crop cycles (Fonteyne et al., 2019). Transitioning from CTB to PB planting can result in several agronomic, socio-economic and ecological benefits, including (i) higher grain yields, (ii) more diverse cropping systems, (iii) improved weed management, (iv) reduced soil erosion and degradation and (v) greater farm net returns (Sayre et al., 2005; Verhulst et al., 2011a; Fonteyne et al., 2019, 2021). Reductions in tillage operations can also improve soil physical, chemical and biological attributes, e.g., aggregate stability, infiltration and microbial biomass-carbon (C), while reducing soil salinity and sodicity (Limon-Ortega et al., 2006; Verhulst et al., 2011b). Others report that residue retention often augments soil organic matter (SOM) by increasing plant-C inputs and slowing microbial-C decomposition (Limon-Ortega et al., 2006; Govaerts et al., 2009).

SOM stability can be evaluated from the quantity and quality of different soil organic C (OC) pools by combining density and particle-size separation schemes (Maillard et al., 2015; Samson et al., 2020a). This approach distinguishes a light fraction (LF) of SOM from a heavy one (HF) that is recovered as sand-, silt-, and clay-size SOM utilizing sodium iodide (NaI) as a heavy liquid (Sohi et al., 2001). Maillard et al. (2015) showed that the LF ($>53 \mu\text{m}$ and $<1.80 \text{ g cm}^{-3}$) is comprised of partly-decomposed plant residues, whereas the sand-size HF resembles particulate organic matter (POM; $>53 \mu\text{m}$ and $>1.80 \text{ g cm}^{-3}$) coating coarse-minerals, i.e., coarse-size mineral-associated organic matter (coarse-MAOM). Accordingly, the LF is closely related to carbohydrate- and aliphatic-like SOM (St. Luce et al., 2011), whereas the HF is mainly comprised of recalcitrant SOM, e.g., alkyl-C (Gregorich et al., 2006), making it a precursor of stabilized SOM (Samson et al., 2020a). Once degraded by soil microbes, by-products of LF and sand-size HF become progressively stabilized as clay- and silt-size SOM, i.e., fine-size mineral-associated organic matter (fine-MAOM; $<53 \mu\text{m}$ and $>1.80 \text{ g cm}^{-3}$) (Lehmann and Kleber, 2015) at C/N ratios ~ 10 (Gregorich et al., 2006; Maillard et al., 2015).

The response of density- and particle-size SOM fractions to agricultural management has been extensively studied in Vertisols as proxies of soil quality (Novelli et al., 2011; Awale et al., 2013; Somasundaram et al., 2017), providing insight on bulk-level C dynamics (Novelli et al., 2011) and soil aggregation (Bravo-Garza et al., 2010). SOM stability may be further investigated by comparing soil OC concentrations with $\delta^{13}\text{C}$ signatures (Balasubramanian et al., 2020). Quantifying natural $\delta^{13}\text{C}$ abundance in soil helps to understand SOM sources, from C_3 or C_4 plants, and may reveal pathways for increased SOM stability after changes in aboveground-C fluxes (Ellert and Janzen, 2006; Balasubramanian et al., 2020). Atere et al. (2020), for example, utilized natural $\delta^{13}\text{C}$ abundance to demonstrate that plant-C, once incorporated into the soil, translocates progressively from coarse- to fine-MAOM. Cotrufo et al. (2015) suggested that coarse plant-C fragments are physically incorporated into the soil matrix as LF pools. These fragments are then progressively converted into coarse-MAOM once they become microbially enriched, resulting in the formation of stable fine-MAOM (Cotrufo et al., 2013, 2015). Little is known, however, about the evolution of MAOM precursors in soil, particularly in croplands influenced by long-term irrigation (Dong et al., 2018). Understanding changes in SOM fractions as proxies of soil OC accumulation is needed to predict the sustainability of irrigated bed planting systems in northern Mexico.

Techniques commonly applied to speciate SOM by functional groups include ^{13}C nuclear magnetic resonance (NMR) and infrared (IR) spectroscopies (Simpson and Preston, 2008; Margenot et al., 2017a). ^{13}C NMR has been widely employed to study SOM responses to cropping systems and fertilization regimes, with most reports relying on the standard, cross-polarization (CP) with magic angle spinning (MAS) sequence (Preston et al., 1994; Dieckow et al., 2005; Boeni et al., 2014). This technique has some known limitations, including non-quantitative signal intensities and ambiguous peak assignments (Mao et al., 2007). By contrast, direct-polarization/MAS (DPMAS) and multiple cross-polarization/MAS (multiCP/MAS) can quantitatively describe SOM

mixtures (Mao et al., 2007). Yet, the acquisition of such ^{13}C NMR spectra is still not routine. Only a few authors applied DPMAS and multiCP to typify SOM (Mao et al., 2000, 2008; Li et al., 2021), and we are not aware of any multiCP studies quantifying individual SOM peaks/bands by spectral deconvolution (Mao et al., 2000). Comprehensive research utilizing new ^{13}C NMR techniques might yield more in-depth insight into SOM chemical stability (Mao et al., 2007) and cycling in agro-ecosystems (Li et al., 2021).

We examined SOM fractions and bulk-level SOM composition in a furrow-irrigated, long-term trial started by the International Maize and Wheat Improvement Center (CIMMYT) in 1992 at its experimental station in the Yaqui Valley, Sonora, Mexico. Treatments ranged from CTB with all crop residues incorporated (CTB-I), to PB with crop residues burned (PB-B), removed (PB-R), and partly (PB-P) or fully retained (PB-K) (Verhulst et al., 2011a). We hypothesized that (i) PB-K would increase plant-derived SOM relative to CTB-I, PB-B and PB-R systems, (ii) such changes would stabilize soil C within the fine-MAOM fraction and (iii) the influence of tillage-residue management is attenuated from fine-MAOM to LF pools.

2. Materials and methods

2.1. Experimental site and study design

The study was conducted at the Campo Experimental Norman E. Borlaug (CENEB) near Ciudad Obregón, Sonora, Mexico ($27^{\circ}33'\text{N}$, $109^{\circ}09'\text{W}$; elevation 38 m). The location has an arid subtropical climate with a mean annual temperature of 23.7°C and average precipitation of 311 mm. Rainfall is summer dominant, with about 47 mm falling during the wheat growing season, i.e., November – May (1991–2019). The soil is a Vertisol, classified as a fine, smectitic Chromic Haplotorrerts (USDA Soil Taxonomy; Soil Survey Staff, 2003) or Hyposodic Vertisol (Calcic, Chromic) (IUSS Working Group WRB, 2006); generally deep with moderate to slow permeability, and derived from fluvial or lacustrine deposits (Verhulst et al., 2009). Selected soil properties (0–15 cm layer) were: pH (H_2O , 1:2): 8.2, cation exchange capacity (CEC): 33.9 meq 100 g^{-1} , total nitrogen (TN): 0.40 g kg^{-1} , SOM: 12.0 g kg^{-1} , and sand, silt and clay content: 320, 180 and 500 g kg^{-1} , respectively (Verhulst et al., 2009).

The field trial consisted of five irrigated tillage-residue management systems replicated three times in a randomized complete block design with a split-plot treatment arrangement, previously described by Verhulst et al. (2011a, 2011b) (Supplementary Fig. S1). The winter crop (wheat (*Triticum durum* L.), cv. Bayoreca Oro, was planted in late November to early December and harvested in May, followed by maize (*Zea mays* L.), cv. P4082W, planted in June and harvested in October. Both crops were grown on 0.75 m wide raised-beds; wheat was sown in two rows (0.24 m apart), while maize was planted in one central row (Supplementary Fig. S2). Main treatments consisted of tillage-residue management systems as follows:

- I. *Conventionally tilled raised beds – residues incorporated* (CTB-I), wheat and maize residues are incorporated (0–20 cm) by disk ploughing after harvest, new beds are formed after each crop;
- II. *Permanent raised beds – residues burned* (PB-B), residues of wheat and maize are burned, permanent beds are reshaped in the furrows after each harvest, while the tops of the beds are not tilled;
- III. *Permanent raised beds – residues removed* (PB-R), residues of wheat that pass through the combine are removed, standing wheat stubble is retained, residues of maize are baled-off for fodder;
- IV. *Permanent raised beds – residues partly retained* (PB-P), residues of maize are baled-off for fodder, residues of wheat are chopped and retained on the soil surface;
- V. *Permanent raised beds – residues fully retained* (PB-K), residues of maize and wheat are chopped and retained on the soil surface.

The split plots had different wheat N-fertilizer rates, of which the basal applications of urea-N at 0, 150 and 300 kg N ha⁻¹ disked on top of the bed, were selected for this study. Maize received a uniform application of 103 kg N ha⁻¹, except in the 0 kg N ha⁻¹ plots. Each year, wheat and maize received 46 and 52 kg of P₂O₅ ha⁻¹, respectively, which was banded in the furrow and incorporated through cultivation when reshaping beds. Split plots, 8-beds wide, measured 6 m × 13 m (Verhulst et al., 2011a).

2.2. Soil sampling and whole-soil chemical analysis

Soil sampling ($n = 45$) occurred in March 2019, approximately three weeks after wheat anthesis, using a 2 cm diameter auger. Eight cores were taken from the 0–15 cm soil layer of each plot, avoiding the central two beds and the border area, and then pooled into a composite sample. All samples were crumbled, sieved at 6.3 mm, and air-dried for 1 week at room temperature. Subsamples of soil were ball-milled to pass through a 0.15 mm mesh. Finely ground samples were acidified with 75 μ L of 6 M HCl to remove carbonate-bound C and analyzed for OC, TN and ¹³C/¹²C isotopic composition ($\delta^{13}\text{C} \text{ ‰}$) using a GC-MS CN analyzer (NC2100, Carlo Erba Instruments, Milan, Italy). Soil $\delta^{13}\text{C}$ was calculated as follows:

$$\delta^{13}\text{C} \text{ ‰} = \left[\left(\frac{R_{\text{sample}}}{R_{\text{standard}}} \right) - 1 \right] \times 1000 \quad (1)$$

where R_{sample} is the ratio of ¹³C/¹²C in the sample and R_{standard} is the ratio of ¹³C/¹²C in the international Pee Dee Belemnite standard. The reproducibility of the analytical procedure was checked with an internal soil reference ($\delta^{13}\text{C} - 21.88 \text{ ‰}$) with a variation of <0.10‰.

2.3. Physical fractionation of SOM

Fractions of SOM were obtained from whole-soil samples using a combined density and particle size fractionation method adapted from Samson et al. (2020a) (Fig. 1). Air-dried soil (25 g) was added to a 250 mL centrifuge bottle with 100 mL of NaI (specific gravity = 1.7 g cm⁻³) and sonicated for 4 min at 450 J g⁻¹ soil, corresponding to a total of

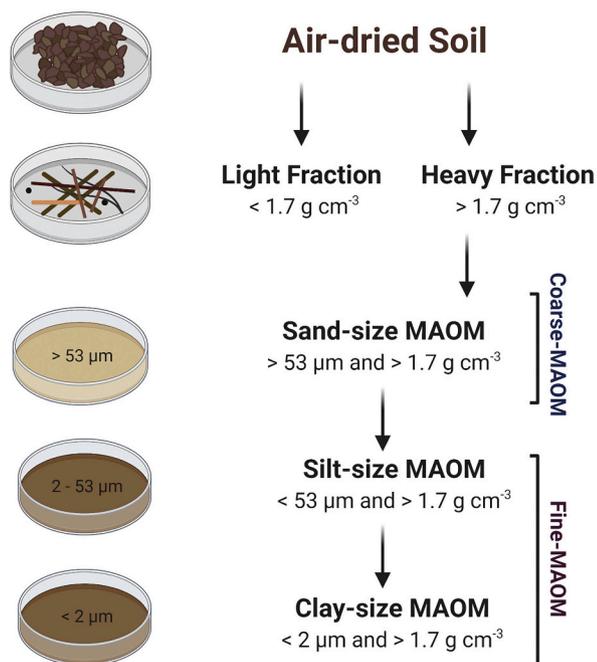


Fig. 1. Scheme of soil organic matter (SOM) fractionation adapted from Samson et al. (2020a). MAOM, mineral associated organic matter. This figure was created using the website <https://app.biorender.com>.

11,250 J. The mixture was then centrifuged at 4100 ×g for 45 min at room temperature (25 °C). The floating LF was vacuum filtered through a 0.45 μ m nylon membrane filter (Sartorius AG, Göttingen, Germany) and then rinsed multiple times with ultrapure Milli-Q® water ($\leq 18.2 \text{ M } \Omega \text{ cm}^{-1}$) to remove residual NaI. The material retained in the centrifuge bottle, i.e., HF of SOM, was augmented with 100 mL of ultrapure Milli-Q® water and 10 glass beads (6 mm) and centrifuged at 15,000 ×g for 10 min at room temperature. The supernatant was then removed by vacuum aspiration. The process was repeated three times to eliminate residual NaI. Subsequently, 100 mL of ultrapure Milli-Q® water was added to the HF, which was then shaken (120 strokes min⁻¹) for 30 min. The soil suspension was passed through a 53 μ m sieve to separate the sand-size HF or coarse-MAOM from smaller particles. The draining fraction (fine-MAOM) was transferred to centrifuge bottles and centrifuged at 285 ×g for 2 min. The top portion of the supernatant, i.e., the clay-size MAOM (<2 μ m), was then removed by vacuum aspiration. The material retained in the centrifuge bottle was considered the silt-size MAOM (2–53 μ m). The process was repeated two times to clear the supernatant after centrifugation. Finally, the clay-size MAOM, recovered in the previous step, was precipitated by centrifugation at 15,000 ×g for 10 min. All material was oven-dried at 55 °C for 24 h and weighed to determine the percentage mass distribution of each SOM fraction within the whole-soil sample. Samples were manually ground and analyzed for soil OC, TN, and $\delta^{13}\text{C} \text{ ‰}$ as described in Section 2.2. To express the content of soil OC and TN of each SOM fraction on a whole-soil basis (g kg⁻¹ soil), the concentration of soil OC and TN in the LF, sand-size MAOM, and silt- and clay-size MAOM (g kg⁻¹ fraction) was multiplied by the mass recovered for each fraction, then divided by the mass of the original whole-soil sample. The reference to “fine-MAOM” later in the text refers to soil OC or TN found in the silt- and clay-size MAOM fractions.

2.4. Solid-state CPMAS ¹³C NMR spectroscopy

Solid-state ¹³C NMR spectroscopy was used to characterize bulk-level SOM composition (Simpson and Preston, 2008). A subset of finely-ground soil samples from CTB-I, PB-B and PB-K fertilized at 300 kg N ha⁻¹ was prepared for solid-state cross polarization with magic angle spinning (CPMAS) ¹³C NMR analysis. All the experiments were carried out at room temperature (25 °C), with powdered samples filled into 4 mm zirconia rotors equipped with Kel-F caps. All measurements were performed on a 500 MHz Bruker Avance III HD NMR Spectrometer (Bruker Corporation, Billerica, MA) outfitted with a Varian Chemagnetics QR T3 HFXY 4 mm MAS probe, employing an 11.05 Tesla superconducting magnet operating at Larmor frequencies of 499.93 and 125.71 MHz for ¹H and ¹³C, respectively. Adamantane ($\geq 99\% \text{ C}_{10}\text{H}_{16}$; Millipore Sigma, St. Louis, MO) was used as a secondary external reference at 1.63 and 38.5 ppm with respect to tetramethylsilane (TMS) for ¹H and ¹³C, respectively. All CPMAS ¹³C NMR spectra were acquired at a spinning speed of 14 kHz using the multiCP sequence by Johnson and Schmidt-Rohr (2014). Our multiCP experiments used a ¹H repolarization delay of 1.2 s using ramped CP, where contact time, recycle delay ¹H and ¹³C powers, and ramp range were calibrated to maximize signal to noise ratio. Each CP period consisted of an 11 pulse composite mixing sequence (150 μ s) repeated five times totaling 8250 μ s; 5 μ s ¹H and 5 μ s ¹³C 90° pulse lengths were used with 14 kHz ¹H decoupling during acquisition and the Hahn Echo delay (Duan and Schmidt-Rohr, 2017). All free-induction decay was apodized with a line broadening of 500 Hz, phase adjusted and linear spine baseline corrected. All spectra were deconvolved into 41 Lorentzian lines with linewidths of 1100 Hz in MestReNova v.12 (Mestrelab Research S.L., Santiago de Compostela, Spain), which were subdivided into four major chemical shift categories, attributed to (i) alkyl-C (0–45 ppm), (ii) O-alkyl-C (45–110 ppm), (iii) aromatic-C (110–160 ppm) and (iv) carboxyl-C (160–220 ppm) (Simpson and Preston, 2008). Peaks were assigned as outlined in Supplementary Table S1 based on “tentative” signal assignments in solid-state ¹³C NMR of plant- and soil-organic matter (OM) (Kögel-Knabner, 2002;

Keeler et al., 2006) (See Supplementary Material S1). Unassigned signal intensities were referred to as “unspecified” SOM compounds. SOM decomposability was measured as alkyl-C/O-alkyl-C (A/O-A ratio) (Bal-dock and Preston, 1995). SOM aromaticity (%) was calculated as aromatic-C/(alkyl-C + O-alkyl-C + aromatic-C) × 100 (Hatcher et al., 1981).

2.5. Statistical analysis

Statistical analysis of tillage-residue management and fertilizer-N rate effects on SOM variables was performed with a linear mixed model (LMM; Method = REML) (Di Rienzo et al., 2017) using the R v. 3.5 interface (R Core Team, 2019) of InfoStat v. 2020 (Di Rienzo et al., 2020). Tillage-residue management and fertilizer-N rate were considered as fixed effects and block as a random factor. Homogeneity of variance was verified through Levene's test. Means were compared using Fisher's LSD, where *F* values were significant at $\alpha = 0.05$. All figures were developed with SigmaPlot v.13 (Systat Software, San Jose, CA). Data for this article is available on CIMMYT's Research Data & Software Repository Network (The Dataverse Project v. 4.20) (Verhulst et al., 2020).

3. Results

3.1. Organic carbon, total N and $\delta^{13}\text{C}$ characterization of whole-soil

Organic C in whole-soil was affected by tillage-residue management ($p = 0.001$) and fertilizer-N rate ($p < 0.001$) (Table 1). Organic C was lowest in CTB-I, and highest in PB-K and PB-B systems, averaging 6.85, 7.64 and 8.31 g kg⁻¹ soil, respectively. Furthermore, it was observed that applying N-fertilizer at 150 and 300 kg N ha⁻¹ increased soil OC contents by 13.3% and 23.5% relative to the unfertilized control, respectively. Total N ranged from 0.55 to 0.93 g kg⁻¹ soil and was only affected ($p < 0.001$) by fertilizer-N rate with 300 > 150 > 0 kg N ha⁻¹. This resulted in a greater C/N ratio under PB-B, which was 14.3% higher relative to CTB-I and the other PB treatments. Soil $\delta^{13}\text{C}$ was marginally affected ($p = 0.047$) by tillage-residue management, ranging from -21.38 to -19.68‰. Retaining crop residues in PB-K enriched soil $\delta^{13}\text{C}$ by +0.36‰ and +0.49‰ relative to PB-P and PB-B systems, respectively.

3.2. Distribution and characteristics of SOM fractions in whole-soil

On average, the LF represented the smallest fraction of the initial soil mass (0.22%) and had the broadest C/N ratio (~15.0) of all SOM pools (Supplementary Table S2; Table 2). The sand-size MAOM had a slightly

lower C/N ratio (~14.6) but accounted for 31.4% of the materials recovered by this fractionation procedure (Fig. 2; Fig. 3). The majority of SOM was comprised of clay-size MAOM (35.2%) at a C/N ratio of ~8.6, while the silt-size MAOM represented 23.8% of the initial soil mass at a C/N ratio of ~11.5.

3.3. Organic carbon, total N and $\delta^{13}\text{C}$ characterization of the light fraction

Neither tillage-residue management nor fertilizer-N rate affected ($p > 0.05$) the soil OC and TN content of the LF pool (Table 2). By contrast, incorporating crop residues in CTB-I augmented the C/N ratio by 24% relative to all PB treatments. The observed LF $\delta^{13}\text{C}$ values ranged from -23.19 to 16.50‰ and were affected by tillage-residue management ($p = 0.009$) and fertilizer-N rate ($p = 0.029$). CTB-I, PB-K, and PB-R systems enriched, on average, LF $\delta^{13}\text{C}$ by +1.61‰ relative to PB-B, while adding fertilizer-N increased it by +1.02‰ relative to the unfertilized control.

3.4. Organic carbon, total N and $\delta^{13}\text{C}$ characterization of sand-, silt- and clay-size MAOM

The results of the ANOVA analysis of MAOM fractions as affected by agronomic practices are provided in Table 3. Soil OC and TN contents in sand-size MAOM were affected ($p < 0.05$) by tillage-residue management and fertilizer-N rate. On average, retaining crop residues in PB-K augmented soil OC and TN contents in sand-size MAOM by 78.4% and 81.8%, respectively, relative to PB-B, CTB-I and PB-R systems, while applying N-fertilizer augmented them by 102.6% and 83.3%, respectively, relative to the unfertilized control (Fig. 2a, b; Fig. 3a, b). Sand-size MAOM $\delta^{13}\text{C}$ values ranged from -21.16 to -17.46‰ and were only affected ($p = 0.005$) by tillage-residue management (Table 4). Planting on PB-B, PB-P and PB-K systems enriched, on average, sand-size MAOM $\delta^{13}\text{C}$ by +0.98‰ relative to CTB-I.

Tillage-residue management and fertilizer-N rate affected ($p < 0.05$) the soil OC content and the C/N ratio in the silt-size MAOM. By contrast, the TN content in this fraction was only affected ($p = 0.001$) by fertilizer-N rate with 300 = 150 > 0 kg N ha⁻¹ (Fig. 3b). Burning crop residues in PB-B augmented soil OC by 43.8% relative to CTB-I and the other PB treatments. This resulted in a greater C/N ratio under PB-B, which was 33.1% higher relative to CTB-I and the other PB treatments (Fig. 2c). Silt-size MAOM $\delta^{13}\text{C}$ values ranged from -21.53 to -19.76‰ and were affected by tillage-residue management ($p < 0.001$) (Table 4). Retaining crop residues in PB-K enriched silt-size MAOM $\delta^{13}\text{C}$ by +0.93‰ relative to PB-B.

Table 1

Tillage-residue management and fertilizer-N rate (0, 150 and 300 kg N ha⁻¹) effects on whole-soil organic carbon (OC), total nitrogen (TN), C/N ratio and $\delta^{13}\text{C}$ values. Data are mean ± SE for tillage-residue management ($n = 9$) and fertilizer-N rate ($n = 15$). CTB-I: conventionally tilled raised beds with all crop residues incorporated; PB-B: permanent raised beds with crop residues burned; PB-R: permanent raised beds with crop residues removed; PB-P: permanent raised beds with crop residues partly retained; and PB-K: permanent raised beds with crop residues fully retained.

	Levels	OC		TN		C/N ratio		$\delta^{13}\text{C}$	
		g kg ⁻¹ soil		g kg ⁻¹ soil				‰	
Tillage-Residue (M) ^d	CTB-I	6.85 ± 0.11 ^c		0.70 ± 0.02		9.74 ± 0.12 ^b		-20.42 ± 0.09 ^{ab}	
	PB-B	8.31 ± 0.57 ^a		0.74 ± 0.05		11.21 ± 0.14 ^a		-20.67 ± 0.09 ^b	
	PB-R	6.95 ± 0.33 ^{bc}		0.72 ± 0.03		9.63 ± 0.14 ^b		-20.43 ± 0.08 ^{ab}	
	PB-P	7.26 ± 0.28 ^{bc}		0.73 ± 0.03		9.90 ± 0.05 ^b		-20.54 ± 0.14 ^b	
	PB-K	7.64 ± 0.23 ^{ab}		0.77 ± 0.03		9.94 ± 0.08 ^b		-20.18 ± 0.11 ^a	
Fertilization (F)	0	6.59 ± 0.20 ^c		0.65 ± 0.02 ^c		10.12 ± 0.15		-20.38 ± 0.06	
	150	7.47 ± 0.21 ^b		0.74 ± 0.02 ^b		10.13 ± 0.17		-20.37 ± 0.09	
	300	8.14 ± 0.31 ^a		0.81 ± 0.02 ^a		10.01 ± 0.20		-20.60 ± 0.10	
ANOVA	df	<i>F</i>	<i>p</i>	<i>F</i>	<i>p</i>	<i>F</i>	<i>p</i>	<i>F</i>	<i>p</i>
M	4	5.74	0.001	1.18	0.342	33.95	<0.001	2.76	0.047
F	2	16.27	<0.001	21.96	<0.001	0.55	0.585	2.38	0.111
M × F	8	1.68	0.148	1.10	0.395	1.22	0.322	0.21	0.986

^d Means followed by a common letter within a column are not significantly different ($p < 0.05$).

Table 2

Tillage-residue management and fertilizer-N rate (0, 150 and 300 kg N ha⁻¹) effects on light fraction (LF) organic carbon (OC), total nitrogen (TN), C/N ratio and $\delta^{13}\text{C}$ values. Data are mean \pm SE for tillage-residue management ($n = 9$) and fertilizer-N rate ($n = 15$). CTB-I: conventionally tilled raised beds with all crop residues incorporated; PB-B: permanent raised beds with crop residues burned; PB-R: permanent raised beds with crop residues removed; PB-P: permanent raised beds with crop residues partly retained; and PB-K: permanent raised beds with crop residues fully retained.

	Levels	OC		TN		C/N ratio		$\delta^{13}\text{C}$	
		g kg ⁻¹ soil		g kg ⁻¹ soil				‰	
Tillage-Residue (M) ^d	CTB-I	0.73 \pm 0.06		0.04 \pm 0.00		17.75 \pm 0.95 ^a		-18.94 \pm 0.44 ^a	
	PB-B	0.66 \pm 0.13		0.04 \pm 0.01		14.24 \pm 0.57 ^b		-21.06 \pm 0.40 ^c	
	PB-R	0.88 \pm 0.17		0.07 \pm 0.01		13.63 \pm 0.38 ^b		-19.77 \pm 0.29 ^{ab}	
	PB-P	0.74 \pm 0.08		0.05 \pm 0.01		14.25 \pm 0.33 ^b		-20.22 \pm 0.50 ^{bc}	
	PB-K	1.02 \pm 0.19		0.07 \pm 0.01		15.14 \pm 0.37 ^b		-19.66 \pm 0.44 ^{ab}	
Fertilization (F)	0	0.62 \pm 0.09		0.04 \pm 0.01		14.69 \pm 0.41		-20.61 \pm 0.33 ^b	
	150	0.84 \pm 0.11		0.05 \pm 0.01		15.48 \pm 0.49		-19.65 \pm 0.26 ^a	
	300	0.96 \pm 0.11		0.07 \pm 0.01		14.83 \pm 0.76		-19.53 \pm 0.43 ^a	
ANOVA	df	F	p	F	p	F	p	F	p
M	4	1.20	0.332	1.63	0.195	8.41	<0.001	4.14	0.009
F	2	2.83	0.076	2.66	0.087	0.95	0.398	4.01	0.029
M \times F	8	0.91	0.521	0.94	0.502	0.87	0.554	1.10	0.391

^d Means followed by a common letter within a column are not significantly different ($p < 0.05$).

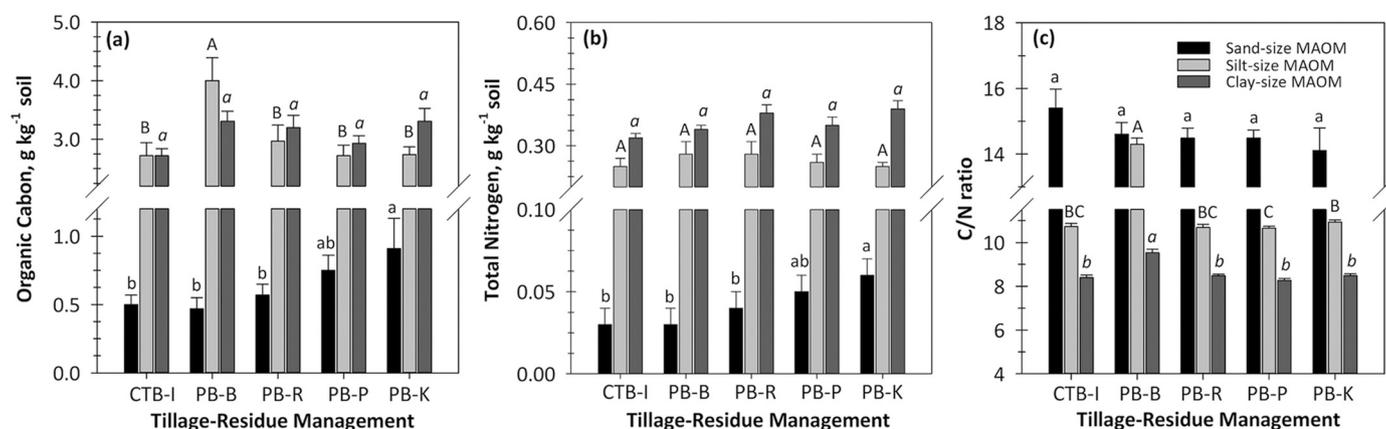


Fig. 2. Effect of tillage-residue management (CTB-I, PB-B, PB-R, PB-P and PB-K) on (a) organic carbon (OC), (b) total nitrogen (TN) concentrations (g kg⁻¹ soil) and (c) the C/N ratio of sand-, silt- and clay-size MAOM. CTB-I: conventionally tilled raised beds with all crop residues incorporated; PB-B: permanent raised beds with crop residues burned; PB-R: permanent raised beds with residue removed; PB-P: permanent raised beds with crop residues partly retained; and PB-K: permanent raised beds with crop residues fully retained. Values with different letters a - b, A - C and a - b were significantly different ($p < 0.05$) for sand-, silt- and clay-size MAOM, respectively. Vertical bars indicate the \pm standard error (SE) ($n = 9$).

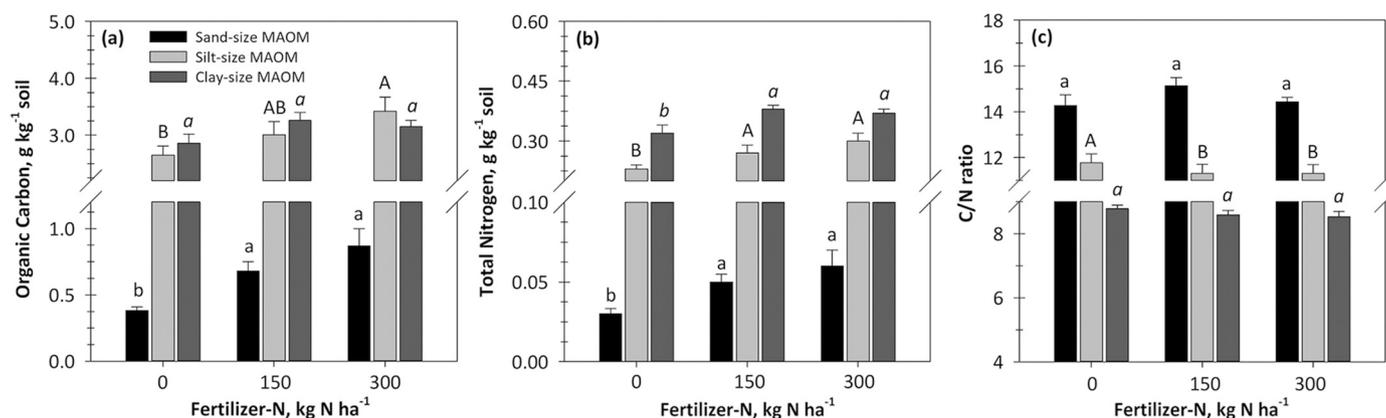


Fig. 3. Effect of fertilizer-N rate (0, 150 and 300 kg N ha⁻¹) on (a) organic carbon (OC), (b) total nitrogen (TN) concentrations (g kg⁻¹ soil) and (c) the C/N ratio of sand-, silt- and clay-size MAOM. Values with different letters a - b, A - B and a - b were significantly different ($p < 0.05$) for sand-, silt- and clay-size MAOM, respectively. Vertical bars indicate the \pm standard error (SE) ($n = 15$).

Table 3

F-statistics and p-values associated with the main effects of tillage-residue management (M) and fertilizer-N rate (F), and their two-way interaction (M × F), on sand-, silt- and clay-size MAOM organic carbon (OC), total nitrogen (TN) and C/N ratio of surface soil (0–15 cm).

Factors	df	OC		TN		C/N ratio	
		F	p	F	p	F	p
<i>Sand-size MAOM</i>							
Tillage-Residue	4	3.23	0.026	4.00	0.010	1.02	0.411
Fertilization	2	9.20	<0.001	10.06	<0.001	1.58	0.224
M × F	8	0.51	0.842	0.56	0.802	0.37	0.927
<i>Silt-size MAOM</i>							
Tillage-Residue	4	7.17	<0.001	0.66	0.625	273.78	<0.001
Fertilization	2	5.81	0.007	8.39	0.001	13.37	<0.001
M × F	8	1.20	0.334	1.30	0.283	0.98	0.475
<i>Clay-size MAOM</i>							
Tillage-Residue	4	2.43	0.071	2.53	0.062	19.66	<0.001
Fertilization	2	2.66	0.087	5.14	0.012	2.12	0.139
M × F	8	0.77	0.632	0.79	0.612	0.50	0.845

Neither tillage-residue management nor fertilizer-N rate affected ($p > 0.05$) the soil OC content in the clay-size MAOM. The TN content in this fraction ranged from 0.25 to 0.53 g kg⁻¹ soil and was only affected ($p = 0.012$) by fertilizer-N rate with 300 = 150 > 0 kg N ha⁻¹ (Fig. 3b). Similar to silt-size MAOM, the C/N ratio was higher under PB-B (~9.5) relative to CTB-I and the other PB treatments (<8.5) (Fig. 2c). Clay-size MAOM $\delta^{13}\text{C}$ values ranged from -20.64 to -18.98‰ and were affected by tillage-residue management ($p = 0.019$) and fertilizer-N rate ($p = 0.005$) (Table 4). Retaining crop residues in PB-K enriched clay-size MAOM $\delta^{13}\text{C}$ by +0.43‰ relative to PB-B. Clay-size MAOM $\delta^{13}\text{C}$ values were depleted by N-fertilization (300 kg N ha⁻¹) by -0.32‰ relative to the unfertilized control.

3.5. Solid-state CPMAS ¹³C NMR spectroscopy of whole-soil

CPMAS ¹³C NMR spectra of PB-K, PB-B and CTB-I systems are presented in Fig. 4 a, b and c, respectively. SOM composition, regardless of tillage-residue management, roughly consisted of 35% O-alkyl-C, 31% alkyl-C, 24% aromatic-C and 10% carboxyl-C (Table 5). Signals dominated the alkyl-C region at 30 and 40 ppm (lipids and cutins) and at 35 and 45 ppm (unspecified alkyl-C compounds) (Fig. 5a). Signals at 24 (lipids and

Table 4

Tillage-residue management (CTB-I, PB-B, PB-R, PB-P and PB-K) and fertilizer-N rate (0, 150 and 300 kg N ha⁻¹) effects on sand-, silt- and clay-size MAOM $\delta^{13}\text{C}$ values. Data are mean ± SE for tillage-residue management ($n = 9$) and fertilizer-N rate ($n = 15$). CTB-I: conventionally tilled raised beds with all crop residues incorporated; PB-B: permanent raised beds with crop residues burned; PB-R: permanent raised beds with residue removed; PB-P: permanent raised beds with crop residues partly retained; and PB-K: permanent raised beds with crop residues fully retained.

	Levels	$\delta^{13}\text{C}$ ‰					
		Sand-size MAOM		Silt-size MAOM		Clay-size MAOM	
Tillage-Residue (M) ^d	CTB-I	-20.33 ± 0.20 ^c		-20.39 ± 0.05 ^b		-19.61 ± 0.09 ^{ab}	
	PB-B	-19.10 ± 0.32 ^a		-20.99 ± 0.10 ^c		-19.87 ± 0.10 ^c	
	PB-R	-19.75 ± 0.16 ^{bc}		-20.42 ± 0.08 ^b		-19.70 ± 0.07 ^{bc}	
	PB-P	-19.58 ± 0.22 ^{ab}		-20.44 ± 0.09 ^b		-19.61 ± 0.08 ^{ab}	
	PB-K	-19.38 ± 0.20 ^{ab}		-20.06 ± 0.07 ^a		-19.44 ± 0.09 ^a	
Fertilization (F)	0	-19.85 ± 0.15		-20.43 ± 0.09		-19.48 ± 0.06 ^a	
	150	-19.42 ± 0.21		-20.39 ± 0.09		-19.66 ± 0.05 ^{ab}	
	300	-19.60 ± 0.23		-20.56 ± 0.11		-19.80 ± 0.08 ^b	
ANOVA	df	F	p	F	p	F	p
M	4	4.60	0.005	16.46	<0.001	3.49	0.019
F	2	1.67	0.206	1.95	0.161	6.42	0.005
M × F	8	1.50	0.202	0.40	0.910	0.21	0.986

^d Means followed by a common letter within a column are not significantly different ($p < 0.05$).

hemicellulose), 18 (unspecified alkyl-C compounds) and 12 ppm (terminal CH₃-groups) further typified alkyl-C features. Signals dominated the O-alkyl-C region at 50 (unspecified O-alkyl-C compounds) and 55 ppm (methoxyl-C) (Fig. 5b). Signals at 72/76 ppm (polysaccharides and lignin), 64, 86 and 91 ppm (polysaccharides), and 105 ppm (lignin) further typified O-alkyl-C features. Contributions from lignin peaks dominated the aromatic-C region at 122 [12% (CTB-I and PB-K) and 14% (PB-B)] and 136 ppm [12% (CTB-I and PB-B) and 8.5% (PB-K)]. The signal intensity of lignin-like OM (protonated-C) at 111 ppm was higher under CTB-I and PB-B (12%) relative to PB-K (6%) (Fig. 5c). Signals at 128, 141, 150, 154 (lignin), 145 (tannin), 116 and 131 ppm (unspecified aromatic-C) further typified aromatic-C features. Signals dominated the carboxyl-C region at 164 (lignin), 174 (esters), 180 (acids), 169 and 186 ppm (unspecified carboxyl-C compounds) (Fig. 5d). Aldehydes (204 ppm) and ketones (211 ppm) accounted for <7% of carboxyl-C features. SOM decomposability averaged 0.82, 0.95, and 0.93 under CTB-I, PB-B and PB-K, respectively (Table 5). SOM aromaticity averaged 27.3, 27.7 and 24.8% under CTB-I, PB-B and PB-K, respectively.

4. Discussion

4.1. Tillage-residue management differentially affects soil OC and TN in whole-soil

Retaining crop residues in PB-K augmented soil OC in the 0–15 cm layer relative to CTB-I. Similar results were reported by Sayre et al. (2005) and Govaerts et al. (2006) at this location 13 years after PB-K adoption. It is recognized that PB-K favors SOM formation and persistence by increasing labile-C pools and soil macroaggregation (Limon-Ortega, 2011; Erenstein et al., 2012) while reducing SOM mineralization (Limon-Ortega et al., 2006; Verachtert et al., 2009). CTB-I brings plant-C into immediate contact with soil enzymes and microbial populations (Chávez-Romero et al., 2016), presumably lowering humic-like SOM retention (Vázquez et al., 2013; Romero et al., 2017). Our results further indicated that burning crop residues in PB-B increased soil OC relative to CTB-I, PB-R and PB-P systems. This response was attributed to black-C inputs favoring the accumulation of stable, refractory SOM in PB-B after each burning, in agreement with Knicker et al. (2012) and Jiménez-González et al. (2016). Soil OC was similar between PB-B and PB-K systems, suggesting that burning crop residues may also maintain SOM levels via root-C depositions of microbial-derived C (Abril et al., 2005; Limon-Ortega et al., 2006).

By contrast, returning crop residues in PB-K did not increase soil TN contents. Mature crop residues in this system likely prompted

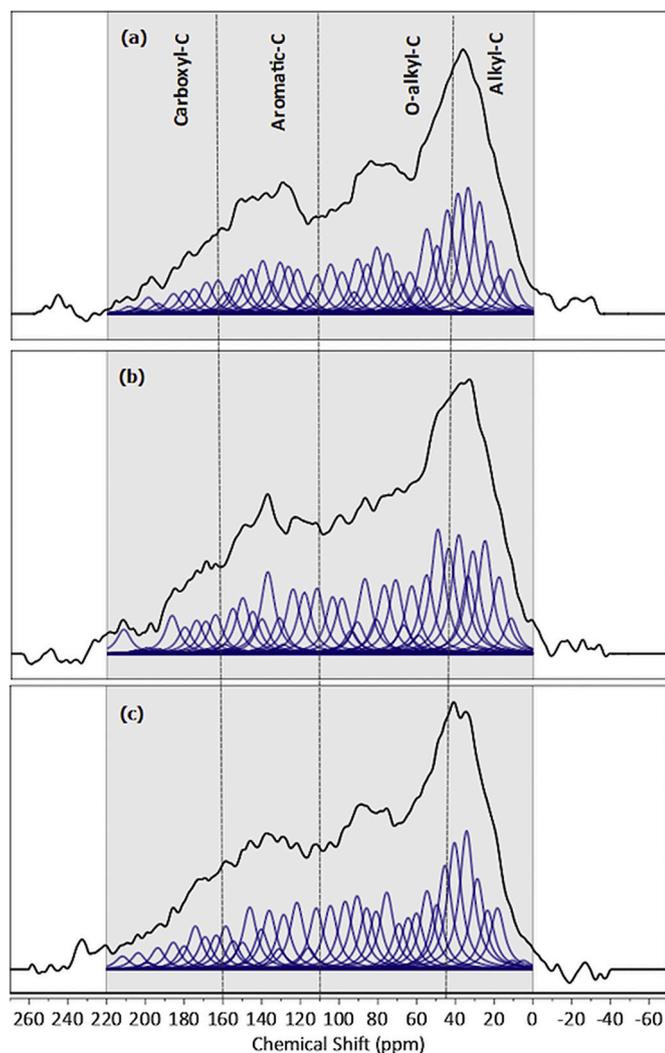


Fig. 4. Solid-state CPMAS ^{13}C NMR spectra of SOM under (a) permanent raised beds with crop residues fully retained (PB-K), (b) permanent raised beds with crop residues burned (PB-B) and (c) conventionally tilled raised beds with all crop residues incorporated (CTB-I) fertilized at 300 kg N ha^{-1} . CPMAS ^{13}C NMR spectra were deconvolved into its constituent lines (blue peaks) with a Lorentzian-Gaussian (L/G 1.00) linewidth of 1100 Hz.

microorganisms to mine indigenous SOM richer in N (Cotrufo et al., 2013), thereby limiting soil TN accumulation. This result is consistent with Conteh et al. (1998), who observed equivalent soil TN levels in irrigated Vertisols (0–30 cm) cropped to cotton (*G. hirsutum* L.) with straw-C either retained by chopping, and further incorporated, or removed by burning. Comparisons of soil C/N ratios among treatments indicated that SOM in PB-B was C-rich and N-depleted; black-C was a significant SOM component in PB-B systems, as previously discussed.

4.2. Tillage-residue management differentially affects soil OC and TN in MAOM fractions

Retaining crop residues in PB-K increased soil OC in sand-size MAOM, but not in LF, or in the silt- and clay-size MAOM fractions. This is consistent with Samson et al. (2020a), who reported that crop residue retention increased soil OC at the bulk level and in coarse-MAOM, but not within the fine-MAOM pool. Because MAOM stabilization is mainly driven by N availability (Cotrufo et al., 2013), our results indicate that straw-C makes a relatively small contribution to soil OC storage within silt- and clay-size MAOM (Kirkby et al., 2014; Samson et al., 2020a). Increased coarse-MAOM under PB-K is expected to

Table 5

Relative signal distributions (%) in the solid-state CPMAS ^{13}C NMR spectra of whole-soil (0–15 cm) under (i) conventionally tilled raised beds with all crop residues incorporated CTB-I, (ii) permanent raised beds with crop residues burned (PB-B) and (iii) permanent raised beds with crop residues fully retained (PB-K) fertilized at 300 kg N ha^{-1} . Data are mean \pm SE ($n = 2$). Multi-CP experiments were repeated twice in the same aliquot of sample.

Tillage-Residue	Chemical shift (ppm)					Aromaticity (%) ^b
	Carboxyl-C 220–160	Aromatic-C 160–110	O-alkyl-C 110–45	Alkyl-C 45–0	A/O-A ^a	
CTB – I	11.02 \pm 0.12	24.35 \pm 0.10	35.43 \pm 0.47	29.21 \pm 0.69	0.82 \pm 0.03	27.36 \pm 0.15
PB – B	9.76 \pm 0.23	25.05 \pm 0.78	33.50 \pm 1.89	31.70 \pm 1.34	0.95 \pm 0.09	27.76 \pm 0.80
PB – K	8.51 \pm 0.68	22.76 \pm 0.21	35.67 \pm 1.44	33.07 \pm 0.55	0.93 \pm 0.05	24.88 \pm 0.41

^a A/O-A, alkyl-C/O-alkyl-C ratio (Baldock and Preston, 1995).

^b Aromaticity (%), aromatic-C/(alkyl-C + O-alkyl-C + aromatic-C) \times 100 (Hatcher et al., 1981).

increase plant-derived carbohydrates, which contributes to soil macro-aggregation (Samson et al., 2020b), but also N immobilization (Lin and Chen, 2014). Recovery of N-fertilizer by maize or wheat at this location increases as crop residues are removed by PB-B, being lowest under CTB-I or PB-K systems (Grahmann et al., 2019). Burning crop residues in PB-B increased soil OC in silt-size MAOM, but not in the LF, sand- or clay-size MAOM fractions. It has been documented that fire-exposed OM, mainly black-C, accumulates as either POM or fine-MAOM (Jiménez-Morillo et al., 2016). For instance, Skjemstad et al. (1996) concluded that black-C is primarily found in soil as physically-protected fine-MAOM, constituting \sim 35% of SOM in Vertisols (Jha et al., 2020).

Overall, two different SOM stabilization mechanisms appear to be important in the studied Vertisols. SOM formed by physically transferring straw-C into coarse-MAOM, *i.e.*, litter-derived SOM (Cotrufo et al., 2015), which likely occurred to some extent in all treatments, was a dominant factor under PB-K and PB-P systems. Under this pathway, coarse-MAOM is stabilized through chemical recalcitrance or physical occlusion, particularly in clayey soils with high shrink/swell capacity (Bravo-Garza et al., 2010; Cotrufo et al., 2015). SOM formed by physically transferring black-C into fine-MAOM, particularly silt-size MAOM, was probably dominant in PB-B systems (Skjemstad et al., 1996). It is important to note, however, that fire-induced SOM losses under PB-B were likely compensated by charred straw-C contributions.

4.3. N-fertilization increased soil OC and TN in whole-soil and MAOM fractions

Fertilizing wheat with urea-N augmented soil OC and TN accumulation at the bulk level. This is consistent with Govaerts et al. (2006), who observed that, after 13 years, soil OC and TN contents at this location were 1.10-times greater in plots with 300 kg N ha^{-1} added relative to the unfertilized control. External N inputs are critical for the cycling of SOM fractions (Manna et al., 2007; Atere et al., 2020); plant-C inputs from aboveground and root biomass, as well as rhizodeposition, are frequently augmented by N-fertilization (Galantini et al., 2014; Engel et al., 2017). Therefore, when crop residues are retained, increasing N-fertilizer rates are associated with elevated soil OC contents (Dalal et al., 2011; Jha et al., 2020).

Increases in soil OC and TN contents were generally reflected in the sand-, silt- and clay-size MAOM fractions, but not in the LF pool. Even though the LF is considered a proxy of straw-C inputs, it is also characterized by fast turnover (Samson et al., 2020a). For example, Neff

et al. (2002) observed that plant-C quickly disappears with N-additions, with LF accumulating as MAOM or HF. Maillard et al. (2015) reported that N-fertilization with NH_4NO_3 at 50 and 100 kg N ha^{-1} increased tall fescue (*F. arundinacea* Schreb) yields, resulting in greater soil OC contents in the silt- and clay-size MAOM, but not in the free LF. It is also plausible that MAOM accumulation in the studied Vertisols was also favoured by greater microbial biomass-C in N-fertilized treatments (Limon-Ortega et al., 2006). Higher microbial biomass-C is expected to increase organo-mineral interactions between LF and mineral surfaces, thereby increasing MAOM accrual (Samson et al., 2020b).

4.4. Residue retention reduced whole-soil SOM and fine-MAOM decomposition degree

Soil $\delta^{13}\text{C}$ in the 0–15 cm layer was higher in PB-K relative to PB-B and PB-P, while CTB-I and PB-R systems exhibited intermediate values. Higher $\delta^{13}\text{C}$ values in PB-K implies a greater ^{13}C contribution from maize- (-13‰ ; C_4 plant) relative to wheat-derived (-27‰ ; C_3 plant) residues (O'Leary, 1988). Fuentes et al. (2010) observed higher soil $\delta^{13}\text{C}$ values (0–5 cm) under maize-only cropping (-16.56‰) relative to maize-wheat rotations (-18.93‰), while Dalal et al. (2011) reported lower ones with increasing wheat/barley straw-C inputs. Under maize-only cropping, soil $\delta^{13}\text{C}$ values are frequently lower under CT (-18.08‰) than ZT (-16.56‰); SOM is more thoroughly mineralized in CT relative to ZT management (Fuentes et al., 2010).

Silt-size MAOM was most $\delta^{13}\text{C}$ enriched under PB-K relative to other treatments, whereas clay-size MAOM exhibited higher soil $\delta^{13}\text{C}$ values

under PB-K, CTB-I and PB-P systems relative to PB-B. Maize, which typically exhibits a wider C/N ratio than wheat, likely decomposed more slowly (Govaerts et al., 2006), stabilizing within soil fractions of slow turnover, i.e., clay- and silt-size MAOM (Atere et al., 2020). Burning crop residues in PB-B also increased soil OC in fine-MAOM but at lower $\delta^{13}\text{C}$ ‰ than PB-K systems. Charred maize-derived C, with lower $\delta^{13}\text{C}$ values than SOM, likely decreased silt- and clay-size MAOM $\delta^{13}\text{C}$ enrichment. This observation is in agreement with Krull et al. (2003), who observed that C_4 -derived OM is prone to ^{13}C losses during burning, whereas C_3 -derived OM is not.

4.5. Tillage-residue management did not affect whole-soil OC composition when combined with N-fertilizer

SOM composition was dominated by O-alkyl-C and alkyl-C, followed by aromatic-C, and considerably lower carboxyl-C contributions. Generally, O-alkyl-C (labile-C) is found in significant quantities in SOM, either as plant-derived POM (Dieckow et al., 2005; Margenot et al., 2017b) or microbially-synthesized MAOM (Helfrich et al., 2006). Alkyl-C (highly recalcitrant-C) and aromatic-C (moderately recalcitrant-C) contributions are variable (Dieckow et al., 2005, 2009) and may depend on litter quality (Ding et al., 2006), but are known to increase at the expense of O-alkyl-C (Mathers and Xu, 2003) following microbial decay of POM (Gregorich et al., 1996; Miller et al., 2018). CPMAS ^{13}C NMR revealed similar structural features in CTB-I, PB-B and PB-K, indicating that tillage-residue management had a minor impact on SOM composition despite changes in soil OC contents. Theoretically,

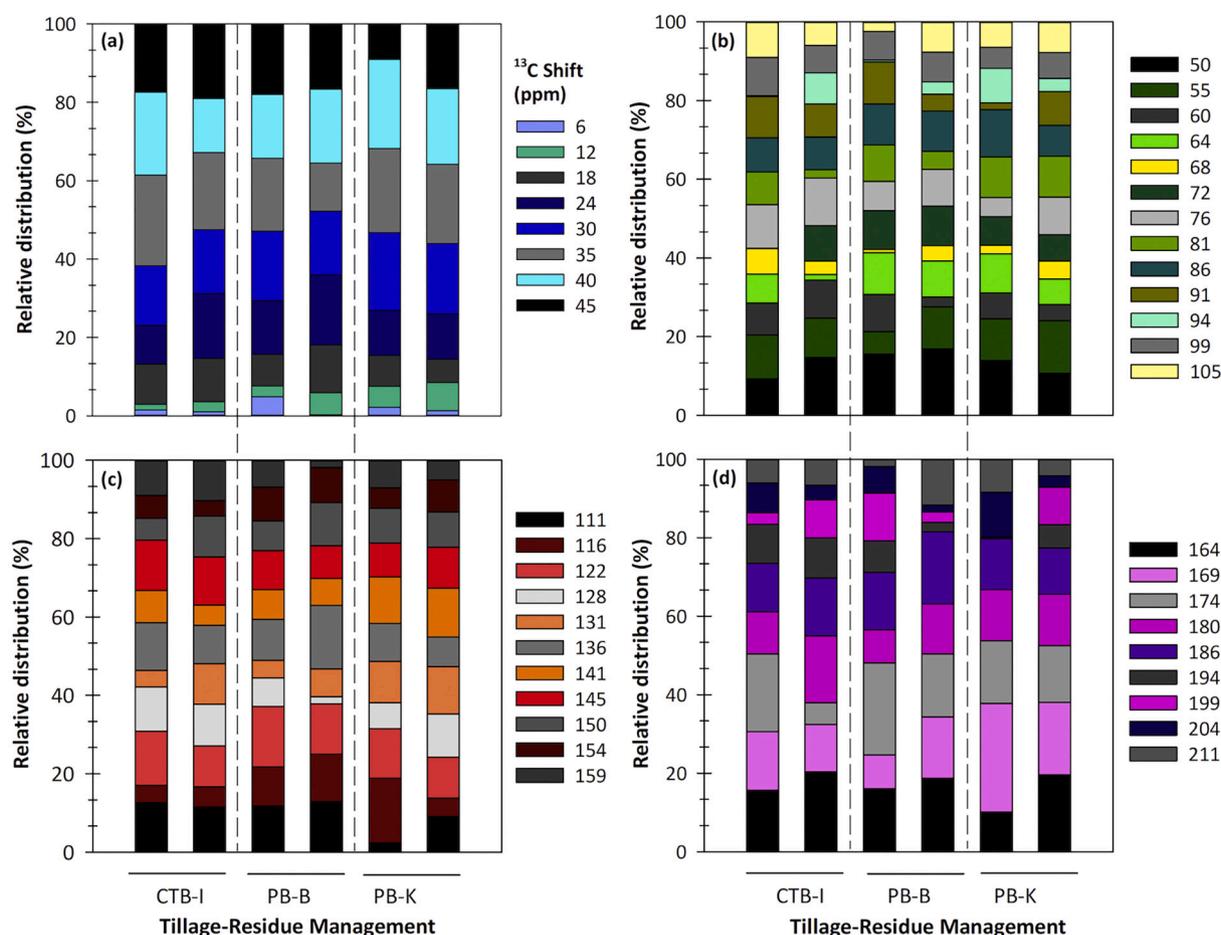


Fig. 5. Relative distribution of ^{13}C chemical shifts (ppm) among (a) alkyl-C, (b) O-alkyl-C, (c) aromatic-C and (d) carboxyl-C main resonance regions. Samples included whole-soil under (i) conventionally tilled raised beds with all crop residues incorporated (CTB-I), (ii) permanent raised beds with crop residues burned (PB-B) and (iii) permanent raised beds with crop residues fully retained (PB-K) fertilized at 300 kg N ha^{-1} . Multi-CP experiments were repeated twice in the same aliquot of sample.

SOM would exhibit a highly humified character as plant-C is either removed by harvest (Mathers and Xu, 2003), burned (Jiménez-González et al., 2016) or incorporated by CT management (Carvalho et al., 2009). Such a response is barely detectable by CPMAS ¹³C NMR, presumably due to the masking of POM chemistry changes by stabilized MAOM fractions (Dieckow et al., 2005), even under continuous burning (Knicker et al., 2012). Increased SOM stability is further expected to delay C degradation in Vertisols (Romero et al., 2019), which are profiles where soil OC is stored mainly as alkyl-C within swelling clay minerals (Leinweber et al., 1999).

The A/O-A ratio is considered an index of the extent of SOM decomposition (Baldock and Preston, 1995) as it has been demonstrated that the relative amount of alkyl-C increases as O-alkyl-C declines during biodegradation (Kögel-Knabner, 1997; Miller et al., 2018). Similarly, Mathers and Xu (2003) utilized aromaticity (%) as a proxy for SOM humification, based on the premise that SOM becomes enriched by aromatic-C during decomposition (Hatcher et al., 1981). The observed A/O-A ratios were >0.80, suggesting that SOM was in an advanced stage of decomposition (Simpson et al., 2008), even under PB-K and PB-B systems. Abe et al. (2009) reported that A/O-A ratios were not descriptive of SOM dynamics, and that A/O-A ratios in a native forest with 2.4% of total C were greater than in agricultural soils with >1.4% of total C. Aromaticity (%) was apparently lower in PB-K relative to PB-B and CTB-I. This is consistent with Dieckow et al. (2009) and may arise from the fact that SOM decomposes more slowly in PB-K than under CTB-I and PB-B systems (Verachtert et al., 2009).

5. Conclusions

Our results reinforce the view that planting on PB with crop residue retention is a suitable alternative to the conventional practice to maintain SOM levels in irrigated Vertisols of northern Mexico. By contrast, CTB-I showed lower amounts of soil OC than PB-K, mainly because crop residues incorporated by ploughing decompose more rapidly than those retained on the soil surface. Interestingly, PB-B resulted in similar soil OC contents as PB-K, even though SOM stabilization pathways likely differed between planting systems. It is inferred that SOM formed by physically transferring straw-C into coarse-MAOM likely occurred to some extent in all treatments but was a dominant factor under PB-K and PB-P, whereas SOM formed by physically transferring black-C into fine-MAOM was probably significant under PB-B. It is important to note, however, that fire-induced SOM losses under PB-B likely occurred but were compensated by the formation and inclusion of recalcitrant, charred straw-C sources. This indicates that other aspects of soil quality need to be considered when deciding how to manage crop residues under PB systems. Although both PB-B and PB-K exhibited similar soil OC contents and bulk-level SOM composition, previous studies showed that burning practices prompt soil degradation and environmental pollution in the Yaqui Valley. Examining whether PB-K increases subsoil SOM in the studied Vertisols is recommended for future research.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at <https://doi.org/10.1016/j.geodrs.2021.e00430>.

References

- Abe, S.S., Mueller, C.W., Steffens, M., Koelbl, A., Knicker, H., Koegel-Knabner, I., 2009. Effects of land-use change on chemical composition of soil organic matter in tropical lowland Bolivia. *Grassl. Sci.* 55, 104–109. <https://doi.org/10.1111/j.1744-697X.2009.00146.x>.
- Abril, A., Bartfeld, P., Bucher, E.H., 2005. The effect of fire and overgrazing disturbs on soil carbon balance in the Dry Chaco forest. *For. Ecol. Manag.* 206, 399–405. <https://doi.org/10.1016/j.foreco.2004.11.014>.
- Atere, C.T., Gunina, A., Zhu, Z., Xiao, M., Liu, S., Kuzyakov, Y., Chen, L., Deng, Y., Wu, J., Ge, T., 2020. Organic matter stabilization in aggregates and density fractions in paddy soil depending on long-term fertilization: tracing of pathways by ¹³C natural abundance. *Soil Biol. Biochem.*, 107931 <https://doi.org/10.1016/j.soilbio.2020.107931>.
- Awale, R., Chatterjee, A., Franzen, D., 2013. Tillage and N-fertilizer influences on selected organic carbon fractions in a North Dakota silty clay soil. *Soil Tillage Res.* 134, 213–222. <https://doi.org/10.1016/j.still.2013.08.006>.
- Balasubramanian, D., Zhang, Y.P., Grace, J., Sha, L.Q., Jin, Y., Zhou, L.G., Lin, Y.X., Zhou, R.W., Gao, J.B., Song, Q.H., Liu, Y.T., 2020. Soil organic matter as affected by the conversion of natural tropical rainforest to monoculture rubber plantations under acric ferral soils. *Catena* 195, 104753. <https://doi.org/10.1016/j.catena.2020.104753>.
- Balbotín, C., Cruz, C.O., Paz, F., Etchevers, J.D., 2009. Soil carbon sequestration in different ecoregions of Mexico. In: Lal, R., Follet, R.F. (Eds.), *Soil Carbon Sequestration and the Greenhouse Effect*, SSSA Special Publication, vol. 57. Soil Science Society of America Inc., Madison, WI, pp. 71–96. <https://doi.org/10.2136/sssaspecpub57.2ed.c5>.
- Baldock, J.A., Preston, C.M., 1995. Chemistry of carbon decomposition processes in forests as revealed by solid-state carbon-13 nuclear magnetic resonance. In: McFee, W.W., Kelly, J.M. (Eds.), *Carbon Forms and Functions in Forest Soils*. Soil Science Society of America Inc., Madison, WI, pp. 89–118.
- Boeni, M., Bayer, C., Dieckow, J., Conceição, P.C., Dick, D.P., Knicker, H., Salton, J.C., Macedo, M.C.M., 2014. Organic matter composition in density fractions of Cerrado Ferral soils as revealed by CPMAS ¹³C NMR: influence of pastureland, cropland and integrated crop-livestock. *Agric. Ecosyst. Environ.* 190, 80–86. <https://doi.org/10.1016/j.agee.2013.09.024>.
- Bravo-Garza, M.R., Voroney, P., Bryan, R.B., 2010. Particulate organic matter in water stable aggregates formed after the addition of ¹⁴C-labeled maize residues and wetting and drying cycles in vertisols. *Soil Biol. Biochem.* 42, 953–959. <https://doi.org/10.1016/j.soilbio.2010.02.012>.
- Carvalho, A.D., Bustamante, M.D.C., Alcántara, F.D., Resck, I.S., Lemos, S.S., 2009. Characterization by solid-state CPMAS ¹³C NMR spectroscopy of decomposing plant residues in conventional and no-tillage systems in Central Brazil. *Soil Tillage Res.* 102, 144–150. <https://doi.org/10.1016/j.still.2008.08.006>.
- Chávez-Romero, Y., Navarro-Noya, Y.E., Reynoso-Martínez, S.C., Sarria-Guzmán, Y., Govaerts, B., Verhulst, N., Dendooven, L., Luna-Guido, M., 2016. 16S metagenomics reveals changes in the soil bacterial community driven by soil organic C, N-fertilizer and tillage-crop residue management. *Soil Tillage Res.* 159, 1–8. <https://doi.org/10.1016/j.still.2016.01.007>.
- Conteh, A., Blair, G.J., Rochester, L.J., 1998. Soil organic carbon fractions in a Vertisol under irrigated cotton production as affected by burning and incorporating cotton stubble. *Soil Res.* 36, 655–668. <https://doi.org/10.1071/S97117>.
- Cotrufo, M.F., Wallenstein, M.D., Boot, C.M., Denef, K., Paul, E., 2013. The Microbial Efficiency-Matrix Stabilization (MEMS) framework integrates plant litter decomposition with soil organic matter stabilization: do labile plant inputs form stable soil organic matter? *Glob. Chang. Biol.* 19, 988–995. <https://doi.org/10.1111/gcb.12113>.
- Cotrufo, M.F., Soong, J.L., Horton, A.J., Campbell, E.E., Haddix, M.L., Wall, D.H., Parton, W.J., 2015. Formation of soil organic matter via biochemical and physical pathways of litter mass loss. *Nat. Geosci.* 8, 776–779. <https://doi.org/10.1038/ngeo2520>.
- Dalal, R.C., Allen, D.E., Wang, W.J., Reeves, S., Gibson, I., 2011. Organic carbon and total nitrogen stocks in a vertisol following 40 years of no-tillage, crop residue retention and nitrogen fertilisation. *Soil Tillage Res.* 112, 133–139. <https://doi.org/10.1016/j.still.2010.12.006>.

- Di Rienzo, J.A., Macchiavelli, R.E., Casanoves, F., 2017. Linear Mixed Models: Applications in InfoStat – 1st Edition. Centro de Transferencia InfoStat, Facultad de Ciencias Agropecuarias, Universidad Nacional de Córdoba, Argentina.
- Di Rienzo, J.A., Casanoves, F., Balzarini, M.G., Gonzalez, L., Tablada, M., Robledo, C.W., 2020. InfoStat versión 2020. Centro de Transferencia InfoStat, Facultad de Ciencias Agropecuarias, Universidad Nacional de Córdoba, Argentina.
- Dieckow, J., Mielniczuk, J., Knicker, H., Bayer, C., Dick, D.P., Kögel-Knabner, I., 2005. Composition of organic matter in a subtropical Acrisol as influenced by land use, cropping and N fertilization, assessed by CPMAS ¹³C NMR spectroscopy. *Eur. J. Soil Sci.* 56, 705–715. <https://doi.org/10.1111/j.1365-2389.2005.00705.x>.
- Dieckow, J., Bayer, C., Conceição, P.C., Zanatta, J.A., Martin-Neto, L., Milori, D.B.M., Salton, J.C., Macedo, M.M., Mielniczuk, J., Hernani, L.C., 2009. Land use, tillage, texture and organic matter stock and composition in tropical and subtropical Brazilian soils. *Eur. J. Soil Sci.* 60, 240–249. <https://doi.org/10.1111/j.1365-2389.2008.01101.x>.
- Ding, G., Liu, X., Herbert, S., Novak, J., Amarasiwardena, D., Xing, B., 2006. Effect of cover crop management on soil organic matter. *Geoderma* 130, 229–239. <https://doi.org/10.1016/j.geoderma.2005.01.019>.
- Dong, L., Zhang, H., Wang, L., Yu, D., Yang, F., Shi, X., Saleem, H., Akhtar, M.S., 2018. Irrigation with sediment-laden river water affects the soil texture and composition of organic matter fractions in arid and semi-arid areas of Northwest China. *Geoderma* 328, 10–19. <https://doi.org/10.1016/j.geoderma.2018.05.002>.
- Duan, P., Schmidt-Rohr, K., 2017. Composite-pulse and partially dipolar dephased multiCP for improved quantitative solid-state ¹³C NMR. *J. Magn. Reson.* 285, 68–78. <https://doi.org/10.1016/j.jmr.2017.10.010>.
- Ellert, B.H., Janzen, H.H., 2006. Long-term biogeochemical cycling in agroecosystems inferred from ¹³C, ¹⁴C and ¹⁵N. *J. Geochim. Explor.* 88, 198–201. <https://doi.org/10.1016/j.gexplo.2005.08.038>.
- Engel, R.E., Miller, P.R., McConkey, B.G., Wallander, R., 2017. Soil organic carbon changes to increasing cropping intensity and no-till in a semiarid climate. *Soil Sci. Soc. Am. J.* 81, 404–413. <https://doi.org/10.2136/sssaj2016.06.0194>.
- Erenstein, O., Sayre, K., Wall, P., Hellin, J., Dixon, J., 2012. Conservation agriculture in maize-and wheat-based systems in the (sub) tropics: lessons from adaptation initiatives in South Asia, Mexico, and Southern Africa. *J. Sustain. Agric.* 36, 180–206. <https://doi.org/10.1080/10440046.2011.620230>.
- Fonteyne, S., Martínez Gamiño, M.A., Saldívar Tejada, A., Verhulst, N., 2019. Conservation agriculture improves long-term yield and soil quality in irrigated maize-oats rotation. *Agronomy* 9, 845. <https://doi.org/10.3390/agronomy9120845>.
- Fonteyne, S., Burguño, J., Albarrán Contreras, B.A., Andrio Enríquez, E., Castillo Villasenor, L., Enyanche Velázquez, F., Escobedo Cruz, H., Espidio Balbuena, J., Espinosa Solorio, A., García Meza, P., González Galindo, F., 2021. Effects of conservation agriculture on physicochemical soil health in 20 maize-based trials in different agro-ecological regions across Mexico. *Land Degrad. Dev.* 32, 2242–2256. <https://doi.org/10.1002/ldr.3894>.
- Fuentes, M., Govaerts, B., Hidalgo, C., Etchevers, J., González-Martín, I., Hernández-Hierro, J.M., Sayre, K.D., Dendooven, L., 2010. Organic carbon and stable ¹³C isotope in conservation agriculture and conventional systems. *Soil Biol. Biochem.* 42, 551–557. <https://doi.org/10.1016/j.soilbio.2009.11.020>.
- Galantini, J.A., Duval, M.E., Iglesias, J.O., Kruger, H., 2014. Continuous wheat in semiarid regions: long-term effects on stock and quality of soil organic carbon. *Soil Sci. Soc. Am. J.* 78, 284–292. <https://doi.org/10.1097/SS.0000000000000072>.
- Govaerts, B., Sayre, K.D., Ceballos-Ramirez, J.M., Luna-Guido, M.L., Limon-Ortega, A., Deckers, J., Dendooven, L., 2006. Conventionally tilled and permanent raised beds with different crop residue management: effects on soil C and N dynamics. *Plant Soil* 280, 143–155. <https://doi.org/10.1007/s11104-005-2854-7>.
- Govaerts, B., Verhulst, N., Castellanos-Navarrete, A., Sayre, K.D., Dixon, J., Dendooven, L., 2009. Conservation agriculture and soil carbon sequestration: between myth and farmer reality. *Crit. Rev. Plant Sci.* 28, 97–122. <https://doi.org/10.1080/07352680902776358>.
- Grahmann, K., Dittert, K., Verhulst, N., Govaerts, B., Buerkert, A., 2019. 15N Fertilizer recovery in different tillage–straw systems on a Vertisol in north-west Mexico. *Soil Use Manag.* 35, 482–491. <https://doi.org/10.1111/sum.12495>.
- Gregorich, E.G., Monreal, C.M., Schnitzer, M., Schulten, H.R., 1996. Transformation of plant residues into soil organic matter: chemical characterization of plant tissue, isolated soil fractions, and whole soils. *Soil Sci. Soc. Am. J.* 60, 680–693.
- Gregorich, E.G., Beare, M.H., McKim, U.F., Skjemstad, J.O., 2006. Chemical and biological characteristics of physically uncomplexed organic matter. *Soil Sci. Soc. Am. J.* 70, 975–985. <https://doi.org/10.2136/sssaj2005.0116>.
- Hatcher, P.G., Schnitzer, M., Dennis, L.W., Maciel, G.E., 1981. Aromaticity of humic substances in soils. *Soil Sci. Soc. Am. J.* 45, 1089–1094. <https://doi.org/10.2136/sssaj1981.03615995004500060016x>.
- Helfrich, M., Ludwig, B., Buurman, P., Flessa, H., 2006. Effect of land use on the composition of soil organic matter in density and aggregate fractions as revealed by solid-state ¹³C NMR spectroscopy. *Geoderma* 136, 331–341. <https://doi.org/10.1016/j.geoderma.2006.03.048>.
- IUSS Working Group WRB, 2006. World Reference Base for Soil Resources 2006, 2nd ed. World Soil Resources Reports No. 103. FAO, Rome, p. 128.
- Jha, P., Hati, K.M., Dalal, R.C., Dang, Y.P., Koppitke, P.M., Menzies, N.W., 2020. Soil carbon and nitrogen dynamics in a Vertisol following 50 years of no-tillage, crop stubble retention and nitrogen fertilization. *Geoderma* 358, 113996. <https://doi.org/10.1016/j.geoderma.2019.11.3996>.
- Jiménez-González, M.A., De la Rosa, J.M., Jiménez-Morillo, N.T., Almendros, G., González-Pérez, J.A., Knicker, H., 2016. Post-fire recovery of soil organic matter in a Cambisol from typical Mediterranean forest in Southwestern Spain. *Soil. Total Environ.* 572, 1414–1421. <https://doi.org/10.1016/j.scitotenv.2016.02.134>.
- Jiménez-Morillo, N.T., José, M., Waggoner, D., Almendros, G., González-Vila, F.J., González-Pérez, J.A., 2016. Fire effects in the molecular structure of soil organic matter fractions under *Quercus subser* cover. *Catena* 145, 266–273. <https://doi.org/10.1016/j.catena.2016.06.022>.
- Johnson, R.L., Schmidt-Rohr, K., 2014. Quantitative solid-state ¹³C NMR with signal enhancement by multiple cross polarization. *J. Magn. Reson.* 239, 44–49. <https://doi.org/10.1016/j.jmr.2013.11.009>.
- Keeler, C., Kelly, E.F., Maciel, G.E., 2006. Chemical-structural information from solid-state ¹³C NMR studies of a suite of humic materials from a lower montane forest soil, Colorado, USA. *Geoderma* 130, 124–140. <https://doi.org/10.1016/j.geoderma.2005.01.015>.
- Kirkby, C.A., Richardson, A.E., Wade, L.J., Passioura, J.B., Batten, G.D., Blanchard, C., Kirkegaard, J.A., 2014. Nutrient availability limits carbon sequestration in arable soils. *Soil Biol. Biochem.* 68, 402–409. <https://doi.org/10.1016/j.soilbio.2013.09.032>.
- Knicker, H., Nikolova, R., Dick, D.P., Dalmolin, R.S.D., 2012. Alteration of quality and stability of organic matter in grassland soils of Southern Brazil highlands after ceasing biannual burning. *Geoderma* 181, 11–21. <https://doi.org/10.1016/j.geoderma.2012.03.001>.
- Kögel-Knabner, I., 1997. ¹³C and ¹⁵N NMR spectroscopy as a tool in soil organic matter studies. *Geoderma* 80, 243–270. [https://doi.org/10.1016/S0016-7061\(97\)00055-4](https://doi.org/10.1016/S0016-7061(97)00055-4).
- Kögel-Knabner, I., 2002. The macromolecular organic composition of plant and microbial residues as inputs to soil organic matter. *Soil Biol. Biochem.* 34, 139–162. [https://doi.org/10.1016/S0038-0717\(01\)00158-4](https://doi.org/10.1016/S0038-0717(01)00158-4).
- Krull, E.S., Skjemstad, J.O., Graetz, D., Grice, K., Dunning, W., Cook, G., Parr, J.F., 2003. ¹³C-depleted charcoal from C₄ grasses and the role of occluded carbon in phytoliths. *Org. Geochem.* 34, 1337–1352. [https://doi.org/10.1016/S0146-6380\(03\)00100-1](https://doi.org/10.1016/S0146-6380(03)00100-1).
- Lehmann, J., Kleber, M., 2015. The contentious nature of soil organic matter. *Nature* 528, 60–68. <https://doi.org/10.1038/nature16069>.
- Leinweber, P., Schulten, H.R., Jancke, H., 1999. New evidence for the molecular composition of soil organic matter in Vertisols. *Soil Sci. Soc. Am. J.* 63, 857–870.
- Li, D., Liu, K., Huang, Q., Zhang, J., Zhao, B., 2021. Different responses of soil carbon chemistry to fertilization regimes in the paddy soil and upland soil were mainly reflected by the opposite shifts of OCH and alkyl C. *Geoderma* 385, 114876. <https://doi.org/10.1016/j.geoderma.2020.11.4876>.
- Limon-Ortega, A., 2011. Planting system on permanent beds; a conservation agriculture alternative for crop production in the Mexican Plateau. In: Godone, D., Stanchi, S. (Eds.), *Soil Erosion Issues in Agriculture*. IntechOpen, Rijeka, Croatia, pp. 183–206.
- Limon-Ortega, A., Sayre, K.D., Francis, C.A., 2000. Wheat and maize yields in response to straw management and nitrogen under a bed planting system. *Agron. J.* 92, 295–302. <https://doi.org/10.2134/agronj2000.922295x>.
- Limon-Ortega, A., Govaerts, B., Deckers, J., Sayre, K.D., 2006. Soil aggregate and microbial biomass in a permanent bed wheat–maize planting system after 12 years. *Field Crop Res.* 97, 302–309. <https://doi.org/10.1016/j.fcr.2005.11.001>.
- Lin, R., Chen, C., 2014. Tillage, crop rotation, and nitrogen management strategies for wheat in Central Montana. *Agron. J.* 106, 475–485. <https://doi.org/10.2134/agronj2013.0316>.
- Maillard, É., Angers, D.A., Chantigny, M., Bittman, S., Rochette, P., Lévesque, G., Hunt, D., Parent, L.E., 2015. Carbon accumulates in organo-mineral complexes after long-term liquid dairy manure application. *Agric. Ecosyst. Environ.* 202, 108–119. <https://doi.org/10.1016/j.agee.2014.12.013>.
- Manna, M.C., Swarup, A., Wanjari, R.H., Mishra, B., Shahi, D.K., 2007. Long-term fertilization, manure and liming effects on soil organic matter and crop yields. *Soil Tillage Res.* 94, 397–409. <https://doi.org/10.1016/j.still.2006.08.013>.
- Mao, J., Olk, D.C., Fang, X., He, Z., Schmidt-Rohr, K., 2008. Influence of animal manure application on the chemical structures of soil organic matter as investigated by advanced solid-state NMR and FT-IR spectroscopy. *Geoderma* 146, 353–362. <https://doi.org/10.1016/j.geoderma.2008.06.003>.
- Mao, J., Cao, X., Olk, D.C., Chu, W., Schmidt-Rohr, K., 2017. Advanced solid-state NMR spectroscopy of natural organic matter. *Prog. Nucl. Magn. Reson. Spectrosc.* 100, 17–51. <https://doi.org/10.1016/j.pnmrs.2016.11.003>.
- Mao, J.D., Hu, W.G., Schmidt-Rohr, K., Davies, G., Ghabbour, E.A., Xing, B., 2000. Quantitative characterization of humic substances by solid-state carbon-13 nuclear magnetic resonance. *Soil Sci. Soc. Am. J.* 64, 873–884. <https://doi.org/10.2136/sssaj2000.643873x>.
- Margenot, A.J., Calderón, F.J., Goyne, K.W., Mukome, F.N.D., Parikh, S.J., 2017a. IR Spectroscopy, Soil Analysis Applications. *Encyclopedia of Spectroscopy and Spectrometry*. Elsevier Inc., pp. 448–454. <https://doi.org/10.1016/B978-0-12-409547-2.12170-5>.
- Margenot, A.J., Calderón, F.J., Magrini, K.A., Evans, R.J., 2017b. Application of DRIFTS, ¹³C NMR, and py-MBMS to characterize the effects of soil science oxidation assays on soil organic matter composition in a Mollic Xerofluvent. *Appl. Spectrosc.* 71, 1506–1518. <https://doi.org/10.1177/0003702817691776>.
- Mathers, N.J., Xu, Z., 2003. Solid-state ¹³C NMR spectroscopy: characterization of soil organic matter under two contrasting residue management regimes in a 2-year-old pine plantation of subtropical Australia. *Geoderma* 114, 19–31. [https://doi.org/10.1016/S0016-7061\(02\)00339-7](https://doi.org/10.1016/S0016-7061(02)00339-7).
- McCullough, E.B., Matson, P.A., 2016. Evolution of the knowledge system for agricultural development in the Yaqui Valley, Sonora, Mexico. *Proc. Natl. Acad. Sci. U. S. A.* 113, 4609–4614. <https://doi.org/10.1073/pnas.2011602108>.
- Miller, J., Hazendonk, P., Drury, C., 2018. Influence of manure type and bedding material on carbon content of particulate organic matter in feedlot amendments using ¹³C NMR-DPMAS. *Compost Sci. Util.* 26, 27–39. <https://doi.org/10.1080/1065657X.2017.1342106>.

- Neff, J.C., Townsend, A.R., Gleixner, G., Lehman, S.J., Turnbull, J., Bowman, W.D., 2002. Variable effects of nitrogen additions on the stability and turnover of soil carbon. *Nature* 419, 915–917. <https://doi.org/10.1038/nature01136>.
- Novelli, L.E., Cavaglia, O.P., Melchiori, R.J.M., 2011. Impact of soybean cropping frequency on soil carbon storage in Mollisols and Vertisols. *Geoderma* 167, 254–260. <https://doi.org/10.1016/j.geoderma.2011.09.015>.
- O'Leary, M.H., 1988. Carbon isotopes in photosynthesis. *Biosci.* 38, 328–336. <https://doi.org/10.2307/1310735>.
- Preston, C.M., Newman, R.H., Rother, P., 1994. Using ^{13}C CPMAS NMR to assess effects of cultivation on the organic matter of particle size fractions in a grassland soil. *Soil Sci.* 157, 26–35.
- Romero, C.M., Engel, R.E., D'Andrilli, J., Chen, C., Zabinski, C., Miller, P.R., Wallander, R., 2017. Bulk optical characterization of dissolved organic matter from semiarid wheat-based cropping systems. *Geoderma* 306, 40–49. <https://doi.org/10.1016/j.geoderma.2017.06.029>.
- Romero, C.M., Engel, R.E., D'Andrilli, J., Miller, P.R., Wallander, R., 2019. Compositional tracking of dissolved organic matter in semiarid wheat-based cropping systems using fluorescence EEMs-PARAFAC and absorbance spectroscopy. *J. Arid Environ.* 167, 34–42. <https://doi.org/10.1016/j.jaridenv.2019.04.013>.
- R Core Team, 2019. *R: A Language and Environment for Statistical Computing*. R Foundation for Statistical Computing, Vienna, Austria.
- Samson, M.É., Chantigny, M.H., Vanasse, A., Menasseri-Aubry, S., Angers, D.A., 2020a. Coarse mineral-associated organic matter is a pivotal fraction for SOM formation and is sensitive to the quality of organic inputs. *Soil Biol. Biochem.* 107935. <https://doi.org/10.1016/j.soilbio.2020.107935>.
- Samson, M.É., Chantigny, M.H., Vanasse, A., Menasseri-Aubry, S., Royer, I., Angers, D.A., 2020b. Management Practices Differently Affect Particulate and Mineral-Associated Organic Matter and their Precursors in Arable Soils. *Soil Biol. Biochem.* p. 107867. <https://doi.org/10.1016/j.soilbio.2020.107867>.
- Sayre, K., Limon, A., Govaerts, B., 2005. Experiences with permanent bed planting systems CIMMYT/MEXICO. In: *International Workshop on Permanent Raised Beds*. Australian Centre for International Agricultural Research, Canberra, Australia, Griffith, NSW, Australia, 1–3 March 2005 (monograph).
- Simpson, M.J., Preston, C.M., 2008. Soil organic matter analysis by solid-state ^{13}C nuclear magnetic resonance spectroscopy. In: Carter, M.R., Gregorich, E.G. (Eds.), *Soil Sampling and Methods of Analysis*. CRC Press, Boca Raton USA, pp. 681–692.
- Simpson, M.J., Otto, A., Feng, X., 2008. Comparison of solid-state carbon-13 nuclear magnetic resonance and organic matter biomarkers for assessing soil organic matter degradation. *Soil Sci. Soc. Am. J.* 72, 268–276. <https://doi.org/10.2136/sssaj2007.0045>.
- Skjemstad, J.O., Clarke, P., Taylor, J.A., Oades, J.M., McClure, S.G., 1996. The chemistry and nature of protected carbon in soil. *Soil Res.* 34, 251–271. <https://doi.org/10.1071/SR9960251>.
- Sohi, S.P., Mahieu, N., Arah, J.R., Powelson, D.S., Madari, B., Gaunt, J.L., 2001. A procedure for isolating soil organic matter fractions suitable for modeling. *Soil Sci. Soc. Am. J.* 65, 1121–1128. <https://doi.org/10.2136/sssaj2001.6541121x>.
- Soil Survey Staff, 2003. *Keys to Soil Taxonomy*. United States Department of Agriculture, Natural Resources Conservation Service, Washington, DC, p. 332.
- Somasundaram, J., Reeves, S., Wang, W., Heenan, M., Dalal, R., 2017. Impact of 47 years of no tillage and stubble retention on soil aggregation and carbon distribution in a vertisol. *Land Degrad. Dev.* 28, 1589–1602. <https://doi.org/10.1002/ldr.2689>.
- St. Luce, M.S., Whalen, J.K., Ziadi, N., Zebbarh, B.J., 2011. Nitrogen dynamics and indices to predict soil nitrogen supply in humid temperate soils. *Adv. Agron.* 112, 55–102. <https://doi.org/10.1016/B978-0-12-385538-1.00002-0>.
- Vázquez, C., Merlo, C., Noé, L., Romero, C., Abril, A., Carranza, C., 2013. Sustainability/resilience of soil organic matter components in an Argentinean arid region. *Spanish J. Soil Sci.* 3, 73–77. <https://doi.org/10.3232/SJSS.2013.V3.N1.05>.
- Verachtert, E., Govaerts, B., Lichter, K., Sayre, K.D., Ceballos-Ramirez, J.M., Luna-Guido, M.L., Deckers, J., Dendooven, L., 2009. Short term changes in dynamics of C and N in soil when crops are cultivated on permanent raised beds. *Plant Soil* 320, 281–293. <https://doi.org/10.1007/s11104-009-9893-4>.
- Verhulst, N., Deckers, J., Govaerts, B., 2009. *Classification of the Soil at CIMMYT's Experimental Station in the Yaqui Valley near Ciudad Obregón, Sonora, Mexico*. CIMMYT Report, CIMMYT, Mexico D.F.
- Verhulst, N., Sayre, K.D., Vargas, M., Crossa, J., Deckers, J., Raes, D., Govaerts, B., 2011a. Wheat yield and tillage-straw management system × year interaction explained by climatic co-variables for an irrigated bed planting system in northwestern Mexico. *Field Crop Res.* 124, 347–356. <https://doi.org/10.1016/j.fcr.2011.07.002>.
- Verhulst, N., Kienle, F., Sayre, K.D., Deckers, J., Raes, D., Limon-Ortega, A., Tijerina-Chavez, L., Govaerts, B., 2011b. Soil quality as affected by tillage-residue management in a wheat-maize irrigated bed planting system. *Plant Soil* 340, 453–466. <https://doi.org/10.1007/s11104-010-0618-5>.
- Verhulst, N., Romero, C.M., Hao, X., Hazendonk, P., Chantigny, M., Fonteyne, S., 2020. *Soil Organic Matter Dynamics after 27 Years of Different Tillage-Residue Management in Northern Mexico*, vol. 1. CIMMYT Research Data & Software Repository Network. <https://data.cimmyt.org/dataset.xhtml?persistentId=hd%3A11529%2F10548541>.